NITROGEN-BEARING TOXINS & THE ENVIRONMENT
FOOD-SAFETY MONITORING SYSTEMS FOR THE QUALITY ASSURANCE OF
VEGETABLE PROTEIN PRODUCTS

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by

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ABSTRACT

Nitrogen-Bearing Toxins & The Environment
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The multitude of food recalls in 2007 clearly demonstrated that total nitrogen-content (ΣN) determination by means of Near Infrared Spectroscopy (NIRS) can be deceived, and should no longer be regarded as a complete quality assurance program for nutritive-protein evaluations. Furthermore, contemporary Canadian-employed analytical tools are precariously limited in their ability to effectively assure a product where there is no *a priori* knowledge of the environmental toxin(s) involved. In light of these challenges, this study explored a number of new analytical techniques used to assess and furthermore assure the quality of Vegetable Protein Products (VPPs). Using LC/MS/MS/MS technologies, a combination of VPP-based samples were analyzed for the presence of nitrogen-bearing environmental toxins. Of the 117 test-runs, involving an assortment of matrices, Melamine (MEL) and Cyanuric Acid (CYA) were positively identified (> 1 PPM) in 22 and 17, respectively. Subsequent HPLC-UV Amino-Acid-Profiling further confirmed the adulteration of those materials contaminated with Melamine-and-Related-Compounds (MARC).

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1.0 INTRODUCTION

1.1 FOOD SAFETY IN CANADA

With a prodigious reserve of land and water resources, Canada's requirement for progressive food safety and nutrient management programs has become a *sine qua non* in recent years. Moreover, Canada's food processing industry is constantly growing, employing well over 250,000 Canadians. In 2005, an additional \$1.81 of gross domestic product was indirectly created in the economy for every \$1.00 of gross domestic product produced within the industry. Following this trend, 2.55 jobs were indirectly created for every job supported by these activities. In 2006 Canada was the world's fifth largest agriculture and agri-food importer, accounting for 2.8% of total global imports. That year Canada was also the fourth largest exporter, representing 3.5% globally (GTA, 2006; AAFC, 2007).

Canadian use of protein meals sourced from cereal grains and vegetable products such as corn, rice, soy and wheat reached record highs in the past 5 years, with a national consumption of over 1.5 megatons (Mt) of soy alone. Unable to source that demand locally, an estimated 80% was imported during that time. This trend has quickly developed, with a marked decline in the ratio of commodity exports to imports over the past five years. For example, the trade balance of vegetable protein products (VPPs) has decreased from close to 70% in favour of exports in 2002, to less than 30% in 2006 (AAFC, 2008). These statistics, coupled with the fact that in 2006 over 65% of consumer products sold in Canada were imported (GOC, 2008), should inspire an increase of large-scale risk assessment research to minimize the likelihood of a food-related catastrophe. Moreover, as the food-web is a highly vulnerable pathway through which environmental toxins can infiltrate Canadian ecosystems, biomagnify and accumulate over time, national environmental infrastructure development is promptly required. In essence, as the complexity of the global agri-food market continues to expand in this direction, so too must the scope of quality assurance programs designed to promote food safety and security.

The core Canadian food safety and security programs are managed by the Canadian Food Inspection Agency (CFIA), in collaboration with several organizations at the federal, provincial and municipal levels. With a mandate "to protect Canadians from preventable food safety

hazards, to ensure that food safety emergencies are effectively managed, and that the public is aware of--and contributes to--food safety," the CFIA has continually evolved over the years to meet the increasing quality demands set by consumer expectations (CFIA, 2008).

Implemented in 1991, the Food Safety Enhancement Program (FSEP) is regarded today as the Canadian foundation of good food safety practices. Governed by the CFIA, the FSEP is designed to "encourage and support the development, implementation and maintenance of Hazard Analysis Critical Control Point (HACCP) systems in all federally registered establishments" (CFIA, 2008).

Further demonstrated with the Canadian Food Safety Adaptation Program (CFSAP 1999), food safety in Canada has a history of partnership among government agencies at the national level and key agri-food industry stakeholders. Also administered by the CFIA, HACCP principles are applied here, in full compliance with international trade standards of food safety as established by the Codex Alimentarius (CFIA, 2008). By providing incentives to national associations involved in all facets of the industry to act in accordance with its policies, from production to marketing of foodstuffs, the inauguration of CFSAP spearheaded the development of risk management strategies designed to strengthen quality assurance measures throughout the food chain. Despite this significant achievement; however, these measures were strictly voluntary, without any real devices of enforceability (CFIA, 2008).

In 2003 CFSAP was replaced by the Canadian Food Safety and Quality Program (CFSQP), adding dimensions of quality and traceability to the national program (AAFC, 2003). Central to this initiative was the consideration of feedback coming from interest groups including the CFIA, Health Canada, provincial and territorial governments, academia, the Canadian Federation of Agriculture and national product organizations (Canadian Horticultural Council, Canadian Produce Marketing Association, the Cold Chain, the Canadian Trucking Alliance, etc.), which served as a mechanism to constantly question and thereby improve food safety practices in Canada (Jol *et al.*, 2003).

Today, HACCP compliance is compulsory in all federally-inspected meat and poultry processing plants (CFIA, 2000; Gagnon *et al.*, 1999). Where not already governed by other bodies of

legislation including the *Meat Inspection Act*, the *Canada Transportation Act*, the *Fish Inspection Act* and the *Canada Agriculture Products Act*, further compliance assessments required by the *Food and Drugs Act* and Regulations are typically exercised by the CFIA; following standards governed by the Canadian Food Inspection System (CFIS) Implementation Group (CFISIG) (Canadian Food Inspection System Implementation Group, 2004; Jol *et al.*, 2003).

Imported cereal products, including VPPs are regulated by the *Canada Grain Act* and its Canada Grain Regulations. Also monitored by the CFIA, along with the Cereal Research Center (CRC), additional safeguards are developed by the Canadian Grains Commission which researches and develops analytical methods for the detection of toxic compounds in grain products (CFIA, 2008).

Most recently, in December of 2007, the federal government unveiled plans for a new program that purports to strengthen enforceable legislation by imposing tougher regulations on food products. The proposed Food and Consumer Safety Action Plan (FCSAP) has promised to improve on the current CFIA programs by:

- Working with industries, provinces and territories to implement preventative food safety control measures along the food chain;
- Putting in place tools to better identify importers, track imports and work with foreign authorities to verify the safety of foods at their country of origin; and
- Increasing authority to monitor risks to Canadians and prevent unsafe products from entering the Canadian marketplace (CFIA, 2008).

Only time can demonstrate the significance of the FCSAP, its ultimate success and whether it will truly address the current lack of legislative powers that exists in Canada to control toxic substances entering the country through the food-web. Here there is a significant requirement for the CFIA to launch itself into the vanguard of environmentally conscious food-safety research, with authoritative powers that dovetail the *Canadian Environmental Protection Act* (CEPA).

1.2. WAKE-UP CALL: FOOD RECALLS

Despite the apparent safeguards of the Canadian agri-food sector, a multitude of food product recalls in 2006 and 2007 spawned a deep-seated lack of consumer confidence in many Canadians. For instance, the unfortunate affliction of 30 Ontarians poisoned by *E. coli*-contaminated lettuce supplies in 2006 raised serious questions about the integrity of the national food safety network (CFIA, 2008). Adding insult to injury, the unprecedented product recalls of 2007 that followed exposed one of the largest-scale food safety failures of our era.

Announced on March 16, 2007, in response to 6 reported dead pets by consumers and an independent feeding trial that killed 4/19 cats within 3-4 days, and produced vomiting and lethargy in 10/10 dogs in a 2-day period, the Canadian-based Menu Foods Income Fund recalled 60,000,000 containers of canned diets co-manufactured for North American pet food companies (MFIF, 2007). As further pet-death reports started to surface in the media, toxicology and food science groups at the University of Cornell, the University of Guelph, the American Veterinary Medical Association, as well as several other industry leaders initiated efforts in search of the source of the contamination. With only kidney failure as a guide, several toxins were suspected: citrinin, aflatoxin, keatoin, T2-H2T, ethylene glycol and oxotoin. As a common ingredient linking the affected brands became apparent, laboratories across Canada and the US began heavily testing wheat gluten samples, without any traces of the suspected compounds. What hastily ensued, fueled by unrelenting demands from media and public interest groups, were restless bouts of rigorous scientific investigation (RCC, 2007).

In collaboration with University of Cornell, samples received at the New York State Food Laboratory were found to contain aminopterin, an antineoplastic agent. However, without reproducibility and/or symptoms consistent with aminopterin poisoning, the United States Food and Drug Administration (USFDA) and Cornell University rejected the findings and continued searching for a more fitting contaminant (USFDA, 2007). In the weeks that followed, melamine, along with its analogues, was identified in raw material wheat gluten samples and finished product samples, as well as in the urine of affected pets. Crystals would also be found in the renal tubules of autopsied pets (UOG, 2007).

As contaminated VPP sources were discovered in the human food supply, on April 3, 2007 the issue spiraled into the mainstream. Traced to thirty-eight farms in Indiana, an estimated 80,000 grain-fed chickens raised for human consumption were found to have consumed melamine-adulterated feed. Swine farms in several states were also identified as having fed contaminated products to approximately 56,000 swine (USFDA, 2007).

Pet owners and consumers demanded explanations as to how such a disaster could have occurred on such a widespread scale, especially in the US which is heavily regulated by government bodies such as the FDA and American Association of Feed Control Officials (AAFCO). Questions in the vein of "how can you assure the safety of your product" were raised throughout the bulk commodities food trade.

1.3. IMPLICATIONS

As the literal livelihoods of those involved in the production, processing, sale and reprocessing of VPPs are dependent on the delivery of quality assured raw materials, the need for time-sensitive and cost-effective monitoring systems has arisen in response to great demand. With a North American economy so heavily reliant on low-cost food supplies coming from foreign countries such as China, the development of such programs fulfills much more than a basic requirement; it has become a critical component of the Canadian food chain. Likewise, with the food-web so closely interrelated to human, animal and environmental health, greater attention is required in several government ministries. For improvements to be made, no longer can assurances from trade partners guide industry practice, but rather, as Jol *et al.* (2006) explain, "each partner in the chain, be it producer, manufacturer, handler or vendor has an important role to play in ensuring that the product is of sound food integrity."

Moreover, in consideration of the ever-increasing role of VPPs in nutrition, the need to accurately monitor environmental contamination by means of nitrogen-bearing toxins such as melamine and its derivatives is crucial. Limited successes have been previously achieved using Gas Chromatography techniques. More advanced tools involving Liquid Chromatography show promise, but not to the required extent. As tremendous negative public opinion towards imported

food supplies sourced from China increases, the technique or combination of techniques best suited to a comprehensive quality assurance program of VPPs is in considerable demand. Scientists who support the industry must do their part in helping the trade and the citizens that rely on it to be better protected in the years ahead.

1.4. THESIS OUTLINE

In light of these challenges, this study will focus on three predominant VPPs that are widely applied in human and animal nutrition: Wheat Gluten (WG), Soy Protein Isolate (SPI) and Rice Protein Concentrate (RPC). Each commodity will be discussed in great detail (**Section 2.1.**), with a focus on the unique functional properties that support their beneficial application in food manufacturing. Along with the increased popularity of these products as a consequence of such advantageous properties comes great risk to the end consumer, as well as the surrounding natural environment.

Other parameters highlighted include the inherent nutritive properties of each VPP, including data pertaining to digestibility, amino acid content and protein density. In the experimental section of this paper (**Section 3**) it will become evident that these parameters are critical factors in effectively assuring a quality product by means of chemical analyses and are the principal targets of falsification in such assessments.

Extraction processes and main fractions yielded are further considered, bringing attention to those physical areas displaying the greatest susceptibility to environmental contamination and/or fraudulent practices. A statistical overview of each commodity's trade data is then presented with the hopes of providing the reader with an understanding of how greatly ingrained in the Canadian infrastructure these VPPs truly are, and as a consequence, the degree to which their continued use demands improved monitoring programs.

In the following section (**Section 2.2.1.**) protein content determination methods will be described for WG, SPI and RPC, in conjunction with the duplicitous role of nitrogen-bearing compounds such as melamine and cyanuric acid in mimicking quality VPPs (**Section 2.2.2.**). The chemistry

of melamine, cyanuric acid and their analogues is further explored, alongside the toxicological profile of each. Drawing on pathological data provided by investigators during the pet food recalls of 2007, the most widely accepted causal theories of pet deaths and poisonings are discussed.

Subsequently, the most widely adopted quality assurance programs for VPPs are reviewed (**Section 2.4.**), followed by a discussion on Canadian legislation requirements for monitoring compliance. This area highlights an overall deficiency in legislative support, enforceability and compliance-incentives for industry stakeholders.

The experimental hypothesis and objective is then stated (**Section 2.3.3.**) which leads into the Materials & Methods (**Section 3**). Subsequently, the Results & Discussion (**Section 4**) are presented, including a look at the economic viability of the required analytical instrumentation (**Section 4.4.**), followed finally by the Conclusion & Recommendations (**Section 5**).

2.0. LITERATURE REVIEW

2.1.0 VEGETABLE PROTEIN PRODUCTS

2.1.1. WHEAT GLUTEN

2.1.1.1. PROPERTIES

Commissioned in 1963 by the Food and Agriculture Organization of the United Nations (FAO) in collaboration with the World Health Organization (WHO) to develop "food standards, guidelines and related texts such as codes of practice under the Joint FAO/WHO Food Standards Programme", the Codex Alimentarius Commission (CODEX-STAN 163-1987 Rev 1-2001) defines wheat gluten products as those "produced by separation from wheat or wheat flour of certain non-protein constituents (starch, other carbohydrates)" (CODEX 2001).

Three categories are given (CODEX 2001):

1. "Vital" wheat gluten is characterized by its property of high viscoelasticity as hydrated;

2. "Devitalized" wheat gluten is characterized by its reduced property of viscoelasticity as hydrated due to denaturation;
3. "Solubilized" wheat proteins are characterized by their reduced property of viscoelasticity as hydrated due to partial hydrolysis of wheat gluten.

Wheat gluten (WG) constitutes an estimated 80-85% of the amorphous mixture of ergastic proteins (gliadin and glutenin) that are present in the whole grain's starchy endosperm. The three main components (Fig. 2.1) of the grain are the bran (aleurone and pericarp), endosperm and germ.

Figure 2.1: Wheat Grain (Surget & Barron, 2005)

Nutritional value aside, functional benefits of WG include (Bietz & Lookhart, 1996; Lens *et al.*, 1999; Maningart *et al.*, 1999; Linares *et al.*, 2000; Popineau *et al.*, 2002; Kong *et al.*, 2007):

- a high water absorption capacity (x2 its own weight in water);
- an ability to improve mixing tolerance and consistency;
- a capability of binding the formation of water-insoluble cohesive masses and adhesive films;

- a conduciveness to thermal coagulation of three-dimensional masses with a structural integrity;
- an anti-staling effect;
- a resistance to freezing.

2.1.1.2. EXTRACTION PROCESS

Vital WG is most commonly produced through industrial wet-milling processes. Although four predominant extraction methods have been identified, with varying water:flour mixtures and step-wise separation fractions, to a large extent the same fundamental principles are applied to all (Sayaslan, 2004).

Wheat is first milled into flour, which is kneaded and slowly hydrated (Fig. 2.2). This process of mixing breaks the protein bonds within the gluten, creating a mass of dough that is separated,

Figure 2.2: Separation of flour into starch and protein fractions (Dik *et al.*, 2002)

washed, and further concentrated. Left to rest, stronger gluten networks begin to form, enhancing the gluten's functional viscoelastic properties. Continuous centrifugation yields the two fractions, one protein and one starch, which are then dried under precise conditions (Van Der Borgh *et al.*, 2005).

The isolated protein is then further fractionated and/or modified through acid or enzymatic hydrolysis, depending on the desired end-product. For instance, certain glutes will be engineered to become soluble at different pH levels, **as well** as tailored to specific emulsification and foaming properties for a given recipe (Tate & Lyle, 2005).

2.1.1.3. MAIN FRACTIONS

The two structural components of vital WG, gliadin and glutenin (Fig. 2.3), complement each other functionally, accounting for 85% of the total protein content in wheat flour. As a consequence it is these elements that are most vulnerable to falsification by means of nitrogen-bearing environmental contaminants such as melamine and its analogues in WG production. By deliberately incorporating such compounds into the production process, manufacturers can maximize profits by greatly reducing the costs associated with using superior raw materials such as gliadin and glutenin proteins.

Gliadin monomers, the lower in molecular weight of the two (25,000 – 100,000 MW), are inelastic and become viscous upon hydration (Veraverbeke & Delcour, 2002). Due to their intramolecular disulfide linkages, this extensibility allows gluten to expand when pressure is applied, a critical component of the leavening process in baking. Glutes form complex networks that trap carbon dioxide produced by yeast, giving breads their characteristic texture and air-bubble appearance (Veraverbeke & Delcour, 2002; Tate & Lyle, 2005).

Glutenin polymers, on the other hand, are highly flexible, much larger in molecular weight (>100,000 MW) and consist of subunits linked by intermolecular disulfide bonds. This provides for strength and resilience due to an elasticity and low extensibility that resists external pressure (Hoseney, 1994; Veraverbeke & Delcour, 2002; Tate & Lyle, 2005).

Other proteins derived from wheat flour are non-gluten, accounting for the remaining 15% of the total protein content: mostly peptides, albumins, globulins, enzymes and amino acids (Fig. 2.3).

Figure 2.3: Main Fractions of Wheat Flour Proteins (Tate & Lyle 2005)

2.1.1.4. NUTRITIVE APPLICATION

Since the first breads were created, WG's role as a functional protein with a host of nutritional benefits has been paramount. Containing extremely low concentrations of indigestible proteins (1-2%), WG is used in products ranging from human nutrition (breads, pastries, nutritional beverages, creamers, fat-filled powders, laminated dough, pastas, breakfast cereals, energy bars, soy sauces, savoury flavourings, meat products and vegetarian meat-alternatives) to pet food and livestock feed applications. The caloric density of WG proteins is roughly 4 kcal per gram of product (Tate & Lyle, 2005).

The apparent digestibility of proteins derived from WG is relatively high (Table 2.1). In fact, no other vegetable protein source contains as little indigestible matter as wheat gluten. Soy Protein Isolate (SPI), the next closest match in these terms is 2-3% less digestible.

Pérez-Conesa *et al.* (2001) observed 96% in vitro protein digestibility (IVPD) in human infants fed WG based formula (Pérez-Conesa *et al.*, 2001). In further digestibility trials that compared spray-dried plasma protein to WG, standard ileal digestibility was 9% higher in piglets fed the WG alternative (Stein & Pedersen, 2005). As a consequence, WG is frequently used in milk replacement products for calves, piglets, chicks and veal, as well as aquaculture feed and pet food.

Table 2.1: Apparent Protein Digestibility of Wheat Gluten

Species	Apparent Protein Digestibility (APD)	Source
<i>Litopenaeus vannamei</i> / Boone	98.00%	Akiyama <i>et al.</i> , 1989
<i>Gadus morhua</i> (Linnaeus, 1758) / Atlantic Cod	99.99%	Tibbetts <i>et al.</i> , 2006
<i>Penaeus setiferus</i> L / White Shrimp	100.00%	Brunson <i>et al.</i> , 1997
<i>Dicentrarchus labrax</i> / European sea bass	99.99%	Robaina <i>et al.</i> , 1999
<i>Oncorhynchus mykiss</i> / Rainbow Trout	97.00%	Pferrer <i>et al.</i> , 1992
<i>B. Taurus</i> / Preruminant Calf	94%	Branco-Pardal <i>et al.</i> , 1995

In addition to its highly digestible properties, WG possesses a rich amino acid profile that is abundant in glutamine, proline, cysteine and methionine, but is low in lysine (Prange *et al.*, 2004). This characteristic amino acid profile is especially useful in preparing dietary formulations, serving well as a complement to meat proteins in nutritional regimes (Table 2.2). Accordingly, this study will argue (Section 4) that amino acid profiling is the most effective standalone means of assessing and subsequently assuring the true quality of VPPs such as WG.

Functionally, WG applications in livestock and pet feeds provide for improved texture, palatability, gelling, mixing, emulsification, fat-binding, elasticity, colouring and gelation, both

in moist-canned and dry-extruded kibble-based regimes. Clearly there is a great responsibility to protect those feeds with the same stringency we have come to expect in support of the human food-chain. Undoubtedly all food waste and digested metabolites are ultimately released to the natural environment, where the risks posed to human health accumulate over time.

Table 2.2: Amino Acid Profile of Wheat Gluten (Branco-Pardal *et al.*, 1995)

Amino Acid	g/100g
ASP	3.17
THR	2.74
SER	4.63
GLU	39.52
PRO	12.73
GLY	3.49
ALA	2.56
CYS	3.27
VAL	4.05
MET	1.75
ILE	4.24
LEU	7.29
TYR	3.28
HIS	1.95
LYS	1.67
ARG	3.65

2.1.1.5. INDUSTRY OVERVIEW

With production levels exceeding 603 million metric tons (MMT), wheat is the most abundant cereal grain in the world (FAO, 2008). Canada is the world's sixth largest producer, with 27 MMT in 2006/07; the majority of Canadian wheat is grown in the western Prairie region of the nation (FAO, 2008; StatsCan., 2008). Despite a 70%+ increase in production levels since 2002, forecasts predict a reverse trend, with an estimated decline to 21 MMT (StatsCan., 2008).

In Canada, WG is categorized under Section 2 (Vegetable Products), Chapter 6 (Products of the milling industry; malt; starches; inulin; wheat gluten), of the Canadian Export Classification 2007 (Stats Can. 2008). Wet-milling of wheat flours is the extraction method most commonly

applied in WG production. These co-products account for roughly 6% of total wheat production (Oleson 1994; Sayaslan 2004). With increasing population growth and consumer demands for higher quality grain-based and vegetable protein products, consumption of vital WG in Canada has risen consistently in the past five years (Table 2.3).

Table 2.3: Canada Trade Balance: Five year wheat gluten (HS Code 1109.00 – Wheat Gluten, Whether or Not Dried) trend; values reported in Canadian dollars.

	2002	2003	2004	2005	2006	2007
Total Exports	24,997,922	20,721,911	18,121,932	11,878,713	9,097,193	9,372,892
Total Imports	6,725,088	8,376,117	10,563,089	6,577,914	6,580,172	8,670,797
Trade Balance	18,272,834	12,345,794	7,558,843	5,300,799	2,517,021	702,095

Data obtained from Statistics Canada and the U.S. Census Bureau (U.S. Department of Commerce) (2008).

Since 2002, overall wheat imports have risen from \$6,725,088 to \$8,670,797 in 2007.

Conversely, exports have dropped considerably, with a 62.50% decrease from \$24,997,922 to \$9,372,892 in that period (Stats Can. 2008). Reflecting this trend, the U.S. Census Bureau (U.S. Department of Commerce) confirms that the industry in the United States has increased from \$91,900,000 in 1997 to \$160,548,000 10-years later (USDC, 2007).

Increased availability of WG from the European Union, Australia, China and other foreign countries has further reduced domestic prices, allowing for greater commercial applications of the ingredient (Table 2.4). Chinese imports alone have increased from \$1,932,000 to \$22,245,000 in that same 10-year period, with unit values being offered at heavily competitive prices (FAO, 2004; StatsCan., 2007).

In view of this dramatic increase in imported commodities over the years, Canada must be better prepared to protect its natural resources, the environmental systems surrounding them and its citizens from the potential contaminants contained within those foreign foodstuffs.

Table 2.4: Top 10 Countries: Wheat Gluten commodity exports sorted by value (FAO, 2004).

	Country	Quantity (MT)	Value (000 US\$)	Unit value (US\$)
1	France	106534	87856	825
2	Germany	66415	63951	963
3	Australia	45671	49497	1084
4	Poland	23453	20471	873
5	Netherlands	22733	19398	853
6	Canada	11717	13954	1191
7	USA	10097	12097	1198
8	United Kingdom	6598	5628	853
9	Italy	3239	4056	1252
10	China	4879	3541	726

By far, the largest consumer of WG is the United States (Table 2.5), with an estimated 2004 import total of 193,703 MT of raw material, valued at \$190,919 (1000 US\$) (FAO, 2007; FAO 2008).

Table 2.5: Top 10 Countries: Wheat Gluten commodity imports sorted by value (FAO, 2004).

	Country	Quantity (MT)	Value (000 US\$)	Unit value (US\$)
1	USA	193703	190917	986
2	Belgium	65148	68783	1056
3	United Kingdom	15108	24100	1595
4	Norway	28757	22380	778
5	Japan	14325	21784	1521
6	France	20335	18670	918
7	Thailand	16394	13848	845
8	Mexico	9615	10635	1106
9	Italy	12375	10494	848
10	Denmark	11432	9948	870

2.1.2.0. SOY PROTEIN ISOLATE

2.1.2.1. PROPERTIES

The classification standard for Soy Protein Isolate (SPI) is established by the Codex Alimentarius Commission standard (CODEX STAN 175-1989) which applies to Vegetable Protein Products (VPP) “prepared from soybeans (seeds of *Glycine max.* L.) by various separation and extraction processes.” As per CODEX STAN 1-1985, Rev 1-1991, Codex Alimentarius Volume 1A, this standard must apply to all prepackaged consumer products containing the raw material (CODEX, 1991).

Defined as “intended for use in foods requiring further preparation and by the food processing industry,” Soy Protein Products (SPP) are “food products produced by the reduction or removal from soybeans of certain of the major non-protein constituents (water, oil, carbohydrates) in a manner to achieve a protein (N x 6.25) content of:

- in the case of soy protein flour (SPF) 50% or more and less than 65%;
- in the case of soy protein concentrate (SPC) 65% or more and less than 90%;
- in the case of soy protein isolate (SPI) 90% or more.”

Nutritional value aside, the physical composition of SPI provides for a host of functional benefits as well, mostly involving solubility, water vapour permeability, viscosity, emulsification, texturizing, fat-binding and gelation. SPIs are also used as adhesives, in cosmetics, paper-coatings, plasticizer combinations, textile fibers, inks, cleaning materials, asphalts, resins and as pesticides/fungicides (Bian, 2003; Lecomte, 1993; Mcklem, 2002).

2.1.2.2. EXTRACTION PROCESS

Two predominant methods are used to extract SPIs from defatted soy flour: precipitation with hydrochloric acid (HCL) and separation with polyisopropylacrylamide gel (Trank *et al.*, 1989; Golbitz, 2004).

Of the two, precipitation with HCL is more commonly applied at an industrial level (Fig. 2.4).

Figure 2.4: Flow diagram: Deak Soy Protein fractionation procedure (Deak, 2007)

The whole beans are cleaned, dried, cracked, de-hulled, conditioned and rolled into flakes. Residual hexane is then removed and the resulting meal is ground (Mcklem, 2002). The remaining defatted flour is then combined with deionized water at a 15:1 water-to-flour ratio, and the pH is adjusted to 8.5-9 with sodium hydroxide (2 N NaOH). The slurry is then stirred and centrifuged to produce the first protein extract (Deak, 2007).

The resultant material is then combined with CaCl_2 and NaHSO_3 , is subsequently stirred and centrifuged again. The curd that precipitates is composed of glycinin-rich fractions. The aqueous extraction is carried out at a pH below 9 (Golbitz, 2004; Deak, 2007).

The second protein extract supernatant is then adjusted to a pH of 4.8 with HCL, is stirred and centrifuged for 30 minutes. A β -conglycinin-rich fraction is obtained, along with a significant whey constituent (Deak, 2007).

The extracted samples are then freeze-dried in 2 steps, with increasing temperatures and decreasing vacuum conditions for transport and quality analysis (Deak, 2007).

2.1.2.3. MAIN FRACTIONS

The two major storage proteins used as specialized protein storage vacuoles (PSV) in soybeans account for 70% of the total seed protein: β -conglycinin (7S globulin or vicillin-type) and glycinin (11S globulin or legumin-type) (Derbyshire, 1976). As the highest protein-contents are contained herein, environmental contaminants such as melamine, cyanuric acid and their analogues can be deliberately incorporated into the production process to mimic quality proteins such as β -conglycinin and glycinin. To a much lesser extent, other proteins present in the soybean include lectins, 2S albumins, defense proteins (i.e. protease and amylase inhibitors), lipoxygenases and vacuolar hydrolytic enzymes (Kinney, 2001; Bennett, 2005).

Glycinins are hexamers with an approximate mass of 320 to 375 kDa, and account for 60% of the total globulin fraction. They are encoded by a gene family designated Gly1 to Gly5 and are synthesized as precursors on the rough endoplasmic reticulum (RER) where they are transported

as trimers through the Golgi Apparatus to PSVs. There they are cleaved into 40 kDa acidic and 20 kDa polypeptides (Bennett, 2005).

Beta-conglycinins are glycoprotein trimers with α' , α , and β subunits (Bennett, 2005). Due to their nutrient-dense, phytochemical and protein-rich compositions, along with non-protein co-products like isoflavones and saponins, great interest in β -conglycinin and glycinin proteins has prevailed (Murphy, 2000). As a consequence these proteins have been propelled to large-scale applications in both human and animal nutrition. Additionally, β -conglycinin globulins have received special attention due to their nutraceutical properties as well (Deak, 2006). Accordingly, this trend poses a great vulnerability in the food chain, as the pervasiveness of soy-based proteins in nutrition continues to expand.

Further differences among the two fractions have been documented; β -conglycinin proteins have been shown to contain considerably fewer sulfur-containing amino acids, with 3 to 4 times less cysteine and methionine per unit of protein than glycinin (Fukushima, 1991). Research has revealed that it is possible, through genetic modification and/or varying environmental conditions (i.e. sulfur deprivation, nitrogen supplementation, etc.), to encourage the seeds themselves to optimize this imbalance (Clark, 1999; El-Shemy *et al.*, 2001). El-Shemy *et al.*, (2001) determined that some mechanism in the seeds is responsible for regulating the varying amounts of each storage protein, which might be used to “selectively increase the type of storage protein” desired (El-Shemy *et al.*, 2001). Thus, by selectively increasing the concentration of glycinin, a more pronounced 11S/7S ratio allows for a more complete amino acid profile in the end product (El-Shemy *et al.*, 2001).

Functionally, β -conglycinin and glycinin protein fractions differ in three areas: emulsification, thermal stability and gel formation. *Beta*-conglycinin proteins are thermally more unstable than glycinins, but possess a more favourable emulsifying and emulsion-stabilizing ability. Glycinin proteins on the other hand produce harder heat and calcium induced coagulum (i.e. tofu gel) than β -conglycinin derivatives (Panizzi, 2005).

Further studies have shown 11S globulins to withstand higher surface pressure and reflectivity of chemical sprays (Sánchez, 2003). Additionally, the hydrophilic nature of SPIs allow for water absorption and retention even when subjected to outside forces such as gravity, centrifugation and heating (Zayas, 1997).

2.1.2.4. NUTRITIVE APPLICATION

Containing exceptionally low concentrations of indigestible proteins (3-4%), SPI is used in a variety of products ranging from human nutrition (infant formulas, soymilk, comminuted meat products, meat analogues, tofu, breakfast cereals, yogurts, cheeses and meal replacement beverages) to pet food and livestock applications (puppy and kitten milk replacement products and highly digestible feeds) (Hosken, 2003). The increasing consumer demand for SPI-fortified products has lead to strong competition in extraction and recovery methods, with several ongoing patented and patent-pending applications. That being the case, what cannot be ignored is the potential for profit-driven manufacturing practices to gain popularity, at the expense of end-consumer safety.

At the same time, although more expensive than Soy Protein Concentrates (SPCs), isolates provide enhanced flavour and colouring to nutritive products (Mcklem, 2002). The caloric density of Soy Protein Isolate is roughly 3.38 kcal per gram of product (CondéNet, 2007).

The apparent digestibility of SPI is quite high (Table 2.6). Next to WG, which is 2-3% more digestible, no other vegetable protein source is comparable in terms of digestibility.

Table 2.6: Digestibility Results of Soy Protein Isolate

Species	Apparent Protein Digestibility	Reference
Rats	95-97%	Gilani and Sepehr 2007
Humans	97%	Kayser <i>et al.</i> , 1992
Pigs	97%	Grala <i>et al.</i> , 1998
Cannulated dogs	83-87%	Zhao <i>et al.</i> , 1997

Gilani and Sepehr (2007) found SPI and mustard flour to be the most digestible vegetable protein sources assessed with young rats, with other sources ranging from 66-83% digestibility. These results were consistent with previous data in human nutrition and in swine studies, which both assessed SPI at a digestibility level of 97% (Kayser, 1992; Grala, 1998).

In addition to delivering highly digestible proteins, SPI further possesses an excellent amino acid profile. The FAO and WHO consider SPI “a complete protein,” given that it meets or exceeds the essential amino acid requirements for humans aged 2-years and older, including children, adolescents, and adults (FAO/WHO, 1991).

As seen in Table 2.7, SPI is truly a complete protein, rich in essential amino acids such as leucine, lysine, phenylalanine, tyrosine, threonine, arginine, valine, isoleucine, tryptophan and histidine. Methionine and cysteine are provided as well, although to a lesser extent.

Table 2.7: Amino Acid Profile of Soy Protein Isolate (SPI).

Amino Acid	g/100g
ASP	12.04
THR	3.65
SER	5.11
GLU	20.68
PHE	6.33
GLY	4.14
ALA	4.14
CYS	5.47
VAL	1.34
MET	1.34
ILE	4.99
LEU	8.27
TYR	3.89
HIS	2.80
LYS	6.33
ARG	8.03
TRP	1.46

Data from Morita and Kiriyaama, 1993.

It is this distinct amino acid profile that typifies SPIs and as pointed out in **Section 4**, should be consulted in quality assessment programs that seek to identify superior raw materials in trade practices.

2.1.2.5. INDUSTRY OVERVIEW

Worldwide soybean production reached 221 MMT in 2006, with the United States, Brazil, Argentina, China and India listed as the top 5 producers. Canada was ranked 9th, contributing 3.5 MMT (FAO, 2008).

In Canada, soy proteins, including SPIs and SPCs, are not classified within the Harmonized System of trade commodities. Regardless, industry estimates can be made in examining the trade balances of the raw materials used to produce these proteins, including soy flour and soybean meal.

In the five years from 2003 to 2007, Canada exhibited increasingly negative trade balances, with a record low in 2007 of -13,332 (1000 Canadian\$) (**Table 2.8**).

Table 2.8: Canadian Trade Balance - HS 1208.10 – Soya Bean Flours and Meals. Value in Thousands of Canadian Dollars.

	2003	2004	2005	2006	2007
Total Exports	936	961	1,763	1,913	2,491
Total Imports	10,104	7,594	8,730	12,833	15,813
Trade Balance	-9,167	-6,633	-6,967	-10,920	-13,322

Data obtained from Statistics Canada and the U.S. Census Bureau (U.S. Department of Commerce) (2008).

The reverse trend is observed in the United States (**Table 2.9**), where annual exports of soy flours and meals consistently counterbalanced foreign dependence on imports.

Table 2.9: United States Trade Balance - HS 1208.10 – Soya Bean Flours and Meals. Value in Thousands of Canadian Dollars.

	2003	2004	2005	2006	2007
Total Exports	120,571	287,011	426,648	415,511	483,013
Total Imports	4,392	7,717	19,104	5,044	6,650
Trade Balance	116,179	279,294	407,544	410,467	476,363

Data obtained from Statistics Canada and the U.S. Census Bureau (U.S. Department of Commerce) (2008).

Whereas soy flours and meals were predominately sourced from the United States, the Netherlands and Japan in 2003 (**Table 2.10**), by 2007, China ranked 2nd, reflecting a 4000%+ increase in its contribution to the Canadian supply (StatsCan, 2008).

Table 2.10: Canadian Imports Products HS 1208.10 – Soya Bean Flours and Meals – by Origin (Top 10 Countries); Units Value in Canadian Dollars.

	2003	2004	2005	2006	2007
United States	9,449,762	6,833,387	8,063,823	10,843,429	11,786,640
China	75,740	203,704	90,870	1,514,526	3,204,437
Netherlands	318,677	236,627	182,391	172,735	498,355
India	33,405	28,149	35,642	57,589	204,614
Japan	124,204	132,377	173,357	40,985	61,889
Sri Lanka	235	147	5,691	29,229	28,276
Belgium	985	--	--	--	9,021
Mexico	--	--	--	--	5,213
Taiwan (Taipei)	59,007	67,110	2,147	2,147	4,676
Korea, South	28,190	2,850	8,760	2,871	3,864
Sub-Total	10,090,205	7,504,351	8,562,681	12,663,511	15,806,985
Others	13,335	89,435	167,210	169,665	6,333
Total (all countries)	10,103,540	7,593,786	8,729,891	12,833,176	15,813,318

Data obtained from Statistics Canada and the U.S. Census Bureau (U.S. Department of Commerce) (2008).

As the responsibility for quality assurance programs increases with the value of imported soy-based commodities, these trends further reflect a strict requirement for the adequate monitoring of materials imported to Canadian soils, the surrounding environment and the food-webs they are ultimately released to.

2.1.3.0. RICE PROTEIN CONCENTRATE

2.1.3.1. PROPERTIES

The Codex Standard For Rice (198-1995) does not include an international definition of “rice protein,” “rice gluten,” “rice protein concentrate,” or “rice bran protein isolate,” nor does it include any further reference to any protein source coming from rice. For that reason regulation of these ingredients is poor, making source security a global issue.

Rice proteins are extracted from different parts of the grain, including the polish and the endosperm, predominantly from areas between starch granules (Cagampang *et al.*, 1966; Lasztity, 1996). Rice proteins are also produced from rice bran, a milling co-product of rice production that is also sought for its oils. Defatted bran residues contain up to 15.4% protein (Hamada, 2000).

Due to their oil binding and air-water interface stabilizing properties, rice bran protein concentrates (RBPC) produce stable emulsions under conditions of high salt and sugar concentrations (Chandi and Sogi, 2006). Nevertheless, their emulsifying properties are lower than that of SPIs (Adebiyi *et al.*, 2007). The macromolecular disulphide complexes formed by glutelin fractions are able to form stable gels at low concentrations (Agboola *et al.*, 2005). For this reason their applications are wide-reaching; they’re found in weaning products, baked goods, whipped toppings, salad dressings and dry mixes (Chandi and Sogi, 2006).

2.1.3.2. EXTRACTION PROCESS

Defatted ground rice is used in protein extractions, as free-fatty-acid-formation and oxidation are common risks associated with the handling and storage of whole grain rice (Adebiyi *et al.*, 2007). Historically, alkaline solutions were regularly employed in RBPC extraction processes (Cagampang *et al.*, 1966; Lynn, 1969; Chen and Houston, 1970; Lew *et al.*, 1975; Connor *et al.*, 1976; Barber and De Barber, 1997; Betschart *et al.*, 1977; Juliano, 1985; Bera and Mukherjee, 1989; Gnanasambandam and Hettiarachchy, 1995). The well accepted Osborne method involved sequential extractions of water, dilute salt, dilute alkali and 70%-ethanol solutions (Padhye and Salunkhe, 1979). These conditions were later found to alter the nutritional integrity of the product, compromising the amino acid profile by producing anti-nutritional factors such as lysinoalanine (Otterburn, 1989; Cheftel *et al.*, 1985).

More recent efforts have shown that methods applying the use of enzymes, typically carbohydrases, α -amylase, hemicellulase, cellulose, pectinase, protease P, phytases and/or xylanases (**Figure 2.5**), are more appropriate for rice bran protein extractions, without detriment to the nutritional qualities of the resultants (Ghose and Halder, 1969; Mudgett *et al.*, 1978; Grossman *et al.* 1980; Ansharullah *et al.*, 1996; Shih and Daigle, 1997; Shih *et al.*, 1999; Hernandez *et al.*, 2000; Tang *et al.*, 2003).

Figure 2.5: Process of extracting protein isolate from defatted rice bran (Wang *et al.*, 1999)

Other extraction processes use defatted rice flour. The Van Der Borgh *et al.* (2006) method employs size-exclusion high-performance-liquid-chromatography (SE-HPLC) with a mobile phase of 2.0% sodium dodecyl sulfate (SDS) (w/v). There, 90.7 mg flour produced 5.0 mg proteins on a dry matter flour basis with: (i) 2.0% (w/v) SDS; (ii) 2.0% SDS/6.0 M urea/0–1.5% (w/v) dithiothreitol (DTT); (iii) 2.0% SDS/6.0 M urea/1.0% 2-ME; (iv) 20–100 mM NaOH (5.0 ml) (Van Der Borgh *et al.*, 2006). At an ambient temperature, suspensions were subjected to 1 hour of shaking (150 rpm). The proteins were recovered by subsequent centrifugation (2800g, 15min) (Van Der Borgh *et al.*, 2006).

2.1.3.3. MAIN FRACTIONS

Rice proteins consist of soluble and insoluble protein/glycoprotein fractions. Within the soluble fractions are water-soluble albumins, along with salt-soluble globulins and ethanol-soluble prolamins. Albumins and globulins are found within the aleurone layer of the bran. The remaining fraction consists of insoluble glutelins (Wang *et al.*, 1999). Agboola *et al.* (2005) found Australian brown rice cultivars to contain (Table 2.11) predominately glutelin, followed by globulin, albumin and prolamine.

Table 2.11: Profile of Rice Protein Fractions.

Fraction	Yield (g/100g flour)	% of total	Protein Content %
Albumin	0.42	6	78.14
Globulin	1.31	15	90.41
Glutelin	5.84	75	95.63
Prolamine	0.21	2.7	92.00

Data obtained from Agboola *et al.*, 2005.

Chandi and Sogi (2007) observed total protein yields of up to 85.71%. Ratios of albumin (24 to 96 kDa), globulin (23 to 119 kDa), prolamin (23 to 28 kDa) and glutelin (22 to 88 kDa) were 11:10:4:75 and 16:13:4:67 for two brown rice cultivars, Basmati 386 and HBC 19 (Chandi and Sogi, 2007).

Owing to its hydrophobic, intermolecular hydrogen and disulfide bonding properties, glutelin is the major storage protein of rice (Hamada, 1996; Juliano, 1985). Two covalently linked major polypeptide subunits have been identified: one basic (19–25 kDa) and one acidic (30–39 kDa) (Juliano, 1985; Kagawa *et al.*, 1988; Kishimoto *et al.*, 1999; Shih *et al.*, 2004; Van Der Borgh *et al.*, 2006). A consequence of these hydrophobic and disulfide bonding interactions is the production of exceedingly large macromolecular complexes that limit their functional uses (Utsumi, 1992; Katsube-Tanaka *et al.*, 2004; Van Der Borgh *et al.*, 2006).

2.1.3.4. NUTRITIVE APPLICATION

Digestibility of rice proteins is greater than 90% (Wang *et al.*, 1999). Consisting of a high protein (70-76%) and lipid (15%) content, rice bran proteins have shown promise with high nutritional value (NRC, 1989; Kennedy & Burlingame, 2003). Extending to fish nutrition for its performance in digestibility trials (Palmegiano *et al.*, 2007; Rónyai and Gál, 2005; Sullivan and Reigh, 1995; Ufodike and Matty, 1983), RBPI surpasses all other cereal-based protein and vegetable protein product sources with the exception of WG (Palmegiano *et al.*, 2006) and SPI. Apparent protein digestibility values of up to 90.93% were reported by Palmegiano *et al.* (2006) in Rainbow Trout (*Oncorhynchus mykiss*). Ufodike and Matty (1983) recorded similar results at 88.00% apparent protein digestibility in Mirror Carp (*Cyprinus carpio*) (Ufodike and Matty, 1983; Palmegiano *et al.*, 2006).

Rice proteins are also considered hypoallergenic and hypocholesterolemic (Chrastil, 1992; Tsuji *et al.*, 2001). For these reasons, rice proteins have been the predominant source of protein in many commercially available infant fortified weaning formulations (Burks and Helm, 1996). High in valine, histidine and tyrosine, RBPI is in accordance with the essential amino acid requirement established by the FAO/WHO/UN Expert Consultation (1985) on human growth (<2-years of age) energy requirements, but is limited by leucine, lysine, isoleucine, threonine and tryptophan. For 2 to 5-year-old children, RBPI is closer to meeting the complete requirements for essential amino acids, limited there by only lysine and threonine (Wang *et al.*, 1999; FAO/WHO/UN 1985). Consequently, rice-based proteins pose a great vulnerability to environmental contamination and/or fraudulent processing practices as their intended end-product consumers are within a highly sensitive segment of the population.

Protein extracts coming from rice bran are higher in lysine content than endosperm protein. According to Juliano (1985), rice bran proteins contain more lysine than any other cereal bran protein source. Chandi and Sogi (2006) further demonstrated that rice bran proteins display a richer lysine profile than casein, whole grain wheat and corn (Kik, 1957; Chandi and Sogi, 2006).

In comparison with SPI and casein (Table 2.12), RPI contained similar or higher levels of histidine, arginine, isoleucine, valine, methionine, tyrosine and tryptophan (Chandi and Sogi, 2006).

Table 2.12: Amino Acid Profiles of Rice Bran Protein Isolate (RBPI), Casein and Soy Protein Isolate (SPI).

Amino Acid	RBPI (g/100g)	Casein (g/100g)	SPI (g/100g)
ASP	9.22	7.57	12.04
THR	4.26	4.44	3.65
SER	4.72	5.53	5.11
GLU	14.40	22.83	20.68
PHE	5.30	5.41	6.33
GLY	6.22	1.92	4.14
ALA	7.03	3.24	4.14
CYS	1.84	0.05	5.47
VAL	7.26	7.21	1.34
MET	2.53	3.12	1.34
ILE	4.49	5.89	4.99
LEU	8.53	10.09	8.27
TYR	3.80	5.29	3.89
HIS	3.34	3.24	2.80
LYS	5.41	8.53	6.33
ARG	10.25	3.96	8.03
TRP	1.38	1.68	1.46

Data obtained from Morita and Kiriya (1993) and Wang *et al.* (1999).

Moreover, as they are colourless and bland to the taste, rice bran proteins are also used to produce protein-based edible films that may serve as **carriers** for nutritional additives, antibiotics and antimicrobials (Chrastil, 1992; Shih *et al.*, 1996; Czechowski & Stoodley, 2002; Adebisi *et al.*, 2006). Because they contain a relatively high asparagine and glutamine content, rice proteins further demonstrate flavour-enhancing properties that might eventually replace monosodium glutamate (MSG) in Asian cuisine (Hamada *et al.*, 1998).

2.1.3.5. INDUSTRY OVERVIEW

As no internationally regulated standard definition for protein extracts coming from rice bran or flour exists, detailed trade data are limited. Moreover, in Canada, rice proteins are not classified within the Harmonized System of trade commodities. Nevertheless, industry estimates can be made by examining the commodities from which rice protein extracts are produced: rice flour and rice bran.

In terms of trade, Thailand and China are the world's greatest exporters of rice flours, with 2005 values of \$54,225.89 (1000 US \$) and 9,261 (1000 US \$), respectively. Top rice bran exporters are the United States and India, estimated at \$14,494 (1000 US \$) and 11,850.15 (1000 US \$) in 2005 (FAO, 2008). Canada is a leading global importer of these products, ranking 6th for rice bran and 7th for rice flour (FAO, 2008).

Import trends from 2002-06 were characterized by negative trade balances in Canada, reflecting a relatively consistent reliance on imported rice products such as rice flour, rice bran and rice sharps (Table 2.13). Conversely, trade balances remained positive within the United States during that time (Table 2.14).

Combined with the overall lack of legislative or regulatory requirements for rice-based protein products, this considerable reliance on foreign suppliers for such products creates a significant Canadian ecological vulnerability to environmental pollutants.

Table 2.13: Canadian Trade Balance - Summation of HS 2302.20 – Rice Bran, Sharps and Other Residues (Whether Pelleted or Not); HS 1102.30 – Rice Flour. Value in Thousands of Canadian Dollars.

Rice Flour (HS 1102.30)	2002	2003	2004	2005	2006
Exports	3,362,383	3,052,241	2,484,245	2,243,324	3,284,923
Imports	5,562,018	5,614,450	6,231,766	5,996,289	5,913,151
Trade Balance	-2,199,635	-2,562,209	-3,747,521	-3,752,965	-2,628,228
Rice Bran (HS 2302.20)					
Exports	31,737	34,756	248,241	543	22,397
Imports	2,691,517	2,412,122	2,196,349	2,028,411	1,724,850
Trade Balance	-2,659,780	-2,377,366	-1,948,108	-2,027,868	-1,702,453
Sub-Total					
Total Exports	3,394,120	3,086,997	2,732,486	2,243,867	3,307,320
Total Imports	8,253,535	8,026,572	8,428,115	8,024,700	7,638,001
Trade Balance	-4,859,415	-4,939,575	-5,695,629	-5,780,833	-4,330,681

Data obtained from Statistics Canada and the U.S. Census Bureau (U.S. Department of Commerce) (2008).

Table 2.14: United States Trade Balance - Summation of HS 2302.20 – Rice Bran, Sharps and Other Residues (Whether Pelleted or Not); HS 1102.30 – Rice Flour. Value in Thousands of Canadian Dollars.

Rice Flour (HS 1102.30)	2002	2003	2004	2005	2006
Exports	5,747,955	4,983,651	6,478,352	5,770,890	5,937,762
Imports	8,990,896	8,065,150	7,316,514	7,540,468	9,335,656
Trade Balance	-3,242,941	-3,081,499	-838,162	-1,769,578	-3,397,894
Rice Bran (HS 2302.20)					
Exports	12,925,000	17,564,346	19,271,978	17,560,465	21,798,198
Imports	4,014,836	3,138,874	2,138,315	449,201	323,055
Trade Balance	8,910,164	14,425,472	17,133,663	17,111,264	21,475,143
Sub-Total					
Total Exports	18,672,955	22,547,997	25,750,330	23,331,355	27,735,960
Total Imports	13,005,732	11,204,024	9,454,829	7,989,669	9,658,711
Trade Balance	5,667,223	11,343,973	16,295,501	15,341,686	18,077,249

Data obtained from Statistics Canada and the U.S. Census Bureau (U.S. Department of Commerce) (2008).

2.2.0. CLASSIFICATION AND SELECTION

2.2.1.0. VEGETABLE PROTEINS: PROTEIN CONTENT DETERMINATION

Success in trade and application of VPPs in foodstuffs is heavily dependent on the quality of the initial raw material. Frequently the total protein content (crude protein) of a given sample is used as the benchmark against which its quality is ultimately assessed. Moreover, the United States Food and Drug Administration's (USFDA) Nutrition Labeling and Education Act (1990) requires that crude protein values be declared on all food labels (Simonne, 1997). Hence, crude protein is often the single most influential factor in determining a raw material's selling price (Delwiche, 1998; Wrigley, 1998).

In today's fast-growing food manufacturing sectors, analytical chemistry has taken a central role in the quality assurance process, providing cost-effective and time-sensitive quantifiable results that may be extrapolated to the value of a given raw material. The associated technologies have evolved to a degree of efficiency never before seen; one where truck-loads of raw materials may be assessed by a prospective buyer simply by analyzing representative samples of the bulk supply, literally while the driver waits outside the receiving zone. Spectroscopic methods including near infrared (NIRS) allow for quality assessment results to be obtained within minutes upon receiving (Williams, 1998). As a direct result of this level of efficiency, organizations that rely on swift execution have adopted these technologies industry-wide in the food and drug sectors.

2.2.1.1. WHEAT GLUTEN

Easily obtaining meaningful crude protein values from VPPs is challenging, due to the well-accepted fact that it is nearly impossible to completely extract and subsequently prepare pure protein for analysis (Williams, 1998). As such, the protein content of gluten samples is typically assessed through an estimation of the total nitrogen content (ΣN) present. Protein and nitrogen contents are especially correlated in methods of analysis pertaining to agriculture products (Sundqvist, 1973). As the total nitrogen content in a foodstuff is the summation of nitrogen groups coming from the protein, amino acid and peptide groups, ΣN is multiplied by a nitrogen-

to-protein conversion factor that is determined by the amino acid profile of the raw material (Chang, 1994).

Through the Dumas Combustion Nitrogen Analysis (CNA) method (Dumas, 1831), samples are incinerated in a combustion tube and the measured nitrogen content is multiplied by a conversion factor of 6.25. As amino acids contribute roughly 16% of the protein weight, the conversion factor for food products is set at 6.25 (Simonne, 1997; FAO, 2005). Due to a slightly greater N-amino contribution (17.54%), 5.7 is the reference ~~conversion~~ factor used in the wheat and flour industry (Simonne, 1997; Williams, 1998). The alternative reference process is the Kjeldahl (1883) method, which involves oxidation by means of boiling corrosive/hazardous chemicals (Jones, 1992; Buckee, 1994; Simonne *et al.*, 1995; Etheridge *et al.*, 1998; Williams, 1998).

Despite non-equivalence in values obtained by the two approaches, with results from the Dumas method consistently displaying levels 1.4% higher, the empirical conversion factors used internationally are based only on the Kjeldahl method (Thompson *et al.*, 2002). The degree to which laboratories employing different methods are at risk for biasing the protein levels they report is currently a pervasive debate.

Accordingly, FAO/WHO/UN approved methods of analysis and sampling include (FAO, 1987):

- Gerhardt semi-micro Kjeldahl method (AOAC 979.09 - 2005) for vital wheat gluten and devitalized wheat gluten (Wheat protein in grain N x 5.7)
- Gerhardt semi-micro Kjeldahl method (AOAC 920.87 - 2005) for solubilized wheat protein (wheat protein in flour N x 5.7).

In recent years the conversion factor of 5.7 has been replaced by 6.25. In the case of vital and devitalized WG, the CODEX-STAN 163-1987 Rev 1-2001 norm sets the minimum protein content (N x 6.25 on dry substance) at 80%, excluding added vitamins, minerals, amino acids and optimal ingredients such as enzymes, carbohydrates, etc.

The final decision following a 2001 proposed revision (Codex Alinorm 01/37A) of the *Codex Standard for Wheat Gluten* was that despite an ongoing debate, that "no consensus was reached

between member countries on the protein conversion factor" and that "although most countries expressed the view that the protein conversion factor of 5.7 was the most accurate for calculating protein content...[that] a review of scientific literature on the issue indicated that...the value of 6.25 could also be accurate for the water soluble proteins." It was thus decided that 6.25 would be retained as the official conversion factor (FAO, 2001).

2.2.1.2. SOY PROTEIN ISOLATE

The same preparation and extraction complications seen in WG-determination regularly affect SPI analyses. Hence, the protein content of a given sample is similarly assessed through an estimation of its total nitrogen content.

According to the Kjeldahl reference method for protein determination, a conversion factor of 5.71 should be applied to SPI materials, while 6.38 should be used for cow's milk proteins and their partial hydrolysates. This discrepancy is the result of an investigation by the International Dairy Federation (IDF) (ref. Codex ALINORM 05/28/26, Appendix IV (A)) that determined the default conversion factor of 6.25 factor to "result in both underestimation by about 2-3 % of the actual protein content for milk, and serious overestimation of the protein content of proteins from plant sources. In the case of soy protein, the actual protein content would be overestimated by approximately 9 %" (CX/NFSDU 05/27/6-Add.2 October 2005).

CODEX STAN 175-1989 for SPPs advocates the standard use of N x 6.25 for raw materials, as well as finished soy-based infant formulas (FAO, 1989).

2.2.1.3. RICE PROTEIN CONCENTRATE

Just as no internationally regulated definitions for rice proteins exist, neither do the analytical standards used to assess their protein determination.

According to the FAO, based on a Kjeldahl conversion factor of 5.95, the unofficial yet commonly assigned protein value of milled rice is 7 percent (WHO 1985; Greenfield & Southgate, 1992; Juliano, 1993). When applied to nutritional studies; however, the factor 6.25 is

used to make diets isonitrogenous with the standard proteins (Juliano, 1993). For that reason, the majority of studies use a conversion factor of 6.25 (Bressani *et al.*, 1971; Eggum & Juliano, 1973; Hegsted & Juliano, 1974; McDonald, 1974; Murata *et al.*, 1978; Juliano, 1993; Palmegiano *et al.*, 2006; Palmegiano *et al.*, 2007).

2.2.2.0 DUPLICITY: NITROGEN-BEARING COMPOUNDS

As VPPs are selected and sold based on their overall protein content, it is in a supplier's interest to bring the highest quality ingredients to sale. Knowing that their goods will most likely be evaluated using the Kjeldahl method to estimate the total organic nitrogen content, the blind assumption of both North American vendors and customers has, until recently, been that only protein and its subunits could be significant contributors to that overall measurement.

The severe limitation of this widely accepted practice was consummately revealed during the global food recalls of 2007. When the University of Guelph Laboratory Services confirmed the presence of melamine and cyanuric acid in raw material samples, as well as in the urine and kidneys of affected pets, several questions were raised, but the most poignant was 'why.' Scientists were baffled in understanding what possible service melamine and cyanuric acid could provide, and how integrating these compounds into raw materials might produce any benefit for a vendor. It was in the chemical analysis of these compounds that would eventually expose their surreptitious roles, and subsequently the criminal actions of those parties who added the contaminating substances.

2.2.2.1. MELAMINE & ITS INTERMEDIATES

Melamine [CAS: 108-78-1] is a chemical substance that is used to produce durable plastics, countertops, fabrics, glues and flame retardants. Melamine is also used to produce fertilizers.

Chemical synonyms include: 1,3,5-triazine-2,4,6(1H,3H,5H)triimine; 1,3,5-Triazine-2,4,6-triamine; 2,4,6-triamino-1,3,5-triazine; 2,4,6-triamino-s-triazine; 2,4,6-triamino sym-triazine; AERO; cyanuramide; cyanuric triamide; cyanurotriamide; cyanurotriamine; Cymel; hicophor pr;

isomelamine; Melamine; s-triaminotriazine; Sym Triaminotriazine; teoharn; theoharn; triaminotriazine; virset 656-4 (Chemfinder, 2008).

Melamine is primarily restricted to industrial chemical manufacturing. The average consumer would be expected to come into contact with melamine only through products where melamine is included in a polymer matrix (OECD, 2002). With a structure of $C_3H_6N_6$ (Fig. 2.6) melamine possesses a relatively high nitrogen content that accounts for 67% of its overall molecular weight (IUCLID, 2000).

To a limited, yet significant degree, melamine-derived compounds coming from the 2-chloro-4,6-di(dialkylamino)-1,3,5-triazines are categorized as a class of organic compounds with a wide range of biological applications (Vanya, 2004): anti-angiogenesis (Ono, 1996), anti-tumour activity for breast (Goldin, 1979) and ovarian (Foster, 1986) cancer treatment, effective treatments for menopausal symptoms and postmenopausal osteoporosis (Cosman, 2002), anti-metastatic activities (Maeda, 2000), herbicidal effects (Drabel, 1991), as well as serving as chemoselective building blocks for dendrimers (Zhang, 2001), porous hydrogen-bonded networks (Sauriat-Dorizon, 2003) and chromophores in probes for the absolute configuration determination by circular dichroism (Mason, 1983).

Figure 2.6: Molecular Structure of Melamine [CAS: 108-78-1] (IUCLID, 2000)

Melamine derivatives are often prepared from 2,4,6-trichloro-1,3,5-s-triazine (cyanuric chloride). Widely applied to supramolecular chemistry, stepwise substitutions are made with appropriate amines (Thurston, 1951).

Other straightforward production methods include prolysis of neat cyanamide (Fig. 3.2.2) which self-reacts to produce dicyandiamide and eventually melamine (Bann, 1958; May, 1959; Finkel'shtein, 1961; Takimoto, 1963; van der Plaats, 1981; Costa, 1988; Pankratov, 1989; Stoner 1991; Belsky *et al.*, 1996).

Figure 2.7: Prolysis of Neat Cyanamide to Produce Melamine (Belsky *et al.*, 1996)

Belsky *et al.* (1996) further produced melamine in high temperature water through a series of steps (Fig. 2.8) involving dimerization of cyanamide to dicyandiamide, cyclization to the cyclic azines and hydrolysis of each compound.

Figure 2.8 Reactions of cyanamide, dicyandiamide & related cyclic azines (Belsky *et al.*, 1996)

Korolev *et al.* (2006) demonstrated complex experimental methods involving the thermal decomposition of *C*-nitro and *N*-nitro-1,2,4-triazoles to produce numerous compounds, including melamine.

2.2.2.2. CYANURIC ACID & ITS INTERMEDIATES

Cyanuric acid [CAS: 108-80-5], an odorless white crystalline solid, has a structure of $\text{C}_3\text{H}_3\text{N}_3\text{O}_3$ that although not as rich as melamine, contains 33% nitrogen by weight (Chemfinder, 2008). Cyanuric acid can exist in two tautomeric forms (Fig. 2.9) (Kuznesof, 2004).

The planar three-fold symmetry of cyanuric acid has been confirmed by neutron diffraction and X-ray crystallographic studies (Coppens & Vos, 1971).

Figure 2.9: Tautomers of Cyanuric Acid (Kuznesof, 2004)

Industrial manufacturing of cyanuric acid involves pyrolyzing urea, which dissociates into isocyanic acid and ammonia. Isocyanic acid trimerizes to cyanuric acid, which is then purified, and subjected to acid digestion, converting acyclic impurities to carbon dioxide and ammonia. Additional reactions between urea and isocyanic acid lead to equilibrium amounts of ammonium cyanate, along with the intermediates biuret and triuret (Wojtowicz, 1993; USFDA, 1997; Kuznesof, 2004).

Amination of biuret and triuret results in cyclic aminotriazine by-products (**Fig. 2.10**), primarily ammelide, ammeline, and minor amounts of melamine (Kuznesof, 2004; Boitsov, 1962).

Figure 2.10: Manufacturing cyanuric acid: aminotriazine by-products (Kuznesof, 2004)

These remaining aminotriazine by-products might account for 20% of the crude recovery and in an industrial manufacturing setting would be re-processed and converted to additional cyanuric acid and ammonia (Kuznesof, 2004).

2.2.2.3. INTERACTIONS BETWEEN MELAMINE & CYANURIC ACID

Toxicological data aside, by no means should melamine, cyanuric acid or any of their intermediates be found in human or animal food products, at levels above those that would be naturally present in the general environment. Moreover, **neither** compound is approved by the USFDA or any governing body, for use in any food products, regardless of industry.

Despite this, melamine and its analogues were found in samples of vegetable proteins and have been directly implicated in the 2007 recalls. Given that neither compound appears especially minacious alone, researchers now believe that reactions between them are the mechanism for toxicity (AVMA, 2007; Puschner *et al.*, 2007; UOG, 2007; USFDA, 2007). Accordingly, the experimental design of this study includes analytical testing protocols for both melamine and cyanuric acid composites in VPP-evaluations and strongly recommends them as part of a comprehensive quality assurance program.

At a pH of 5.8, melamine has been shown to react with cyanuric acid, forming an insoluble compound consisting of 50.58% cyanuric acid by weight (Nelson, 1967). Whitesides *et al.* (1995) confirmed this reaction to be a non-covalent synthesis of 1:1 hydrogen-bonded adducts between melamine and cyanuric acid to form rosette-shaped structures. Moreover, this adduct is a highly hydrogen-bonded solid of limited solubility in organic solvents (Ranganathan, 1999).

As Mathias *et al.* (1994) and Whitesides *et al.* (1995) demonstrated, yielding adducts in water or alcohol solutions will indeed precipitate the desired result, but not crystals appropriate for x-ray crystallography. Hence, following the work of Rao *et al.* (1993), Ranganathan *et al.* (1999) used hydrothermal conditions, up to 180°C to separate out representative rectangular plate-like crystals. There the 1:1 adduct had an asymmetric unit consisting of melamine and cyanuric acid molecules, with a respective molecular structure of $(C_3H_3N_3O_3) : (C_3H_6N_6)$. There, N-H...O and N-H...N hydrogen bonds yielded a clear rosette-shaped hexamer (Ranganathan *et al.*, 1999).

Later supported by Herbstein (2003), this observation amended the work of Wang *et al.* (1990), whose concept of a melamine/cyanuric acid complex displayed one-dimensional tapes rather than rosette structures, with a formula of “melamine cyanuric acid trihydrochloride” ($\text{MCA} \cdot 3\text{HCl}$). Herbstein (2003) represented the interaction as “diprotonated melamine cyanuric acid dichloride dehydrate,” with a molecular structure of $(\text{C}_3\text{H}_8\text{N}_6)^{2+} \cdot \text{C}_3\text{H}_3\text{N}_3\text{O}_3 \cdot 2\text{Cl}^- \cdot 2\text{H}_2\text{O}$ (Ranganathan *et al.*, 1999; Herbstein, 2003).

Perdigão *et al.* (2006) prepared a hydrogen-bonded bimolecular network formed between melamine and cyanuric acid by a surface-based self-assembly process. The complex formed was held together by hydrogen bonds (Fig. 2.11) (Perdigão *et al.*, 2006).

Figure 2.11: Cyanuric Acid/Melamine H-Bonded Bimolecular Network (Perdigão *et al.*, 2006)

Under different conditions, Perdigão *et al.* (2006) showed that melamine/cyanuric acid bimolecular arrays could be formed on $\text{Ag/Si}(111)\sqrt{3} \times \sqrt{3}$ surfaces, giving a more distinctive honeycomb array than a rosette. Staniec *et al.* (2007) reproduced these results and further

managed to create a larger superstructure with reduced “pore” dimensions on gold $\text{Au}(111)$ film surfaces. Similar results were achieved by Xu *et al.* (2007).

2.2.2.4. TOXICOLOGICAL PROFILE & FOOD RECALLS

In response to the 2007 outbreak of illnesses caused by VPPs contaminated with melamine, cyanuric acid, and related compounds such as ammelide and ammeline, a risk assessment outlining the implications to human health was prepared by the United States Food and Drug Administration’s Center for Food Safety and Applied Nutrition, working in collaboration with the Food Safety and Inspection Service (FSIS) of the Department of Agriculture, the Centers for Disease Control and Prevention (CDC), the Environmental Protection Agency (EPA), and the Department of Homeland Security (DHS). The report evaluated the dangers associated with eating contaminated foodstuffs, including chicken, pork, eggs and fish, that either directly consumed adulterated protein samples and/or were coming from parent animals that had consumed tainted products (USFDA, 2007).

According to the USFDA, the results of those efforts confirmed that (USFDA, 2007):

...Based on currently available data and information, the results of the safety/risk assessment indicate that the consumption of pork, chicken, domestic fish, and eggs from animals inadvertently fed animal feed contaminated with melamine and its analogues is very unlikely to pose a human health risk...

Melamine toxicity in mammals has been described as low, with a lowest lethal dose (LD_{50}) of 3161 mg/kg in rats and 3900 mg/kg in mice (USFDA, 2007; IUCLID, 2000; OECD, 2002; Melnick *et al.*, 1984). Moreover, no metabolism or tissue accumulation of melamine has been demonstrated in mammals. Lipschitz and Stokey (1945) recovered up to 85% of orally-administered melamine (approximately 120 mg/kg) in canine urine samples collected within 24 hours. Further studies with rats demonstrated 90% recovery of orally-administered ^{14}C -labeled melamine in urine samples collected within 24 hours (Mast *et al.*, 1983; Puschner *et al.*, 2007). Reproductive and developmental toxicity no-observed-adverse-effect-levels (NOAELs) of 1060 mg/kg bw/day were identified in orally-administered fetal rats and 400 mg/kg bw/day in maternal rats (USFDA, 2007; IUCLID, 2000; OECD, 2002).

To the contrary, older research suggests that melamine might pose great risks than assessed today. For instance, Mackenzie (1966) found that doses of 250 mg/kg fed to sheep lead to complete food refusals and weight loss in the majority of animals, while a smaller group who were also fed low-quality hay as a complement, died. Newton and Utley (1978) also observed melamine-induced food refusal in 4/6 steers at concentrations of 100 mg/kg. Further research by Clark (1966) confirmed that ingestion of 250 mg/kg of melamine was lethal to some sheep in the study; necropsies of affected animals revealed aggregates of densely-packed melamine crystals in the kidneys, along with severe tubular damage.

According to the USFDA, “observed effects include: reduced food consumption, body weight loss, bladder stones, crystalluria, epithelial hyperplasia of urinary bladder, and lowered survival rate. However, no kidney failure or clinical symptoms of kidney failure were observed from these studies, including in a dog study” (USFDA, 2007; Clayton, 1982; USDHHS, 1983).

Worst-case scenario assumptions were assessed with intake levels of 150-µg of melamine compounds per person per day or 2.5 µg/ kg bw/day for a 60 kg individual. These values were based on the following parameters: melamine compounds are present in all solid food at a level of 100 ppb (µg/kg); a person consumes 3000 g (or 3 kg) of food per day, of which 1500 g is from solid food (USFDA, 2007).

In comparison to the calculated Tolerable Daily Intake (TDI) of 0.63 mg/kg bw/day for melamine, the report confirms that (USFDA, 2007):

In light of the calculation that the highest estimate of exposure (i.e., 2.5 µg/ kg bw/day for a 60 kg individual) is more than two orders of magnitude lower than the TDI (0.63 mg per kg per day), which are (*sic*) based on reasonable assumptions and a 100-fold safety factor, the consumption of pork, poultry, eggs and domestic fish is very unlikely to pose a human health risk.

Similar risk assessments and toxicological studies were preformed at various animal research facilities in Canada, the United States and Europe to determine the associated health implications to dogs and cats eating pet foods containing VPPs polluted with melamine and its analogues (UOG, 2007; UCD, 2007; AVMA, 2007; MSU, 2007; AAVLD, 2007; EFSA, 2007).

Researchers at the University of Guelph Laboratory Services confirmed severe toxicological agents at play, with several cases of acute renal failure in cats that had consumed tainted pet food. Further analyses involving microscopic imaging techniques revealed a series of insoluble crystals in the urine samples of those cats (**Fig 2.12**) (UOG, 2007).

Figure 2.12: Microscopic images of crystals found in urine of affected pets (UOG, 2007)

Subsequently, by adding melamine and cyanuric acid to samples of clean cat urine, University of Guelph Laboratory Services successfully produced insoluble crystals *in vitro* (**Fig. 2.13**) that Fourier Transform Infrared Spectroscopy (FTIR) found to be consistent with those found in the urine of affected cats (UOG, 2007).

Figure 2.13: Melamine/Cyanuric Acid crystals produce in vitro (UOG, 2007)

Cyanuric Acid has been shown to pose no risks to human health at concentrations of 20-25 mg per liter, a pH within 7.2-7.7, along with free available chlorine concentrations in the range of 1.0-1.5 mg per liter. Acute oral toxicity LD₅₀ values for rats have been identified at 0.75 gm per kg and higher. Cyanuric acid supplementation in rat and mice studies caused bladder stones and associated complications at 700 mg/kg and 2,200 mg/kg, respectively (Hammond *et al.*, 1986). Comparatively, a 50 kg human ingesting 100 ml of water, with a concentration of 25 mg per liter of cyanuric acid would ingest less than 1/100,000 that LD₅₀ amount (Canelli, 1974; Mazaev, 1964).

Researchers postulated that the above described cyanuric acid/melamine adduct reactions were the likely origin of the insoluble crystals found within the kidneys and urine of euthanized cats, as neither compound alone lead to similar symptoms at those concentrations present (UOG, 2007).

The USFDA safety/risk assessment confirmed this theory in stating (USFDA, 2007):

...It has been hypothesized that melamine may interact synergistically with its three analogues, but no studies have been conducted that specifically test this hypothesis. Very preliminary work suggests that if it does occur, the formation of lattice crystals, particularly between melamine and cyanuric acid, takes place at very high dose levels and is a threshold and concentration dependent phenomenon that would not be relevant to low levels of exposure. Although still under investigation, it now appears that the

combination of melamine and cyanuric acid has been linked to the acute renal failure in cats and dogs that have eaten the suspect pet foods...

However, one area where the USFDA's conclusion was inconsistent with empirical findings was in its assumption that melamine and its analogues were to be present in equal levels of potency (USFDA, 2007):

...Melamine and melamine analogues (cyanuric acid, ammelide and ammeline) are assumed to be of equal potency and will be referred to collectively in this assessment as melamine compounds (MC). While it is entirely possible that the analogues are more or less potent than the parent compound, melamine, we have no information that assesses the relative potency of the three analogues as compared to melamine; therefore, for the purpose of this interim assessment, we have made an assumption of equal potency...

As discussed above, this does not appear to be the case, with cyanuric acid frequently outweighing the parent melamine compound (USFDA, 2007). Correspondingly, toxicologists at the American Veterinary Medical Association (AVMA) maintained that crystal analyses revealed approximate melamine:cyanuric acid ratios of 30:70 (AVMA, 2007).

A study conducted within the California Animal Health and Food Safety Laboratory of the UC Davis School of Veterinary Medicine, further confirmed that incidences of acute renal failure occurred only when both melamine and cyanuric acid were present together (Puschner *et al.*, 2007). Concentrations within the kidneys were as follows: 496 to 734 mg/kg wet weight of melamine and 487 to 690 mg/kg wet weight of cyanuric acid. Those cases of renal failure were associated with the formation of similar urinary tract-laden fan-shaped birefringent crystals that lead to extensive renal trauma within 12 hours of consuming tainted samples. Those cats were euthanized within 48 hours due to an impossibility of reversing those deleterious effects. Although the LD₅₀ was not determined, histopathologic findings included "crystals primarily within tubules of the distal nephron, severe renal interstitial edema, and hemorrhage at the corticomedullary junction." Moreover, the authors suggested that the combination of melamine and cyanuric acid, each at individual concentrations as low as 32 mg/kg could lead to crystallization and acute renal failure in cats (Puschner *et al.*, 2007).

Hypotheses from the literature suggest that bacterial or photocatalytic degradation might explain the presence of melamine and cyanuric acid in tainted VPPs, helping to explain how both compounds might be present despite the initial inclusion of only one of them. Another possibility has been identified by El-Sayed *et al.* (2006) who isolated novel bacterial *Micrococcus* sp. strain MF-1 that was capable of metabolizing melamine formaldehyde resin as a main carbon and nitrogen source. Biodegradation sequences (Fig. 2.14) revealed general triazine catabolism of melamine, followed by successive deamination, resulting in the formation of cyanuric acid. Decarboxylation produced biuret, which hydrolyzed to produce urea and ammonia, a characteristic of melamine degradation (Cook, 1987; Eaton & Karns, 1991; Wackett *et al.*, 2002).

Similarly, Goutailler *et al.* (2001) applied irradiation and photocatalytic treatments to cyromazine (N-cyclopropyl-1,3,5-triazine-2,4,6-triamine), an insect growth regulator. The first oxidative degradation produced amide-triazines and melamine as the predominant photoproducts. Successive deaminations transformed the melamine into cyanuric acid.

Figure 2.14: Proposed biodegradation pathway of MF by sp. MF-1 (El-Sayed *et al.*, 2006)

Bozzi *et al.* (2004) further observed the production of (Fig 2.15) ammeline, ammelide and cyanuric acid under oxidative conditions of UV-irradiation in the presence of H_2O_2 from melamine.

Figure 2.15: Identified intermediates in the photocatalytic degradation of melamine (Bozzi *et al.*, 2004)

2.3.0. QUALITY ASSURANCE PROGRAMS

2.3.1. ANALYTICAL ANALYSIS OF VEGETABLE PROTEIN PRODUCTS

Prior to the food recalls of 2007, widespread VPP quality assurance programs were limited to careful selection based primarily on protein determination results. The extrapolation of results coming from the Gerhardt semi-micro Kjeldahl and Combustion Nitrogen Analysis DUMAS methods was regarded by many food and drug industry leaders as the standard benchmark against which quality evaluations could be assessed. Quality in that sense was blindly regarded as encompassing a combination of safety, consistency, security, functionality, traceability and sanitation, without considering the limited nature of the tests being employed to actually measure it. Operating from that framework made the idea of adulterated results or the possibility of a deceptive result inconceivable in North America and was thus not considered to any significant extent by the governing bodies overseeing food safety and security. Literature reviews that followed in the months subsequent to the pet food recalls of 2007 revealed that similar instances of fraudulent melamine use were in fact reported by Italian scientists at levels as high as 56% of fish feed samples analyzed (Cattaneo and Cantoni, 1982).

2.3.1.1. NIRS

Near-infrared reflectance (NIRR) technologies have been used extensively in quality assessments of VPPs since the early 1970s. Due to their fast, non-destructive, reproducible and accurate results, the Canadian Grain Commission's Grain Research Laboratory (GRL) employed NIRR methods that could be carried out at terminal elevators on individual railcars carrying commodity loads (Williams, 1998). By the mid-1980s near-infrared reflectance spectroscopy (NIRS) became widely applied throughout agricultural and food production sectors. Based on the distinctive reflection spectra produced by samples exposed to light within a range of 700 to 2500 nm, fast and accurate information could be obtained relating to nutrient composition and digestibility (Murray & Hall, 1983; Murray & Williams, 1987; Murray, 1992; Osborne *et al.*, 1993; van Kempen *et al.*, 1997; Williams, 1999; Deaville & Flinn, 2000; Cozzolino *et al.*, 2002).

Fundamental to the success of these processes is the proper calibration of NIRS instrumentation, through a combination of multivariate mathematical modeling and computerized reference

libraries of previously analyzed samples (Beebe & Kowalsky, 1987; Martens & Naes, 1989; Murray, 1992; Alomar *et al.*, 2003; Johnson, 2001; Cozzolino *et al.*, 2002). In the past 10 years, advances in chemometric software, spectral data transformation and spectrophotometers have minimized interferences such as water and particle size within the NIRS system (Griggs *et al.*, 1999; Murray & Cowe, 2004; Cozzolino *et al.*, 2006).

NIRSs are commonly calibrated to measure nitrogen levels consistent with those obtained by the Kjeldahl or Dumas methods. NIRS results are then extrapolated to crude protein values, an accepted measure of nutritional value. However, van Kempen *et al.* (1997) demonstrated that protein content measured by $N \times 6.25$ does not necessarily correlate with true nutrient digestibility. Concerns were raised over the lack of information provided by NIRS pertaining to the required essential amino acid profile of a given species. Calibrations were successfully made that provided insight in predicting true ileal digestibility (van Kempen *et al.*, 1997). Similar NIRS calibrated nutritive evaluations were performed on various species including ruminants, swine, poultry and rabbits (Aufrère *et al.*, 1996; Park *et al.*, 1997; Givens & Deaville, 1999; Van Barneveld *et al.*, 1999; Xiccato *et al.*, 1999; Garnsworthy *et al.*, 2000; De Boever *et al.*, 2003; Yan & Agnew, 2004). Castrillo *et al.* (2005) used NIRS to predict the chemical composition, apparent digestibility, digestible nutrients and energy content of commercially extruded compound foods for dogs.

Hence NIRSs are powerful tools that are relied upon for fast and accurate quality assurance evaluations of incoming raw materials. Given the natural variation in nutritional composition of those materials from one year to the next, it is imperative that NIRS calibrations are constantly rechecked and updated where necessary (Reeves, 2007). How data are interpreted must also be considered mindfully; as van Kempen *et al.* (1997) demonstrated, blind assumptions of simple nitrogen levels do not necessarily correlate with subsequent fulfillment of a species' true nutritive requirement.

2.3.1.2. GC/MS/MS: USFDA

The first gas chromatographic (GC) column was developed in 1951-1952 by James and Martin. By the late 1950s, GCs were coupled with mass spectrometers (MS), creating a powerful tool for

compound identification (Holmes & Morrell, 1957; Gohlke, 1959). Extensively used today in agricultural, drug testing and environmental sectors, GC/MS principles rely on the behaviour of various compounds within a sample after being vaporized by the instrument. Factors affecting the speed at which a compound will travel through the GC column include its volatility and polarity. Unique spectral peaks are captured by the MS which measures the time elapsed between initial injection and elution, known as the retention time. Spectrograph peak-size is indicative of the quantity of the compound captured. However GC/MS is typically used as a simple confirmation/identification test, rather than a quantification system (QUASU, 2007).

Gas chromatography techniques have been employed in the identification and determination of melamine and cyanuric acid in various matrixes. For the determination of volatile compounds arising from epoxy powder paints during the curing process, Peltonen (1986) successfully identified melamine through GC/MS. Bardalaye *et al.* (1987) used GC in the determination of cyromazine and melamine in Chinese cabbage. Similarly, Toth and Bardalaye (1987) applied capillary gas chromatographic separation with mass spectrometric in the further detection of cyromazine and melamine. Additional work by Yokley and Cheung (2000) employed GC techniques to determine cyromazine and melamine residues in soil samples.

For the determination of cyanuric acid in swimming pool water, surface water, human urine, and simulated air filter samples, Fiamegos *et al.* (2003) developed a highly sensitive, almost-free-from-interferences, GC/MS method.

In response to the urgency surrounding the pet food recalls of March, 2007, the USFDA's Center for Veterinary Medicine rapidly developed testing protocols for the presence of melamine in contaminated wheat gluten samples. In April 2007, the method was extended to identify the presence of cyanuric acid (USFDA, 2007). Aminopterin, the first suspected compound of interest, mostly as a result of unsubstantiated media claims, was shortly thereafter completely eliminated and no longer tested for. With not even the time to properly validate the method, gas chromatography was selected, with tandem mass spectrometry (GC/MS) for identification, supplemented with HPLC-UV for compound quantification (USFDA, 2007). The procedure was applied to 3 substrates: pure raw material wheat gluten samples, dry finished good pet food

products and wet finished goods pet food products. Instrumental parameters permitted a turnaround time of 10.5 minutes per analysis (USFDA, 2007).

To validate the method, however limited, uncontaminated composites of pet food and wheat gluten were spiked with a range of 3-TMS melamine derivatives at various concentrations and analyzed using ~0.500 g samples with splitless injection (USFDA, 2007).

Melamine was successfully detected at 7 µg/g in the pet food and 14 µg/g in wheat gluten composites. The mass spectrum (Fig. 2.16) revealed the molecular ion at $m/z=342$, a methyl group loss at $m/z=327$, and significant ions at $m/z=171$, 99, and 73.

The GC/MS method further successfully detected melamine in the three substrates, detecting melamine at levels as low as 0.01% by weight. At 7.1 minutes, 0.02% melamine-3TMS eluted (Fig. 2.17) (USFDA, 2007).

Figure 2.16: Melamine 3-TMS Derivative Mass Spectrum (USFDA, 2007)

Contaminated raw material samples of wheat gluten revealed 1.2% melamine by HPLC-UV (Fig. 4.3) (USFDA, 2007).

Figure 2.17: GC/MS Chromatograph of Contaminated Cat Food (USFDA, 2007)

Given that melamine displays limited solubility in methanol and is only slightly more soluble in methanol/water mixtures, dimethyl sulfoxide (DMSO) was recommended as a primary solvent followed by methanol or acetonitrile, for more concentrated solutions of melamine. Due to those solubility limitations of melamine in methanol, this USFDA method was described as “intended to be a qualitative screen only” (USFDA, 2007).

Figure 2.18: GC/MS Chromatograph of Contaminated Wheat Gluten (USFDA, 2007)

These methods were expanded in May, 2007, to further screen for cyanuric acid, ammeline and ammeline. The established minimum reporting level (MRL) was set at 10 µg/g for an expanded set of matrixes: wheat gluten, rice protein, corn gluten, and soy protein, wet and dry pet foods, and dry animal feeds (USFDA, 2007).

Rather than using methanol, samples were extracted using a mixture of acetonitrile/water/diethylamine. Moreover, the analytes were subsequently converted to trimethylsilyl derivatives for analysis (USFDA, 2007). Retention times of those compounds were measured at 9.2 minutes for the cyanuric acid tri-TMS derivative, 10.0 minutes for the ammeline tri-TMS derivative, 10.7 minutes for the ammeline tri-TMS derivative and 11.2 minutes for the melamine tri-TMS derivative (**Fig. 2.19**) (USFDA, 2007).

Figure 2.19: GC/MS Chromatogram of TMS derivations of melamine analogues (USFDA, 2007)

2.3.1.3. GC/MS/MS: LIMITATIONS

Gas chromatography presents several limitations, most notably that it is heavily temperature dependent, requiring thermally stable, volatile compounds. Consequently, in the context of monitoring VPPs for melamine and its analogues, gas chromatography presents significant challenges.

From the outset, in preparing samples for the gas chromatograph, the sample must first undergo a process of extraction into an organic solvent. As discussed above, the USFDA's methanol extract / gas chromatograph combination is challenging as compounds like melamine and aminopterin are not easily extractable; melamine for instance is insoluble in methanol (USFDA, 2007).

Moreover, injecting aqueous samples into a gas chromatograph presents further challenges as aqueous or very polar materials are aggressive to the gas chromatographic columns and will degrade quickly. As one is forced to rely on a sample's volatility and its behaviour in a chromatographic column when subjected to heat, if the compound is not volatile, and is rather polar or thermally labile, it will break down (Katz & Strusz, 1969; Spengler & Hamroll, 1970; Games, 1981; Tamiri & Zitrin, 1987; Croley *et al.*, 2000; Fialkov *et al.*, 2003).

Finally, in addition to the physical challenges faced in applying GC/MS systems in this context, little information is provided that might be used constructively by food safety regulators. A simple positive or negative result does not indicate the extent to which a given compound like melamine is present, at what concentration, and consequently how that might be extrapolated to assess the associated health implications.

2.3.1.4. LC/MS/MS: ADVANTAGES

With the significant limitations presented by GC/MS in the context of screening vegetable protein products for melamine and its analogues, alternative analytical techniques were investigated. As commonly seen in the drug testing industry where they are finding more and more applications, as well as in conventional testing areas such as food safety and environmental monitoring of soil, biological matrices and water quality, liquid chromatographs were considered, (Tswett, 1903; Martin & Synge, 1941; Arpino & Guiochon, 1982; Cairns, 1983; Covey *et al.*, 1986; Garcia & Barcelo, 1993; Le *et al.*, 1996; Careri *et al.*, 1998; Hartig *et al.*, 1999; Lee and Kerns, 1999; Croley *et al.*, 2000; Bobeldijk *et al.*, 2001; Simona *et al.*, 2004; Leermakers *et al.*, 2006), with tandem mass spectrometers (LC/MS/MS).

Based on analysis by liquid chromatography/electrospray ionisation mass spectrometry, Mamone *et al.* (2000) developed a new method for fast and sensitive fingerprinting of gliadins and glutenins in wheat flour. These techniques successfully provided sufficient qualitative and quantitative information about wheat gluten proteins to provide gluten in diets selected for celiac patients (Mamone *et al.*, 2000).

Following the work of Rossi *et al.* (2002), Potter *et al.* (2001) and Rutherford *et al.* (2001), Zhang *et al.* (2004) used robotic liquid-handling technologies in high throughput LC-MS/MS assays to quantify a new bone integrin antagonist in human plasma and urine with great success. Similarly, Patel and Jones (2007) applied LC/MS methods for the quantitative determination of cyanuric acid as the degradation product of sodium dichloroisocyanurate in urine samples. LC/MS/MS screenings for melamine and cyanuric acid follow the same rationale, but because of the nature of the compounds, require different extractions and clean-up protocols due to the molecular structures of the two; one is a basic compound and the other is an acidic compound. Hence, although independent analyses must be run at different times, the ultimate analysis of the extract is nonetheless still LC/MS/MS.

Since liquid chromatography is not performed in the gas phase but rather at ambient temperature and in a liquid phase, the extraction process is much simpler than in gas chromatography (Glad and Grubb, 1978; Cairns *et al.*, 1983; Croley, 2000). By gradually adding water to a methanol extraction, the solubility of melamine in methanol is increased, improving its tendency to release from the sample into the liquid phase (Aurand, 2007). Moreover, extracting a contaminant of interest from a given sample into a medium, whether aqueous or organic, becomes easier when applied to the liquid chromatograph as it doesn't degrade as quickly as in a gas chromatograph. A clear advantage of liquid chromatography: one can simply inject an extracted sample onto the liquid chromatograph, clean out interferences, and simply identify the compound using the tandem MS/MS (Aurand, 2007; QUASU, 2007).

Additionally, LC/MS/MS provides both the identification as well as the quantitative information required. The GC/MS screen on the other hand involves a difficult extraction followed by a simple positive or negative result that requires an additional LC/MS/MS or HPLC-UV step to then identify the compound in the end.

2.3.1.5. LC/MS/MS: UCD

With a goal of detecting melamine in tissue samples from hogs affected by adulterated food, on April 30th, 2007, the California Health and Food Safety Laboratory in the University of

California at Davis released methods for the determination of melamine in porcine muscle tissue (Filigenzi *et al.*, 2007). Using solid-phase extraction (SPE) and high-performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS) the authors achieved a mean recovery for the seven replicates tested of 83% with a 6.5% relative standard deviation and a calculated method detection limit of 1.7 ng/g (1.7 PPB). Quantitative determinations of melamine concentrations in tissue samples were attained using a triple stage quadrupole mass spectrometer (Filigenzi *et al.*, 2007). Clear advantages of solid phase extraction (SPE) versus liquid-liquid extraction (LLE) have been identified and as such were using in the experimental design of this study: less solvent is required, analysis times are shorter and working products are more amenable to automation (Zhao *et al.*, 1994). Further inefficiencies with LLE have been documented by Dufresne *et al.* (2001).

In consultation with University of Guelph scientist Dr. Martos, Filigenzi *et al.* (2007) used a mobile phase of ammonium acetate/acetic acid and acetonitrile to prevent carryover in the HPLC/MS/MS system. This eliminated the frequent melamine contamination in blank analyses. Melamine was extracted in 50% acetonitrile in water (Filigenzi, 2007).

The precursor ion for melamine was identified at m/z 127. Collision-induced dissociation (CID) product ions (**Fig 2.20**) of melamine were consistent with those previously reported by Ju *et al.* (1999): m/z 85 and 68 (Filigenzi, 2007).

Figure 2.20: CID product ions of melamine (Filigenzi, 2007)

2.3.1.6. LC/MS/MS: USFDA

Based of the UC Davis method, Andersen *et al.* (2007) of the USFDA, released a modified variation in May, 2007: the Determination of Melamine Residues in Catfish Tissue by Triple Quadrupole LC-MS-MS with HILIC Chromatography. There, catfish tissue was extracted with 50:50 acetonitrile:water and 1 N hydrochloric acid, followed by LC/MS/MS analysis with HILIC chromatography and electrospray ionization in positive ion mode. Catfish tissue was fortified at 10, 25, 50, 100, and 500 ng/g (ppb), with an average melamine recovery for the 17 samples of 76.3 %, with a relative standard deviation of 14.3 % (Andersen *et al.*, 2007).

Similarly to Filigenzi *et al.* (2007), quantitative determination was performed using a triple quadrupole mass spectrometer (Andersen *et al.*, 2007). Further consistent with Ju *et al.* (1999) and Filigenzi *et al.* (2007), CID product ions of melamine were identified at m/z 85 and 68. The precursor ion for melamine was m/z 127 (Fig 2.21).

Figure 2.21: SRM chromatograms: control & fortified catfish tissues (Andersen *et al.*, 2007)

Table 2.15: CID product ions of melamine and its analogues (Andersen *et al.*, 2007)

Compound	Retention Time (min)	Precursor Ion (m/z)	Quantitative SRM transition (m/z) (Collision Energy)	Confirmatory SRM transition (m/z) (Collision Energy)
Melamine	2.7	127	85 (7)	68 (23)
Ammeline	3.1	128	86 (5)	69 (26)
Ammelide	2.0	129	87 (14)	70 (27)

2.3.1.7. LC/MS/MS: CFIA

From May to June of 2007, the CFIA released 3 methods using Liquid Chromatography Electrospray Ionization Mass Spectrometry (LC/ESI-MS/MS): 1. The Determination of Melamine; 2. The Screening for Cyanuric Acid; 3. The Determination of Melamine and Cyanuric Acid. The methods were presented as applicable to the “quantification and confirmation of melamine in vegetable protein products including wheat, rice, soy, and corn” in addition to rice bran, corn meal, fish meal and fish feed. Serving as guidelines for other laboratories, the drafts outlined the materials and methods required for successful analyses, without providing any data or demonstrations from actual runs (CFIA, 2007c-e).

Sample extraction took place in acetonitrile and water. In line with the USFDA, reporting levels were set at 10 µg/g (PPM), with an analytical range of 10-20000 PPM (CFIA, 2007c-e).

2.3.1.8. HPLC-UV

To avoid the now well-established danger of presuming the total nitrogen content in a standard crude protein analysis could be the summation of only a sample's protein, amino acid and peptide groups, amino acid profiling has become a critical component in developing suitable monitoring programs for vegetable protein products. Amino acid analyzers are essentially highly sophisticated liquid chromatographs: High Performance Liquid Chromatographs (HPLC). Used extensively in pharmaceutical drug and physiological testing, amino acid profiling by means of HPLC technology has been applied to the food sciences as well.

For instance, Pastorello *et al.* (2000) determined that the major allergen responsible for food-induced allergic reactions in maize was a lipid transfer protein. The allergen identified was purified by HPLC and characterized by mass spectrometry, determination of the isoelectric point value, and N-terminal amino acid sequencing (Pastorello *et al.*, 2000). Wieser and Kieffer (2001) analyzed amino acid profile of α -gliadines from different wheat species in an effort to isolate those sequences potentially toxic for celiac patients (Wieser and Kieffer, 2001; Wieser *et al.*, 1998).

Similar techniques were applied by Penã *et al.* (2006) who investigated the role of tyrosine-rich crosslinks in the formation of the gluten networks in wheat (*Triticum aestivum L.*) dough.

Reverse phase HPLC was used to determine the quantity of dityrosine and isodityrosine derivatives produced under different treatments during gluten formation (Penã *et al.*, 2006).

Amino acids cannot be easily analyzed; HPLC-profiling requires a 3 step extraction sample work-up process, involving a derivatization step before the extract goes onto the liquid chromatograph. As Aristoy and Toldra (1991) discovered in their examination of the various deproteinization techniques for HPLC amino acid analysis in pork muscle and dry-cured ham, amino acid profiling requires a more complex extraction process than standard LC systems.

Fekkes (1996) applied various pre-column derivatization techniques involving *o*-phthaldialdehyde for primary amino acids in plasma, and phenylisothiocyanate for secondary amino acids and cystine. The authors confirmed that HPLC methods were capable of separating over forty physiological amino acids, lending support to their use in the analysis of urine samples (Fekkes, 1996).

Hence, in HPLC analyses, amino acids must first be cleaved in a 3 step process of hydrolysis to break off all of the amino acids from the parent protein. From there, a carbonyl derivative must be formed in order for the amino acids to be amenable to successive analysis. Reverse phase C18 columns with 254-nm UV are commonly employed for that analysis and are the key to the qualification process of raw materials coming in. In order to discern that a certain percentage of protein is genuinely coming from a given vegetable protein, and was not adulterated with compounds like melamine or cyanuric acid, amino acid profiling is vital to the qualification process.

Hence LC/MS/MS technologies coupled with HPLC-UV-driven amino acid profiling provide an excellent monitoring system for both the qualification of protein samples, as well as the determination of melamine and its analogues.

Other advantages of combining the two methods were identified by Mamone *et al.* (2000). In analyzing wheat gluten proteins by HPLC combined with standard liquid chromatography/electrospray ionization mass spectrometry (LC/ESI-MS/MS) to separate the two protein fractions from seven durum wheat varieties, N-terminal amino acid sequences of the gliadin and glutenin fractions could be determined, along with accurate molecular mass data (Mamone *et al.*, 2000).

Although HPLC methods are fast and straightforward to run, according to Patel and Jones (2007), they are limited by themselves as their spectrophotometric detectors typically operate within the 200 nm range, where absorption interferences are common, especially when applied to biological matrices. The authors recommend additional selectivity to be gained from combined mass spectrometry methods such as LC/MS/MS (Patel and Jones, 2007).

2.3.2. LEGAL REQUIREMENTS

After discovering melamine-contaminated ingredients in imported Canadian fish feeds, on May 11, 2007, the Canadian Food Inspection Agency (CFIA) issued a complete border lockout of selected vegetable protein products of Chinese origin: wheat gluten, wheat protein, wheat protein concentrate; rice gluten, rice protein, rice protein concentrate; corn gluten, corn proteins, corn protein concentrate, corn-by products (corn meal, corn gluten meal); soy protein concentrate, soy protein isolate; mung bean protein; protein hydrolysates; amino acids (and their salts – which include MSG-monosodium glutamate) (CFIA, 2007e). In addition to the lockout, systematic CFIA testing of all such products for the presence of melamine began, along with thorough tracking investigations to determine which materials made their way into Canada.

On June 4, 2007, a compliance verification strategy was implemented, requiring importers of vegetable protein products to “demonstrate that their imports are free from melamine and cyanuric acid (free from melamine and cyanuric acid means that neither chemical is detected at a level of 10 mg per kg or greater)” in order to pass into Canada for use “in or as food or livestock feed” (CFIA, 2007e).

This expanded the previous CFIA program to all countries of origin, requiring certificates of analysis from acceptable laboratories (Table 2.16), using acceptable methods of analysis (Table 2.17), to be present for all non-Chinese imports. Products sourced from China were treated with stricter measures; requiring immediate lockouts until in-house testing results could assure their safety (CFIA, 2007e).

Table 2.16: CFIA (2007e) - Acceptable Laboratories for Analytical Testing

1. The CFIA laboratory in Calgary;
2. Canadian private sector laboratories accredited by the Standards Council of Canada to ISO 17025 (www.scc.ca);
3. United States Food and Drug Administration (USFDA) laboratories;
4. USFDA Food Emergency Response Network laboratories; and
5. United States Customs and Border Protection laboratories

- Acceptability of other laboratories will be assessed by the Science Branch of the CFIA on a case-by-case basis.

Where no acceptable certificates are produced, products are held and tested by the CFIA at higher sampling frequencies than those products accompanied by approved certificates. If those tests show two lots of the same product to be free of contamination, they will be held and tested at a lower frequency. Products that are analyzed and found not to contain detectable levels of melamine will be released for use. Products that are found to contain detectable levels of melamine will need to be disposed of in an appropriate manner (CFIA, 2007e).

Table 2.17: CFIA (2007g) - Acceptable Analytical Methods for Melamine/Cyanuric Acid

1. Determination of Melamine using Liquid Chromatography Electrospray Ionization Mass Spectrometry (LC/ESI-MS/MS) and Screening for Cyanuric Acid using Liquid Chromatography Electrospray Ionization Mass Spectrometry (LC/ESI-MS/MS) (CFIA methods);
2. GC-MS Method for Screening and Confirmation of Melamine and Related Analogues (USFDA method); and
3. A method that is based on mass spectrometry methods, has sensitivity to 10 mg per kg or less and is used by a Canadian private sector laboratory that is accredited by the Standards Council of Canada to ISO 17025.

2.3.3. EXPERIMENTAL HYPOTHESIS & OBJECTIVE

Although LC/MS/MS technologies have been widely applied in the design of monitoring systems intended for critical toxicological pathways in the environmental, few have targeted the agri-food chain as a point source of contamination. The widespread food recalls of 2007 clearly demonstrate that greater attention is required in this area, along with the firm recognition that NIRS-screening for nitrogen is a false assurance of overall nutritive protein value, and can no longer be regarded as a complete quality assurance program for such measures. Selecting accurate, efficient and cost-effective tools for the monitoring of environmental contaminants entering via the food-web thus poses a considerable challenge. The formal recommendations and legislative support of the CFIA might be sufficient for the identification of melamine and its analogues, but is precariously limited in its ability to effectively assure a quality product where there is no *a priori* knowledge of the toxin(s) involved. Therefore it is crucial that new techniques or new combinations of techniques that are best suited to the enhancement of safety programs be investigated.

In light of these challenges, this study explored a number of analytical techniques currently being employed in Canadian food safety laboratories to assess and furthermore assure the quality of VPPs: GC/MS, LC/MS/MS, NIRS and HPLC-UV. The overall goal was to provide an analytical framework from which recommendations could be made attesting to the empirical methods most

suited to the agri-food industry, both nationally and internationally. Based on their cost-effectiveness, time-effectiveness and practicality, conclusions were drawn to identify the most appropriate monitoring system as applied to raw-material and finished product samples containing WG, SPI and RPC.

The hypothesis is that LC/MS/MS technologies, when combined with HPLC-UV amino acid profilers, provide for the most comprehensive quality assurance VPP-screening program for nitrogen-bearing toxins found in the environment. Moreover, as LC/MS/MS instrumentation is quite costly and consequently limited in widespread installation, amino acid profiling by means of HPLC-UV is the most effective stand-alone quality-assessment-tool for VPP protein-content-determination.

3.0. MATERIALS & METHODS

3.1. RAW MATERIALS & FINISHED GOOD SAMPLES

Twenty-one human-grade wheat gluten (WG), rice protein concentrate (RPC) and soy protein isolate (SPI) samples were purchased from prominent internationally regulated food product suppliers as raw materials (Table 3.1). All samples were screened and selected by the purchaser for their determined high quality N-content profiles (i.e. N x 6.25 >70-90%) using Near Infrared Spectroscopy (NIRS).

Table 3.1: Sources of Raw Material Vegetable Protein Products & Finished Product Samples

Wheat Gluten (7 samples)	Rice Protein Concentrate (7 samples)	Soy Protein Isolate (7 samples)	Dry Products (19 diets)	Wet Products (12 diets)
Asia: - Supplier 1 (3 samples) Europe: - Supplier 2 (1 sample) - Supplier 3 (2 samples) - Supplier 4 (1 sample)	Asia: - Supplier 1 (5 samples) - Supplier 2 (1 sample) Europe: - Supplier 3 (1 sample)	Asia: - Supplier 1 (7 samples)	Canada: - Supplier 1 (17 samples) USA: - Supplier 2 (2 samples)	USA: - Supplier 3 (12 samples)

Thirty-one finished-product samples were received directly from pet food manufacturing facilities in Canada and the United States. Both dry extruded (19 diets) and canned/wet (12 diets) diets were selected from a wide portfolio of canine and feline products containing WG, RPC and/or SPI. Similar to raw material sample selection, VPP-based diets used in this study were initially screened by the vendor and determined by NIRS methods to be of superior quality. Only commercially available products destined for veterinary clinics, pet-stores, and professional breeder networks were chosen. All samples were coded for further reference as shown in tables 7.1, 7.2 and 7.3 (appendices A, B & C respectively).

3.2. MELAMINE (MEL) & AMINOPTERIN (APT) STANDARDS

Melamine (CAS 108-78-1) and aminopterin (CAS 54-62-6) were obtained in powder form (Sigma-Aldrich Canada Ltd., ON, Canada) and prepared as separate stock solutions of 1000 ug/ml. A working solution (50 ug/ml) was prepared from the individual stock solution by dilution in methanol (CAS 67-56-1) purchased from Fisher Scientific Co. (Ottawa, ON, Canada). All of the stock standard solutions are stable for six months stored in the refrigerator at 1 to 4 °C.

3.3. CYANURIC ACID (CYA) SOLUTIONS

The elution solution for cyanuric acid (CAS 108-80-5) was prepared with (50:50 v/v) methanol-THF (CAS 109-99-9) and 0.1% formic acid (CAS 64-18-6) purchased from Fisher Scientific Co. (Ottawa, ON, Canada) and Sigma-Aldrich Canada Ltd. (Oakville, ON, Canada), respectively. The prepared cyanuric acid stock solution (1000 ug/ml) is stable for six months stored in the refrigerator at 1 to 4 °C. The working standard solution (50 ug/ml) was prepared by diluting the cyanuric acid stock solution with methanol. This standard is stable for one month at 1 to 4 °C.

3.4. RAW MATERIAL & FINISHED GOOD SAMPLE PREPARATION

All samples were finely ground with a Sumeet 950 model heavy-duty mixer/grinder (Sumeet Power Control Co., Chennai, India) and stored in individual, clearly labeled tubes. All plastic tubing was clean, disposable polypropylene and polyethylene, which contained no melamine or its analogues. Blanks were prepared using previously tested food that was negative for the presence of melamine, aminopterin and cyanuric acid.

3.4.1. MELAMINE & AMINOPTERIN

To each tube of sample, 10 mM of HCL was added. For homogeneity, all samples, calibration standards S1-S6 and spiked blank samples, were shaken at 50 rpm for 10 minutes on a variable speed controller (Leeson Speedmaster Motor Control, Wisconsin, USA) followed by centrifugation (Heraeus Multifuge 3 General Purpose Tabletop Centrifuge; Thermo Fisher Scientific Inc., ON, Canada) at 4000 rpm for 5 minutes.

To remove any fats, hexane was added to new tubes followed by shaking and further centrifugation (Heraeus Multifuge 3 General Purpose Tabletop Centrifuge; Thermo Fisher Scientific Inc., ON, Canada) at 4000 rpm for 5 minutes. The hexane was aspirated to waste.

3.4.2. CYANURIC ACID

To each tube of sample, 15 mL of D_2O was added, vortexed for 15 minutes at a high speed, followed of centrifugation for 10 minutes, at 4000 RPM. The resulting clean supernatant was transferred into 50 mL centrifuge tube, where 10 mL of hexane was added, along with further vortexing for 5 minutes, followed by 5 minutes of centrifugation at 3500 RPM. All standards were extracted along with the samples and blanks.

Table 3.2: Composition of Standard and Spiked Solutions

Standard Solution Level	Concentration (ppb)	Amount of Matrix (g)	Volume of composite standard (50 ug/ml) μl
0	0	1	0
1	500	1	10
2	1000	1	20
3	1500	1	30
4	2500	1	50
5	4000	1	80
6	5000	1	100

3.4.3. CALIBRATION STANDARD & SPIKED BLANK SAMPLES

Calibration standards ranging from 500 ppb to 5000 ppb and spiked blank samples were prepared by adding appropriate amounts of spiking standard solutions to 1 g of previously screened blank matrix (Table 3.2). An additional high level standard(s) (S7 & S7A) was included with a high concentration (100,000 ppb) dilution quality check to account for extraordinarily high levels of contamination: exceeding an upper range of 5000 ppb. In such an event S7 was diluted by 1/100th with reconstitution solvent and reinserted with the samples for reanalysis. Since the more a sample is diluted and the more solvent is added to it, eventually no matrix remains. This was

essentially an enhancement effect; based on the quantitation of S7, a correction could be applied to ensure the correct amount was eluted.

3.5. SOLID PHASE EXTRACTION

This method required separate solid phase extractions as the columns could not accommodate both melamine ($-\text{NH}_2$ groups) and cyanuric acid ($-\text{OH}$ groups) at the same time. Although solid phase extraction prevented simultaneous analyses for MEL and CYA, it was selected due to its efficiency in allowing for one method to be applied to several matrices (i.e. dry food, wet food, premix materials, etc.)

3.5.1. MELAMINE & AMINOPTERIN EXTRACTION

Solid phase extraction was performed by cation exchange using a mixed mode UCT Clean Screen DAU SPE column (United Chemical Technologies, Inc., PA, USA). To prepare the Solid Phase Extraction (SPE) column, a Supelco Visiprep 24 Port-DL Solid Phase Extraction manifold (Supelco, PA, USA) was preconditioned with 3 ml of MeOH and 10 mM of HCL by gravity flow. The sample was applied to the column and allowed to pass through by gravity, then washed with deionized water followed by a methanol wash. No vacuum conditions were applied other than for subsequent drying; special attention was made to avoid excess water, leading to shorter drying times.

To elute the melamine from the sorbent in the solid phase, 4 ml of 5% ammonium hydroxide in methanol was added. To prepare the sample for analysis, the elute was left for approximately 30 minutes to adequately dry. A nitrogen spray evaporator (ReactiTherm; Pierce, Rockford, IL, USA) was used, without applying heat, to facilitate evaporation of the solvent at room temperature.

The residue was reconstituted in 0.5 ml of acetonitrile–deionized ultra-filtered water (70:30 v/v) solution. The extracts were vortex mixed by hand for 10 seconds (Barnstead, Maxi Mix II on high setting), and transferred through PVDF syringe filters (Millipore Corp., MA, USA) to remove particulates that might clog up the LC/MS system, to LC vials containing inserts.

3.5.2. CYANURIC ACID EXTRACTION

After the hexane from the sample preparation was aspirated to waste, SPE cartridges were preconditioned in a similar manner as with melamine, with 5 mL of methanol, although due to the acidic nature of cyanuric acid, was followed by 5 mL of DiH₂O. After the waste passed by gravity flow, 5 mL of the sample was loaded onto the cartridges and allowed to pass to waste yet again by gravity. Low vacuum conditions were applied where gravity flow was not sufficient for this step. The cartridges were then washed with 5 mL of DiH₂O and dried under vacuum after the wash.

Once 5 mL of elution solution could be collected in a 15 mL glass centrifuge tubes, it was evaporated to dryness under a gentle stream of heated nitrogen (approx 50°C), followed by a reconstitution with 0.4 mL of DiH₂O. The remaining material was vortexed and filtered through a PVDF 13mm, 0.22 µm syringe filter into a vial for analytical determination.

3.6. LC/MS/MS/MS ANALYSIS

Analysis of raw material and finished good samples was performed using a LC/MS/MS instrument (API 4000 MDS/Sciex Triple quadrupole mass spectrometer; Applied Biosystems, Foster City, CA, USA) coupled to a LC pump and degasser (Agilent 1100; Agilent, CA, USA), with an autosampler (MPS; Gerstel Inc., MD, USA). Analysis of melamine and aminopterin was simultaneously performed, while cyanuric acid was determined in an independent analysis with different extraction and analysis conditions.

For all samples, a matrix blank sample was included leveling each run. Six matrix calibration standards were included in the analysis run along with 2 quality control samples (S7 & S7A). Spikes were essentially the same as the standards: duplicates of the lowest range of 500 ppb (Table 3.3). In large runs additional Quality Controls were prepared and these would be staggered throughout the run.

Table 3.3: LC/MS/MS/MS: Sample Testing Scheme

	Sample Name	Sample Type	Analyte Peak Area (Counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)
1	Rinse	Solvent	0.00	0.00	N/A	N/A	0.00
2	Blk	Blank	0.00	0.00	N/A	N/A	0.00
3	Std 1	Standard	44900	500	524	105	1.81
4	Std 2	Standard	85500	1000	903	90.3	1.81
5	Std 3	Standard	145000	1500	1460	97.5	1.81
6	Std 4	Standard	255000	2500	2480	99.2	1.81
7	Std 5	Standard	451000	4000	4310	108	1.81
8	Std 6	Standard	527000	5000	5020	100	1.81
9	S7	Quality Control	55200	500	620	124	1.81
10	Sample	Unknown	N/A	N/A	N/A	N/A	N/A
11	S7A	Quality Control	55100	500	620	124	1.81

Standard Operating Procedures (SOP) required that after 22 samples, a spike was analyzed to ensure that quality control criteria were being met (i.e. nothing changed throughout the course of the run (i.e. signal, retention time, etc.)). For large runs where the possibility of drift of signal could occur, the curve would be analyzed both at the beginning of the run and at the end of the run. For runs with fewer than 10 samples, only one calibration curve was analyzed and spikes were placed before and after the sample.

3.6.1. MELAMINE & AMINOPTERIN ANALYSIS

Separation was performed by a Primesep AB (Sielc, IL, USA) reverse-phase column with embedded acidic and basic ion-pairing groups, with isocratic elution of 32.5% 25 mM ammonium acetate + 0.3% formic acid for mobile phase A and 67.5% acetonitrile for mobile phase B. A standard buffer of ammonium acetate with formic acid helped ionize the compounds and achieved decent retention with a good peak shape on the column.

The LC flow rate was 1 ml/min. A rinse sample containing the mobile phase was analyzed before every run to ensure that there was no carryover. The autosampler wash cycles involved washing (10 washes) with methanol–deionized water (80:20, v/v) between samples; the injector was washed 6 times and the syringe 4 times. Run time per sample was 5 minutes.

3.6.2. CYANURIC ACID ANALYSIS

A Zorbax Stable Bond-Phenyl (Agilent, Santa Clara, CA, USA) column (4.6 x 75 mm, 3.5 μ m) was employed for separation. Mobile phases were of 0.2% formic acid in DiH₂O for A and methanol for B.

Injection volume of cyanuric acid was 25 μ l. The elution mode gradient followed the profile seen in **Table 3.4**.

Table 3.4: Elution Mode Gradient

Step	Time	%A	%B	Flow rate (ml/min)
0	0.00	98	2	0.65
1	2.50	98	2	0.65
2	2.51	10	90	1.00
3	6.00	10	90	1.00
4	6.01	98	2	1.00
5	10.00	98	2	1.00

3.7. MASS SPECTROMETRIC CONDITIONS

Mass spectrometry was applied in multiple reaction-monitoring mode (MRM) for all samples. MRM transitions were monitored according to the parameters outlined in **Table 3.5**. Nitrogen gas was used as desolvation gas.

Table 3.5: MS Detection parameters for Melamine, Aminopterin and Cyanuric Acid

Compound	Average Retention Time (min)	Q1 (amu)	Q3 (amu)
MEL	1.81	127	85
		127	68
		127	60
APT	2.24	441	294
		441	175
		441	120
CYA	2.01	128	42
		128	85

For melamine and aminopterin Turbo Ionspray Ionization (TIS) was operated in positive mode for MEL & APT, with a voltage of 5500 V, at a temperature of 550°C. Peak Widths for Q1 and Q2 were 1 amu. Ion-Source Gases G1 and G2 were 55 psi and 50 psi, respectively, with a Collision Gas Thickness of 5 psi and Curtain Gas at 15 psi. Entrance Potential was set to 10 V, with a Collision Exit Potential of 10 V. Collision Energy was 25 V for melamine and 26 & 46 V for aminopterin. Declustering Potential was 81 for Melamine, and 84 & 87 for aminopterin.

For cyanuric acid Turbo Ionspray was set to negative polarity for CYA, with a voltage of -4200 V at 550°C. Peak Widths for Q1 and Q2 were 1 amu. Nitrogen Ion-Source Gases G1 and G2 were 50 psi, with a Collision Gas Thickness of 10 psi and Curtain Gas at 15 psi. Entrance Potential was set to -10 V, with a Collision Exit Potential of -6.0 V. Collision Energy was -14 V for 128→85 and -27 V for 128→42. Declustering Potential was -41.

3.8. STATISTICAL ANALYSIS & INTERPRETATION

Quantitative data were obtained using the area counts of the chromatographic peaks observed in MRM mode of the m/z 127→85, m/z 127→68, m/z 127→60, m/z 441→294, m/z 441→175, m/z 441→120, m/z 128→42 and m/z 128→85 transitions. Applied Biosystems Analyst® 1.4.1 software was used for data collection and interpretation. Concentration data were obtained by

linear regression with $1/(x \cdot x)$ weighting): i.e. $y = 146x + 2.47e+003$ ($r=0.9992$). Calibration curves were plotted using a weighted ($1/x \cdot x$) linear regression model with 6 concentration points (not including blank solution) ranging from 500 to 5000 ng/mL (ppb) in the mobile phase. Consistent with ISO 17025, information on every run was retained for reference: date, time, compound, method, mobile phase(s), columns employed and the instrument used.

3.9. OUTSOURCED HPLC-UV AMINO ACID PROFILING

Due to the significant levels of contamination found in samples of Rice Protein Concentrate (RPC), additional analyzes were performed on Dry-Food-Rice-Protein-Concentrate-11 (DFRPC11), whose RPC-source was traced to Asian supplier "RPC-Supplier-1." Amino acid profiling by means of HPLC-UV¹ was outsourced at a facility better equipped for the analyses.

¹ Method Reference: AOAC 982.30

4.0. RESULTS & DISCUSSION

Of the 21 NIRS-prescreened raw material samples, as well as the 19 NIRS-prescreened dry and 12 NIRS-prescreened wet-finished product samples analyzed, melamine (MEL), aminopterin (APT) and cyanuric acid (CYA) were successfully monitored in all matrices tested (**Table 4.1**). In the interest of consistency with the USFDA GC/MS method in March of 2007, reporting levels were set at >10 PPM.

Quantitative data returned a high level of confidence, with satisfactory reproducibility. Acceptable calibration curves (i.e. **Fig 4.1**) were a linear fit, not forced through zero, with correlation coefficients of 0.9878 or greater. Where required, $1/(x \cdot x)$ weighting was used to improve the accuracy of reporting at lower concentrations. At least 6 standards were used to define each curve.

Accuracy was measured through % recovery (%RV) of fortified standards 1-6, ranging from 500 ppb to 5000 ppb, along with 1-2 additional quality controls (Spk 1 & Spk 1A), depending on the sample. Concentrations that exceeded the highest reference standard were diluted with blank matrix. No contaminants were found in method blanks or rinses. Typical %RV values were between 96 and 102%. Signal-to-noise (STN) ratios were high, ranging from 3.95:1 at the lowest levels (blanks/rinses) up to 477000:1.

Overall, MEL was identified in 22 of the 49 test-runs at concentrations above 1 PPM (**Table 4.1**). Calibrations curves over the range of 500 to 5000 ppb displayed high levels of accuracy (90-130%). Only 2 samples (Wet-Food-Wheat-Gluten-4 (WFWG4) & Wet-Food-Wheat-Gluten-5 (WFWG5)) were identified at levels above 1000 PPM, containing 1120 PPM and 1090 PPM of MEL, respectively. These concentrations did not exceed the LD₅₀ of 3161 PPM in rats, yet did slightly exceed the reproductive and developmental toxicity no-observed-adverse-effect-levels (NOAELs) of 1060 mg/kg bw/day identified in orally administered fetal rats and 400 mg/kg bw/day in maternal rats (USFDA, 2007; IUCLID, 2000; OECD, 2002). Whether these findings might be extrapolated to human health is debatable, as no metabolism or tissue accumulation of melamine has been demonstrated in mammals. Of concern however is the fact that these levels did also exceed the concentration of MEL found in the kidneys of autopsied cats, 496 to 734

PPM. Furthermore, the overall risk to the environment remains unknown, especially when found in combination with CYA. Being cognizant of the 2007 food recalls and the magnitude of the 60,000,000 containers of wet food alone that were disposed of and destroyed, a multidisciplinary investigation is required to elucidate the multifarious effects of the complex biochemical pathways of MEL released into local ecosystems.

Table 4.1: Number of Positive samples tested for Melamine, Aminopterin & Cyanuric Acid

Compound	Total Analyses	% contaminated (>1 PPM)	% contaminated (>10 PPM)	% contaminated (>100 PPM)	% contaminated (>1000 PPM)
MEL	49	44.89	18.37	8.16	4.08
CYA	33	51.52	42.42	36.36	33.33
APT	35	0	0	0	0

Consistent with previous findings by the USFDA (2007) and University of Cornell (2007), APT was not found in any samples tested at levels above 1 PPM, lending further support to the theory suggesting that its initial suspected role was nothing more than an artifact of analytical limitations associated with GC.

Of the 33 CYA analyses completed, 17 tested positive, at levels exceeding 1 PPM. Raw-Material-Rice-Protein-Concentrate-7 (RMRPC7) was the only sample that exceeded contamination levels of 5000 PPM with a recovery of 5400 PPM. Indeed those levels did exceed the LD₅₀ for rats, which was assessed at 750 PPM, as well as the no-risk-to-human health level of 20-25 mg per liter of water (Canelli, 1974). As with MEL, more research into the potential human health and environmental risks associated with CYA discharges into the natural surroundings is needed.

Additionally, the confirmed presence of MEL along with CYA contamination in this study confirmed that the source of initial pollution was not pure MEL altogether, but rather its waste-stream of analogues. This fact further highlights the degree of immorality and foolhardiness in which those individuals responsible for such criminal acts engaged for financial gain.

4.1.0. LC/MS/MS/MS ANALYSIS

In working with the above-mentioned matrices such as finished product animal feeds (wet & dry), reduced electrospray sensitivity due to the presence of endogenous components such as excess fats, salts, starches, etc. was observed.

Although ion-exchange chromatography initially separated the compounds of interest from endogenous compounds, at times dilutions were required for samples containing higher levels of contamination to minimize the electrospray sensitivity. Shifts in retention times for the same compound in different test runs can be explained by this effect. As a consequence, calibration curves were applied to each trial to ensure acceptable consistency with previous runs.

The use of dilution factors ranging from 1:500 to 1:20000 were indeed sufficient to eliminate these matrix enhancement effects, providing statistically significant results. Results displayed in bold (i.e. RMRPC1, RMRPC7, etc.) represent columns that may have been overloaded by such residues, possibly leading to lower results being obtained. In those cases actual MEL and/or CYA concentrations may be higher than reported.

4.1.1. RAW MATERIALS

All 14 samples of wheat gluten (RMWG1 to RMWG7) and soy protein isolate (RMSPI1 to RMSPI7) tested negative for the presence of MEL and CYA. Rice protein concentrate (RPC) was unequivocally the greatest source of contamination, with high levels of CYA: up to 5400 PPM. Of those RPC samples assessed, 3 tested positive for both MEL and CYA (**Table 4.2**). Where both compounds were present, CYA was found in significantly larger proportions than MEL. In RMRPC7 for instance, CYA levels (5400 PPM) were 524 times greater than MEL (10.3

PPM). These findings were consistent with reports by the American Veterinary Medical Association (AVMA) that also observed higher CYA concentrations, albeit to a lesser extent: MEL:CYA (30:70 v/v) (AVMA, 2007).

Table 4.2: Concentrations of Melamine, Aminopterin & Cyanuric Acid in Raw Materials

Sample	[MEL] (ppm)	[MEL] (%)	[APT] (ppm)	[APT] (%)	[CYA] (ppm)	[CYA] (%)	Supplier
RMWG1	<1	ND	<1	ND	--	--	WGSUP2
RMWG2	<1	ND	<1	ND	<1	ND	WGSUP1
RMWG3	<1	ND	<1	ND	<1	ND	WGSUP1
RMWG4	<1	ND	<1	ND	<1	ND	WGSUP1
RMWG5	<1	ND	<1	ND	--	--	WGSUP3
RMWG6	<1	ND	<1	ND	--	--	WGSUP3
RMWG7	<1	ND	<1	ND	<1	ND	WGSUP4
RMSP11	<1	ND	--	--	<1	ND	SPISUP1
RMSP12	<1	ND	--	--	<1	ND	SPISUP1
RMSP13	<1	ND	<1	ND	--	--	SPISUP1
RMSP14	<1	ND	--	--	<1	ND	SPISUP1
RMSP15	<1	ND	--	--	<1	ND	SPISUP1
RMSP16	<1	ND	--	--	<1	ND	SPISUP1
SMSP17	<1	ND	--	--	<1	ND	SPISUP1
RMRPC1	16.2	0.0016	<1	ND	4780	0.478	RPCSUP1
RMRPC2	<1	ND	<1	ND	38.3	0.0038	RPCSUP1
RMRPC3	<1	ND	<1	ND	56.0	0.0056	RPCSUP1
RMRPC4	3.8	ND	<1	ND	637	0.0637	RPCSUP1
RMRPC5	<1	ND	--	--	<1	ND	RPCSUP2
RMRPC6	<1	ND	--	--	<1	ND	RPCSUP3
RMRPC7	10.3	0.0010	<1	ND	5400	0.54	RPCSUP2

- ❖ ND = not detected above 0.0001%;
- ❖ -- = testing not performed for this compound;
- ❖ Actual concentrations of CYA may be higher.

Although the LD₅₀ was not determined in cats poisoned with adulterated foodstuffs, the CYA concentrations measured here significantly exceeded those found within the autopsied kidneys: 487 to 690 PPM. On the contrary, the MEL contamination of these materials was substantially less in comparison to those levels found within the kidneys of affected cats: 496 to 734 PPM.

However, as Puschner *et al.* (2007) demonstrated, concentrations as low as 32 mg/kg each of MEL and CYA were shown to cause renal crystallization in cats. Hence, herein lies a great opportunity for further research into the toxicological factors leading up to and surrounding the formation of such renal-blocking fan-shaped birefringent MEL-CYA lattice crystals.

Analyte retention times acquired from MRM scans for CYA were observed at 1.93 minutes (**Fig. 4.1**). Signal intensity was strong with a height of 5.09e+006 cps and an initial signal-to-noise ratio of 477000:1. Subsequent reanalyzes with dilutions of 1:1000, 1:10000 and 1:20000 decreased signal-to-noise ratios to 2020:1, 503:1 and 387:1, respectively. The signal intensity at 1:20000 was reduced to 4.02e+004 cps (**Fig. 4.2**). Linear regression was observed for CYA analyses of RPC samples, where $y = 84.1x + 6.2e+003$ ($r=0.9973$).

Figure 4.1: Chromatographic peak of CYA (5400 PPM) from sample RMRPC7

Figure 4.2: Linearity of RPC7 CYA calibration curve (range: 500 ppb to 5000 ppb)

Retention times for MEL were observed at 1.87 minutes (**Fig. 4.3**). RMRPC7's (16.2 PPM) initial signal-to-noise ratio was 8040:1. Following a dilution of 1:20, signal-to-noise was reduced to 1580:1.

Linear regression was observed, where $y = 84.1x + 6.2e+003$ ($r=0.9878$) (**Fig 4.4**).

Figure 4.3: Chromatographic peak of MEL (16.2 PPM) from sample RMRPC7

Figure 4.4: Linearity of RPC MEL calibration curve (range: 500 ppb to 5000 ppb)

4.1.2. LC/MS/MS/MS: DRY FOOD

Finished product dry food results were consistent with those observed in raw material screenings. The most concentrated sources of contamination were RPC-based products. Moreover, nine samples contained both MEL and CYA contamination, with proportionally higher CYA levels, lending further support to the AVMA's findings. RPC-based sample DFRPC6 contained CYA in concentrations exceeding MEL by a factor of 753.85 (Table 4.3).

Peaks for CYA were observed at 1.93 – 1.99 minutes (see Appendix B). The highest level of contamination was observed in DFRPC2, with a CYA concentration of 3580 PPM peaking (Fig. 4.5) at 1.93 minutes. Once again, these levels exceeded the LD₅₀ of 750 PPM in rats and require further investigation to determine potential risks to the environment and human health (Canelli, 1974).

Enhancement effects were calculated at 3110%, based on a 1:20000 dilution². The MS-calculated analyte concentration was 1150 PPB, which considered with the enhancement percent, provided a CYA level of 3580 PPM³. Linear regression was seen (Fig. 4.6) where $y = 101x + 8.24e+003$ ($r = 0.9970$).

Analyte retention times for MEL were seen in a range of 1.76 – 1.81 minutes. The most concentrated source was DFRPC9, whose level of 11.8 PPM was significantly lower than those MEL concentrations determined to be of any significant risk to human or animal health (USFDA, 2007; IUCLID, 2000; OECD, 2002). However, given that CYA was also present in this sample at a concentration exceeding the 32 mg/kg dose assessed by Puschner *et al.* (2007) as sufficient for kidney blockage, once again further research is required to help better understand the interplay between MEL and CYA in birefringent-lattice-crystal formation.

Peaks were observed at 1.81 (Fig. 4.7) minutes with a dilution factor of 1:5 and a signal-to-noise ratio of 5210:1. Linear regression (see Appendix B & Fig. 4.8) was high ($r = 0.9992$ to $r = 0.9996$).

² Percent enhancement = { dilution factor } / { % accuracy of QC / 100 }

³ [CYA] = { [MS-calculated analyte] x percent enhancement } / 1000 PPM

Table 4.3: Concentrations of Melamine, Aminopterine & Cyanuric Acid in Dry Food Samples

Diet VPP Source	Indication	[MEL] (ppm)	[MEL] (%)	[APT] (ppm)	[APT] (%)	[CYA] (ppm)	[CYA] (%)
DFWG1	CAN	<1	ND	<1	ND	--	--
DFWG2	FEL	<1	ND	<1	ND	--	--
DFWG3	FEL	<1	ND	<1	ND	--	--
DFWG4	FEL	<1	ND	<1	ND	--	--
DFWG5	FEL	<1	ND	<1	ND	--	--
DFWG6	FEL	<1	ND	<1	ND	--	--
DFWG7	CAN	--	--	--	--	3.4	ND
DFSPI1	FEL	<1	ND	--	--	<1	ND
DFRPC1	FEL	<1	ND	<1	ND	--	--
DFRPC2	CAN	8.6	ND	<1	ND	3580	0.358
DFRPC3	FEL	4.0	ND	<1	ND	1170	0.117
DFRPC4	CAN	3.7	ND	<1	ND	2360	0.236
DFRPC5	CAN	7.5	ND	<1	ND	2650	0.265
DFRPC6	CAN	2.6	ND	<1	ND	1960	0.196
DFRPC7	CAN	8.5	ND	<1	ND	1890	0.189
DFRPC8	CAN	5.1	ND	<1	ND	1980	0.198
DFRPC9	CAN	11.8	0.0012	<1	ND	1770	0.177
DFRPC10	CAN	11.5	0.0012	<1	ND	--	--
DFRPC11	FEL	8.6	ND	<1	ND	3270	0.327

❖ ND = not detected above 0.0001%;

❖ -- = testing not performed for this compound;

❖ FEL = Feline Diet; CAN = Canine Diet;

❖ Actual concentrations of CYA may be higher.

Sample Name: "DFRPC2(dil 1:20000)" Sample ID: "" File: "Dry Food"
Peak Name: "CYA-1" Mass(es): "127.9/42.0 amu"

Figure 4.5: Chromatographic peak of CYA (3580 PPM) from sample DFRPC2

Sample Name: "DFRPC9(dil 1:5)" Sample ID: "" File: "Dry Food.wiff"
Peak Name: "MEL-1" Mass(es): "126.8/85.2 amu"

Figure 4.7: Chromatographic peak of MEL (11.8 PPM) from sample DFRPC9

Untitled 9 (CYA-1): "Linear" Regression ("1 / (x * x)" weighting): $y = 101x + 8.24e+003$ ($r = 0.9970$)

Figure 4.6: Linearity of dry food CYA calibration curve (range: 500 ppb to 5000 ppb)

Untitled 7 (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): $y = 167x + -3.55e+003$ ($r = 0.9996$)

Figure 4.8: Linearity of dry food MEL calibration curve (range: 500 ppb to 5000 ppb)

4.1.3. LC/MS/MS/MS: WET FOOD

Contrary to raw material and dry food sampling, the highest levels of contamination in wet food screenings (Table 4.4) were reserved to WG-based diets, containing 1120 PPM and 1090 PPM of MEL. Although lower than the LD₅₀ of 3161 mg/kg in rats, these diets were implicated in the recalls of March 2007, and were found to have directly lead to chronic renal failure in cats (IUCLID, 2000; USFDA, 2007). Unfortunately, as no *a priori* knowledge of MEL-analogues existed at that time, CYA was not tested for in those wet diet samples. However, based on the documented symptoms (i.e. chronic renal failure) observed in pets who had consumed WG-based diets coming from similarly sourced wet food, it seems quite fair to assume that CYA was present in proportionally higher concentrations than MEL. Knowing that only 32 mg/kg of each compound can lead to crystal precipitation, if this scenario was indeed the case, contaminated feline wet food diets would represent the most significant instances of renal-blocking lattice-crystals thus far.

The two SPI-based (Wet-Food-Soy-Protein-Isolate-1 (WFSPI1) & Wet-Food-Soy-Protein-Isolate-2 (WFSPI2)) and four RPC-based diets (Wet-Food-Rice-Protein-Concentrate-1 (WFRPC1) to Wet-Food-Rice-Protein-Concentrate-4 (WFRPC4)) analyzed tested negative for the presence of MEL and CYA (<10 PPM).

Table 4.4: Concentrations of Melamine, Aminopterin & Cyanuric Acid in Wet Food Samples

Diet VPP Source	Indication	[MEL] (ppm)	[MEL] (%)	[APT] (ppm)	[APT] (%)	[CYA] (ppm)	[CYA] (%)
WFWG1	FEL	970	0.0970	<1	ND	--	--
WFWG2	FEL	<1	ND	<1	ND	--	--
WFWG3	FEL	530	0.0530	<1	ND	--	--
WFWG4	FEL	1120	0.1120	<1	ND	--	--
WFWG5	FEL	1090	0.1090	<1	ND	--	--
WFWG6	FEL	23.8	0.0024	<1	ND	--	--
WFSPI1	CAN	2.6	ND	<1	ND	<1	ND
WFSPI2	CAN	2.6	ND	--	--	<1	ND
WFRPC1	FEL	--	--	--	--	1.7	0.00017
WFRPC2	FEL	--	--	--	--	1.3	0.00013
WFRPC3	CAN	2.7	ND	--	--	<1	ND
WFRPC4	CAN	3.2	ND	--	--	<1	ND

- ❖ ND = not detected above 0.0001%
- ❖ -- = testing not performed for this compound
- ❖ FEL = Feline Diet; CAN = Canine Diet

The greatest contamination was observed in WFWG4, with MEL concentrations of 1120 PPM. Initial duplicate readings showed spikes at 1.67 and 1.68 minutes, with heights of 1.30e+006 cps and 1.21e+006 cps. Dilution down to 100:1 displayed a retention time of 1.73 minutes with a peak intensity of 1.08e+005 cps. Finally, with a dilution factor of 500:1, retention times (Fig. 4.9) occurred at 1.73 minutes with an intensity of 3.57e+004 cps. Linear regression (Fig. 4.10) displayed where $y = 99.8x + 2.71e+003$ ($r = 0.9972$).

Sample Name: "WFWG4 (dil 1:500)" Sample ID: "" File: "Test samples-wet food.wiff"
Peak Name: "MEL-1" Mass(es): "126.8/85.2 amu"

Figure 4.9: Chromatographic peak of MEL (1120 PPM) from sample WFWG4

ApplMel-wet food_070409.rdb (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): $y = 99.8x + 2.71e+003$ ($r = 0.9972$)

Figure 4.10: Linearity of wet food MEL calibration curve (range: 500 ppb to 5000 ppb)

4.2. HPLC-UV RESULTS

Outsourced amino acid profiling by means of HPLC-UV⁴ further confirmed the adulteration of materials tested. Given that all samples were screened by NIRS at the outset and successfully selected for their target protein content, contradictory results by HPLC-UV were unexpected.

The most pronounced deviation was observed in a raw RPC sample obtained from RPC Supplier 2. Using the Kjeldhal method⁵ for protein determination, 70% crude protein (CP) was assessed by NIRS, representative of a total N-content of 11.20% ($N \times 6.25$). Conversely, subsequent HPLC-UV analysis revealed a total amino acid content of 12%, a significant digression from the expected 70%.

⁴ Method Reference: AOAC 982.30

⁵ Method Reference: AOAC 955.04, 979.09

Control samples displayed a typical RPC nutrient profile (**Table 4.5**) of 70% crude protein, 8% starch⁶, 4% ash⁷, 4% total dietary fiber (TDF)⁸, 8% moisture and 6% crude fat (CF)⁹.

Comparatively, core nutritional analyses of a contaminated sample containing 4590 PPM of CYA (RPC-HPLC) revealed a nutritional profile not typically associated with a VPP sample of any kind: 12% CP, 42% starch, 0.5% TDF and 10% moisture (**Table 4.5**). This relatively large difference in nutrient profiles can be explained by yet another level of vendor duplicity, whereby the sample labeled as RPC was in fact closer in composition to rice flour than anything else and consequently was not a VPP altogether.

Melamine and its derivatives aside, the core nutritional profile assessed in Table 4.5 would not meet the minimal canine or feline nutrient profiles for growth, reproduction or maintenance as established by the National Research Council (NRC) or the American Association of Feed Control Officials (AAFCO).

These findings, in combination with those above attesting to the impurity of the nitrogen-bearing toxins themselves, further demonstrate the proclivity towards financial gains above all for those individuals responsible. The implication of such actions is astounding, especially in consideration of the fact that RPCs and RBPIs are often applied to such nutritive products as infant fortified weaning formulations (Chrastil, 1992; Helm and Burks, 1996; Tsuji *et al.*, 2001).

4.3. SUCCESSIVE SCREENINGS

In the interest of identifying the 35.5% unknown constituent, successive MEL and CYA screenings were performed, along with tests for urea and ammonia¹⁰, as well as open characterizations using GC/MS scans.

⁶ Method Reference: AOAC 996.11

⁷ Method Reference: AOAC 923.03

⁸ Method Reference: AOAC 991.43

⁹ Method Reference: ISO 1211, AOAC 905.02, AOAC 989.05

¹⁰ Method Reference: AOAC 941.04, 967.07

Table 4.5: Nutrient profiles: Typical RPC vs. sample from Asian-Sourced RPC-Supplier-1

Unadulterated Control Sample	Nitrogen Contribution	Rice Protein Concentrate (RCP-Supplier-1)	Nitrogen Contribution
Protein Assessed by NIRS (N x 6.25): 70%	Σ N = 11.20%	Protein Assessed by NIRS (N x 6.25): 70%	Σ N = 11.20%
Amino Acid (70%)	→ 11.20% N	Amino Acid (12%)	→ 1.92% N
		Unknown (35.50%)	→ 9.28% N
Starch (8%)	→ 0% N	Starch (42%)	→ 0% N
Ash (4%)			
TDF (4%)			
Fat (6%)			
Moisture (8%)			
		TDF (0.5%)	
		Moisture (10%)	

Results (Table 4.6) confirmed <5 PPM MEL and 4590 PPM CYA, accounting for a mere 0.93% of the unknown N-protein content. Urea was insignificant at <0.5 PPM and 292 PPM of ammonia was revealed, contributing 0.15% to the nitrogen content. The remaining 8.2% residual source of N remains unidentified.

Open characterizations by GC/MS revealed the presence of N-(Bromomethyl)phthalimide (C₉H₆BrNO₂; CAS 5332-26-3) in all samples analyzed, as well as a number of long chain amides, possibly indicative of nylon manufacture. Due to their chemical conformation and nitrogen-density, likely candidates for further research include biuret (C₂H₅N₃O₂; CAS 108-19-0) and dicyandiamide (C₂H₄N₄; CAS 461-58-5).

Table 4.6: Potential Nitrogen Sources: Sample from RPC Supplier 1

Potential N-source	Levels (PPM)	Contribution to N-protein (%)
MEL	< 0.5	Not Significant
CYA	4590	0.93 ¹¹
Ammonia	292	0.15 ¹²
Urea	< 0.5	Not Significant

HPLC-UV further demonstrated a similar disparity between the total amino acid and NIRS protein-determined contents of DFRPC11. Core nutritional profiling (Table 4.7) showed a protein level of 32.35% in the RPC-based finished product.

¹¹ Contribution = 6.25(0.4590) x { 14(3) / 129.0740 }

¹² Contribution = 6.25(0.0292) x { 14(1) / 17.0306 }

Table 4.7: Core nutrient profile of sample DFRPC11

Nutritional Component	Units	Amount
KJ ¹³	/100g	1702
Ash	g/100g	8.30
Fat	g/100g	14.30
Calories ¹⁴	/100g	407
Protein	g/100g	32.35
Carbohydrates ¹⁵	g/100g	37.30
Starch	g/100g	31.23
Moisture ¹⁶	g/100g	7.90

To the contrary, amino acid analysis by HPLC-UV revealed a total amino acid content (Table 4.8) of 23.77%. Amino acids such as valine, histidine and tyrosine were compromised, producing an overall profile that would not have met the essential amino acid requirement established by the FAO/WHO/UN Expert Consultation (1985) on human growth (<2-years of age) energy requirements, nor for 2 to 5-year-old children (Wang *et al.*, 1999; FAO/WHO/UN 1985). Regrettably this is not the first time Chinese infant formulas were implicated in such a catastrophe. In 2004, the New York State Department of Agriculture and Markets food laboratory discovered nutrient-lacking samples to contain “less than 1/7 of the federally required minimal amount of protein per serving, approximately 1/4 the required amount of fat and only minute amounts of declared calcium and magnesium.” Consumption of those products lead to infant malnutrition and eventually deaths in some cases (USFDA, 2004).

¹³ Method Reference: 21 CFR 101.9

¹⁴ Method Reference: 21 CFR 101.9

¹⁵ Method Reference: 21 CFR 101.9

¹⁶ Method Reference: AOAC 925.09, 926.08

Table 4.8: HPLC-UV Amino acid profile of Dry-Food-Rice-Protein-Concentrate-11 (DFRPC11)

Amino Acid	Concentration (g/100g)
ASP	2.12
THR	0.94
SER	1.06
GLU	3.88
PHE	1.16
GLY	2.12
ALA	1.66
CYS	0.28
VAL	1.23
MET	0.53
ILE	1.00
LEU	1.85
TYR	0.09
HIS	0.57
LYS	1.58
ARG	1.75
TRP	0.36
PRO	1.59
Total Content	23.77

Moreover, at the levels of contamination identified in DFRPC11 (8.6 PPM & 3270 PPM), nitrogen contributions from MEL and CYA accounted for 0.036% and 0.665% of the protein content, respectively. As in the previous case, the remaining 7.88% has yet to be identified. More work is required to successfully identify and isolate those nitrogen-bearing toxins responsible for this additional contribution. Unfortunately, this lack of knowledge is indicative of how much more erudite the USFDA and similar governing bodies must become in order to circumvent further food safety calamities.

4.4. ECONOMICS & WIDESPREAD VIABILITY

Looking at the actual costs associated with the above-mentioned instrumentation, the reasoning behind customary industry method-selection becomes clear. With a NIRS system running in the 20,000-50,000 USD range (Reeves III, 2007), it is the least expensive of all methods employed. This remains the case even when considering the need for properly trained operators, constant recalibrations and general maintenance. Following NIRS, a high-quality GC/MS, similar to what the USFDA was first using, can be purchased from \$50,000 USD and up.

A LC/MS/MS system such as the API 3000, 4000 or 5000, is considerably more costly, being offered in a \$300,000-\$500,000 USD range. As a consequence, most companies are forced to outsource samples to independent laboratories. Associated costs are cheaper short-term, but can add up quickly if routine testing programs are implemented. A 24-hour turn around time, under emergency/reactive conditions such as those seen in March of 2007, can range from \$1000-\$1500 per test. Under more typical conditions, where no rush is required, these tests can be performed for substantially less, costing around \$200 per analysis (GMI, 2008).

In a similar light, HPLC systems capable of amino acid analysis such as the Waters KiloPrep 250 EF, Hitachi L-8800 Amino Acid Analyzer, Beckman 6300 Amino Acid Analyzer, are available at an asking price of over \$100,000 USD. Tandem MS/MS capability can bring the system cost up to and above the \$500,000 mark. For that reason, few private laboratories possess the capital to bring these units in-house and often outsource amino acid profiles at \$165-\$400 per test (GMI, 2008).

5.0. RECOMMENDATION/CONCLUSION

The food recalls of 2007 adumbrated the erroneous proclivity with which analytical *modus operandi* such as NIRS technologies have been commonly applied to agri-food quality assurance programs. Indeed their beneficial applications in food safety programs remain unquestionable, when applied correctly, monitored closely and effectively calibrated on a regular basis. What can no longer be accepted however is the assumption that nitrogen-protein determination by NIRS is sufficiently representative of the totality of a sample's quality. With that in mind, the methods employed in this study were successful in providing **auxiliary** tools, whose reliability and time-sensitive efficiencies were conducive to the on-going monitoring of VPPs for the presence of MEL and its analogues. In this context LC/MS/MS/MS was highly robust, enabling a similar work-up process to be applied to both MEL and CYA with only a slight shift in extraction techniques. LC by itself was a powerful identification tool, while the tandem MS/MS/MS allowed for fast and precise quantification. By comparison, GC/MS has been shown to be quite limited here, posing operational difficulties and reporting constraints, despite its considerably cheaper operating costs.

All too often governing bodies such as the CFIA and the USFDA are forced to react to crises in an *ad hoc* manner, without even the requisite prescience to do so efficiently. The mass food recalls of 2007 were clearly indicative of this constraint. In order to better avoid these situations in the years ahead, proper tools must be in place to promote preparedness. In this study amino acid analysis by means of HPLC-UV proved to be an invaluable component to any VPP food safety program. Despite a more complicated sample work-up process prior to analysis than experienced with a standard LC/MS/MS/MS, HPLC-UV was duly able to provide critical quality information where other methods could not. Unequivocally, HPLC-UV was the only tool investigated capable of qualifying a raw material without any *a priori* understanding of a given adulteration. Knowing after the fact that some of raw materials tested above were not VPPs altogether, and that the contaminants were not pure in themselves, highlights the need for a broader scope in how nutritive protein sources are monitored for safety. Moreover, with the majority of the nitrogen content in the samples tested still unaccounted for, it is clear that further research is required. For this reason the author recommends the prompt adoption of this tool in nationwide industry-standard protein-assessment quality assurance programs. HPLC-UV is

exemplary for this task, capable of directly and single-handedly evaluating the requirement for additional steps such as the identification and/or quantification of a particular contaminant by means of LC/MS/MS/MS.

In conclusion, based on the evidence presented herein, LC/MS/MS/MS, in combination with HPLC-UV provides for a reliable food safety monitoring program as applied to VPPs. HPLC-UV is indispensable as a stand-alone first level of screening to assess the integrity of a VPP or any nutritive protein-based sample. Future research and development is required to bring the associated instrumentation costs down to a level where they can be adopted on a widespread basis. The economic cost of compliance is one of the most important barriers to overcome. Moreover, how and where other food safety analytical techniques may be deceived in a similar manner are further questions deserving great attention.

Finally, the federal government, in collaboration with the CFIA and its key associations, must implement the proper programs to not only require the instatement of approved quality programs, but the legislative authority to monitor compliance with strength and power of enforcement. In essence, Canadian authorities need to agree on a combined approach of command-and-control and economic incentive programs for food safety abatement policies. Hopefully the proposed Food and Consumer Safety Action Plan (FCSAP) will deliver this type of change, in the vanguard of North American food safety and environmental protection legislation. Without these tools and information systems in place, the future integrity and sustainability of the food-web is in great jeopardy, as is the heavily reliant environmental and economic infrastructures that surround it. This issue extends to the most serious levels of public health, with great consequences to sensitive populations, especially for children.

6.0. REFERENCES

- AAFC: Agriculture and Agri-Food Canada (2003). Canadian Food Safety and Quality Program: systems development component. Delivering safe, high-quality food at home and abroad. Available from http://www.agr.gc.ca/fd_al/cfsqp-pcsqa/brochure/pdf/sdc_e.pdf.
- AAVLD (2007): American Association of Veterinary Laboratory Diagnosticians. Protocol for Suspected Pet Food Associated Nephrotoxicity Available from http://data.memberclicks.com/site/aavld/Protocol_Suspected_Pet_Food_Toxicity.pdf.
- Adebiyi A.P., Adebiyi A.O., Jin D.H., Ogawa T. and Muramoto K. (2007). Preparation and Characterization of high-quality rice bran proteins. edible films. *Journal of Food Science and Agriculture*. **87**: 1219-1227.
- Adebiyi A.P., Adebiyi A.O., Jin D.H., Ogawa T. and Muramoto K. (2008). Rice bran protein-based edible films. *International Journal of Food Science and Technology*. **43**: 476-483.
- Agboola S., Ng D. and Mills D. (2005). Characterization and functional properties of Australian rice protein isolates. *Journal of Cereal Science*. **41**: 283-290.
- Akiyama D.M., Coelho S.R., Lawrence A.L. and Robinson E.H. (1989). Apparent digestibility of feedstuffs by the marine shrimp *Penaeus Iannamei* Boone. *Nippon Suisan Gakkaishi*. **55**: 91-98.
- Alomar D., Gallo C., Castaneda M. and Fuchslocher R. (2003): Chemical and discriminant analysis of bovine meat by near infrared reflectance spectroscopy (NIRS). *Meat Science*, **63**: 441-450.
- Andersen W.C., Turnipseed S.B., Karbiwnyk C.M. and Madson M.R. (2007). Determination of Melamine Residues in Catfish Tissue by Triple Quadrupole LC-MS-MS with HILIC Chromatography. USFDA Laboratory Information Bulletin No. 4396, Volume 23. Available from <http://www.cfsan.fda.gov/~frf/lib4396.html>.
- Ansharullah J.A. (1992). Studies of nitrogen extraction of extruded full fat rice bran by alkaline and enzymatic methods. MSc (hons) thesis, School of Food Sciences, University of Western Sydney, Hawkesbury, Richmond, NSW, Australia.
- Ansharullah J.A., Hourigan C.F. and Chesterman (1997). Application of Carbohydrases in Extracting Protein from Rice Bran. *Journal of the Science of Food and Agriculture*. **74**(2): 141-146.
- AOAC (1990). *Official Methods of Analysis* (15th ed.). Washington, DC, USA: Association of Official Analytical Chemists.
- AOAC (1995a). Official Methods of Analysis of AOAC International, Method 920.87, Protein (Total) in Flour Final Action.
- AOAC (1995b). Official Method of Analysis of AOAC International, Method 922.06, Fat in Flour—Acid Hydrolysis Method—Final Action.
- AOAC (2002). Official Method 979.09, Protein in Grains, Official Methods of Analysis, 17th edition. AOAC International, Washington, DC, Rev. 2002.
- AOAC (1995c). Official Methods of Analysis of AOAC International, Method 923.03, Ash of Flour—Direct Method— Final Action.
- AOAC (1995d). Official Methods of Analysis of AOAC International. Method 925.10, Solids and Moisture in Flour—Air Oven Method—Final Action.
- AOAC (1995e). Official Methods of Analysis of AOAC International. Method 979.10 Starch in Cereals—Glucoamylase Method—First Action 1979.
- AOAC (1995f). Official Methods of Analysis of AOAC International. Method 991.43 Total, Soluble, and Insoluble Dietary Fiber in Foods—Enzymatic-gravimetric method, MES-TRIS buffer—Final Action 1994.
- Aristoy M.C. and Toldra F. (1991). Deproteinization techniques for HPLC amino acid analysis in fresh pork muscle and dry-cured ham. *Journal of Agricultural and Food Chemistry*, **39**(10): 1792-1795.
- Arpino P.J. and Guiochon G. (1982). Why the solvent should not be removed in liquid chromatography interface methods. *J. Chromatogr.* **251**: 153.
- Aufrère J., Graviou D., Demarquilly C. and Perez J. (1996). Near infrared reflectance spectroscopy to predict energy value of compound feeds for swine and ruminants. *Animal Feed Science and Technology*, **62**: 77-90.
- Aurand K., Seifert B. and Wegner J. (1982). [Air quality in rooms] Stuttgart: Gustav-Fischer-Verlag, (Ed.) pp. 91-128 (in German).
- AVMA: American Veterinary Medical Association (2007). Melamine and Cyanuric Acid Interaction May Play Part in Illness and Death from Recalled Pet Food. *American Veterinary Medical Association*. Available from http://www.avma.org/press/releases/070501_petfoodrecall.asp.
- Bann B. and Miller S. (1958). Melamine and derivatives of melamine. *Chemical Reviews*. **58**(1): 131.

- Barber S. and De Barber C.B. (1977). "Basic and applied food and feed." In: *Proc Rice by Product Utilisation* (Int Conf, Valencia, Spain, Vol IV), eds Barber S. and Tortosa E. Inst Agroquim Technol Aliment, Valencia, Spain, p.1.
- Bardalaye P.C., Wheeler W.B. and Meister C.W. (1987). Gas chromatographic determination of cyromazine and its degradation product, melamine, in Chinese cabbage. *J. Assoc. Off. Anal. Chem.*, **70**: 455-457.
- Baskin H.A (1966). Production of Cyanuric Acid from Urea. U.S. Patent 3,236,845.
- Belsky A.J., Li Tong-Jin and Brill T.B. (1997). Reactions of cyanamide, dicyandiamide and related cyclic azines in high temperature water. *The Journal of Supercritical Fluids*. **10**(3): 201-208.
- Bennett, J.O. (2005). Soybean seed components as affected by nodal position, environmental conditions, and irrigation. MS thesis. University of Missouri-Columbia.
- Bera M.B. and Mukherjee R. K. (1989). Solubility, emulsifying and foaming properties of rice bran protein concentrates. *Journal of Food Science*. **54**: 142-143.
- Betschart A.A., Fong R.Y. and Saunders R.M. (1977). Rice by products: Comparative extraction and precipitation nitrogen from US and Spanish bran and germ. *Journal of Food Science*. **42**: 1088-1089.
- Bian Y., Myers D.J., Dias K., Lihono M.A., Wu S. and Murphy P.A. (2003). Functional properties of soy protein fractions produced using a pilot plant-scale process. *Journal of the American Oil Chemists' Society*. **80**(6): 545-549.
- Bietz J.A. and Lookhart G.L. (1996). Properties and non-food potential of gluten. *Cereal Foods World*. **41**(5): 376-382.
- Bobeldijk I., Vissers J.P.C., Kearney G., Major H. and van Leerdam J.A. (2001). Screening and identification of unknown contaminants in water with liquid chromatography and quadrupole-orthogonal acceleration-time-of-flight tandem mass spectrometry. *Journal of Chromatography A*, **929**: 63-74.
- Boitsov E.N., Finkel'Shtein A.I. and Petukhov V.A. (1962). Dependence of the Vacuum Ultraviolet Spectra of Sym-Triazine Derivatives on Their Molecular Structure. Optics and Spectroscopy, Vol. 13, p.151.
- Bozzi A., Dhananjeyan M., Guasaquillo I., Parra S., Pulgarin C., Weins C., Kiwi J. (2004). Evolution of toxicity during melamine photocatalysis with TiO₂ suspensions. *J. Photochem. Photobiol. A: Chem.* **162**(1): 179-185.
- Branco-Pardal P., Lalles J.P., Formal M., Guilloteau P. and Toullec R. (1995). Digestion of wheat gluten and potato protein by the preruminant calf: digestibility, amino acid

composition and immunoreactive proteins in ileal digesta. *Reproduction Nutrition Development*. **35**: 639-654.

- Bressani R., Elias L.G. and Juliano B.O. (1971). Evaluation of the protein quality of milled rices differing in protein content. *J. Agric. Food Chem.* **19**: 1028-1034.
- Brunson J.F., Romaine R.P. and Reigh R.C. (1997). Apparent digestibility of selected ingredients in diets for white shrimp *Penaeus setiferus* L. *Aquaculture*. **8**: 9-16.
- Buckee G.K. (1994). Determination of total nitrogen in barley, malt and beer by Kjeldahl procedures and the Dumas Combustion Method. *J. Inst. Brewing*. **100**: 57-64.
- Cagampang G.B., Cruz L.J., Espiritu S.G., Santiago R.G. and Juliano B.O. (1966). Studies on the extraction and composition of rice proteins. *Cereal Chemistry*. **43**: 145-155.
- Cairns T., Siegmund E.G., Stamp J.J. and Skelly J.P. (1983). Liquid chromatography mass spectrometry of dexamethasone and betamethasone *Biomed. Mass Spectrom.* **10**(3): 203-208.
- Canelli E. (1974). Chemical, Bacteriological, and Toxicological Properties of Cyanuric Acid and Chlorinated Isocyanurates as Applied to Swimming Pool Disinfection. *American Journal of Public Health*, **64**: 2.
- Careri M., Mangia A. and Musci M. (1998). Overview of the applications of liquid chromatography-mass spectrometry interfacing systems in food analysis: naturally occurring substances in food. *Journal of Chromatography A*, **794**(1-2): 263-297.
- Castrillo C., Baucells M., Vicente F., Muñoz F. and Andueza D. (2005). Energy evaluation of extruded compound foods for dogs by near-infrared spectroscopy. *Journal of Animal Physiology and Animal Nutrition*, **89**: 194-198.
- Castrillo C., Vicente F. and Guada J.A. (2001). The effect of crude fibre on apparent digestibility and digestible energy content of extruded dog foods. *Journal of Animal Physiology and Animal Nutrition* **85**: 231-236.
- Cattaneo P. and Cantoni C (1982). Presenza di melammina in farina di pesce. *Tecnica Molitoria*. **6**: 17-18.
- CFIA (2007a): Canadian Food Inspection Agency. Food Safety Enhancement Program—HACCP Generic Models. Available from <http://www.inspection.gc.ca/english/fssa/polstrat/haccp/modele.shtml>.
- CFIA (2008): Canadian Food Inspection Agency. Quality Management Program. Available from <http://www.inspection.gc.ca/english/anima/fispoi/qmp/qmppgqe.shtml>.
- CFIA (2000): Canadian Food Inspection Agency. Food Safety Enhancement Program, Implementation Manual, Vol. 2: Guidelines and principles for the development of HACCP

generic models, 2nd edition. Available from
<http://www.inspection.gc.ca/english/fssa/polstrat/haccp/manu/vol2/vol2e.shtml>

CFIA (2007b). Determination of Melamine using Liquid Chromatography Electrospray Ionization Mass Spectrometry (LC/ESI-MS/MS). Calgary Laboratory, Version 1.1.: May 17, 2007.

CFIA (2007c). Determination of Melamine and Cyanuric Acid using Liquid Chromatography Electrospray Ionization Mass Spectrometry (LC/ESI-MS/MS). Calgary Laboratory, Version 1.0.: June 4, 2007.

CFIA (2007d). Screening for Cyanuric Acid using Liquid Chromatography Electrospray Ionization Mass Spectrometry (LC/ESI-MS/MS). Calgary Laboratory, Version 1.0.: May 11, 2007.

CFIA (2007e). Vegetable Protein Concentrate Compliance Verification Strategy. Industry Notice: June 4, 2007. Available from
<http://www.inspection.gc.ca/english/fssa/invenq/inform/20070604e.shtml>.

Chandi G.K. and Sogi D.S. (2007). Biochemical caharacterisation of rice protein fractions. *International Journal of Food Science and Technology*. **42**: 1357-1362.

Chandi G.K. and Sogi D.S. (2007). Functional properties of rice bran concentrates. *Journal of Food Engineering*. **79**: 592-597.

Chang S.K.C. (1994). "Protein analysis" in S.S. Nielsen (ed.). *Chemical Analysis of Foods*. Jones and Bartlett, Boston: 207-219.

Chapman B. and Powell D (2007). Fresh Threat: what's lurking in your salad bowl. *Journal of the Science of Food and Agriculture*. **87**: 1799-1801.

Cheftel J.C., Cuq J.L. and Lorient D. (1985). "Amino acids, peptides and proteins." In: *Food Chemistry* (2nd edn), ed Fennema O.R. Marcel Dekker, New York, USA p.245.

Chemfinder (2008). Chemical Database. Available from:
<http://chemfinder.cambridgesoft.com/chemicals/>.

Chen L. and Houston D.F. (1970). Solubilization and recovery of protein from defatted rice bran. *Cereal Chemistry*. **47**: 72-74.

Chrastil J. (1992). Correlation between the physicochemical and functional properties of rice. *Journal of Agricultural and Food Chemistry*. **40**: 1683-1686.

Clark D.C. and Wilde P.J. (1998). "Mobility of adsorbed protein molecules as studied by fluorescence recovery after photobleaching (FRAP)". In D. Möbius & R. Miller (Eds.), *Proteins at liquid interfaces* (pp. 267-301). Amsterdam: Elsevier.

Clark R. (1966). Melamine crystalluria in sheep. *J S Afr Vet Med Assoc*. **37**: 349-351.

Clayton G.D. and Clayton F.E. (eds.) (1982). *Patty's Industrial Hygiene and Toxicology*. Vol. 2A, 2B, 2C: Toxicology. 3rd Ed. New York: John Wiley Sons: 2771.

CODEX: Codex Alimentarius Commission (1985). General Standard for The Labelling of Prepackaged Foods. *CODEX STAN 1-1985, Rev. 1-1991*. Available from
http://www.caobisco.com/doc_uploads/legislation/STAN1-EN.pdf.

CODEX: Codex Alimentarius Commission (1985). Standard for Rice. Available from
http://www.codexalimentarius.net/download/standards/61/CXS_198e.pdf.

CODEX: Codex Alimentarius Commission (1989). General Standard for Soy Protein Products. *CODEX STAN 175-1989*. Available from
www.codexalimentarius.net/download/standards/325/CXS_175e.pdf.

CODEX: Codex Alimentarius Commission (2001). Alinorm 01/37A: Draft Standard for Wheat Protein Products to Draft Codex Standard for Wheat Protein Products Including Wheat Gluten. Available from <ftp://ftp.fao.org/codex/ALINORM01/al0137ae.pdf>.

CODEX: Codex Alimentarius Commission (2001). Standard for Wheat Protein Products Including Wheat Gluten. *CODEX STAN 163-1987, Rev. 1-2001*. Available from
http://www.codexalimentarius.net/download/standards/323/CXS_163e.pdf.

CODEX: Codex Alimentarius Commission (2005). Alinorm 05/28/26 – Appendix IV (A): Codex Committee on Nutrition and Foods for Special Dietary Uses. Available from
ftp://ftp.fao.org/codex/Alinorm05/al28_26e.pdf.

CODEX: Codex Alimentarius Commission (2005). CX/NFSDU 05/27/6-Add.2 October 2005: Draft revised standard for infant formula and formulas for special medical purposes intended for infants. Available from <ftp://ftp.fao.org/codex/ccnfsdu27/nf2706be.pdf>.

CondéNet (2007). NutritionData. Available from <http://www.nutritiondata.com/>.

Connor M.A., Saunders R.M. and Kohler G.O. (1976). Rice bran protein concentrates obtained by wet alkaline extraction. *Cereal Chemistry*. **53**: 488-492.

Cook A.M. (1987). Biodegradation of s-triazine xenobiotics. *FEMS Microbiol. Rev*. **46**: 93-116.

Coppens P. and Vos A. (1971). Electron Density Distribution in Cyanuric Acid. II. Neutron Diffraction Study at Liquid Nitrogen Temperature and Comparison of X-ray and Neutron Diffraction Results. *Acta Cryst. B27*, 146-158 and references therein.

Cosman F. and Lindsay R. (1999). Selective Estrogen Receptor Modulators: Clinical Spectrum *Endocrinology Reviews*. **20**: 418-434.

Costa L. and Camino (1988). Thermal behavior of melamine. *Journal of Thermal Analysis*. **34**: 423-429.

Covey T.R., Lee E.D. and Henion J.D. (1986). High-speed liquid chromatography/tandem mass spectrometry for the determination of drugs in biological samples. *Anal. Chem.*, **58**: 2453-2460.

Cozzolino D., Damberg R.G., Cynkar W.U., Janik L. and Gishen M. (2006). Review: Analysis of grapes and wine by near infrared spectroscopy. *Journal Near Infrared Spectroscopy* **14**: 279-290.

Cozzolino D., Martins V. and Murray I. (2002). Visible and near infrared spectroscopy of beef longissimus dorsi muscle as a means of discriminating between pasture and corn silage feeding regimes. *Journal of Near Infrared Spectroscopy*, **10**: 187-193.

Croley T.R., Hughes R.J., Koenig B.G. and Metcalfe C.D. (2000). MS Applied to the analysis of estrogens in the environment. *Rapid Commun. Mass Spectrom.* **14**: 1087.

Czechowski M.H. and Stoodley P. (2002). Biofilms and biocides. *Journal of Industrial Microbiology and Biotechnology*. **29**: 325.

De Boever J.L., Vanacker J.M. and De Brabander D.L. (2003). Rumen degradation characteristics of nutrients in compound feeds and the evaluation of tables, laboratory methods and NIRS as predictors. *Animal Feed Science and Technology*, **107**: 29-43.

Deak N.A. and Johnson L.A. (2006). Functional Properties of Protein Ingredients Prepared from High-sucrose/Low-stachyose Soybeans. *Journal of the American Oil Chemists' Society*. **83**(9): 811-818.

Deak N.A., Murphy P.A. and Johnson L.A. (2007). Characterization of fractionated soy proteins produced by a new simplified procedure. *Journal of the American Oil Chemists' Society*. **84**: 137-149.

Deville E.R. and Flinn P.C. (2000) Near infrared (NIR) spectroscopy: an alternative approach for the estimation of forage quality and voluntary intake. In: *Givens D.I., Owen E., Axford R.F.E. and Omedi H.M. (eds) Forage Evaluation in Ruminant Nutrition*. pp. 301 - 320. CAB International.

Delwiche S.R., Graybosch R.A. and Peterson J.C. (1998). Predicting Protein Composition, Biochemical Properties, and Dough-Handling Properties of Hard Red Winter Wheat Flour by Near-Infrared Reflectance. *Cereal Chemistry*. **75**(4): 412-416.

Derbyshire E., Wright D.J. and Boulter D. (1976). Legumin and vicilin storage protein of legume seeds. *Journal of Phytochemistry*. **15**: 3-24.

Dik T., Yöndem-Makascioglu F., Aytaç C.H., Kincal N.S. (2002). Wet separation of wheat flours into starch and gluten fractions: the combined effects of water to flour ratio-dough maturation time and the effects of flour aging and ascorbic acid addition. *Journal of the Science of Food and Agriculture*. **82**: 405-413.

Draber W., Tietjen K., Kluth J.F. and Trebst A. (1991). Herbicides in photosynthesis research. *Angewandte Chemie International. Ed. English*. **30**: 1621-1633.

Dufresne C., Favetta P., Paradis C. and Bouliou R. (2001). Comparative Study of Liquid-Liquid Extraction and Solid-Phase Extraction Methods for the Separation of Sufentanil from Plasma before Gas Chromatographic-Mass Spectrometric Analysis. *Clinical Chemistry*. **47**: 600-602.

Dumas J.B.A. (1831). « Procédés de l'analyse organique ». *Ann. Chim. Phys.* **2**: 198-213.

Eaton R.W. and J.S. Karns (1991). Cloning and comparison of the DNA encoding ammelide aminohydrolase and cyanuric acid amidohydrolase from three *s*-triazine-degrading bacterial strains. *J. Bacteriol.* **173**: 1363-1366.

EFSA (2007): European Food Safety Authority. Provisional Statement on a Request from the European Commission related to Melamine and Structurally related compounds such as Cyanuric Acid in Protein-Rich Ingredients Used for Feed and Food. Available from http://www.efsa.europa.eu/EFSA/Statement/efsa_statement_melamine_en_rev1.0.pdf.

Eggum B.O. and Juliano B.O. (1973). Nitrogen balance in rats fed rices differing in protein content. *J. Sci. Food. Agric.* **24**: 921-927.

El-Sayed W.S., El-Baz A.F. and Othman A.M. (2006). Biodegradation of melamine formaldehyde by *Micrococcus* sp. strain MF-1 isolated from aminoplastic wastewater effluent. *International Biodeterioration & Biodegradation*, **57**(2): 75-81.

El-Shemy H.A., Ahmed S.H., Saneoka H. and Fujita K. (2001). Differences in composition of glycinin and β -conglycinin globulins in some legume cultivars. *American biotechnology laboratory*. **19**(4): 46-48.

El-Shemy H.A., Teraishi M., Khalafalla M.M., Katsube-Tanaka T., Utsumi S. and Ishimoto M. (2004). Isolation of soybean plants with stable transgene expression by visual selection based on green fluorescent protein. *Molecular Breeding*. **14**: 227-238.

Etheridge R.D., Pestib G.M. and Foster E.H. (1998). A comparison of nitrogen values obtained utilizing the Kjeldahl nitrogen and Dumas combustion methodologies (Leco CNS 2000) on samples typical of an animal nutrition analytical laboratory. *Animal Feed Science and Technology*. **73**: 21-28.

FAO (1998). Carbohydrates in human nutrition. Report of a Joint FAO/WHO Expert Consultation. FAO Food and Nutrition Paper 66. Rome, Italy.

- FAO (1999). The state of food insecurity in the world. Rome, Italy: Food and Agriculture Organization of the United Nations.
- FAO (2000). FAO rice information (Vol. 2). Rome, Italy: Food and Agriculture Organization of the United Nations.
- FAO (2001). Specialty rices of the world. Food and Agriculture Organization of the United Nations, Rome, Italy.
- FAO/WHO. (1990) Report of the Joint FAO/WHO Expert Consultation on Protein Quality Evaluation. Food and Agriculture Organization/World Health Organization, Rome, Italy.
- FAO: Food and Agriculture Organization of the United Nations (2004-2008). FAOSTAT Database. Available from <http://faostat.fao.org/>.
- FDA (2007). Import Alert #99-29, Detention without physical examination of all vegetable protein products from China for animal or human food use due to the presence of melamine and/or melamine analogs. Available from http://www.fda.gov/ora/fiars/ora_import_ia9929.html.
- Fekkes D. (1996). State-of-the-art of high-performance liquid chromatographic analysis of amino acids in physiological samples. *Journal of Chromatography B: Biomedical Sciences and Applications*, **682(1)**: 3-22.
- Fialkov A.B., Gordin A. and Amirav A. (2003). Extending the range of compounds amenable for gas chromatography-mass spectrometric analysis. *J Chromatogr A*. **4(2)**: 217-40.
- Fiamegos Y.C., Konidari C.N. and Stalikas C.D. (2003). Cyanuric acid trace analysis by extractive methylation via phase-transfer catalysis and capillary gas chromatography coupled with flame thermoionic and mass-selective detection. Process parameter studies and kinetics. *Analytical Chemistry*, **75(16)**: 4034-4042.
- Filigenzi M.S., Tor R.E., Poppenga R.H., Aston L.A. and Puschner B. (2007). The determination of melamine in muscle tissue by liquid chromatography/tandem mass spectrometry. *Rapid Communications in Mass Spectrometry*. **21(24)**: 4027-4032.
- Finkel'shtein A.I. (1961). The molecular structure of the product of low temperature deamination of melamine. *Journal Gen. Chem. (Russ.)*. **31**: 1046.
- Foster B.J., Harding B.J., Leyland-Jones B. and Hoth D. (1986). Hexamethylmelamine: a critical review of an active drug. *Cancer Treatment Reviews*. **13**: 197-217.
- Fukushima D. (1991). Recent progress of soybean protein foods: chemistry, technology and nutrition. *Food Review International*. **7**: 323-351.
- Gagnon B., McEachern V., & Bray S. (1999). The role of the Canadian government agency in assessing HACCP. *Food Control*, **11**: 359-364.

- Games D.E. (1981). Combined high performance liquid chromatography mass spectrometry. *Biomedical Mass Spectrometry*, **8(9)**: 454-462.
- Garcia J.F. and Barceló D. (1993). An Overview of LC-MS Interfacing Systems with Selected Applications., *J.High.Res.Chromatogr*. **16**: 633-641.
- Garnsworthy P.C., Wiseman J., Feregos K. (2000). Prediction of chemical, nutritive and agronomic characteristics of wheat by near infrared spectroscopy. *Journal of Agricultural Science*, **135**: 409-417.
- Ghose K.C. and Haldar D.P. (1969). Application of cellulase II. Extraction of protein from soybean with fungal cellulases. *Journal of Food Science and Technology (India)*. **6**: 205.
- Gilani G.S. and Sepehr E. (2003). Protein Digestibility and Quality in Products Containing Antinutritional Factors are Adversely Affected by Old Age in Rats. *Journal of Nutrition*. **133**: 220-225.
- Givens D.I. and Deaville E.R. (1999). The current and future role of near infrared reflectance spectroscopy in animal nutrition: a review. *Australian Journal of Agricultural Research*, **50**: 1131-1145.
- Glad C. and Grubb A.O. (1978). Immunocapillary migration—A new method for immunochemical quantitation. *Analytical Biochemistry*, **85(1)**: 180-187.
- GMI (2008): Global Medical Instrumentation Product Catalogue. Available from: <http://www.gmi-inc.com/products.htm>.
- Gnanasambandam R. and Hettiarachchy N.S. (1995). Protein concentrates from unstabilised rice bran: preparation and properties. *Journal of Food Science*. **60**: 1066-1074.
- Gnanasambandam R., Hettiarachchy N.S. and Coleman M. (1997). Mechanical and barrier properties of rice bran films. *Journal of Food Science*. **62**: 395-398.
- GOC (2007): Government of Canada, available from <http://canada.gc.ca/home.html>.
- Gohlke R.S. (1959). Time-of-flight mass spectrometry and gas-liquid partition chromatography. *Anal. Chem.*, **31**: 535.
- Golbitz P. (1995). Traditional soyfoods: processing and products. *Journal of Nutrition*. **125**: 570-572.
- Golbitz P. (2003). Whole Soybeans as Food Ingredient: Soyfoods and Beyond, Practical Short Course on Soyfoods: Ingredients, Preparation and Utilization, held in conjunction with the annual meeting of the American Oil Chemists' Society, Kansas City, MO.

- Goldin A. and Wolpert-Defilippes M.K. (1979). Nude mouse models as predictors of chemotherapy in man: thymidine and pyrimidines. *Bulletin du Cancer (Paris)*. **66**: 61-66.
- Goutailler G., Valette J.C., Guillard C., Paissé O. and Faure R. (2001). Photocatalysed degradation of cyromazine in aqueous titanium dioxide suspensions: comparison with photolysis. *J. Photochem. Photobiol. A: Chem.* **141**(1): 79-84.
- Grala W., Verstegen M.W., Jansman A.J., Huisman J. and Wasilewko J. (1998). Nitrogen utilization in pigs fed diets with soybean and rapeseed products leading to different ileal endogenous nitrogen losses. *Journal of Animal Science*. **76**: 569-577.
- Greenfield H. and Southgate D.A.T. (1992). Food composition data. production, management and use. London: Elsevier Applied Science.
- Griggs T.G., Lobos K.B. and Kingery P.E. (1999). Digestibility analysis of undried, unground and dry ground herbage by near infrared reflectance spectroscopy. *Crop Sci.*, **39**: 1164-1170.
- Grossman M.V., Rao C.S. and Da Silva R.S.F. (1980). Extraction of proteins from buckwheat bran: Application of enzymes. *Journal of Food Biochemistry*. **4**:181.
- Hamada J.S. (1997). Characterization of protein fractions of rice bran to devise effective methods of protein solubilization. *Cereal Chemistry*. **74**: 662-668.
- Hamada J.S. (2000). Characterization and functional properties of rice bran proteins modified by commercial exoproteases and endoproteases. *Journal of Food Science*. **65**(2): 305-310.
- Hammond B.G., Barbee S.J., Inoue T., Ishida N., Levinskas G.J., Stevens M.J., Wheeler A.G. and Cascieri T. (1986) A review of toxicology studies on cyanurate and its chlorinated derivatives. *Environ. Health Perspect.*, **69**, 287-292.
- Hartig C., Storm T. and Jekel M. (1999). Detection and identification of sulphonamide drugs in municipal waste water by liquid chromatography coupled with electrospray ionisation tandem mass spectrometry. *Journal of Chromatography A*, **854**:163-173.
- Haydon, R.M. and Hosken, R.W., 1998. Thermal gelling of soy proteins during canning. 10th Australian Soybean Research Conference, Brisbane, p. 175-179.
- Heck H.D. and Tyl R.W. (1985). The induction of bladder stones by terephthalic acid, dimethyl terephthalate, and melamine (2,4,6-triamino-s-triazine) and its relevance to risk assessment. *Regul Toxicol Pharmacol.* **5**(3): 294-313.
- Hegsted D.M. and Juliano B.O. (1974). Difficulties in assessing the nutritional quality of rice protein. *J. Nutr.* **104**: 772-781.
- Helm R.M. and Burks A.W. (1996). Hypoallergenicity of rice bran protein. *Cereal Foods World*. **41**(11): 839-843.

- Herbstein F.H. (2003). Purported "melamine cyanuric acid trihydrochloride" $C_3H_6N_6 \cdot C_3H_3N_3O_3 \cdot 3HCl$ is actually "diprotonated-melamine cyanuric acid dichloride dihydrate" $(C_3H_8N_6)_2^{2+} \cdot C_3H_3N_3O_3 \cdot 2Cl^- \cdot 2H_2O$. *Journal of Chemical Crystallography* **33**(7): 527-529.
- Hernandez N., Rodriguez-Alegría M.E., Gonzalez F. and Lopez-Munguia A. (2000). Enzymatic treatment of rice bran to improve processing. *Journal of the American Oil Chemists' Society*. **77**(2): 177-180.
- Hoeck J., Fehr W., Murphy P., Welke G (2000). Influence of genotype and environment on isoflavone contents of soybean. *Crop Science*. **40**: 48-51.
- Holmes J.C. and Morrell F.A. (1957). Oscillographic Mass Spectrometric Monitoring of Gas Chromatography. *Appl. Spectrosc.* **11**: 86-87.
- Hoseney R.C. (1994). Principles of Cereal Science and Technology, second edition. Association of Cereal Chemists, St Paul, MN.
- IUCLID (2000): International Uniform Chemical Information Database. Chemical Data Sheet: Substance ID 108-78-1: 30-98.
- Jol S., Kassianenko A., Wszol K. and Oggel J. (2007). The Cold Chain, one link in Canada's food safety initiatives. *Food Control*, **18**: 713-715.
- Jones J.B. (1992). Methods of nitrogen determination in soils and plant tissue. *Commun. Soil Sci. Plant Anal.* **23**: 17-20.
- Ju S.S., Han C.C., Wu C.J., Mebel A.M. and Chen Y.T. (1999). The Fragmentation of Melamine: A Study via Electron-Impact Ionization, Laser-Desorption Ionization, Collision-Induced Dissociation, and Density Functional Calculations of Potential Energy Surface. *J. Phys. Chem. B.*, **103**(3): 582-596.
- Juliano B.O. (1985) "Rice bran." In: Rice: Chemistry and Technology, ed Juliano B.O. American Association of Cereal Chemists, St Paul, MN, USA p.647.
- Juliano B.O. and Hicks P.A. (1993). Utilization of rice functional properties to produce rice food products with modern processing technologies. *Int. Rice Comm. Newsl. (Special Issue: Proc. 17th Session Intl. Rice Comm., 1990)*, **39**: 163-179.
- Kagawa H., Hirano H. and Kikuchi F. (1988). Variation in glutelin seed storage protein in rice (*Oryza sativa* L.). *Japanese Journal of Breeding*. **38**: 327-332.
- Katsube-Tanaka T., Duldulao J.B.A., Kimura Y., Iida S., Yamaguchi T., Nakano J. and Utsumi S. (2004a). The two subfamilies of rice glutelin differ in both primary and higher-order structures. *Biochimica et Biophysica Acta*. **1699**: 95-102.

- Katsube-Tanaka T., Endo T., Iida S., Yamaguchi T. and Nakano J. (2004b). Quality assessment of rice seed protein using the capillary electrophoresis method. Abstracts of the Fourth International Crop Science Congress, Brisbane, Australia, p. 304.
- Katz S.E. and Strusz R.F. (1969). Gas chromatographic separation of several urea herbicides and their metabolites. *J. Agr. Food Chem.* **17**: 1409-1411.
- Kayser B., Acheson K., Decombaz J., Fern E. and Cerretelli P. (1992). Protein absorption and energy digestibility at high altitude. *Journal Applied Physiology*. **73**: 2425-2431.
- Kennedy G. and Burlingame B. (2003). Analysis of food composition data on rice from a plant genetic resources perspective. *Food Chemistry*. **80**: 589-596.
- Kinney A.J., Jung R. and Herman E.M. (2001) Cosuppression of the alpha -subunits of beta -conglycinin in transgenic soybean seeds induces the formation of endoplasmic reticulum-derived protein bodies. *The Plant Cell* **13**: 1165-1178.
- Kishimoto T., Watanabe M., Mitsui T. and Hori H. (1999). Glutelin basic subunits have mammalian mucin-type O-linked disaccharide side chain. *Archives of Biochemistry and Biophysics*. **370**: 271-277.
- Kjeldahl J.T. (1883). *Z. Anal Chem.* **22**: 366.
- Kong X., Zhou H., Qian H. (2007). Enzymatic preparation and functional properties of wheat gluten hydrolysates. *Food Chemistry*. **101**(2): 615-620.
- Korolev V.L., Petukhova T.V., Pivina T.S., Porollo A.A., Sheremetev A.B., Suponitskii K. Yu and Ivshin V.P. (2006). Thermal decomposition mechanisms of nitro-1,2,4-triazoles: A theoretical study. *Russian Chemical Bulletin*. **55**(8): 1388-1410.
- Kurteva V.B. and Afonso C.A.M (2004). Solvent-free synthesis of melamines under microwave irradiation. *Green Chemistry*. **6**: 183-187.
- Kuznesof P.M. (2004) *Sodium dichloroisocyanurate (NaDCC — anhydrous and dihydrate)*. Prepared for the 61st meeting of the Joint FAO/WHO Committee on Food Additives, 10-19 June 2003, Rome.
- Labservices (2007). University of Guelph. *Pet food recall*. Available from <http://www.labservices.uoguelph.ca/urgent.cfm#crystals>.
- Lahalih M.L. and Absi-Halabi M. (1989). Recovery of Solids from Melamine Waste Effluents and Their Conversion to Useful Products. *Industrial & Engineering Chemistry Research*. **28**: 500-504.
- Lasztity R. (1996). *The Chemistry of Cereal Proteins*, second ed. CRC Press, Boca Raton.

- Le X.C., Mingsheng M.A. and Wong N.A. (1996). Speciation of arsenic compounds using high performance liquid chromatography. *Anal. Chem.*, **68**: 4501.
- Lecomte N.B., Zayas J.F. and Kastner C.L. (1993). Soya Proteins Functional and Sensory Characteristics Improved in Cornminuted bleats. *Journal of Food Science*. **58**(3), 464-466.
- Lee M.S. and Kerns E.H. (1999). LC/MS Applications in Drug Development. *Mass Spectrometry Reviews*. **18**(3-4): 187-279.
- Leermakers M., Baeyens W., De Gieter M., Smedts B., Meert C., De Bisschop H.C., Morabito R. and Quevauviller Ph. (2006). Toxic arsenic compounds in environmental samples: Speciation and validation. *Trends in Analytical Chemistry*, **25**(1): 1-10.
- Lens J.P., Mulder W.J. and Kolster P. (1999). Modification of wheat gluten for nonfood applications. *Cereal Foods World*. **44**: 5-9.
- Lew E.J.L., Houston D.F. and Fellers D.A. (1975). A note on protein concentrate from full fat rice bran. *Cereal Chemistry*. **52**: 748.
- Lim L.L., Scherer S.J., Shuler K.D. and Toth, J.P. (1990). Disposition of Cyromazine in Plants under Environmental Conditions. *J. Agric. Food Chem.* **38**: 860-864.
- Linarès, E., Larré, C., Lemeste, M. and Popineau, Y. (2000). Emulsifying and foaming properties of gluten hydrolysates with an increasing degree of hydrolysis: role of soluble and insoluble fractions. *Cereal Chemistry*. **77**(4): 414-420.
- Lipschitz W.L. and Stokey E. (1945), The mode of action of three new diuretics: melamine, adenine and formoguanamine, *J.Pharmacol.Exp.Ther.* **83**: 235-249.
- Lynn J. (1969). "Edible rice bran foods." In: *Protein-Enriched Cereal Foods for World Foods*, ed Milner M. American Association of Cereal Chemists, St Paul, MN, USA, p.154.
- Mackenzie HI (1966). Melamine for sheep. *J S Afr Vet Med Assoc*. **37**: 153-157.
- Maeda M., Ligo M., Tsuda H., Fujita H., Yonemura Y., Nakagawa K., Endo Y. and Sasaki T. (2000). Antimetastatic and antitumor effects of 2,4-diamino-6-(pyridine-4-yl)-1,3,5-triazine (4PyDAT) on the high lung metastatic colon 26 tumor in mice. *Anti-Cancer Drug Design*. **15**: 217-223.
- Mamone G., Ferranti P., Chianese L., Scafuri L. and Addeo F. (2000). Qualitative and quantitative analysis of wheat gluten proteins by liquid chromatography and electrospray mass spectrometry. *Rapid Commun Mass Spectrom*, **14**(10): 897-904.
- Maningart C.C., Demeritt G.K., Chinnaswamy R. and Bassi S.D. (1999). Properties and applications of texturized wheat gluten. *Cereal Foods World*. **44**(9): 650-655.

Martin A.J.P. and Synge R.L.M. (1941). New form of chromatogram employing two liquid phases. *Biochem J.*, **35**: 1358.

Mason S.F. (1982). *Molecular Optical Activity and the Chiral Discriminations*. Cambridge University Press, Cambridge, UK.

Mast R.W. *et al* (1983). Metabolism, disposition and excretion of [¹⁴C]melamine in male Fischer 344 rats. *Food and Chemical Toxicology*. **21**(6): 807-810.

Mast R.W. *et al*. (1983). Metabolism, disposition and excretion of ¹⁴C-melamine in male in male Fischer 344 rats, *Fd. Chem. Toxic.* **21**(6): 807-810.

Mathias J.P., Simanek E.E., Zerkowski J.A., Seto C.T. and Whitesides G.M. (1994). Structural Preferences of Hydrogen Bonding Networks in Organic Solution-- The Cyclic CA₃•M₃ 'Rosette.' *J. Am. Chem. Soc.* **116**: 4316.

MAXXAM Analytics (2007a). Melamine In Pet Foods And Related Products, The Determination of Melamine by LC/MS/MS. Available from http://www.maxxam.ca/content_man/admin/document/Melamine_in_Pet_Food.pdf.

MAXXAM Analytics (2007b). Aminopterin In Pet Foods And Related Products, The Determination of Aminopterin by LC/MS/MS. Available from http://www.maxxam.ca/content_man/admin/document/Aminopterin_in_Pet_Food.pdf.

May H. (1959). Prolysis of melamine. *Journal of Applied Chemistry*. **9**: 340.

Mazaev V.T. (1964). Experimental determination of the maximum permissible concentrations of cyanuric acid, simazine, and a 2-hydroxy derivative of simazine in water reservoirs. *Chem. Abstr.* **62**: 15304.

McKlem L. (2002). *Investigation of molecular forces involved in gelation of commercially prepared soy protein isolates*. MS thesis, NCSU.

McKlem L.K., Lanier T.C., Kwanyuen P. (2002). Thermal reversibility and reformability of commercial soy protein isolate gels. To be submitted to *J. Agric. Food Chem.*

Melnick R.L. *et al*. (1984). Urolithiasis and bladder carcinogenicity of melamine in rodents, *Toxicol. Appl. Pharmacol.* **72**: 292-303.

MFIF: Menu Foods Income Fund (2007). Recall Information. Available from <http://www.menufoods.com/recall/index.html>.

Morita T. and Kiriya S. (1993). Mass production method for rice protein isolate and nutritional evaluation. *Journal of Food Science*. **58**: 1393-1397.

MSU (2007): Michigan State University; Diagnostic Center for Population and Animal Health. Available from <http://newsroom.msu.edu/site/indexer/3263/content.htm>.

Mudgett R.E., Rufner R., Bajracharya R., Kim K. and Rajagopalan K. (1978). Enzymatic effects on cell rupture in plant protein recovery. *Journal of Food Biochemistry*. **2**: 185.

Murata K., Kitagawa T. and Juliano B.O. (1978). Protein quality of high protein rice in rats. *Agric. Biol. Chem.* **42**: 565-570.

Murphy P.A., Barua K. and Hauck C.C. (2002). Solvent extraction selection in the determination of isoflavones in soy foods. *Journal of Chromatography B-Analytical Technologies in the Biomedical and Life Sciences*. **777**: 129-138.

Murray I. and Cowe I. (1992). *Making Light Work: Advances in Near Infrared Spectroscopy* VCH. London.

Murray I. and Cowe I. (2004). Sample Preparation. In: Roberts, C.A., Workman, J., Reeves, III J.B., (Eds.), *Near Infrared Spectroscopy in Agriculture*, ASA, CSSA, SSSA, Madison, USA, pp. 75-115, Chapter 5.

Murray I. and Hall A.P. (1983). Animal feed evaluation by use of near infrared reflectance (NIR) spectrocomputer. *Anal. Proc.* **20**: 75-79.

Murray I. and Williams P.C. (1987). Chemical principles of near-infrared technology. Pages 17-34 in: *Near-Infrared Technology in the Agricultural and Food Industries*. P. C Williams and K. Norris, ed. American Association of Cereal Chemists, Inc., St. Paul, MN.

Nelson G.D. (1967). Swimming pool disinfection with chlorinated isocyanurates. Special report 6862. Monsanto Chemical Co., St. Louis, Mo.

Newton G.L. and Utley P.R. (1978). Melamine as a dietary nitrogen source for ruminants. *Journal of Animal Science*. **47**: 1338-44.

NRC: National Research Council (1993): Nutrient Requirements of Fish. Committee on Animal Nutrition Board on Agriculture, National Academy Press, Washington, DC, p.114.

OECD: Organisation for Economic Co-operation and Development (2002). Screening Information Data Set (SIDS): Melamine CAS N°: 108-78-1. Available from: <http://www.inchem.org/documents/sids/sids/108781.pdf>.

Oleson B.T. (1994). World wheat production, utilization, and trade. In W. Bushuk & V. F. Rasper (Eds.), *Wheat production, properties and quality* (pp. 1-11). New York: Chapman & Hall.

Ono M., Kawahara N., Goto D., Wakabayashi Y., Ushiro S., Yoshida S., Izumi H., Kuwano M. and Sato Y. (1996). Inhibition of Tumor Growth and Neovascularization by an Anti-Gastric Ulcer Agent, Irsogladine. *Cancer Research*. **56**: 1512-1516.

- Osborne B.G., Fearn T. and Hindle P.H. (1993). Practical *NIR Spectroscopy with applications in food and beverage analysis*. Longman Scientific and Technical 2nd edition.
- Otterburn M.S. (1989). "Protein crosslinking." In: *Protein Quality and the Effects of Processing*, eds Phillips R.D. and Finley J.W. Marcel Dekker Inc, New York, USA, p.247.
- Padhye V.W., Salunkhe D.K. (1979). Extraction and characterization of rice proteins. *Cereal Chemistry*. **56**(5): 389-393.
- Palmegiano G. B., Boccignone M., Forneris G., Salvo F., Signorino D., Ziino M., Sicuro B., Gasco L. and Zoccarato I. (2000). Effect of feeding level on nutritional quality of rainbow trout (*Oncorhynchus mykiss*) flesh. *Journal of Agromedicine*. **6**: 69-81.
- Palmegiano G.B., Agradi E., Forneris G., Gai, F., Gasco L., Rigamonti E., Sicuro, B. and Zoccarato, I. (2005). Spirulina as a nutrient source in diets for growing sturgeon (*Acipenser baeri*). *Aquaculture Research*. **16**: 188-195.
- Palmegiano G.B., Costanzo M.T., Daprà F., Gai F., Galletta M.G., Maricchiolo G., Micale V., Peiretti P.G. and Genovese L. (2007). Rice protein concentrate meal as potential dietary ingredient in practical diets for blackspot seabream (*Pagellus bogaraveo*). *Journal of Animal Physiology and Animal Nutrition*. **91**: 235-239.
- Palmegiano G.B., Dapra F., Forneris G., Gai F., Gasco L., Guo K., Peiretti P.G., Sicuro B. and Zoccarato I. (2006). Rice protein concentrate meal as a potential ingredient in practical diets for rainbow trout (*Oncorhynchus mykiss*). *Aquaculture*. **258**: 357-367.
- Panizzi M., Kwanyuen P., Erhan S.Z. (2005). Environmental and genetic variation of beta-conglycinin and glycinin content in brazilian soybean cultivars. Annual Meeting and Expo of the American Oil Chemists' Society; p. 100.
- Pankratov V.A. and Chesnokova A.E. (1989). Polycyclotrimerization of cyanamides. *Russian Chemistry Reviews*. **58**: 879.
- Park P.S., Agnew R.E., Gordon F.J. and Steen R.W.J. (1997). The use of near infrared reflectance spectroscopy (NIRS) on undried samples of grass silage to predict chemical composition and digestibility parameters. *Anim. Feed Sci. Technol.*, **72**: 155-167.
- Pastorello E.A., Farioli L., Pravettoni V., Ispano M., Scibola E., Trambaioli C., Giuffrida M.G., Ansaloni R., Godovac-Zimmermann J., Conti A., Fortunato D. and Ortolani C. (2000). The maize major allergen, which is responsible for food-induced allergic reactions, is a lipid transfer protein. *J. Allergy Clin. Immunol.*, **106**(4): 744-51.
- Patel K. and Jones K. (2007). Analytical method for the quantitative determination of cyanuric acid as the degradation product of sodium dichloroisocyanurate in urine by liquid chromatography mass spectrometry. *Journal of Chromatography B*, **853**(1-2): 360-363.

- Peltonen K. (1986). Gas chromatographic - mass spectrometric determination of volatile compounds arising from epoxy powder paints during the curing process. *Analyst*, **111**: 819-822.
- Penã E., Bernardo A., Soler C. and Jouve N. (2006). Do tyrosine crosslinks contribute to the formation of the gluten network in common wheat (*Triticum aestivum* L.) dough? *Journal of Cereal Science*, **44**(2): 144-153.
- Perdigão L.M.A., Champness N.R. and Beton P.H. (2006). Surface self-assembly of the cyanuric acid-melamine hydrogen bonded network. *Chem. Commun.* 538-540.
- Pérez-Conesa D., Ros G. and Periago M. J. (2002). Protein nutritional quality of infant cereals during processing. *Journal of Cereal Science*. **36**: 125-133.
- Pfeffer E.B., Henrichfreise J.B., Jansen H.D. (1991). Effect of extrusion on efficiency of utilization of maize starch by rainbow trout *Oncorhynchus mykiss*. *Aquaculture*. **96**: 293-303.
- Popineau, Y., Huchet, B., Larré, C. and Bérot, S. (2002). Foaming and emulsifying properties of fractions of gluten peptides obtained by limited enzymatic hydrolysis and ultrafiltration. *Journal of Cereal Science*. **35**: 327-335.
- Potter J.A. (2001). Laboratory 2000 - The challenge of achieving efficiency and compliance. *Journal of Automated Methods & Management in Chemistry*, **23**(6): 89-190.
- Prange A., Birzele B., Modrow H., Hormes J. and Koehler P. (2004). Analysis of sulphur in gluten proteins by X-ray absorption near-edge structure (XANES) spectroscopy. *Royal Society of Chemistry*. **295**: 327-330.
- Puschner B., Poppenga R.H., Lowenstine L.J., Filigenzi M.S. and Pesavento A.P. (2007). Assessment of melamine and cyanuric acid toxicity in cats. *Journal of Veterinary Diagnostic Investigation*. **19**: 616-624.
- QUASU (2007): Queen's University Analytical Services Unit Spectrometry Facility. Available from <http://www.queensu.ca/envst/asu/index.php/publisher/articleview/?PHXSESSID=b9b8a6e8c2f0c6792ffb30e2ff41a4b1&/1/fmArticleID/16/staticId/55/>.
- Ranganathan A., Pedireddi V.R. and Rao C.N.R. (1999). Hydrothermal Synthesis of Organic Channel Structures: 1:1 Hydrogen-Bonded Adducts of Melamine with Cyanuric and Trithiocyanuric Acids. *J. Am. Chem. Soc.*, **121** (8), 1752 -1753, 1999.
- Rao C.N.R. (1993). *Chemical Approaches to the Synthesis of Inorganic Materials*; John Wiley: New York.

- Reeves J.B. III. (2007). The present status of "quick tests" for on-farm analysis with emphasis on manures and soil: What is available and what is lacking? *Livestock Science*, **112**: 224–231.
- Robaina L., Corraze G., Aguirre P., Blanc D., Melcion J.P. and Kaushik S. (1999). Digestibility, postprandial ammonia excretion and selected plasma metabolites in European sea bass (*Dicentrarchus labrax*) fed pelleted or extruded diets with or without wheat gluten. *Aquaculture*, **179**: 45–56.
- Rónyai A. and Gál D. (2005). "Effect of different proportion of rice bran in the diets on the production performance of common carp (*Ciprinus carpio* L)." In: Howell B. and Flos R. (Eds.), *Lessons from the Past to Optimise the Future, Aquaculture Europe 2005*, 5–9 August 2005, Trondheim, Norway EAS Special Publication, vol. 35, pp. 388–389.
- Rossi G. (2002). Set-based Nondeterministic Declarative Programming in Singleton. *Electronic Notes in Theoretical Computer Science*, **76**: 1–17.
- Royal Canin Canada (2007). Royal Canin Canada Voluntarily Recalls Pet Food Products That May Contain Contaminated Rice Protein Concentrate. Available from http://www.royalcanin.ca/release_april20_en.php.
- Sánchez C.C., Rodríguez Niño M.R., Molina Ortiz S.E., Añón M.C. and Rodríguez Patino J.M. (2004). Soy globulin spread films at the air–water interface. *Food Hydrocolloids*, **18**: 335–347.
- Sancho J.V., Ibanez M., Grimalt S., Pozo O.J. and Hernandez F. (2005). Residue determination of cyromazine and its metabolite melamine in chard samples by ion-pair liquid chromatography coupled to electrospray tandem mass spectrometry. *Analytica Chimica Acta*, **530**: 237–243.
- Sauriat-Dorizon H., Maris T. and Wuest J. (2003). Molecular Tectonics. Construction of Porous Hydrogen-Bonded Networks from Bisketals of Pentaerythritol. *Journal of Organic Chemistry*, **68**: 240–246.
- Sayaslan, A. (2004). Wet-milling of wheat flour: Industrial processes and small-scale test methods. *Lebensmittel-Wissenschaft und-Technologie (Food Science and Technology)*, **37**: 499–515.
- Sepehr E., Cooke G., Robertson P., Gilani G.S. (2007). Bioavailability of soy isoflavones in rats Part I: Application of accurate methodology for studying the effects of gender and source of isoflavones. **51**(7): 799–812.
- Shelton D.R., Karns J.S., McCarty G.W. and Durham D.R. (1997). Metabolism of Melamine by *Klebsiella terrigena*. *Applied and Environmental Microbiology*, **63**(7): 2832–2835.
- Shih F.F. (1996). Edible films from rice protein concentrate and pullulan. *Cereal Chemistry*, **73**: 406–409.

- Shih F.F. and Daigle K. (1997). Use of enzymes for the separation of protein from rice flour. *Cereal Chemistry*, **74**: 437–441.
- Shih F.F. and Daigle K.W. (2000). Preparation and characterization of rice protein isolates. *Journal of the American Oil Chemists' Society*, **77**: 885–889.
- Shih F.F., Champagne E.T., Daigle K. and Zarins Z. (1999). Use of enzymes in the processing of protein products from rice bran and rice flour. *Nahrung*, **43**: 14–18.
- Simanek E.E., Li X., Choi L.S. and Whitesides G.M. (1996). Cyanuric Acid and Melamine: A Platform for the Construction of Soluble Aggregates and Crystalline Materials. *Comprehensive supramolecular chemistry*, **9**: 495.
- Simona S., Lobos G., Pannier F., De Gregori I., Pinochet H. and Potin-gautier M. (2004). Speciation analysis of organoarsenical compounds in biological matrices by coupling ion chromatography to atomic fluorescence spectrometry with on-line photooxidation and hydride generation. *Analytica Chimica Acta*, **521**: 99–108.
- Simonne A.H., Simonne E.H., Eitenmiller R.R., Mills H.A. and Cresman C.P. (1997). Could the Dumas Method Replace the Kjeldahl Digestion for Nitrogen and Crude Protein Determinations in Foods? *Journal of the Science of Food and Agriculture*, **73**(1): 39–45.
- Simonne E.H., McCrimmon J.N., Mills H.A., Scoggins-Mantero H.L. and Cresman III C.P. (1995). Adjustments of sufficiency ranges of selected ornamentals and turfgrasses for assessing nitrogen status with Dumas-N data. *Commun. Soil Sci. Plant Anal.*, **26**: 2243–2251.
- Spengler D. and Hamroll B. (1970). Separation and determination of carbamate and urea herbicides by reaction gas chromatography. *J Chromatogr.*, **49**(2): 205–14.
- Staniec P.A., Perdigão L.M.A., Rogers B.L., Champness N.R. and Beton P.H. (2007). Honeycomb Networks and Chiral Superstructures Formed by Cyanuric Acid and Melamine on Au(111). *J. Phys. Chem. C*, **111**(2): 886–893.
- StatsCan: Statistics Canada. Import and Exports (International Trade Statistics) Database. Available from <http://www.statcan.ca/english/tradedata/tradedata.htm>.
- Stein H.H., Pederson C. and Boersma M.G. (2005). Energy and nutrient digestibility in dried distillers grain with solubles by growing pigs. *Journal of Animal Science*, **83**(2): 79.
- Stoner C.E. Jr. and Brill T.B. (1991). Thermal decomposition of energetic materials 46. The formation of melamine-like cyclic azines as a mechanism for ballistic modification of composite propellants by DCD, DAG and DAF. *Combust. Flame*, **83**: 302.
- Sullivan J.A. and Reigh R.C. (1995). Apparent digestibility of selected feedstuffs in diet for hybrid striped bass (*Morone saxatilis*—*Morone chrysops*). *Aquaculture*, **138**: 313–322.

- Surget A., Barron C. (2005). Histologie du grain de blé. *Industrie des Céréales*. **145**: 3-7.
- Takimoto M. and Funakawa T. (1963). Formation of melamine and other cyanamide compounds by polymerization and condensation of dicyandiamide I. The effect of heat on dicyandiamide and melamine studied by the differential thermal analysis under atmospheric and superatmospheric pressure. *Kogyo Kagaku Zasshi*. **66**: 797.
- Tamiri T. and Zitrin S. (1987). Gas chromatography mass spectrometry of some thermally labile urea pesticides. *Biomedical and Environmental Mass Spectrometry*, **14(1)**: 39-42.
- Tang S., Hettiarachchy N.S., Horax R. and Eswaranandam S. (2003). Physiochemical Properties and Functionality of Rice bran Protein Hydrolyzate Prepared from Heat-Stabilized Defatted rice Bran With the Aid of Enzyme. *Journal of Food Science*. **68**: 152-157.
- Tate and Lyle Food & Industrial Ingredients Europe (2005). Wheat Proteins. Available from <http://www.tateandlyle.com>.
- Thompson M., Owen L., Wilkinson K., Wood R. and Damant A. (2002). A comparison of the Kjeldahl and Dumas methods for the determination of protein in foods, using data from a proficiency testing scheme. *The Analyst*. **127**: 1666-1668
- Thurston J., Dudley J., Kaiser D., Hechenbleikner I., Schaefer F. and Holm-Hansen D. (1951). Cyanuric Chloride Derivatives. I. Aminochloro-s-triazines & Reaction of Chloro-s-triazines with Sodium Aryloxides in Aqueous Systems. *Journal of the American Chemical Society*. **73(7)**: 2981-2992.
- Tibbetts S.M., Milley J.E. and Lall S.P. (2006). Apparent protein and energy digestibility of common and alternative feed ingredients by Atlantic cod, *Gadus morhua* (Linnaeus, 1758). *Aquaculture*. **261**: 1314-1327.
- Toth J.P. and Bardalaye P.C. (1987). Capillary gas chromatographic separation and mass spectrometric detection of cyromazine and its metabolite melamine. *J. Chromatogr.*, **408**: 335-340.
- Trank S.J., Johnson D.W. and Cussler E.L. (1989). Isolated Soy Protein Production Using Temperature-Sensitive Gels. *Food Technology*. **43(6)**: 78.
- Tsuji H., Kimoto M. and Natori Y. (2001). Allergens in major crops. *Nutrition Research*. **21**: 925-934.
- Tswett M. (1903). On a new category of adsorption phenomena and its application in biochemical analysis. *Proc. Warsaw Soc. Nat. Sci. (Biol.)*, **14(6)**.
- UCD (2007); University of California Davis. Study Identifies Deadly Chemical Duo in Contaminated Pet Food. Available from http://www.news.ucdavis.edu/search/news_detail.lasso?id=8414.

- Ufodike E.B.C. and Matty A.J. (1983). Growth responses and nutrient digestibility in mirror carp (*Ciprinus carpio*) fed different levels of cassava and rice. *Aquaculture*. **31**: 41-50.
- UOG (2007): University of Guelph Laboratory Services. Urgent Bulletins: Pet Food Recall. Available from <http://www.labservices.uoguelph.ca/urgent.cfm>.
- USDC: United States Department of Commerce (2007). TradeStats Database. Available from <http://tse.export.gov/>.
- USDHHS (1983): U.S. Department of Health and Human Services: NTP Technical Report, TR 245.
- USFDA (1997). Food Additive Petition no. 8B4571 submitted to the U.S. Food and Drug Administration by Occidental Chemical Corporation for "Use of Sodium Dichloroisocyanurate/Sodium Bromide as a Slimicide for the Manufacture of Food Contact Paper."
- USFDA: United States Food and Drug Administration (2007). Pet Food Recall (Melamine)/Tainted Animal Feed. Available from <http://www.fda.gov/oc/opacom/hottopics/petfood.html>.
- Utsumi S., 1992. Plant food protein engineering. *Advances in Food Nutrition Research* **36**: 89-208.
- Van Barneveld R.J., Nuttal J.D., Flinn P.C. and Osborne B.G. (1999). Near infrared reflectance measurements of the digestible energy content of cereals for growing pigs. *Journal of Near Infrared Spectroscopy*, **7**: 1-7.
- Van Der Borgh A., Goesart H., Veraverbeke W.S. and Delcour J.A. (2005). Fractionation of wheat and wheat flour into starch and gluten: overview of the main processes and factors involved. *Journal of Cereal Science*. **41**: 221-237.
- Van Der Borgh A., Vandeputte G.E., Derycke V., Brijs K., Daenen G., and Delcour J.A. (2006). Extractability and chromatographic separation of rice endosperm proteins. *Journal of Cereal Science*. **44**: 68-74.
- Van der Plaats G., Soons H. and Snellings R. (1981). "The thermal Behaviour of Melamine," in: Dollimore D. (Ed.), Proceedings of the Second European Symposium on Thermal Analysis. Heyden, London: p.215.
- Van Kempen T.A.T.G. and Simmons P.H. (1997). Near infrared reflectance spectroscopy in precision feed formulation. *Journal of Applied Poultry Research*, **6**: 471-477.
- Veraverbeke W.S. and Delcour J.A. (2002). Wheat protein composition and properties of wheat glutenin in relation to breadmaking functionality. *Critical Reviews in Food Science and Nutrition*. **42**: 179-208.

- Wackett L.P., Sadowsky M.J., Martinez B. and Shapir N. (2002). Biodegradation of atrazine and related s-triazine compounds: from enzymes to field studies. *Appl Microbiol Biotechnol.* **58**(1): 39-45. [javascript:PopUpMenu2_Set\(Menu11831474\);](#)
- Wang M., Hettiarachchy N.S., Qi M., Burks W. and Siebenmorgen, T. (1999). Preparation and functional properties of rice bran protein isolate. *Journal of Agricultural and Food Chemistry.* **47**(2): 411-416.
- Wang Y., Wei B. and Wang Q. (1990) Crystal structure of melamine cyanuric acid complex (1:1) trihydrochloride, MCA·3HCl. *Journal of Chemical Crystallography* **20**(1): 79-84.
- Whitesides G.M., Simanek E.E., Mathias J.P., Seto C.T., Chin D.N., Mammen M. and Gordon D.M. (1995). Noncovalent Synthesis: Using Physical-Organic Chemistry To Make Aggregates. *Accounts of Chemical Research* **28**: 37-44.
- WHO: World Health Organization (1985). Energy and protein requirements. Report of a Joint FAO/WHO/UNU Expert Consultation WHO Tech. Rep. Ser. 724. Geneva WHO: pp. 206.
- Wieser H., Antes S. and Seilmeier W. (1998). Quantitative determination of gluten protein types in wheat flour by reverse-phase high-performance liquid chromatography. *Cereal Chemistry*, **75**: 644-650.
- Wieser H. and Kieffer R. (2001). Correlations of the amount of gluten protein types to the technological properties of wheat flour determined on microscale. *Journal of Cereal Science*, **34**: 19-27.
- Williams P.C. (1987). Chapter 7 "Variables affecting Near-infrared Reflectance Spectroscopic Analysis" in "Near-infrared Technology in the Agriculture and Food Industries: The American Association of Cereal Chemists, St. Paul, MN. Phil Williams and Karl Norris Co-Editors, pp 143 - 167.
- Williams P., Sobering D. and Antoniszyn J. (1998). "Protein testing methods at the Canadian Grain Commission." Proceedings of the Wheat Protein Symposium. Saskatoon, Sask. March 9 and 10, 1998 [conference paper online]. Available from: http://epe.lac-bac.gc.ca/100/200/301/cgc-ccg/protein_testing_cgc/html/protein1-e2.htm.
- Williams P.C. (1999). Grain-grading in Canada by electronics. *NIR News* **10**(6): 10-12.
- Wojtowicz J. (1993). Cyanuric and Isocyanuric Acids. In Kirk-Othmer Encyclopedia of Chemical Technology, 4th ed., John Wiley & Sons, Inc., New York, vol. 7, pp. 834-850.
- Wrigley C.W. (1998). Potential methodologies and strategies for the rapid assessment of feed-grain quality. *Australian Journal of Agricultural Research.* **50**(5) 789 - 806.

- Xiccato G., Trocino A., Carazzolo A., Meurens M., Maertens L. and Carabaño R. (1999). Nutritive and ingredient prediction of compound feeds for rabbits by nearinfrared reflectance spectroscopy (NIRS). *Animal Feed Science and Technology* **77**, 201-212. St. Paul, MN.
- Xu W., Dong M., Gersen H., Rauls E., Vázquez-Campos S., Crego-Calama M., Reinhoudt D.N., Stensgaard I., Laegsgaard E., Linderroth T.R. and Besenbacher F. (2007). Cyanuric Acid and Melamine on Au(111): Structure and Energetics of Hydrogen-Bonded Networks. *Small* **3**(5): 854-858.
- Yan T. and Agnew R.E. (2004). Prediction of nutritive values in grass silages: I. Nutrient digestibility and energy concentrations using nutrient compositions and fermentation characteristics. *J. Anim. Sci.*, **82**: 1367-1379.
- Yokley R.A. and Cheung M.W. (2000). Analytical Method for the Determination of Atrazine and Its Dealkylated Chlorotriazine Metabolites in Water Using Gas Chromatography/Mass Selective Detection. *J. Agric. Food Chem.*, **48**(10): 4500-4507.
- Zayas J.F. (1997). *Functionality of proteins in food*. Berlin/Heidelberg: Springer.
- Zhang J., Zeng W., Kitchen C., Wang A.Q. and Musson D.G. (2004). High-throughput sample preparation procedures for the quantitation of a new bone integrin $\alpha_5\beta_3$ antagonist in human plasma and urine using liquid chromatography-tandem mass spectrometry. *Journal of Chromatography B, Analytical Technologies in the Biomedical and Life Sciences*, **806**(2): 67-175.
- Zhang W., Nowlan III D.T., Thomson L.M., Lackowski W. and Simanek E. (2001). Orthogonal, Convergent Syntheses of Dendrimers Based on Melamine with One or Two Unique Surface Sites for Manipulation. *Journal of the American Chemical Society.* **123**: 8914-8922.
- Zhao X.T., McCamish M.A., Miller R.H., Wang L. and Lin H.C. (1997). Intestinal transit and absorption of soy protein in dogs depend on load and degree of protein hydrolysis. *Journal of Nutrition.* **127**: 2350-2356.
- Zhao Y., Ware G.M., Kuan S.S. and Carman A.S. (1994). Isolation of Mycotoxins using Extracting Disk: USFDA Laboratory Information Bulletin: DFS/ORL No. 3972. Available from http://www.fda.gov/ora/science_ref/bulletins/lib3972.html.

7.0.1. APPENDIX A

Table 7.1.: Raw Material Data

Study Sample	Coded Sample Name
RMWG1	R58207
RMWG2	T51068
RMWG3	T51089
RMWG4	R58210
RMWG5	R58211
RMWG6	R60254
RMWG7	R60255
RMSP11	W32480
RMSP12	V71834
RMSP13	R77219
RMSP14	T51090
RMSP15	T70822
RMSP16	V85497
RMSP17	W00855
RMRPC1	R79905
RMRPC2	R94777
RMRPC3	R94804
RMRPC4	R94805
RMRPC5	T51086
RMRPC6	T51058
RMRPC7	R77222

Results Name: Untitled 6
Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\Untitled 6
Printing File: 03126-23
Printing Date: Tuesday, May 01, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Rinse	Solvent	CYA-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	127.942.0 amu	2007/05/01 12:14:29 PM
2 Blk 1	Blank	CYA-1	1790.	0.00	N/A	N/A		☐	1.00	46.0	1.91	127.942.0 amu	2007/05/01 12:26:38 PM
3 Blk 2	Blank	CYA-1	2500.	0.00	N/A	N/A		☐	1.00	56.0	1.91	127.942.0 amu	2007/05/01 12:37:21 PM
4 Std 1	Standard	CYA-1	48400.	500.	498.	99.6	☒	☐	1.00	1580.	1.91	127.942.0 amu	2007/05/01 12:48:07 PM
5 Std 2	Standard	CYA-1	90500.	1000.	1010.	101.	☒	☐	1.00	1930.	1.91	127.942.0 amu	2007/05/01 12:58:53 PM
6 Std 3	Standard	CYA-1	132000.	1500.	1510.	101.	☒	☐	1.00	3240.	1.91	127.942.0 amu	2007/05/01 01:09:40 PM
7 Std 4	Standard	CYA-1	215000.	2500.	2500.	100.	☒	☐	1.00	4350.	1.92	127.942.0 amu	2007/05/01 01:20:26 PM
8 Std 5	Standard	CYA-1	336000.	4000.	3970.	99.2	☒	☐	1.00	6600.	1.92	127.942.0 amu	2007/05/01 01:31:13 PM
9 Std 6	Standard	CYA-1	328000.	5000.	3870.	77.5	☐	☐	1.00	8200.	1.90	127.942.0 amu	2007/05/01 01:42:00 PM
10 Std 7 (dil 1:500)	Quality Control	CYA-1	85500.	50000.	945.	1.89	☒	☐	1.00	2470.	1.93	127.942.0 amu	2007/05/01 01:52:46 PM
11 R77222 *	Unknown	CYA-1	44400000.	N/A	535000.	N/A		☐	1.00	462000.	1.93	127.942.0 amu	2007/05/01 02:03:32 PM
12 R79905 *	Unknown	CYA-1	47600000.	N/A	577000.	N/A		☐	1.00	30900.	1.93	127.942.0 amu	2007/05/01 02:14:19 PM
13 R77222 (dil 1:1000)	Unknown	CYA-1	3020000.	N/A	36400.	N/A		☐	1.00	2020.	1.93	127.942.0 amu	2007/05/01 02:25:06 PM
14 R79905 (dil 1:1000)	Unknown	CYA-1	2850000.	N/A	34300.	N/A		☐	1.00	2570.	1.93	127.942.0 amu	2007/05/01 02:35:52 PM
15 R77222 (dil 1:10000)	Unknown	CYA-1	4290000.	N/A	4990.	N/A		☐	1.00	425.	1.93	127.942.0 amu	2007/05/01 02:46:39 PM
16 R79905 (dil 1:10000)	Unknown	CYA-1	394000.	N/A	4670.	N/A		☐	1.00	444.	1.93	127.942.0 amu	2007/05/01 02:57:26 PM
17 R77222 (dil 1:20000)	Unknown	CYA-1	219000.	N/A	2550.	N/A		☐	1.00	462.	1.93	127.942.0 amu	2007/05/01 03:08:13 PM
18 R79905 (dil 1:20000)	Unknown	CYA-1	194000.	N/A	2260.	N/A		☐	1.00	245.	1.92	127.942.0 amu	2007/05/01 03:18:58 PM

$\frac{20000}{945} = 2116$ $\frac{20000}{100} = 200$ $\frac{20000}{100} = 200$ $\frac{20000}{100} = 200$

actual concn = $2116 \times 2550 = 5400$ ppm

actual concn = $47800 \times 2260 = 4780$ ppm

■ Untitled 6 (CYA-1): "Linear" Regression ("1 / (x * x)" weighting): y = 84.1 x + 6.2e+003 (r = 0.9973)

Results Name: CYA-Val_Rice_ptn_070501.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\CYA-Val_Rice_ptn_070501.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\CVA-Val_Rice_ptn_070501.rdb

Results Name: CVA-Val_Rice_ptn_070501.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\CVA-Val_Rice_ptn_070501.rdb

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Rinse	Solvent	CYA-2	0.00	N/A	N/A		☐	1.00	N/A	0.00	127.9/85.0 amu	2007/05/01 12:14:29 PM
2 Blk 1	Blank	CYA-2	0.00	N/A	N/A		☐	1.00	N/A	0.00	127.9/85.0 amu	2007/05/01 12:26:38 PM
3 Blk 2	Blank	CYA-2	0.00	N/A	N/A		☐	1.00	N/A	0.00	127.9/85.0 amu	2007/05/01 12:37:21 PM
4 Std 1	Standard	CYA-2	26900.	488.	97.7	☒	☐	1.00	254.	1.92	127.9/85.0 amu	2007/05/01 12:48:07 PM
5 Std 2	Standard	CYA-2	52100.	997.	99.7	☒	☐	1.00	694.	1.92	127.9/85.0 amu	2007/05/01 12:58:53 PM
6 Std 3	Standard	CYA-2	75100.	1460.	97.3	☒	☐	1.00	826.	1.92	127.9/85.0 amu	2007/05/01 1:09:40 PM
7 Std 4	Standard	CYA-2	122000.	2410.	96.4	☒	☐	1.00	933.	1.92	127.9/85.0 amu	2007/05/01 1:20:28 PM
8 Std 5	Standard	CYA-2	195000.	3890.	97.2	☒	☐	1.00	2060.	1.92	127.9/85.0 amu	2007/05/01 1:31:13 PM
9 Std 6	Standard	CYA-2	189000.	3750.	75.0	☐	☐	1.00	1860.	1.91	127.9/85.0 amu	2007/05/01 1:42:00 PM
10 Std 7 (dil 1:500)	Quality Control	CYA-2	49600.	946.	1.89	☒	☐	1.00	508.	1.94	127.9/85.0 amu	2007/05/01 1:52:48 PM
11 R77222	Unknown	CYA-2	29100000.	587000.	N/A		☐	1.00	114000.	1.93	127.9/85.0 amu	2007/05/01 2:03:32 PM
12 R79905	Unknown	CYA-2	31000000.	624000.	N/A		☐	1.00	42500.	1.93	127.9/85.0 amu	2007/05/01 2:14:19 PM
13 R77222 (dil 1:1000)	Unknown	CYA-2	1790000.	36100.	N/A		☐	1.00	3150.	1.93	127.9/85.0 amu	2007/05/01 2:25:06 PM
14 R79905 (dil 1:1000)	Unknown	CYA-2	1710000.	34300.	N/A		☐	1.00	3340.	1.93	127.9/85.0 amu	2007/05/01 2:35:52 PM
15 R77222 (dil 1:10000)	Unknown	CYA-2	245000.	4880.	N/A		☐	1.00	645.	1.94	127.9/85.0 amu	2007/05/01 2:46:39 PM
16 R79905 (dil 1:10000)	Unknown	CYA-2	230000.	4570.	N/A		☐	1.00	645.	1.94	127.9/85.0 amu	2007/05/01 2:57:26 PM
17 R77222 (dil 1:20000)	Unknown	CYA-2	127000.	2510.	N/A		☐	1.00	355.	1.93	127.9/85.0 amu	2007/05/01 3:08:13 PM
18 R79905 (dil 1:20000)	Unknown	CYA-2	112000.	2200.	N/A		☐	1.00	269.	1.93	127.9/85.0 amu	2007/05/01 3:18:58 PM
19 LOD 1	Quality Control	CYA-2	28400.	519.	104.	☒	☐	1.00	97.5	1.93	127.9/85.0 amu	2007/05/01 3:29:46 PM
20 LOD 2	Quality Control	CYA-2	28300.	515.	103.	☒	☐	1.00	120.	1.93	127.9/85.0 amu	2007/05/01 3:40:31 PM
21 LOD 3	Quality Control	CYA-2	28400.	518.	104.	☒	☐	1.00	65.4	1.93	127.9/85.0 amu	2007/05/01 3:51:17 PM
22 LOD 4	Quality Control	CYA-2	29000.	530.	106.	☒	☐	1.00	84.5	1.93	127.9/85.0 amu	2007/05/01 4:02:03 PM
23 LOD 5	Quality Control	CYA-2	30100.	552.	110.	☒	☐	1.00	102.	1.93	127.9/85.0 amu	2007/05/01 4:12:49 PM
24 LOD 6	Quality Control	CYA-2	30200.	554.	111.	☒	☐	1.00	110.	1.93	127.9/85.0 amu	2007/05/01 4:23:37 PM
25 LOD 7	Quality Control	CYA-2	31600.	583.	117.	☒	☐	1.00	96.6	1.93	127.9/85.0 amu	2007/05/01 4:34:22 PM
26 LOD 8	Quality Control	CYA-2	26900.	487.	97.5	☒	☐	1.00	69.5	1.93	127.9/85.0 amu	2007/05/01 4:45:09 PM
27 Spk 2	Quality Control	CYA-2	82000.	1600.	107.	☒	☐	1.00	310.	1.93	127.9/85.0 amu	2007/05/01 4:55:57 PM
28 Spk 2A	Quality Control	CYA-2	81900.	1600.	106.	☒	☐	1.00	301.	1.93	127.9/85.0 amu	2007/05/01 5:06:43 PM
29 Spk 2B	Quality Control	CYA-2	82300.	1600.	107.	☒	☐	1.00	227.	1.93	127.9/85.0 amu	2007/05/01 5:17:29 PM
30 Spk 3	Quality Control	CYA-2	197000.	3910.	97.7	☒	☐	1.00	555.	1.93	127.9/85.0 amu	2007/05/01 5:28:15 PM
31 Spk 3A	Quality Control	CYA-2	12600.	201.	5.02	☒	☐	1.00	32.4	1.97	127.9/85.0 amu	2007/05/01 5:39:01 PM
32 Spk 3B	Quality Control	CYA-2	214000.	4260.	107.	☒	☐	1.00	1270.	1.93	127.9/85.0 amu	2007/05/01 5:49:46 PM
33 Std 1	Standard	CYA-2	27100.	493.	98.6	☒	☐	1.00	197.	1.93	127.9/85.0 amu	2007/05/01 6:00:32 PM
34 Std 2	Standard	CYA-2	57900.	1110.	111.	☒	☐	1.00	195.	1.93	127.9/85.0 amu	2007/05/01 6:11:20 PM
35 Std 3	Standard	CYA-2	76500.	1490.	99.2	☒	☐	1.00	317.	1.93	127.9/85.0 amu	2007/05/01 6:22:06 PM
36 Std 4	Standard	CYA-2	128000.	2530.	101.	☒	☐	1.00	545.	1.93	127.9/85.0 amu	2007/05/01 6:32:53 PM
37 Std 5	Standard	CYA-2	207000.	4130.	103.	☒	☐	1.00	713.	1.93	127.9/85.0 amu	2007/05/01 6:43:40 PM
38 Std 6	Standard	CYA-2	246000.	4910.	98.2	☒	☐	1.00	1080.	1.93	127.9/85.0 amu	2007/05/01 6:54:27 PM

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/24 12:42:34 PM
2 Blk	Blank	MEL-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/24 12:50:05 PM
3 Std 1	Standard	MEL-1	105000.	500.	553.	111.	☒	☐	1.00	1020.	1.86	2007/04/24 12:55:40 PM
4 Std 2	Standard	MEL-1	198000.	1000.	1100.	110.	☒	☐	1.00	2870.	1.86	2007/04/24 01:01:14 PM
5 Std 3	Standard	MEL-1	284000.	1500.	1600.	107.	☒	☐	1.00	2290.	1.86	2007/04/24 01:06:48 PM
6 Std 4	Standard	MEL-1	456000.	2500.	2610.	104.	☒	☐	1.00	4700.	1.86	2007/04/24 01:12:23 PM
7 Std 5	Standard	MEL-1	717000.	4000.	4130.	103.	☒	☐	1.00	7180.	1.86	2007/04/24 01:17:58 PM
8 Std 6	Standard	MEL-1	847000.	5000.	4900.	97.9	☒	☐	1.00	7420.	1.86	2007/04/24 01:23:33 PM
9 R79805	Unknown	MEL-1	1350000.	N/A	7840.	N/A	☐	☐	1.00	10500.	1.87	2007/04/24 01:29:08 PM
10 R77222	Unknown	MEL-1	2000000.	N/A	11700.	N/A	☐	☐	1.00	8040.	1.87	2007/04/24 01:34:43 PM
11 Spk 1	Quality Control	MEL-1	90400.	500.	488.	93.5	☒	☐	1.00	352.	1.86	2007/04/24 01:40:18 PM
13 R94777	Unknown	MEL-1	31700.	N/A	124.	N/A	☐	☐	1.00	177.	1.87	2007/04/24 01:51:28 PM
14 Spk 1A	Quality Control	MEL-1	81400.	500.	414.	82.9	☒	☐	1.00	629.	1.87	2007/04/24 01:57:03 PM
15 R94804	Unknown	MEL-1	7120.	N/A	< 0	N/A	☐	☐	1.00	66.2	1.87	2007/04/24 02:02:38 PM
16 R94805	Unknown	MEL-1	657000.	N/A	3790.	N/A	☐	☐	1.00	6260.	1.87	2007/04/24 02:08:12 PM
17 Std 1	Standard	MEL-1	81100.	500.	413.	82.6	☒	☐	1.00	475.	1.87	2007/04/24 02:13:47 PM
18 Std 6	Standard	MEL-1	732000.	5000.	4220.	84.5	☒	☐	1.00	7910.	1.87	2007/04/24 02:19:22 PM
19 Dil QC (1:20)	Quality Control	MEL-1	698000.	50000.	4030.	8.05	☒	☐	1.00	6010.	1.85	2007/04/24 02:24:58 PM
20 R79805 (dil 1:20)	Unknown	MEL-1	153000.	N/A	831.	N/A	☐	☐	1.00	1100.	1.87	2007/04/24 02:30:33 PM
21 R77222 (dil 1:20)	Unknown	MEL-1	235000.	N/A	1310.	N/A	☐	☐	1.00	1590.	1.86	2007/04/24 02:36:08 PM

$R79905 = 831 \text{ ppb} \times 20 = 16620 \text{ ppb} \times 12.4 = 10.3 \text{ ppm}$

$R77222 = 1310 \text{ ppb} \times 20 = 26200 \text{ ppb} \times 12.4 = 16.2 \text{ ppm}$

CF for enhancement = $\frac{20}{\frac{8.05 \times 20}{100}} = 12.4$

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-rice ptn_070424.rdb

Printing Date: Tuesday, April 24, 200

Method Name: APT-MEL_070409.qmf

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Results Name: AptMel-rice ptn_070424.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-rice ptn_070424.rdb

Printing Time: 02:54:53

Printing Date: Tuesday, April 24, 200

■ AptMel-rice ptn_070424.rdb (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): y = 171 x + 1.05e+004 (r = 0.9878)

Method Name: APT-MEL_070409.qmf

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Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-rice ptn_070424.rdb

Printing Date: Tuesday, April 24, 2007

	Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1	Rinse	Solvent	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/24 12:42:34 PM
2	Blk	Blank	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/24 12:50:05 PM
3	Std 1	Standard	MEL-3	7760.	500.	564.	113.	☒	☐	1.00	39.3	1.85	2007/04/24 12:55:40 PM
4	Std 2	Standard	MEL-3	13900.	1000.	1060.	106.	☒	☐	1.00	794.	1.85	2007/04/24 01:01:14 PM
5	Std 3	Standard	MEL-3	20500.	1500.	1600.	107.	☒	☐	1.00	1150.	1.86	2007/04/24 01:06:48 PM
6	Std 4	Standard	MEL-3	32600.	2500.	2570.	103.	☒	☐	1.00	1220.	1.86	2007/04/24 01:12:23 PM
7	Std 5	Standard	MEL-3	52900.	4000.	4220.	106.	☒	☐	1.00	1430.	1.86	2007/04/24 01:17:58 PM
8	Std 6	Standard	MEL-3	61800.	5000.	4940.	98.8	☒	☐	1.00	1970.	1.86	2007/04/24 01:23:33 PM
9	R79905	Unknown	MEL-3	97800.	N/A	7850.	N/A		☐	1.00	2700.	1.86	2007/04/24 01:28:08 PM
10	R77222	Unknown	MEL-3	147000.	N/A	11800.	N/A		☐	1.00	2780.	1.86	2007/04/24 01:34:43 PM
11	Spk 1	Quality Control	MEL-3	6190.	500.	437.	87.4	☒	☐	1.00	80.3	1.85	2007/04/24 01:40:18 PM
13	R94777	Unknown	MEL-3	2420.	N/A	132.	N/A		☐	1.00	86.3	1.88	2007/04/24 01:51:28 PM
14	Spk 1A	Quality Control	MEL-3	5970.	500.	419.	83.8	☒	☐	1.00	145.	1.86	2007/04/24 01:57:03 PM
15	R94804	Unknown	MEL-3	723.	N/A	< 0	N/A		☐	1.00	19.4	1.87	2007/04/24 02:02:38 PM
16	R94805	Unknown	MEL-3	48600.	N/A	3870.	N/A		☐	1.00	1170.	1.87	2007/04/24 02:08:12 PM
17	Std 1	Standard	MEL-3	5680.	500.	412.	82.4	☒	☐	1.00	163.	1.87	2007/04/24 02:13:47 PM
18	Std 6	Standard	MEL-3	53400.	5000.	4260.	85.2	☒	☐	1.00	1250.	1.86	2007/04/24 02:19:22 PM
19	Dil QC (1:20)	Quality Control	MEL-3	51900.	0.00	4140.	N/A	☒	☐	1.00	2290.	1.85	2007/04/24 02:24:58 PM
20	R79905 (dil 1:20)	Unknown	MEL-3	11300.	N/A	851.	N/A		☐	1.00	351.	1.86	2007/04/24 02:30:33 PM
21	R77222 (dil 1:20)	Unknown	MEL-3	17500.	N/A	1350.	N/A		☐	1.00	630.	1.86	2007/04/24 02:36:08 PM

Method Name: APT-MEL_070409.qmf

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Results Name: AptMel-WG_070718.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-WG_070718.rdb

Printing Time: 8:53:15

Printing Date: Thursday, July 19, 2007

	Sample Name	Use Record	Sample Type	Analyte Peak Name	Analyte Mass Ranges (amu)	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Analyte Retention Time (min)	Accuracy (%)	Record Modified	Acquisition Date
1	Blk		Blank	MEL-1	126.8/85.2 amu	1200.	0.00	N/A	2.04	N/A	☐	2007/07/18 5:38:19 PM
2	Std 1	☒	Standard	MEL-1	126.8/85.2 amu	179000.	500.	501.	2.04	100.	☐	2007/07/18 5:43:54 PM
3	Std 2	☒	Standard	MEL-1	126.8/85.2 amu	317000.	1000.	960.	2.04	96.0	☐	2007/07/18 5:49:29 PM
4	Std 3	☒	Standard	MEL-1	126.8/85.2 amu	495000.	1500.	1550.	2.04	103.	☐	2007/07/18 5:55:04 PM
5	Std 4	☒	Standard	MEL-1	126.8/85.2 amu	840000.	2500.	2700.	2.04	108.	☐	2007/07/18 6:00:39 PM
6	Std 5	☒	Standard	MEL-1	126.8/85.2 amu	1170000.	4000.	3790.	2.04	94.9	☐	2007/07/18 6:06:14 PM
7	Std 6	☒	Standard	MEL-1	126.8/85.2 amu	1500000.	5000.	4880.	2.03	97.6	☐	2007/07/18 6:11:49 PM
8	Spk 1	☒	Quality Control	MEL-1	126.8/85.2 amu	164000.	500.	451.	2.04	90.3	☐	2007/07/18 6:17:24 PM
9	T51068		Unknown	MEL-1	126.8/85.2 amu	3610.	N/A	< 0	2.04	N/A	☐	2007/07/18 6:22:59 PM
10	T51089		Unknown	MEL-1	126.8/85.2 amu	1440.	N/A	< 0	2.04	N/A	☐	2007/07/18 6:28:34 PM
11	Spk 1A	☒	Quality Control	MEL-1	126.8/85.2 amu	153000.	500.	414.	2.04	82.7	☐	2007/07/18 6:34:09 PM

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■ Untitled 5 (MEL-1): "Linear" Regression ("1 / (x * x)" weighting); y = 301 x + 2.83e+004 (r = 0.9975)

Results Name: AptMel-WG_070718.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-WG_070718.rdb

Printing Time: 8:52:08

Printing Date: Thursday, July 19, 200

Sample Name	Sample Comment	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Blk		Blank	CYA-1	524	0.00	N/A	N/A			2.05	127.9/42.0 amu	2007/07/19 8:38:57 PM
2 Std 1		Standard	CYA-1	47100	500	498	99.7	☒		2.06	127.9/42.0 amu	2007/07/19 8:48:44 PM
3 Std 2		Standard	CYA-1	94100	1000	963	96.3	☒		2.06	127.9/42.0 amu	2007/07/19 9:00:31 PM
4 Std 3		Standard	CYA-1	158000	1500	1600	106	☒		2.06	127.9/42.0 amu	2007/07/19 9:11:18 PM
5 Std 4		Standard	CYA-1	257000	2500	2580	103	☒		2.06	127.9/42.0 amu	2007/07/19 9:22:05 PM
6 Std 5		Standard	CYA-1	404000	4000	4030	101	☒		2.06	127.9/42.0 amu	2007/07/19 9:32:52 PM
7 Std 6		Standard	CYA-1	472000	5000	4700	94.0	☒		2.06	127.9/42.0 amu	2007/07/19 9:43:39 PM
8 Spk 1	A773799	Quality Control	CYA-1	51900	500	546	109	☒		2.06	127.9/42.0 amu	2007/07/19 9:54:26 PM
9 T51068	A773799	Unknown	CYA-1	2820	N/A	60.8	N/A			2.07	127.9/42.0 amu	2007/07/19 10:05:13 P
10 T51089	A773804	Unknown	CYA-1	2250	N/A	55.1	N/A			2.12	127.9/42.0 amu	2007/07/19 10:16:00 P
11 Spk 1A		Quality Control	CYA-1	36200	500	391	78.1	☒		2.09	127.9/42.0 amu	2007/07/19 10:26:47 P

■ Untitled 3 (CYA-1): "Linear" Regression ("1 / (x * x)" weighting): y = 101 x + -3.33e+003 (r = 0.9979)

Results Path: R:\Projects\Residue\Aminopterin\Results\CYA-WG_070719.rdb

Printing Date: Friday, July 20, 2007

Results Name: CYA-WG_070719.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\CYA-WG_070719.rdb

Printing Time: 2:08:44 PM

Printing Date: Friday, July 20, 2007

Sample Name	Sample Comment	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1	Blk	Blank	CYA-2	0.00	0.00	N/A	N/A			0.00	127.9/85.0 amu	2007/07/19 8:38:57 PM
2	Std 1	Standard	CYA-2	27900.	500.	495.	99.0	☒		2.07	127.9/85.0 amu	2007/07/19 8:49:44 PM
3	Std 2	Standard	CYA-2	56200.	1000.	987.	96.7	☒		2.06	127.9/85.0 amu	2007/07/19 9:00:31 PM
4	Std 3	Standard	CYA-2	95100.	1500.	1620.	108.	☒		2.07	127.9/85.0 amu	2007/07/19 9:11:18 PM
5	Std 4	Standard	CYA-2	156000.	2500.	2630.	105.	☒		2.07	127.9/85.0 amu	2007/07/19 9:22:05 PM
6	Std 5	Standard	CYA-2	229000.	4000.	3860.	96.5	☒		2.07	127.9/85.0 amu	2007/07/19 9:32:52 PM
7	Std 6	Standard	CYA-2	282000.	5000.	4740.	94.9	☒		2.07	127.9/85.0 amu	2007/07/19 9:43:39 PM
8	Spk 1	Quality Control	CYA-2	31100.	500.	547.	109.	☒		2.07	127.9/85.0 amu	2007/07/19 9:54:26 PM
9	T51068	A773789	CYA-2	1630.	N/A	55.7	N/A			2.08	127.9/85.0 amu	2007/07/19 10:05:13 PM
10	T51089	A773804	CYA-2	1030.	N/A	45.9	N/A			2.14	127.9/85.0 amu	2007/07/19 10:16:00 PM
11	Spk 1A	Quality Control	CYA-2	21100.	500.	380.	76.1	☒		2.10	127.9/85.0 amu	2007/07/19 10:26:47 PM

Sample Name	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Rinse	Solvent	CYA-1	0.00	0.00	N/A	N/A			N/A	0.00	127.942.0 amu	2007/04/30 2:04:51 PM
2 Blk	Blank	CYA-1	465	0.00	N/A	N/A			9.81	1.91	127.942.0 amu	2007/04/30 2:17:19 PM
3 Std 1	Standard	CYA-1	29500	500	495	99.0	☒		725	1.91	127.942.0 amu	2007/04/30 2:28:00 PM
4 Std 2	Standard	CYA-1	60800	1000	940	94.0	☒		1560	1.91	127.942.0 amu	2007/04/30 2:38:41 PM
5 Std 3	Standard	CYA-1	145000	2000	2130	107	☒		3960	1.92	127.942.0 amu	2007/04/30 2:49:22 PM
6 Std 4	Standard	CYA-1	205000	3000	2980	99.4	☒		3030	1.91	127.942.0 amu	2007/04/30 3:00:03 PM
7 Std 5	Standard	CYA-1	329000	5000	4760	95.1	☒		4830	1.91	127.942.0 amu	2007/04/30 3:10:44 PM
8 Std 6	Standard	CYA-1	450000	8000	6470	80.9	☐		7280	1.91	127.942.0 amu	2007/04/30 3:21:25 PM
9 R94777	Unknown	CYA-1	1300000	N/A	18500	N/A			25300	1.92	127.942.0 amu	2007/04/30 3:32:06 PM
10 R94804	Unknown	CYA-1	9280000	N/A	132000	N/A			120000	1.92	127.942.0 amu	2007/04/30 3:42:47 PM
11 R94805	Unknown	CYA-1	2100000	N/A	30000	N/A			9050	1.92	127.942.0 amu	2007/04/30 3:53:28 PM
12 R94777 (dil 1:100)	Unknown	CYA-1	145000	N/A	2130	N/A			913	1.92	127.942.0 amu	2007/04/30 4:04:09 PM
13 R94804 (dil 1:500)	Unknown	CYA-1	482000	N/A	6820	N/A			4350	1.92	127.942.0 amu	2007/04/30 4:14:52 PM
14 R94805 (dil 1:100)	Unknown	CYA-1	213000	N/A	3110	N/A			2160	1.92	127.942.0 amu	2007/04/30 4:25:34 PM
15 Std 5 (dil 1:50)	Quality Control	CYA-1	33800	5000	556	11.1	☒		322	1.93	127.942.0 amu	2007/04/30 4:36:16 PM
16 R94804 (dil 1:1000)	Unknown	CYA-1	243000	N/A	3540	N/A			3290	1.93	127.942.0 amu	2007/04/30 4:46:58 PM
17 Std 2	Standard	CYA-1	69000	1000	1060	106	☒		881	1.92	127.942.0 amu	2007/04/30 4:57:40 PM
18 Spk 1	Quality Control	CYA-1	41300	500	663	133	☒		462	1.92	127.942.0 amu	2007/04/30 5:08:21 PM
19 Spk 2	Quality Control	CYA-1	96100	1000	1020	102	☒		1130	1.92	127.942.0 amu	2007/04/30 5:19:02 PM

actual dilution factor = dil fact
% acc of spike = $\frac{50 \times 556}{5000} = 5.56\%$
% acc of spike = $\frac{556}{100} = 5.56\%$
% actual dilution $\rightarrow 1:100$ dilution = $\frac{100}{556} = 18$
factors for $\rightarrow 1:1000$ dilution = $\frac{1000}{556} = 180$

% actual concn $294777 = 2130 \text{ ppb} \times 18 = 38340 \text{ ppb} = 38.34 \text{ ppm}$
 $294805 = 3110 \text{ ppb} \times 18 = 55980 \text{ ppb} = 56.0 \text{ ppm}$
 $294804 = 3540 \text{ ppb} \times 180 = 637200 \text{ ppb} = 637.2 \text{ ppm}$

■ CYA-rice ptn_070430.rdb (CYA-1): "Linear" Regression ("1 / (x * x)" weighting): y = 70.3 x + -5.35e+003 (r = 0.9967)

Sample Name	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Rinse	Solvent	CYA-2	0.00	0.00	N/A	N/A			N/A	0.00	127.9/85.0 amu	2007/04/30 2:04:51 PM
2 Blk	Blank	CYA-2	0.00	0.00	N/A	N/A			N/A	0.00	127.9/85.0 amu	2007/04/30 2:17:19 PM
3 Std 1	Standard	CYA-2	15600	500	495	99.0	☒		92.6	1.92	127.9/85.0 amu	2007/04/30 2:28:00 PM
4 Std 2	Standard	CYA-2	34700	1000	943	94.3	☒		236	1.92	127.9/85.0 amu	2007/04/30 2:38:41 PM
5 Std 3	Standard	CYA-2	84900	2000	2120	106	☒		483	1.92	127.9/85.0 amu	2007/04/30 2:49:22 PM
6 Std 4	Standard	CYA-2	121000	3000	2970	99.0	☒		1470	1.92	127.9/85.0 amu	2007/04/30 3:00:03 PM
7 Std 5	Standard	CYA-2	196000	5000	4790	95.7	☒		1630	1.92	127.9/85.0 amu	2007/04/30 3:10:44 PM
8 Std 6	Standard	CYA-2	267000	8000	6410	80.1	☐		1280	1.92	127.9/85.0 amu	2007/04/30 3:21:25 PM
9 R94777	Unknown	CYA-2	787000	N/A	19600	N/A			3560	1.93	127.9/85.0 amu	2007/04/30 3:32:06 PM
10 R94804	Unknown	CYA-2	5680000	N/A	134000	N/A			20400	1.93	127.9/85.0 amu	2007/04/30 3:42:47 PM
11 R94805	Unknown	CYA-2	1270000	N/A	30000	N/A			4480	1.92	127.9/85.0 amu	2007/04/30 3:53:28 PM
12 R94777 (dil 1:100)	Unknown	CYA-2	85800	N/A	2140	N/A			299	1.93	127.9/85.0 amu	2007/04/30 4:04:09 PM
13 R94804 (dil 1:500)	Unknown	CYA-2	283000	N/A	6790	N/A			1360	1.93	127.9/85.0 amu	2007/04/30 4:14:52 PM
14 R94805 (dil 1:100)	Unknown	CYA-2	130000	N/A	3180	N/A			836	1.93	127.9/85.0 amu	2007/04/30 4:25:34 PM
15 Std 5 (dil 1:50)	Quality Control	CYA-2	20700	0.00	613	N/A	☒		90.5	1.94	127.9/85.0 amu	2007/04/30 4:36:16 PM
16 R94804 (dil 1:1000)	Unknown	CYA-2	143000	N/A	3490	N/A			965	1.93	127.9/85.0 amu	2007/04/30 4:46:58 PM
17 Std 2	Standard	CYA-2	39600	1000	1060	106	☒		286	1.92	127.9/85.0 amu	2007/04/30 4:57:40 PM
18 Spk 1	Quality Control	CYA-2	24200	500	697	139	☒		154	1.92	127.9/85.0 amu	2007/04/30 5:08:21 PM
19 Spk 2	Quality Control	CYA-2	40800	1000	1090	109	☒		416	1.93	127.9/85.0 amu	2007/04/30 5:19:02 PM

Sample Name	Use Record	Sample Type	Analyte Peak Name	Analyte Mass Ranges (amu)	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Record Modified	Acquisition Date
1 Rinse		Solvent	MEL-1	126.8/85.2 amu	490.	0.00	N/A	N/A	2.01	☐	2007/07/18 3:32:08 PM
2 Blk		Blank	MEL-1	126.8/85.2 amu	4100.	0.00	N/A	N/A	2.03	☐	2007/07/18 3:41:05 PM
3 Std 1	☒	Standard	MEL-1	126.8/85.2 amu	327000.	500.	484.	96.8	2.02	☐	2007/07/18 3:46:40 PM
4 Std 2	☒	Standard	MEL-1	126.8/85.2 amu	607000.	1000.	1040.	104.	2.02	☐	2007/07/18 3:52:15 PM
5 Std 3	☒	Standard	MEL-1	126.8/85.2 amu	887000.	1500.	1600.	106.	2.03	☐	2007/07/18 4:03:24 PM
6 Std 4	☒	Standard	MEL-1	126.8/85.2 amu	1320000.	2500.	2460.	98.6	2.03	☐	2007/07/18 4:08:59 PM
7 Std 5	☒	Standard	MEL-1	126.8/85.2 amu	2010000.	4000.	3840.	96.0	2.02	☐	2007/07/18 4:14:34 PM
8 Std 6	☒	Standard	MEL-1	126.8/85.2 amu	2550000.	5000.	4910.	98.1	2.03	☐	2007/07/18 4:20:09 PM
9 Spk 1	☒	Quality Control	MEL-1	126.8/85.2 amu	285000.	500.	401.	80.1	2.03	☐	2007/07/18 4:25:44 PM
10 T51058		Unknown	MEL-1	126.8/85.2 amu	1790.	N/A	< 0	N/A	2.03	☐	2007/07/18 4:31:19 PM
11 T51086		Unknown	MEL-1	126.8/85.2 amu	6120.	N/A	< 0	N/A	2.02	☐	2007/07/18 4:36:54 PM
12 Spk 1A	☒	Quality Control	MEL-1	126.8/85.2 amu	283000.	500.	396.	79.2	2.03	☐	2007/07/18 4:36:54 PM

■ Untitled 4 (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): y = 503 x + 8.38e+004 (r = 0.9981)

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-rice ptn_070718.rdb

Printing Date: Thursday, July 19, 2007

Results Name: CYA-rice ptn_070719.rdb

Printing Time: 2:00:18

Results Path: R:\Projects\Residue\Aminopterin\Results\CYA-rice ptn_070719.rdb

Printing Date: Friday, July 20, 2007

Sample Name	Sample Comment	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Rinse		Solvent	CYA-1	0.00	0.00	N/A	N/A			0.00	127.9/42.0 amu	2007/07/19 4:27:32 PM
2 Rinse		Solvent	CYA-1	0.00	0.00	N/A	N/A			0.00	127.9/42.0 amu	2007/07/19 4:41:43 PM
3 Blk		Blank	CYA-1	1590	0.00	N/A	N/A			2.04	127.9/42.0 amu	2007/07/19 4:52:30 PM
4 Std 1		Standard	CYA-1	33000	500	484	96.8	☒		2.04	127.9/42.0 amu	2007/07/19 5:03:17 PM
5 Std 2		Standard	CYA-1	62200	1000	1060	106	☒		2.03	127.9/42.0 amu	2007/07/19 5:14:04 PM
6 Std 3		Standard	CYA-1	85700	1500	1520	102	☒		2.04	127.9/42.0 amu	2007/07/19 5:24:51 PM
7 Std 4		Standard	CYA-1	137000	2500	2540	102	☒		2.04	127.9/42.0 amu	2007/07/19 5:35:38 PM
8 Std 5		Standard	CYA-1	198000	4000	3730	93.4	☒		2.04	127.9/42.0 amu	2007/07/19 5:46:25 PM
9 Std 6		Standard	CYA-1	262000	5000	5010	100	☒		2.04	127.9/42.0 amu	2007/07/19 5:57:12 PM
10 Spk 1		Quality Control	CYA-1	29300	500	410	82.0	☒		2.04	127.9/42.0 amu	2007/07/19 6:07:59 PM
11 T51058	A773792	Unknown	CYA-1	1200	N/A	< 0	N/A			2.04	127.9/42.0 amu	2007/07/19 6:18:46 PM
12 T51086	A773802	Unknown	CYA-1	4970	N/A	< 0	N/A			2.04	127.9/42.0 amu	2007/07/19 6:29:33 PM
13 Spk 1A		Quality Control	CYA-1	34600	500	516	103	☒		2.04	127.9/42.0 amu	2007/07/19 6:40:20 PM

■ Untitled1 (CYA-1): "Linear" Regression ("1 / (x * x)" weighting) y = 50.6 x + 8.49e+003 (r = 0.9980)

Results Name: CYA-rice ptn_070719.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\CYA-rice ptn_070719.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\CYA-rice ptn_070719.rdb

Printing Date: Friday, July 20, 2007

Sample Name: "CYA-rice ptn_070719.rdb" Sample ID: "127.942.0 amu"
Peak Name: "CYA-rice ptn_070719.rdb" Mass: "127.942.0 amu"
Comment: "A773792"
Sample Index: 11
Sample Type: Unknown
Concentration: 500.00 ppb
Calculated Conc: No Intercept
Acq. Date: 2007/07/19
Acq. Time: 6:13:46 PM
Modified: No
Peak: Algorithm: Analyst Classic
Baseline Threshold: 30.00 cps
Area Threshold: 100.00 cps
Exp. Peak Ratio: 1.0000
Exp. Adj. Ratio: 1.0000
Exp. Val. Ratio: 1.0000
Exp. Window: 30.0 sec
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 2.04 min
Height: 2.85e+002 cps
Start Time: 1.76 min
End Time: 2.22 min

Sample Name: "CYA-rice ptn_070719.rdb" Sample ID: "127.942.0 amu"
Peak Name: "CYA-rice ptn_070719.rdb" Mass: "127.942.0 amu"
Comment: "A773802"
Sample Index: 12
Sample Type: Unknown
Concentration: 500.00 ppb
Calculated Conc: No Intercept
Acq. Date: 2007/07/19
Acq. Time: 6:23:33 PM
Modified: No
Peak: Algorithm: Analyst Classic
Baseline Threshold: 30.00 cps
Area Threshold: 100.00 cps
Exp. Peak Ratio: 1.0000
Exp. Adj. Ratio: 1.0000
Exp. Val. Ratio: 1.0000
Exp. Window: 30.0 sec
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 2.04 min
Height: 2.85e+002 cps
Start Time: 1.76 min
End Time: 2.22 min

Sample Name: "CYA-rice ptn_070719.rdb" Sample ID: "127.942.0 amu"
Peak Name: "CYA-rice ptn_070719.rdb" Mass: "127.942.0 amu"
Comment: "A773792"
Sample Index: 11
Sample Type: Unknown
Concentration: 500.00 ppb
Calculated Conc: No Intercept
Acq. Date: 2007/07/19
Acq. Time: 6:13:46 PM
Modified: No
Peak: Algorithm: Analyst Classic
Baseline Threshold: 30.00 cps
Area Threshold: 100.00 cps
Exp. Peak Ratio: 1.0000
Exp. Adj. Ratio: 1.0000
Exp. Val. Ratio: 1.0000
Exp. Window: 30.0 sec
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 2.04 min
Height: 2.85e+002 cps
Start Time: 1.76 min
End Time: 2.22 min

Sample Name: "CYA-rice ptn_070719.rdb" Sample ID: "127.942.0 amu"
Peak Name: "CYA-rice ptn_070719.rdb" Mass: "127.942.0 amu"
Comment: "A773802"
Sample Index: 12
Sample Type: Unknown
Concentration: 500.00 ppb
Calculated Conc: No Intercept
Acq. Date: 2007/07/19
Acq. Time: 6:23:33 PM
Modified: No
Peak: Algorithm: Analyst Classic
Baseline Threshold: 30.00 cps
Area Threshold: 100.00 cps
Exp. Peak Ratio: 1.0000
Exp. Adj. Ratio: 1.0000
Exp. Val. Ratio: 1.0000
Exp. Window: 30.0 sec
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 2.04 min
Height: 2.85e+002 cps
Start Time: 1.76 min
End Time: 2.22 min

Results Name: CYA-rice ptn_070719.rdb

Printing Time: 2:01:20

Results Path: R:\Projects\Residue\Aminopterin\Results\CYA-rice ptn_070719.rdb

Printing Date: Friday, July 20, 2007

Sample Name	Sample Comment	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Rinse		Solvent	CYA-2	0.00	0.00	N/A	N/A		☐	0.00	127.9/85.0 amu	2007/07/19 4:27:32 PM
2 Rinse		Solvent	CYA-2	0.00	0.00	N/A	N/A		☐	0.00	127.9/85.0 amu	2007/07/19 4:41:43 PM
3 Bk		Blank	CYA-2	1080.	0.00	N/A	N/A		☐	2.05	127.9/85.0 amu	2007/07/19 4:52:30 PM
4 Std 1		Standard	CYA-2	20300.	500.	500.	99.9	☒	☐	2.04	127.9/85.0 amu	2007/07/19 5:03:17 PM
5 Std 2		Standard	CYA-2	35200.	1000.	1010.	101.	☒	☐	2.04	127.9/85.0 amu	2007/07/19 5:14:04 PM
6 Std 3		Standard	CYA-2	48400.	1500.	1470.	97.8	☒	☐	2.05	127.9/85.0 amu	2007/07/19 5:35:38 PM
7 Std 4		Standard	CYA-2	80300.	2500.	2570.	103.	☒	☐	2.05	127.9/85.0 amu	2007/07/19 5:46:25 PM
8 Std 5		Standard	CYA-2	118000.	4000.	3850.	96.2	☒	☐	2.04	127.9/85.0 amu	2007/07/19 5:57:12 PM
9 Std 6		Standard	CYA-2	155000.	5000.	5120.	102.	☒	☐	2.04	127.9/85.0 amu	2007/07/19 6:07:59 PM
10 Spk 1		Quality Control	CYA-2	16800.	500.	378.	75.7	☒	☐	2.04	127.9/85.0 amu	2007/07/19 6:18:46 PM
11 T51058	A773792	Unknown	CYA-2	581.	N/A	< 0	N/A		☐	2.05	127.9/85.0 amu	2007/07/19 6:29:33 PM
12 T51086	A773802	Unknown	CYA-2	2680.	N/A	< 0	N/A		☐	2.04	127.9/85.0 amu	2007/07/19 6:40:20 PM
13 Spk 1A		Quality Control	CYA-2	19700.	500.	478.	95.5	☒	☐	2.05	127.9/85.0 amu	2007/07/19 6:40:20 PM

Sample Name	Sample Comment	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Blk		Blank	CYA-1	1220.	0.00	N/A	N/A			2.05	127.9/42.0 amu	2007/07/19 6:51:07 PM
2 Std 1		Standard	CYA-1	60800.	500.	501.	100.			2.05	127.9/42.0 amu	2007/07/19 7:01:54 PM
3 Std 2		Standard	CYA-1	129000.	1000.	1020.	102.			2.05	127.9/42.0 amu	2007/07/19 7:12:41 PM
4 Std 3		Standard	CYA-1	182000.	1500.	1420.	94.5			2.05	127.9/42.0 amu	2007/07/19 7:23:28 PM
5 Std 4		Standard	CYA-1	330000.	2500.	2540.	102.			2.05	127.9/42.0 amu	2007/07/19 7:34:15 PM
6 Std 5		Standard	CYA-1	534000.	4000.	4090.	102.			2.05	127.9/42.0 amu	2007/07/19 7:45:02 PM
7 Std 6		Standard	CYA-1	649000.	5000.	4960.	98.2			2.05	127.9/42.0 amu	2007/07/19 7:55:49 PM
8 Spk 1		Quality Control	CYA-1	66500.	500.	546.	109.			2.05	127.9/42.0 amu	2007/07/19 8:06:36 PM
9 T51090	A773805	Unknown	CYA-1	1610.	N/A	53.9	N/A			2.05	127.9/42.0 amu	2007/07/19 8:17:23 PM
10 Spk 1A		Quality Control	CYA-1	64700.	500.	533.	107.			2.05	127.9/42.0 amu	2007/07/19 8:28:10 PM

■ Untitled 2 (CYA-1): "Linear" Regression ("1 / (x * x)" weighting): y = 132 x + -5.51e+003 (r = 0.9991)

Results Path: R:\Projects\Residue\Aminopterin\Results\CVA-Soy_070719.rdb

Results Name: CVA-Soy_070719.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\CVA-Soy_070719.rdb

Sample Name	Sample Comment	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1	Blk		CYA-2	717.	0.00	N/A	N/A			2.05	127.9/85.0 amu	2007/07/19 6:51:07 PM
2	Std 1	Blank	CYA-2	36100.	500.	502.	100.	☒		2.05	127.9/85.0 amu	2007/07/19 7:01:54 PM
3	Std 2	Standard	CYA-2	76800.	1000.	1020.	102.	☒		2.05	127.9/85.0 amu	2007/07/19 7:12:41 PM
4	Std 3	Standard	CYA-2	107000.	1500.	1400.	93.3	☒		2.05	127.9/85.0 amu	2007/07/19 7:23:28 PM
5	Std 4	Standard	CYA-2	202000.	2500.	2610.	104.	☒		2.06	127.9/85.0 amu	2007/07/19 7:34:15 PM
6	Std 5	Standard	CYA-2	315000.	4000.	4040.	101.	☒		2.06	127.9/85.0 amu	2007/07/19 7:45:02 PM
7	Std 6	Standard	CYA-2	386000.	5000.	4950.	99.1	☒		2.06	127.9/85.0 amu	2007/07/19 7:55:49 PM
8	Spk 1	Quality Control	CYA-2	40300.	500.	555.	111.	☒		2.06	127.9/85.0 amu	2007/07/19 8:06:36 PM
9	T51050	A773805	CYA-2	680.	N/A	51.3	N/A	☒		2.06	127.9/85.0 amu	2007/07/19 8:17:23 PM
10	Spk 1A	Quality Control	CYA-2	39200.	500.	541.	108.	☒		2.06	127.9/85.0 amu	2007/07/19 8:28:10 PM

Sample Name	Use Record	Sample Type	Analyte Peak Name	Analyte Mass Ranges (amu)	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Record Modified	Acquisition Date
1 Blk		Blank	MEL-1	126.8/85.2 amu	276	0.00	N/A	N/A	2.00	☐	2007/07/18 4:42:29 PM
2 Std 1	☒	Standard	MEL-1	126.8/85.2 amu	368000	500	488	97.6	2.03	☐	2007/07/18 4:48:04 PM
3 Std 2	☒	Standard	MEL-1	126.8/85.2 amu	725000	1000	1020	102	2.03	☐	2007/07/18 4:53:39 PM
4 Std 3	☒	Standard	MEL-1	126.8/85.2 amu	1120000	1500	1600	107	2.03	☐	2007/07/18 4:59:14 PM
5 Std 4	☒	Standard	MEL-1	126.8/85.2 amu	1720000	2500	2500	99.8	2.03	☐	2007/07/18 5:04:49 PM
6 Std 5	☒	Standard	MEL-1	126.8/85.2 amu	2560000	4000	3760	93.9	2.03	☐	2007/07/18 5:10:24 PM
7 Std 6	☐	Standard	MEL-1	126.8/85.2 amu	4570000	5000	6750	135	2.03	☐	2007/07/18 5:15:59 PM
8 Spk 1	☒	Quality Control	MEL-1	126.8/85.2 amu	378000	500	503	101	2.03	☐	2007/07/18 5:21:34 PM
9 T51090	☒	Unknown	MEL-1	126.8/85.2 amu	3120	N/A	< 0	N/A	2.03	☐	2007/07/18 5:27:09 PM
10 Spk 1A	☒	Quality Control	MEL-1	126.8/85.2 amu	377000	500	502	100	2.04	☐	2007/07/18 5:32:44 PM

■ Untitled 3 (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): y = 672 x + 3.97e+004 (r = 0.9973)

Sample Name	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-1	411.	0.00	N/A	N/A			3.00	1.70	2007/04/18 07:39:38 PM
2 Blk (WG)	Blank	MEL-1	655.	0.00	N/A	N/A			5.75	1.83	2007/04/18 07:46:12 PM
3 Blk (Soy)	Blank	MEL-1	7070.	0.00	N/A	N/A			51.2	1.81	2007/04/18 07:50:47 PM
4 Std 1	Standard	MEL-1	50700.	500.	484.	96.8	☒		360.	1.81	2007/04/18 07:56:22 PM
5 Std 3	Standard	MEL-1	157000.	1500.	1690.	113.	☒		946.	1.82	2007/04/18 08:01:57 PM
6 Std 4	Standard	MEL-1	259000.	2500.	2850.	114.	☒		1030.	1.82	2007/04/18 08:07:32 PM
7 Std 5	Standard	MEL-1	317000.	4000.	3510.	87.7	☒		1090.	1.81	2007/04/18 08:13:07 PM
8 Std 6	Standard	MEL-1	336000.	5000.	3730.	74.5	☒		1450.	1.81	2007/04/18 08:18:42 PM
9 Spk 1 (WG)	Quality Control	MEL-1	51200.	500.	489.	97.8	☒		250.	1.82	2007/04/18 08:24:17 PM
10 Spk 1 (soy)	Quality Control	MEL-1	875000.	500.	9860.	1970.	☒		3650.	1.83	2007/04/18 08:29:52 PM
11 R79905	Unknown	MEL-1	1360000.	N/A	15400.	N/A	☒		8200.	1.81	2007/04/18 08:35:27 PM
12 R79906	Unknown	MEL-1	688.	N/A	< 0	N/A			2.75	1.83	2007/04/18 08:41:02 PM
13 R79907	Unknown	MEL-1	878.	N/A	< 0	N/A			5.78	1.81	2007/04/18 08:46:37 PM
14 R79908	Unknown	MEL-1	623.	N/A	< 0	N/A			5.18	1.81	2007/04/18 08:52:12 PM
15 R77219	Unknown	MEL-1	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 08:57:47 PM
16 R77220	Unknown	MEL-1	1170.	N/A	< 0	N/A			9.61	1.86	2007/04/18 09:03:22 PM
17 R77221	Unknown	MEL-1	1160.	N/A	< 0	N/A			8.67	1.87	2007/04/18 09:08:57 PM
18 R77222	Unknown	MEL-1	1760000.	N/A	19900.	N/A			13800.	1.81	2007/04/18 09:14:32 PM
19 R77223	Unknown	MEL-1	890.	N/A	< 0	N/A			5.94	1.83	2007/04/18 09:20:07 PM
20 R81577	Unknown	MEL-1	25900.	N/A	201.	N/A			101.	1.82	2007/04/18 09:25:42 PM
21 Spk 1A (WG)	Quality Control	MEL-1	48400.	500.	457.	91.5	☒		328.	1.82	2007/04/18 09:31:17 PM
22 Spk 1A (soy)	Quality Control	MEL-1	943000.	500.	10600.	2130.	☒		5200.	1.83	2007/04/18 09:36:52 PM
23 Std 1	Standard	MEL-1	51100.	500.	488.	97.6	☒		288.	1.82	2007/04/18 09:42:27 PM
24 Std 3	Standard	MEL-1	147000.	1500.	1580.	106.	☒		753.	1.82	2007/04/18 09:48:02 PM
25 Std 4	Standard	MEL-1	276000.	2500.	3050.	122.	☒		1400.	1.82	2007/04/18 09:53:37 PM
26 Std 5	Standard	MEL-1	308000.	4000.	3420.	85.4	☒		1400.	1.82	2007/04/18 09:59:12 PM
27 Std 6	Standard	MEL-1	332000.	5000.	3690.	73.8	☒		986.	1.82	2007/04/18 10:04:47 PM

■ Untitled 6 (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): y = 87.9 x + 8.21e+003 (r = 0.9863)

Method Name: APT-MEL_070409.qmf

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Results Name: AptMel-WG_070418.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-WG_070418.rdb

Sample Name: "Std 1" Sample ID: "1" File: "Pre-mix.wif"
Peak Name: "MEL-1" Mass(es): "126.0652 amu"
Comment: " Annotation: "

Sample Name: "Std 3" Sample ID: "3" File: "Pre-mix.wif"
Peak Name: "MEL-1" Mass(es): "126.0652 amu"
Comment: " Annotation: "

Method Name: APT-MEL_070409.qmf

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Results Name: AptMel-WG_070418.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-WG_070418.rdb

Printing Time: 08:55:13

Printing Date: Thursday, April 19, 200

Method Name: APT-MEL_070409.qmf

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Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-WG_070418.rdb

Printing Date: Thursday, April 19, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Peak Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-3	0.00	0.00	N/A	N/A			N/A	0.00	2007/04/18 07:39:38 PM
2 Blk (WG)	Blank	MEL-3	0.00	0.00	N/A	N/A			N/A	0.00	2007/04/18 07:45:12 PM
3 Blk (Soy)	Blank	MEL-3	281	0.00	N/A	N/A			13.8	1.79	2007/04/18 07:50:47 PM
4 Std 1	Standard	MEL-3	3620	500	511	102	☒		88.0	1.81	2007/04/18 07:56:22 PM
5 Std 3	Standard	MEL-3	10700	1500	1610	108	☒		366	1.81	2007/04/18 08:01:57 PM
6 Std 4	Standard	MEL-3	17400	2500	2650	106	☒		384	1.81	2007/04/18 08:07:32 PM
7 Std 5	Standard	MEL-3	22600	4000	3460	86.5	☒		528	1.81	2007/04/18 08:13:07 PM
8 Std 6	Standard	MEL-3	22900	5000	3520	70.3	☐		730	1.81	2007/04/18 08:18:42 PM
9 Spk 1 (WG)	Quality Control	MEL-3	3390	500	474	94.9	☒		60.7	1.81	2007/04/18 08:24:17 PM
10 Spk 1 (soy)	Quality Control	MEL-3	63600	500	9650	1970	☒		2620	1.83	2007/04/18 08:29:52 PM
11 R79905	Unknown	MEL-3	96600	N/A	15000	N/A			3290	1.81	2007/04/18 08:35:27 PM
12 R79906	Unknown	MEL-3	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 08:41:02 PM
13 R79907	Unknown	MEL-3	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 08:46:37 PM
14 R79908	Unknown	MEL-3	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 08:52:12 PM
15 R77219	Unknown	MEL-3	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 08:57:47 PM
16 R77220	Unknown	MEL-3	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 09:03:22 PM
17 R77221	Unknown	MEL-3	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 09:08:57 PM
18 R77222	Unknown	MEL-3	124000	N/A	19300	N/A			2830	1.81	2007/04/18 09:14:32 PM
19 R77223	Unknown	MEL-3	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 09:20:07 PM
20 R81577	Unknown	MEL-3	1220	N/A	137	N/A			41.3	1.81	2007/04/18 09:25:42 PM
21 Spk 1A (WG)	Quality Control	MEL-3	3250	500	454	90.8	☒		64.6	1.80	2007/04/18 09:31:17 PM
22 Spk 1A (soy)	Quality Control	MEL-3	69000	500	10700	2140	☒		2510	1.83	2007/04/18 09:36:52 PM
23 Std 1	Standard	MEL-3	3270	500	457	91.4	☒		50.5	1.82	2007/04/18 09:42:27 PM
24 Std 3	Standard	MEL-3	11300	1500	1710	114	☒		253	1.83	2007/04/18 09:48:02 PM
25 Std 4	Standard	MEL-3	17900	2500	2730	109	☒		260	1.81	2007/04/18 09:53:37 PM
26 Std 5	Standard	MEL-3	21800	4000	3340	83.5	☒		450	1.82	2007/04/18 09:59:12 PM
27 Std 6	Standard	MEL-3	23000	5000	3530	70.6	☐		433	1.82	2007/04/18 10:04:47 PM

Method Name: APT-MEL-WG_070409.qmf

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Results Name: AptMel-WG_070418.rdb

Printing Time: 08:55:44

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-WG_070418.rdb

Printing Date: Thursday, April 19, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Peak Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	APT-1	0.00	0.00	N/A	N/A			N/A	0.00	2007/04/18 07:39:38 PM
2 Blk (WG)	Blank	APT-1	0.00	0.00	N/A	N/A			N/A	0.00	2007/04/18 07:45:12 PM
3 Blk (Soy)	Blank	APT-1	0.00	0.00	N/A	N/A			N/A	0.00	2007/04/18 07:50:47 PM
4 Std 1	Standard	APT-1	156000	100	94.6	94.6	☒		2420	2.17	2007/04/18 07:56:22 PM
5 Std 3	Standard	APT-1	505000	300	327	109	☒		5430	2.17	2007/04/18 08:01:57 PM
6 Std 4	Standard	APT-1	856000	500	560	112	☒		12800	2.17	2007/04/18 08:07:32 PM
7 Std 5	Standard	APT-1	1220000	800	804	100	☒		15100	2.16	2007/04/18 08:13:07 PM
8 Std 6	Standard	APT-1	1370000	1000	905	90.5	☒		17900	2.17	2007/04/18 08:18:42 PM
9 Spk 1 (WG)	Quality Control	APT-1	169000	100	103	103	☒		1860	2.18	2007/04/18 08:24:17 PM
10 Spk 1 (soy)	Quality Control	APT-1	58800	100	29.7	29.7	☒		720	2.18	2007/04/18 08:29:52 PM
11 R79905	Unknown	APT-1	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 08:35:27 PM
12 R79906	Unknown	APT-1	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 08:41:02 PM
13 R79907	Unknown	APT-1	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 08:46:37 PM
14 R79908	Unknown	APT-1	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 08:52:12 PM
15 R77219	Unknown	APT-1	925	N/A	< 0	N/A			N/A	0.00	2007/04/18 08:57:47 PM
16 R77220	Unknown	APT-1	0.00	N/A	No Peak	N/A			0.698	2.08	2007/04/18 09:03:22 PM
17 R77221	Unknown	APT-1	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 09:08:57 PM
18 R77222	Unknown	APT-1	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 09:14:32 PM
19 R77223	Unknown	APT-1	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 09:20:07 PM
20 R81577	Unknown	APT-1	0.00	N/A	No Peak	N/A			N/A	0.00	2007/04/18 09:25:42 PM
21 Spk 1A (WG)	Quality Control	APT-1	160000	100	97.0	97.0	☒		2510	2.17	2007/04/18 09:31:17 PM
22 Spk 1A (soy)	Quality Control	APT-1	69200	100	36.6	36.6	☒		984	2.19	2007/04/18 09:36:52 PM
23 Std 1	Standard	APT-1	163000	100	99.2	99.2	☒		2390	2.17	2007/04/18 09:42:27 PM
24 Std 3	Standard	APT-1	493000	300	319	106	☒		14500	2.17	2007/04/18 09:48:02 PM
25 Std 4	Standard	APT-1	834000	500	546	109	☒		23600	2.17	2007/04/18 09:53:37 PM
26 Std 5	Standard	APT-1	1150000	800	753	94.1	☒		12900	2.17	2007/04/18 09:59:12 PM
27 Std 6	Standard	APT-1	1290000	1000	846	84.6	☒		10900	2.18	2007/04/18 10:04:47 PM

Method Name: APT-MEL-WG_070409.qmf

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Table 7.2.: Dry Food Data

Study Sample	Coded Sample Name
DFWG1	R75279
DFWG2	R75276
DFWG3	R75280
DFWG4	R75281
DFWG5	R84609
DFWG6	R75283
DFWG7	S34738
DFSPI1	S39194
DFRPC1	R76720
DFRPC2	R82930
DFRPC3	R82950
DFRPC4	R82962
DFRPC5	R82966
DFRPC6	R82971
DFRPC7	R82982
DFRPC8	R82988
DFRPC9	R82997
DFRPC10	R83003
DFRPC11	R84548

Results Name: AptMel-dry food_070418.rdb
Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-dry food_070418.rdb

Printing Time: 08:44:34
Printing Date: Thursday, April 19, 2007

	Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1	Rinse	Solvent	MEL-1	0.00	0.00	N/A	N/A		☐	N/A	0.00	2007/04/18 03:20:49 PM
2	Blk	Blank	MEL-1	1020.	0.00	N/A	N/A		☐	10.3	1.79	2007/04/18 03:28:23 PM
3	Std 1	Standard	MEL-1	74800.	500.	496.	99.1	☒	☐	540.	1.78	2007/04/18 03:33:59 PM
4	Std 2	Standard	MEL-1	144000.	1000.	972.	97.2	☒	☐	1210.	1.78	2007/04/18 03:39:34 PM
5	Std 3	Standard	MEL-1	215000.	1500.	1460.	97.2	☒	☐	1540.	1.77	2007/04/18 03:45:09 PM
6	Std 4	Standard	MEL-1	361000.	2500.	2460.	98.2	☒	☐	3340.	1.78	2007/04/18 03:50:44 PM
7	Std 5	Standard	MEL-1	576000.	4000.	3930.	98.3	☒	☐	4150.	1.77	2007/04/18 03:56:19 PM
8	Std 6	Standard	MEL-1	739000.	5000.	5050.	101.	☒	☐	7030.	1.77	2007/04/18 04:01:54 PM
9	R75276	Unknown	MEL-1	4390.	N/A	13.2	N/A		☐	40.2	1.77	2007/04/18 04:07:29 PM
10	R75278	Unknown	MEL-1	1930.	N/A	< 0	N/A		☐	14.0	1.79	2007/04/18 04:13:04 PM
11	R75279	Unknown	MEL-1	1270.	N/A	< 0	N/A		☐	9.20	1.79	2007/04/18 04:18:39 PM
12	R75280	Unknown	MEL-1	934.	N/A	< 0	N/A		☐	6.06	1.76	2007/04/18 04:24:14 PM
13	R75281	Unknown	MEL-1	1490.	N/A	< 0	N/A		☐	17.5	1.76	2007/04/18 04:29:49 PM
14	R71483	Unknown	MEL-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:35:24 PM
15	R75283	Unknown	MEL-1	5060.	N/A	17.7	N/A		☐	40.1	1.78	2007/04/18 04:40:58 PM
16	R75285	Unknown	MEL-1	979.	N/A	< 0	N/A		☐	10.8	1.77	2007/04/18 04:46:33 PM
17	R76720	Unknown	MEL-1	409.	N/A	< 0	N/A		☐	5.24	1.78	2007/04/18 04:52:08 PM
18	R75301	Unknown	MEL-1	1480.	N/A	< 0	N/A		☐	15.7	1.78	2007/04/18 04:57:43 PM
19	R73313	Unknown	MEL-1	6250.	N/A	25.9	N/A		☐	77.1	1.76	2007/04/18 05:03:18 PM
20	R75303	Unknown	MEL-1	1350.	N/A	< 0	N/A		☐	15.9	1.79	2007/04/18 05:08:53 PM
21	Spk 1	Quality Control	MEL-1	72800.	500.	482.	96.4	☒	☐	650.	1.78	2007/04/18 05:14:28 PM
22	R81574	Unknown	MEL-1	7890.	N/A	37.2	N/A		☐	82.8	1.76	2007/04/18 05:20:03 PM
23	R73494	Unknown	MEL-1	1920.	N/A	< 0	N/A		☐	18.4	1.76	2007/04/18 05:25:38 PM
24	R78938	Unknown	MEL-1	389.	N/A	< 0	N/A		☐	5.62	1.76	2007/04/18 05:31:13 PM
25	R79936	Unknown	MEL-1	1970.	N/A	< 0	N/A		☐	29.0	1.77	2007/04/18 05:36:48 PM
26	R84548	Unknown	MEL-1	1380000.	N/A	9420.	N/A		☐	10300.	1.78	2007/04/18 05:42:23 PM
27	R84544	Unknown	MEL-1	1980.	N/A	< 0	N/A		☐	15.1	1.78	2007/04/18 05:47:58 PM
28	R84556	Unknown	MEL-1	1220.	N/A	< 0	N/A		☐	6.75	1.77	2007/04/18 05:53:33 PM
29	R84590	Unknown	MEL-1	936.	N/A	< 0	N/A		☐	7.22	1.76	2007/04/18 05:59:08 PM
30	R84591	Unknown	MEL-1	1340.	N/A	< 0	N/A		☐	13.6	1.79	2007/04/18 06:04:43 PM
31	R84594	Unknown	MEL-1	1170.	N/A	< 0	N/A		☐	9.36	1.77	2007/04/18 06:10:18 PM
32	R84595	Unknown	MEL-1	1690.	N/A	< 0	N/A		☐	9.33	1.77	2007/04/18 06:15:53 PM
33	R84596	Unknown	MEL-1	1810.	N/A	< 0	N/A		☐	10.7	1.78	2007/04/18 06:21:29 PM
34	Spk 1A	Quality Control	MEL-1	77200.	500.	512.	102.	☒	☐	615.	1.78	2007/04/18 06:27:02 PM
35	R84597	Unknown	MEL-1	627.	N/A	< 0	N/A		☐	6.08	1.78	2007/04/18 06:32:38 PM
36	R84598	Unknown	MEL-1	1130.	N/A	< 0	N/A		☐	23.1	1.79	2007/04/18 06:38:11 PM
37	R84599	Unknown	MEL-1	1210.	N/A	< 0	N/A		☐	8.15	1.78	2007/04/18 06:43:46 PM

■ Untitled 7 (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): y = 167 x + -3.55e+003 (r = 0.9996)

■ Untitled 5 (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): y = 146 x + 2.47e+003 (r = 0.9992)

Sample Name: "Rins" Sample ID: " " File: "Dry Pet Food.wif"
Peak Name: "MEL-1" Mass(es): "126.865.2 amu"
Comment: " Annotation: "
Sample Index: 2
Concentration: 0.00 PPB
Calculated Conc: 2097.04/18
Acq. Time: 03:20:49 PM
Modified: NO

Sample Name: "Std 1" Sample ID: " " File: "Dry Pet Food.wif"
Peak Name: "MEL-1" Mass(es): "126.865.2 amu"
Comment: " Annotation: "

Method Name: APT-MEL_070409.qmf

Method Name: APT-MEL_070409.qmf

Method Name: APT-MEL_070409.gmf

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Results Name: AptMcl-dry food_070418.rdb

Printing Time: 08:45:12

Results Path: D:\Analyst Data\Residue\Aminopterin\Results\AptMEL-dry food_070418.rdb

Printing Date: Thursday, April 19, 200

Method Name: APT-MEL_070409.qmc

00000000

Sample Name	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Peak Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-3	0.00	0.00	N/A	N/A		☐	N/A	0.00	2007/04/18 03:20:49 PM
2 Blk	Blank	MEL-3	0.00	0.00	N/A	N/A		☐	N/A	0.00	2007/04/18 03:28:23 PM
3 Std 1	Standard	MEL-3	4980.	500.	466.	93.1	☒	☐	115.	1.77	2007/04/18 03:33:59 PM
4 Std 2	Standard	MEL-3	10100.	1000.	952.	95.2	☒	☐	509.	1.78	2007/04/18 03:39:34 PM
5 Std 3	Standard	MEL-3	15800.	1500.	1490.	99.3	☒	☐	571.	1.77	2007/04/18 03:45:09 PM
6 Std 4	Standard	MEL-3	25800.	2500.	2440.	97.6	☒	☐	789.	1.77	2007/04/18 03:50:44 PM
7 Std 5	Standard	MEL-3	41300.	4000.	3910.	97.8	☒	☐	1350.	1.77	2007/04/18 03:56:19 PM
8 Std 6	Standard	MEL-3	54000.	5000.	5120.	102.	☒	☐	1190.	1.77	2007/04/18 04:01:54 PM
9 R75276	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:07:39 PM
10 R75278	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:13:04 PM
11 R75279	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:18:39 PM
12 R75280	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:24:14 PM
13 R75281	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:29:49 PM
14 R71483	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:35:24 PM
15 R75283	Unknown	MEL-3	354.	N/A	26.4	N/A		☐	22.8	1.76	2007/04/18 04:40:58 PM
16 R75285	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:46:33 PM
17 R76720	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:52:08 PM
18 R75301	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:57:43 PM
19 R73313	Unknown	MEL-3	298.	N/A	21.1	N/A		☐	13.9	1.74	2007/04/18 05:03:18 PM
20 R75303	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:08:53 PM
21 Spk 1	Quality Control	MEL-3	5240.	500.	491.	96.1	☒	☐	243.	1.78	2007/04/18 05:14:28 PM
22 R81574	Unknown	MEL-3	495.	N/A	39.8	N/A		☐	15.8	1.74	2007/04/18 05:20:03 PM
23 R73494	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:25:38 PM
24 R79938	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:31:13 PM
25 R79936	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:36:48 PM
26 R84548	Unknown	MEL-3	98100.	N/A	9120.	N/A		☐	3380.	1.77	2007/04/18 05:42:23 PM
27 R84544	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:47:58 PM
28 R84556	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:53:33 PM
29 R84590	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:59:08 PM
30 R84591	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:04:43 PM
31 R84594	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:10:18 PM
32 R84595	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:15:53 PM
33 R84596	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:21:29 PM
34 Spk 1A	Quality Control	MEL-3	5830.	500.	546.	109.	☒	☐	274.	1.78	2007/04/18 06:27:02 PM
35 R84597	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:32:38 PM
36 R84598	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:38:11 PM
37 R84599	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:43:46 PM

Sample Name	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Peak Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
38 R84601	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:49:22 PM
39 R84602	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:54:56 PM
40 R84604	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 07:00:32 PM
41 R84607	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 07:06:06 PM
42 R84608	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 07:11:42 PM
43 R84609	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 07:17:16 PM
44 Std 1	Standard	MEL-3	5840.	500.	547.	109.	☒	☐	285.	1.78	2007/04/18 07:22:52 PM
45 Std 3	Standard	MEL-3	16000.	1500.	1510.	101.	☒	☐	553.	1.78	2007/04/18 07:28:27 PM
46 Std 6	Standard	MEL-3	55100.	5000.	5220.	104.	☒	☐	2430.	1.78	2007/04/18 07:34:03 PM

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	APT-1	0.00	0.00	N/A	N/A		☐	N/A	0.00	2007/04/18 03:20:49 PM
2 Blk	Blank	APT-1	0.00	0.00	N/A	N/A		☐	N/A	0.00	2007/04/18 03:28:23 PM
3 Std 1	Standard	APT-1	76200	100	102	102	☒	☐	992	2.19	2007/04/18 03:33:59 PM
4 Std 2	Standard	APT-1	159000	200	208	104	☒	☐	2050	2.20	2007/04/18 03:39:34 PM
5 Std 3	Standard	APT-1	226000	300	292	97.5	☒	☐	3250	2.19	2007/04/18 03:45:09 PM
6 Std 4	Standard	APT-1	390000	500	501	100	☒	☐	3990	2.18	2007/04/18 03:50:44 PM
7 Std 5	Standard	APT-1	651000	800	835	104	☒	☐	5840	2.19	2007/04/18 03:56:19 PM
8 Std 6	Standard	APT-1	783000	1000	1000	100	☒	☐	5230	2.18	2007/04/18 04:01:54 PM
9 R75276	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:07:29 PM
10 R75278	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:13:04 PM
11 R75279	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:18:39 PM
12 R75280	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:24:14 PM
13 R75281	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:29:49 PM
14 R71483	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:35:24 PM
15 R75283	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:40:58 PM
16 R75285	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:46:33 PM
17 R76720	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:52:08 PM
18 R75301	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 04:57:43 PM
19 R73313	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:03:18 PM
20 R75303	Unknown	APT-1	1470	N/A	6.64	N/A		☐	17.9	2.12	2007/04/18 05:08:53 PM
21 Spk 1	Quality Control	APT-1	85200	100	113	113	☒	☐	762	2.19	2007/04/18 05:14:28 PM
22 R81574	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:20:03 PM
23 R73494	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:25:38 PM
24 R79938	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:31:13 PM
25 R79936	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:36:48 PM
26 R84548	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:42:23 PM
27 R84544	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:47:58 PM
28 R84556	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:53:33 PM
29 R84590	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 05:59:08 PM
30 R84591	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:04:43 PM
31 R84594	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:10:18 PM
32 R84595	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:15:53 PM
33 R84596	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:21:29 PM
34 Spk 1A	Quality Control	APT-1	69700	100	93.6	93.6	☒	☐	804	2.19	2007/04/18 06:27:02 PM
35 R84597	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:32:38 PM
36 R84598	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:38:11 PM
37 R84599	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	2007/04/18 06:43:46 PM

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-1	359	0.00	N/A	N/A		☐	1.00	3.95	1.74	2007/04/19 10:37:47 AM
2 Blk	Blank	MEL-1	766	0.00	N/A	N/A		☐	1.00	8.23	1.79	2007/04/19 10:43:34 AM
3 Std 1	Standard	MEL-1	80400	500	504	101	☒	☐	1.00	1720	1.79	2007/04/19 10:49:09 AM
4 Std 2	Standard	MEL-1	164000	1000	1010	101	☒	☐	1.00	1260	1.79	2007/04/19 10:54:44 AM
5 Std 3	Standard	MEL-1	237000	1500	1450	96.4	☒	☐	1.00	2150	1.79	2007/04/19 11:00:19 AM
6 Std 4	Standard	MEL-1	408000	2500	2470	98.7	☒	☐	1.00	3300	1.79	2007/04/19 11:05:55 AM
7 Std 5	Standard	MEL-1	674000	4000	4070	102	☒	☐	1.00	7510	1.79	2007/04/19 11:11:30 AM
8 Std 6	Standard	MEL-1	845000	5000	5090	102	☒	☐	1.00	6710	1.79	2007/04/19 11:17:05 AM
9 Std 6 (dil 1:5)	Quality Control	MEL-1	445000	5000	2590	53.9	☒	☐	1.00	3080	1.80	2007/04/19 11:22:40 AM
10 R84548 (dil 1:5)	Unknown	MEL-1	764000	N/A	4610	N/A		☐	1.00	6030	1.80	2007/04/19 11:28:15 AM

enhancement of the signal is seen.

dilution factor is

$$2690 \times 5 = 13450 \text{ ppb}$$
$$\% \text{acc} = 269\%$$

$$\text{enhancement} = \frac{\text{dil factor} \times 5}{\% \text{acc} / 100}$$
$$= \frac{5}{269 / 100} = 1.86$$

∴ signal enhancement is 1.86 x

so concⁿ in sample is $4610 \text{ ppb} \times 1.86$

$$= 8570 \text{ ppb}$$

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-dry food_070418-dil.rdb

Printing Date: Thursday, April 19, 200

Method Name: APT-MEL_070409.qmf

Page 1 of 2

Results Name: AptMel-dry food_070418-dil.rdb

Printing Time: 11:45:20 A

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-dry food_070418-dil.rdb

Printing Date: Thursday, April 19, 200

Method Name: APT-MEL_070409.qmf

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Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-3	0.00	0.00	N/A	N/A			1.00	N/A	0.00	2007/04/19 10:37:47 AM
2 Blk	Blank	MEL-3	0.00	0.00	N/A	N/A			1.00	N/A	0.00	2007/04/19 10:43:34 AM
3 Std 1	Standard	MEL-3	6280	500	499	99.9	☒		1.00	348	1.78	2007/04/19 10:49:09 AM
4 Std 2	Standard	MEL-3	12100	1000	1020	102	☒		1.00	540	1.78	2007/04/19 10:54:44 AM
5 Std 3	Standard	MEL-3	17300	1500	1470	97.8	☒		1.00	640	1.78	2007/04/19 11:00:19 AM
6 Std 4	Standard	MEL-3	29000	2500	2500	100	☒		1.00	1180	1.78	2007/04/19 11:05:55 AM
7 Std 5	Standard	MEL-3	46400	4000	4030	101	☒		1.00	914	1.78	2007/04/19 11:11:30 AM
8 Std 6	Standard	MEL-3	57500	5000	5000	100	☒		1.00	2040	1.79	2007/04/19 11:17:05 AM
9 Std 6 (dil 1:5)	Quality Control	MEL-3	30800	5000	2660	53.1	☒		1.00	1640	1.80	2007/04/19 11:22:40 AM
10 R64548 (dil 1:5)	Unknown	MEL-3	54500	N/A	4740	N/A			1.00	2010	1.80	2007/04/19 11:28:15 AM

Method Name: APT-MEL_070409.qmf

Results Name: CYA-Dry_070522.sdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Dry_070522.rdb

Sample Name	Sample Type	Analyte Peak Name	Analyte Mass Ranges (amu)	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Dilution Factor	Analyte Retention Time (min)	Analyte Signal To Noise	Record Modified	Acquisition Method	Acquisition Date
1 Rinse	Solvent	CYA-1	127.942.0 amu	0.00	0.00	N/A	N/A	1.00	0.00	N/A		CYA-MRM_070423.dam	2007/05/22 04:54:39 PM
2 Blk	Blank	CYA-1	127.942.0 amu	5120	0.00	N/A	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 05:07:45 PM
3 Std 1	Standard	CYA-1	127.942.0 amu	64000	500	516	103	1.00	1.97	N/A	☒	CYA-MRM_070423.dam	2007/05/22 05:18:30 PM
4 Std 2	Standard	CYA-1	127.942.0 amu	125000	1000	1070	107	1.00	1.97	N/A	☒	CYA-MRM_070423.dam	2007/05/22 05:28:15 PM
5 Std 3	Standard	CYA-1	127.942.0 amu	199000	1500	1750	116	1.00	1.97	N/A	☒	CYA-MRM_070423.dam	2007/05/22 05:40:00 PM
6 Std 4	Standard	CYA-1	127.942.0 amu	262000	2500	2600	100	1.00	1.97	N/A	☒	CYA-MRM_070423.dam	2007/05/22 05:50:45 PM
7 Std 5	Standard	CYA-1	127.942.0 amu	409000	4000	3660	91.6	1.00	1.97	N/A	☒	CYA-MRM_070423.dam	2007/05/22 06:01:31 PM
8 Std 6	Standard	CYA-1	127.942.0 amu	547000	5000	4930	98.6	1.00	1.98	N/A	☒	CYA-MRM_070423.dam	2007/05/22 06:12:15 PM
9 S34717	Unknown	CYA-1	127.942.0 amu	2230000	N/A	20300	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 06:23:01 PM
10 S34733	Unknown	CYA-1	127.942.0 amu	1400000	N/A	12700	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 06:33:48 PM
11 S34737	Unknown	CYA-1	127.942.0 amu	1270000	N/A	11500	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 06:44:31 PM
12 S34738	Unknown	CYA-1	127.942.0 amu	379000	N/A	3400	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 06:55:15 PM
13 Spk 1	Quality Control	CYA-1	127.942.0 amu	55200	500	435	87.1	1.00	1.98	N/A	☒	CYA-MRM_070423.dam	2007/05/22 07:06:01 PM
14 S34744	Unknown	CYA-1	127.942.0 amu	4250000	N/A	38700	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 07:16:48 PM
15 S34745	Unknown	CYA-1	127.942.0 amu	2130000	N/A	19400	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 07:27:33 PM
16 S34750	Unknown	CYA-1	127.942.0 amu	7590000	N/A	68200	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 07:38:19 PM
17 S34751	Unknown	CYA-1	127.942.0 amu	8610000	N/A	78600	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 07:49:05 PM
18 S34762	Unknown	CYA-1	127.942.0 amu	6670000	N/A	62700	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 07:59:50 PM
19 S39194	Unknown	CYA-1	127.942.0 amu	376	N/A	< 0	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 08:10:35 PM
20 S39197	Unknown	CYA-1	127.942.0 amu	10700	N/A	28.9	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 08:21:05 PM
21 S39546	Unknown	CYA-1	127.942.0 amu	52000	500	406	81.3	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 08:32:05 PM
22 Spk 1A	Quality Control	CYA-1	127.942.0 amu	22100	N/A	134	N/A	1.00	1.97	N/A	☒	CYA-MRM_070423.dam	2007/05/22 08:42:50 PM
23 S18197	Unknown	CYA-1	127.942.0 amu	12500	N/A	45.2	N/A	1.00	1.97	N/A		CYA-MRM_070423.dam	2007/05/22 08:53:35 PM
24 S18177	Unknown	CYA-1	127.942.0 amu	10800	N/A	681	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 09:04:20 PM
25 S08770	Unknown	CYA-1	127.942.0 amu	12500	N/A	29.7	N/A	1.00	1.97	N/A		CYA-MRM_070423.dam	2007/05/22 09:15:06 PM
26 S22957	Unknown	CYA-1	127.942.0 amu	82000	N/A	485	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 09:25:51 PM
27 S22968	Unknown	CYA-1	127.942.0 amu	2190	N/A	455	N/A	1.00	1.98	N/A		CYA-MRM_070423.dam	2007/05/22 09:36:36 PM
28 Std 1	Standard	CYA-1	127.942.0 amu	57300	500	931	91.0	1.00	1.98	N/A	☒	CYA-MRM_070423.dam	2007/05/22 09:47:31 PM
29 Std 2	Standard	CYA-1	127.942.0 amu	109000	1000	931	93.1	1.00	1.98	N/A	☒	CYA-MRM_070423.dam	2007/05/22 09:58:07 PM
30 Std 3	Standard	CYA-1	127.942.0 amu	185000	1500	1620	106	1.00	1.98	N/A	☒	CYA-MRM_070423.dam	2007/05/22 10:08:52 PM
31 Std 4	Standard	CYA-1	127.942.0 amu	273000	2500	2420	96.9	1.00	1.98	N/A	☒	CYA-MRM_070423.dam	2007/05/22 10:19:37 PM
32 Std 5	Standard	CYA-1	127.942.0 amu	425000	4000	3820	95.4	1.00	1.98	N/A	☒	CYA-MRM_070423.dam	2007/05/22 10:30:23 PM
33 Std 6	Standard	CYA-1	127.942.0 amu	545000	5000	4810	98.2	1.00	1.96	N/A	☒	CYA-MRM_070423.dam	2007/05/22 10:41:09 PM
34 Spk 1	Quality Control	CYA-1	127.942.0 amu	53600	500	422	84.3	1.00	1.96	N/A	☒	CYA-MRM_070423.dam	2007/05/22 10:52:17 AM
35 Std 7 Dil 1/100	Quality Control	CYA-1	127.942.0 amu	339000	50000	29500	58.9	100	1.95	N/A	☒	CYA-MRM_070423.dam	2007/05/23 10:52:17 AM
36 S34717 Dil 1/100	Unknown	CYA-1	127.942.0 amu	154000	N/A	1340	N/A	1.00	1.95	N/A		CYA-MRM_070423.dam	2007/05/23 11:03:03 AM
37 S34733 Dil 1/100	Unknown	CYA-1	127.942.0 amu	99000	N/A	836	N/A	1.00	1.96	N/A		CYA-MRM_070423.dam	2007/05/23 11:13:48 AM
38 S34737 Dil 1/100	Unknown	CYA-1	127.942.0 amu	99400	N/A	830	N/A	1.00	1.96	N/A		CYA-MRM_070423.dam	2007/05/23 11:24:36 AM
39 S34745 Dil 1/100	Unknown	CYA-1	127.942.0 amu	202000	N/A	1770	N/A	1.00	1.96	N/A		CYA-MRM_070423.dam	2007/05/23 11:35:23 AM
40 S34744 Dil 1/500	Unknown	CYA-1	127.942.0 amu	91700	N/A	769	N/A	1.00	1.95	N/A		CYA-MRM_070423.dam	2007/05/23 11:56:56 AM
41 S34750 Dil Recon Dil 1/250	Equiv 1/500	CYA-1	127.942.0 amu	195000	N/A	1630	N/A	1.00	1.95	N/A		CYA-MRM_070423.dam	2007/05/23 12:07:42 PM
42 S34751 Dil 1/500	Unknown	CYA-1	127.942.0 amu	344000	N/A	3060	N/A	1.00	1.96	N/A		CYA-MRM_070423.dam	2007/05/23 12:18:28 PM
43 S34762 Dil 1/500	Unknown	CYA-1	127.942.0 amu	207000	N/A	1820	N/A	1.00	1.96	N/A		CYA-MRM_070423.dam	2007/05/23 12:29:14 PM
44 Std 7 Dil 1/500	Quality Control	CYA-1	127.942.0 amu	72800	50000	28600	56.6	500	1.96	N/A	☒	CYA-MRM_070423.dam	2007/05/23 01:31:42 PM

Method Name: CYA-MRM_20070427.qmf

1:100 5x enhancement
actual dilution factor = $\frac{100}{599} = 17$

1:500 15x enhancement
actual dilution factor = $\frac{500}{599} = 84$

934717 = 1340 x 17 = 22780 ppb
934733 836 x 17 = 14,212 ppb
934737 = 830 x 17 = 14,110 ppb
934745 = 1770 x 17 = 30,090 ppb

934747 = 769 x 84 = 64,596 ppb
934750 = 1630 x 84 = 136,920 ppb
934751 = 3080 x 84 = 258,720 ppb
934762 = 1820 x 84 = 152,880 ppb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Dry_070522.rdb

Sample Name	Sample Type	Analyste Peak Name	Analyste Mass Ranges (amu)	Analyste Peak Area (counts)	Analyste Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Dilution Factor	Analyste Retention Time (min)	Analyste Signal To Noise	Use Record	Record Modified	Acquisition Method	Acquisition Date
1 Rinse	Solvent	CYA-2	127.985.0 amu	0.00	0.00	N/A	N/A	1.00	0.00	N/A			CYA-MRM_070423.dam	2007/05/22 04:54:39 PM
2 Blk	Blank	CYA-2	127.985.0 amu	2280	0.00	N/A	N/A	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 05:07:45 PM
3 Sid 1	Standard	CYA-2	127.985.0 amu	34200	500	513	103	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 05:16:30 PM
4 Sid 2	Standard	CYA-2	127.985.0 amu	68900	1000	1070	107	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 05:29:15 PM
5 Sid 3	Standard	CYA-2	127.985.0 amu	112000	1500	1760	117	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 05:40:00 PM
6 Sid 4	Standard	CYA-2	127.985.0 amu	156000	2500	2450	97.9	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 05:50:45 PM
7 Sid 5	Standard	CYA-2	127.985.0 amu	237000	4000	3740	93.4	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 06:01:31 PM
8 Sid 6	Standard	CYA-2	127.985.0 amu	312000	5000	4930	98.6	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 06:12:16 PM
9 S34733	Unknown	CYA-2	127.985.0 amu	1310000	N/A	20800	N/A	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 06:23:01 PM
10 S34733	Unknown	CYA-2	127.985.0 amu	818000	N/A	13000	N/A	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 06:33:46 PM
11 S34737	Unknown	CYA-2	127.985.0 amu	740000	N/A	11700	N/A	1.00	1.99	N/A			CYA-MRM_070423.dam	2007/05/22 06:44:31 PM
12 S34738	Unknown	CYA-2	127.985.0 amu	222000	N/A	3500	N/A	1.00	1.99	N/A			CYA-MRM_070423.dam	2007/05/22 06:55:18 PM
13 Spk 1	Quality Control	CYA-2	127.985.0 amu	296000	500	439	87.9	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 07:06:01 PM
14 S34744	Unknown	CYA-2	127.985.0 amu	2490000	N/A	39600	N/A	1.00	1.99	N/A			CYA-MRM_070423.dam	2007/05/22 07:16:46 PM
15 S34745	Unknown	CYA-2	127.985.0 amu	1250000	N/A	19800	N/A	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 07:27:33 PM
16 S34750	Unknown	CYA-2	127.985.0 amu	4490000	N/A	71500	N/A	1.00	1.99	N/A			CYA-MRM_070423.dam	2007/05/22 07:38:19 PM
17 S34751	Unknown	CYA-2	127.985.0 amu	5100000	N/A	81100	N/A	1.00	1.99	N/A			CYA-MRM_070423.dam	2007/05/22 07:48:05 PM
18 S34762	Unknown	CYA-2	127.985.0 amu	4070000	N/A	64800	N/A	1.00	1.99	N/A			CYA-MRM_070423.dam	2007/05/22 07:59:50 PM
19 S39194	Unknown	CYA-2	127.985.0 amu	439	N/A	< 0	N/A	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 08:10:35 PM
20 S39197	Unknown	CYA-2	127.985.0 amu	5860	N/A	62.0	N/A	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 08:21:20 PM
21 S39049	Unknown	CYA-2	127.985.0 amu	325	N/A	< 0	N/A	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 08:32:05 PM
22 Spk 1A	Quality Control	CYA-2	127.985.0 amu	28400	500	421	84.2	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 08:42:50 PM
23 S16197	Unknown	CYA-2	127.985.0 amu	12600	N/A	170	N/A	1.00	1.99	N/A			CYA-MRM_070423.dam	2007/05/22 08:53:35 PM
24 S16177	Unknown	CYA-2	127.985.0 amu	6860	N/A	79.6	N/A	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 09:04:20 PM
25 S09770	Unknown	CYA-2	127.985.0 amu	45000	N/A	686	N/A	1.00	1.99	N/A			CYA-MRM_070423.dam	2007/05/22 09:15:06 PM
26 S22957	Unknown	CYA-2	127.985.0 amu	6060	N/A	65.1	N/A	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 09:25:51 PM
27 S22956	Unknown	CYA-2	127.985.0 amu	1760	N/A	< 0	N/A	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 09:36:36 PM
28 Sid 1	Standard	CYA-2	127.985.0 amu	30700	500	457	91.4	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 09:47:21 PM
29 Sid 2	Standard	CYA-2	127.985.0 amu	60700	1000	935	93.5	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 09:58:07 PM
30 Sid 3	Standard	CYA-2	127.985.0 amu	105000	1500	1640	110	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 10:08:52 PM
31 Sid 4	Standard	CYA-2	127.985.0 amu	154000	2500	2420	96.8	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 10:19:37 PM
32 Sid 5	Standard	CYA-2	127.985.0 amu	241000	4000	3810	95.1	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 10:30:23 PM
33 Sid 6	Standard	CYA-2	127.985.0 amu	308000	5000	4870	97.5	1.00	1.98	N/A			CYA-MRM_070423.dam	2007/05/22 10:41:09 PM
34 Spk 1	Quality Control	CYA-2	127.985.0 amu	29200	0.00	433	N/A	1.00	1.96	N/A			CYA-MRM_070423.dam	2007/05/23 10:52:17 AM
35 Sid 7 Dil 1/100	Unknown	CYA-2	127.985.0 amu	196000	0.00	505000	N/A	1.00	1.96	N/A			CYA-MRM_070423.dam	2007/05/23 11:03:03 AM
36 S34717 Dil 1/100	Unknown	CYA-2	127.985.0 amu	91200	N/A	1420	N/A	1.00	1.96	N/A			CYA-MRM_070423.dam	2007/05/23 11:13:49 AM
37 S34733 Dil 1/100	Unknown	CYA-2	127.985.0 amu	56200	N/A	864	N/A	1.00	1.96	N/A			CYA-MRM_070423.dam	2007/05/23 11:24:36 AM
38 S34737 Dil 1/100	Unknown	CYA-2	127.985.0 amu	57000	N/A	876	N/A	1.00	1.96	N/A			CYA-MRM_070423.dam	2007/05/23 11:35:23 AM
39 S34745 Dil 1/100	Unknown	CYA-2	127.985.0 amu	51600	N/A	1750	N/A	1.00	1.96	N/A			CYA-MRM_070423.dam	2007/05/23 11:56:56 AM
40 S34744 Dil 1/500	Unknown	CYA-2	127.985.0 amu	13000	N/A	790	N/A	1.00	1.96	N/A			CYA-MRM_070423.dam	2007/05/23 12:07:42 PM
41 S34750 DBI Recan Dil 1/250 Equiv 1/500	Unknown	CYA-2	127.985.0 amu	12000	N/A	1760	N/A	1.00	1.96	N/A			CYA-MRM_070423.dam	2007/05/23 12:18:28 PM
42 S34751 Dil 1/500	Unknown	CYA-2	127.985.0 amu	202000	N/A	3180	N/A	1.00	1.97	N/A			CYA-MRM_070423.dam	2007/05/23 12:29:14 PM
43 S34762 Dil 1/500	Unknown	CYA-2	127.985.0 amu	122000	N/A	1910	N/A	1.00	1.96	N/A			CYA-MRM_070423.dam	2007/05/23 12:39:14 PM
44 Sid 7 Dil 1/500	Quality Control	CYA-2	127.985.0 amu	43300	0.00	320000	N/A	500	1.97	N/A			CYA-MRM_070423.dam	2007/05/23 01:31:42 PM

Method Name: CYA-MRM_20070427.qmf

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Results Name: CYA-Dry_070522.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Dry_070522.rdb

■ CYA-Dry_070522.rdb (CYA-1): "Linear" Regression ("1 / (x * x)" weighting): y = 109 x + 7.51e+003 (r = 0.9935)

Method Name: CYA-MRM_20070427.qmf

Page 1 of 1

CYA-Dry_070522.rdb (CYA-2): "Linear" Regression ("1 / (x * x)" weighting): y = 62.8 x + 1.97e+003 (r = 0.9935)

Method Name: CYA-NRM_20070427.qmf

Page 1 of 1

Results Name: CYA-Dry_070522.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Dry_070522.rdb

Printing Time: 01:58:15

Printing Date: Wednesday, May 23, 2007

Method Name: CYA-NRM_20070427.qmf

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Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Dry_070522.rdb

Method Name: CYA-MRM_20070427.qmf

Results Name: CYA-Dry_070522.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Dry_070522.rdb

Method Name: CYA-MRM_20070427.qmf

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CVA-Dry_070522.rdb

Method Name: CVA-MRX_20070427.qmf

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Results Name: CVA-Val_Dry_070502.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CVA-Val_Dry_070502.rdb

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Rinse	Solvent	CYA-1	786	0.00	N/A	N/A			1.00	16.9	1.75	127.942.0 amu	2007/05/03 01:37:54 AM
2 Blk 1	Blank	CYA-1	28000	0.00	N/A	N/A			1.00	472	1.94	127.942.0 amu	2007/05/03 01:48:40 AM
3 Blk 2	Blank	CYA-1	4050	0.00	N/A	N/A			1.00	52.6	1.95	127.942.0 amu	2007/05/03 01:59:26 AM
4 Std 1	Standard	CYA-1	56800	500	479	95.9	☒		1.00	668	1.94	127.942.0 amu	2007/05/03 02:10:12 AM
5 Std 2	Standard	CYA-1	110000	1000	1000	100	☒		1.00	1550	1.94	127.942.0 amu	2007/05/03 02:20:59 AM
6 Std 3	Standard	CYA-1	170000	1500	1590	106	☒		1.00	2890	1.94	127.942.0 amu	2007/05/03 02:31:46 AM
7 Std 4	Standard	CYA-1	207000	2500	1980	78.4	☒		1.00	2660	1.94	127.942.0 amu	2007/05/03 02:42:32 AM
8 Std 5	Standard	CYA-1	398000	4000	3850	96.3	☒		1.00	8010	1.94	127.942.0 amu	2007/05/03 02:53:18 AM
9 Std 6	Standard	CYA-1	507000	5000	4920	98.4	☒		1.00	6620	1.94	127.942.0 amu	2007/05/03 03:04:05 AM
10 LOD 1	Quality Control	CYA-1	58000	500	491	98.2	☒		1.00	939	1.95	127.942.0 amu	2007/05/03 03:14:51 AM
11 LOD 2	Quality Control	CYA-1	67000	500	491	98.2	☒		1.00	516	1.95	127.942.0 amu	2007/05/03 03:25:37 AM
12 LOD 3	Quality Control	CYA-1	67000	500	579	116	☒		1.00	2180	1.94	127.942.0 amu	2007/05/03 03:36:24 AM
13 LOD 4	Quality Control	CYA-1	60400	500	515	103	☒		1.00	874	1.95	127.942.0 amu	2007/05/03 03:47:09 AM
14 LOD 5	Quality Control	CYA-1	63200	500	542	108	☒		1.00	704	1.95	127.942.0 amu	2007/05/03 03:57:55 AM
15 LOD 6	Quality Control	CYA-1	59800	500	509	102	☒		1.00	802	1.95	127.942.0 amu	2007/05/03 04:08:41 AM
16 LOD 7	Quality Control	CYA-1	60000	500	511	102	☒		1.00	578	1.95	127.942.0 amu	2007/05/03 04:19:29 AM
17 LOD 8	Quality Control	CYA-1	67300	500	583	117	☒		1.00	1080	1.95	127.942.0 amu	2007/05/03 04:30:15 AM
18 Spk 2	Quality Control	CYA-1	170000	1500	1590	106	☒		1.00	4280	1.95	127.942.0 amu	2007/05/03 04:41:01 AM
19 Spk 2A	Quality Control	CYA-1	155000	1500	1450	96.6	☒		1.00	1630	1.95	127.942.0 amu	2007/05/03 04:51:47 AM
20 Spk 2B	Quality Control	CYA-1	161000	1500	1510	101	☒		1.00	3360	1.95	127.942.0 amu	2007/05/03 05:02:34 AM
21 Spk 3	Quality Control	CYA-1	419000	4000	4060	101	☒		1.00	3860	1.96	127.942.0 amu	2007/05/03 05:13:20 AM
22 Spk 3A	Quality Control	CYA-1	447000	4000	4330	108	☒		1.00	8570	1.96	127.942.0 amu	2007/05/03 05:24:06 AM
23 Spk 3B	Quality Control	CYA-1	409000	4000	3950	98.9	☒		1.00	5060	1.96	127.942.0 amu	2007/05/03 05:34:52 AM
24 R84590	Unknown	CYA-1	1130	N/A	< 0	N/A	☒		1.00	19.8	1.96	127.942.0 amu	2007/05/03 05:45:38 AM
25 R84591	Unknown	CYA-1	44000	N/A	352	N/A			1.00	599	1.96	127.942.0 amu	2007/05/03 05:56:23 AM
26 R84594	Unknown	CYA-1	5760	N/A	< 0	N/A			1.00	80.6	1.97	127.942.0 amu	2007/05/03 06:07:10 AM
27 R84595	Unknown	CYA-1	49600	N/A	408	N/A			1.00	879	1.96	127.942.0 amu	2007/05/03 06:17:55 AM
28 R84596	Unknown	CYA-1	804	N/A	< 0	N/A			1.00	23.3	1.95	127.942.0 amu	2007/05/03 06:28:43 AM
29 R84597	Unknown	CYA-1	1510	N/A	< 0	N/A			1.00	20.7	1.97	127.942.0 amu	2007/05/03 06:39:29 AM
30 R84598	Unknown	CYA-1	8100	N/A	< 0	N/A			1.00	158	1.97	127.942.0 amu	2007/05/03 06:50:15 AM
31 R84599	Unknown	CYA-1	9810	N/A	15.5	N/A			1.00	184	1.96	127.942.0 amu	2007/05/03 07:01:01 AM
32 R84601	Unknown	CYA-1	4830	N/A	< 0	N/A			1.00	41.0	2.00	127.942.0 amu	2007/05/03 07:11:48 AM
33 R84602	Unknown	CYA-1	6320	N/A	< 0	N/A			1.00	67.2	1.97	127.942.0 amu	2007/05/03 07:22:35 AM
34 R84604	Unknown	CYA-1	3960	N/A	< 0	N/A			1.00	95.2	1.96	127.942.0 amu	2007/05/03 07:33:21 AM
35 R84607	Unknown	CYA-1	933	N/A	< 0	N/A			1.00	63.3	1.96	127.942.0 amu	2007/05/03 07:44:08 AM
36 R84608	Unknown	CYA-1	4180	N/A	< 0	N/A			1.00	19.0	1.97	127.942.0 amu	2007/05/03 07:54:54 AM
37 R82930	Unknown	CYA-1	36500000	N/A	360000	N/A			1.00	227000	1.95	127.942.0 amu	2007/05/03 08:05:40 AM
38 R82950	Unknown	CYA-1	20800000	N/A	205000	N/A			1.00	18500	1.95	127.942.0 amu	2007/05/03 08:16:26 AM
39 R82962	Unknown	CYA-1	29700000	N/A	293000	N/A			1.00	15800	1.95	127.942.0 amu	2007/05/03 08:27:12 AM
40 R82966	Unknown	CYA-1	28400000	N/A	281000	N/A			1.00	16000	1.95	127.942.0 amu	2007/05/03 08:37:59 AM
41 R82971	Unknown	CYA-1	29200000	N/A	288000	N/A			1.00	11300	1.95	127.942.0 amu	2007/05/03 08:48:45 AM
42 R82982	Unknown	CYA-1	33500000	N/A	331000	N/A			1.00	13100	1.95	127.942.0 amu	2007/05/03 08:59:31 AM
43 R82988	Unknown	CYA-1	33700000	N/A	333000	N/A			1.00	11800	1.94	127.942.0 amu	2007/05/03 09:10:18 AM
44 R82997	Unknown	CYA-1	24100000	N/A	238000	N/A			1.00	8360	1.94	127.942.0 amu	2007/05/03 09:21:05 AM
45 R84548	Unknown	CYA-1	31500000	N/A	311000	N/A			1.00	7960	1.93	127.942.0 amu	2007/05/03 09:31:51 AM
46 Std 1	Standard	CYA-1	75400	500	672	134	☒		1.00	25.4	1.94	127.942.0 amu	2007/05/03 09:42:37 AM
47 Std 2	Standard	CYA-1	129000	1000	1190	119	☒		1.00	75.1	1.94	127.942.0 amu	2007/05/03 09:53:24 AM

Method Name: CVA-MRX_20070427.qmf

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Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
48 Std 3	Standard	CYA-1	176000	1500	1660	111	☒	☐	1.00	114	1.94	127.9420 amu	2007/05/03 10:04:11 AM
49 Std 4	Standard	CYA-1	211000	2500	2000	80.1	☐	☐	1.00	152	1.93	127.9420 amu	2007/05/03 10:14:57 AM
50 Std 5	Standard	CYA-1	392000	4000	3760	94.6	☒	☐	1.00	380	1.93	127.9420 amu	2007/05/03 10:25:43 AM
51 Std 6	Standard	CYA-1	505000	5000	4900	98.1	☒	☐	1.00	572	1.93	127.9420 amu	2007/05/03 10:36:29 AM
52 Std 7 (dil 1:1000)	Quality Control	CYA-1	40800	50000	322000	643	☒	☐	1000	49.6	1.93	127.9420 amu	2007/05/03 10:47:15 AM
53 R82930 (dil 1:20000)	Unknown	CYA-1	124000	N/A	1150	N/A	☐	☐	1.00	211	1.93	127.9420 amu	2007/05/03 10:58:22 AM
54 R82950 (dil 1:20000)	Unknown	CYA-1	46500	N/A	377	N/A	☐	☐	1.00	46.5	1.93	127.9420 amu	2007/05/03 11:09:08 AM
55 R82962 (dil 1:20000)	Unknown	CYA-1	85100	N/A	758	N/A	☐	☐	1.00	151	1.93	127.9420 amu	2007/05/03 11:19:54 AM
56 R82966 (dil 1:20000)	Unknown	CYA-1	94500	N/A	829	N/A	☐	☐	1.00	154	1.92	127.9420 amu	2007/05/03 11:41:28 AM
57 R82971 (dil 1:20000)	Unknown	CYA-1	72000	N/A	607	N/A	☐	☐	1.00	305	1.93	127.9420 amu	2007/05/03 11:52:14 AM
58 R82982 (dil 1:20000)	Unknown	CYA-1	69700	N/A	638	N/A	☐	☐	1.00	148	1.92	127.9420 amu	2007/05/03 12:03:01 PM
59 R82988 (dil 1:20000)	Unknown	CYA-1	72900	N/A	569	N/A	☐	☐	1.00	144	1.93	127.9420 amu	2007/05/03 12:13:47 PM
60 R82997 (dil 1:20000)	Unknown	CYA-1	65500	N/A	1050	N/A	☐	☐	1.00	319	1.92	127.9420 amu	2007/05/03 12:24:33 PM
61 R84548 (dil 1:20000)	Unknown	CYA-1	114000	N/A			☐	☐	1.00				

% enhancement per spike = $\frac{\text{dil factor}}{\% \text{acc}} = \frac{1000}{643} = 156$

is 6x
% actual dil factor

% enhancement for sample = $\frac{\text{dil factor}}{\% \text{acc of spike}} = \frac{30000}{\frac{643}{100}} = 3110$
is 6x
% actual dilution factor

% samples

R82930 = $1150 \times 3110 = 3576500 \text{ ppb} = 3577 \text{ ppm}$
R82950 = $377 \times 3110 = 1172470 \text{ ppb} = 1172 \text{ ppm}$
R82962 = $758 \times 3110 = 2357380 \text{ ppb} = 2357 \text{ ppm}$
R82966 = $851 \times 3110 = 2646610 \text{ ppb} = 2647 \text{ ppm}$
R82971 = $607 \times 3110 = 1956900 \text{ ppb} = 1956 \text{ ppm}$
R82982 = $607 \times 3110 = 1888900 \text{ ppb} = 1888 \text{ ppm}$
R82988 = $638 \times 3110 = 1984000 \text{ ppb} = 1984 \text{ ppm}$
R82997 = $569 \times 3110 = 1769590 \text{ ppb} = 1770 \text{ ppm}$
R84548 = $1050 \times 3110 = 3266500 \text{ ppb} = 3266 \text{ ppm}$

Method Name: CYA-MRM_20070427.qmz

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Results Name: CYA-Val_Dry_070502.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Val_Dry_070502.rdb

Printing Time: 01:54:27 PM

Printing Date: Thursday, May 03, 2007

Method Name: CYA-MRM_20070427.qmz

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Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Val_Dry_070502.rdb

Method Name: CYA-NRM_20070427.qmf

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Results Name: CYA-Val_Dry_070503.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Val_Dry_070503.rdb

Method Name: CYA-NRM_20070427.qmf

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Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Val_Dry_070502.rdb

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Rinse	Solvent	CYA-2	112	0.00	N/A	N/A		☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 01:37:54 AM
2 Blk 1	Blank	CYA-2	16300	0.00	N/A	N/A		☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 01:48:40 AM
3 Blk 2	Blank	CYA-2	3620	0.00	N/A	N/A		☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 01:59:26 AM
4 Std 1	Standard	CYA-2	33600	500	444	88.7	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 02:10:12 AM
5 Std 2	Standard	CYA-2	65700	1000	1020	102	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 02:20:59 AM
6 Std 3	Standard	CYA-2	102000	1500	1690	112	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 02:31:46 AM
7 Std 4	Standard	CYA-2	123000	2500	2070	82.8	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 02:42:32 AM
8 Std 5	Standard	CYA-2	233000	4000	4060	102	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 02:53:18 AM
9 Std 6	Standard	CYA-2	298000	5000	5230	105	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 03:04:05 AM
10 LOD 1	Quality Control	CYA-2	34200	500	454	90.8	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 03:14:51 AM
11 LOD 2	Quality Control	CYA-2	35300	500	474	94.7	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 03:25:37 AM
12 LOD 3	Quality Control	CYA-2	39300	500	547	109	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 03:36:24 AM
13 LOD 4	Quality Control	CYA-2	35900	500	485	96.9	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 03:47:09 AM
14 LOD 5	Quality Control	CYA-2	37200	500	509	102	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 03:57:55 AM
15 LOD 6	Quality Control	CYA-2	34700	500	464	92.8	☒	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 04:08:41 AM
16 LOD 7	Quality Control	CYA-2	36700	500	499	99.9	☒	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 04:19:29 AM
17 LOD 8	Quality Control	CYA-2	39400	500	549	110	☒	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 04:30:15 AM
18 Spk 2	Quality Control	CYA-2	99000	1500	1630	109	☒	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 04:41:01 AM
19 Spk 2A	Quality Control	CYA-2	89600	1500	1460	97.3	☒	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 04:51:47 AM
20 Spk 2B	Quality Control	CYA-2	97700	1500	1610	107	☒	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 05:02:34 AM
21 Spk 3	Quality Control	CYA-2	239000	4000	4170	104	☒	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 05:24:06 AM
22 Spk 3A	Quality Control	CYA-2	256000	4000	4470	112	☒	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 05:34:52 AM
23 Spk 3B	Quality Control	CYA-2	239000	4000	4170	104	☒	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 05:45:38 AM
24 R84590	Unknown	CYA-2	1630	N/A	< 0	N/A	☒	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 05:56:23 AM
25 R84591	Unknown	CYA-2	2690	N/A	321	N/A	☐	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 06:07:10 AM
26 R84594	Unknown	CYA-2	5080	N/A	< 0	N/A	☐	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 06:17:55 AM
27 R84595	Unknown	CYA-2	27300	N/A	328	N/A	☐	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 06:28:43 AM
28 R84596	Unknown	CYA-2	1080	N/A	< 0	N/A	☐	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 06:39:29 AM
29 R84597	Unknown	CYA-2	3820	N/A	< 0	N/A	☐	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 06:50:15 AM
30 R84598	Unknown	CYA-2	5610	N/A	< 0	N/A	☐	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 07:01:01 AM
31 R84599	Unknown	CYA-2	7100	N/A	< 0	N/A	☐	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 07:11:48 AM
32 R84601	Unknown	CYA-2	2990	N/A	< 0	N/A	☐	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 07:22:35 AM
33 R84602	Unknown	CYA-2	4050	N/A	< 0	N/A	☐	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 07:33:21 AM
34 R84604	Unknown	CYA-2	2560	N/A	< 0	N/A	☐	☐	1.00	N/A	1.97	127.9/85.0 amu	2007/05/03 07:44:08 AM
35 R84607	Unknown	CYA-2	1050	N/A	< 0	N/A	☐	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 07:54:54 AM
36 R84608	Unknown	CYA-2	4680	N/A	< 0	N/A	☐	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 08:05:40 AM
37 R82930	Unknown	CYA-2	23600000	N/A	429000	N/A	☐	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 08:16:26 AM
38 R82950	Unknown	CYA-2	19000000	N/A	236000	N/A	☐	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 08:27:12 AM
39 R82952	Unknown	CYA-2	18900000	N/A	343000	N/A	☐	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 08:37:59 AM
40 R82956	Unknown	CYA-2	18100000	N/A	328000	N/A	☐	☐	1.00	N/A	1.96	127.9/85.0 amu	2007/05/03 08:48:45 AM
41 R82971	Unknown	CYA-2	18700000	N/A	339000	N/A	☐	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 08:59:31 AM
42 R82982	Unknown	CYA-2	21500000	N/A	390000	N/A	☐	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 09:10:18 AM
43 R82988	Unknown	CYA-2	21900000	N/A	397000	N/A	☐	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 09:21:05 AM
44 R82997	Unknown	CYA-2	15100000	N/A	273000	N/A	☐	☐	1.00	N/A	1.94	127.9/85.0 amu	2007/05/03 09:31:51 AM
45 R84548	Unknown	CYA-2	20200000	N/A	366000	N/A	☐	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 09:42:37 AM
46 Std 1	Standard	CYA-2	37700	500	518	104	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 09:53:24 AM
47 Std 2	Standard	CYA-2	70600	1000	1120	112	☒	☐	1.00	N/A	1.95	127.9/85.0 amu	2007/05/03 09:53:24 AM

Method Name: CYA-NRM_20070427.qmf

Results Name: CYA-Val_Dry_070502.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Val_Dry_070502.rdb

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
48 Std 3	Standard	CYA-2	99200	1500	1630	109	☒	☐	1.00	N/A	1.94	127.9/85.0 amu	2007/05/03 10:04:11 AM
49 Std 4	Standard	CYA-2	121000	2500	2030	81.2	☒	☐	1.00	N/A	1.94	127.9/85.0 amu	2007/05/03 10:14:57 AM
50 Std 5	Standard	CYA-2	224000	4000	3890	97.3	☒	☐	1.00	N/A	1.94	127.9/85.0 amu	2007/05/03 10:25:43 AM
51 Std 6	Standard	CYA-2	299000	5000	5250	105	☒	☐	1.00	N/A	1.94	127.9/85.0 amu	2007/05/03 10:36:29 AM
52 Std 7 (dil 1:1000)	Quality Control	CYA-2	23300	0.00	256000	N/A	☒	☐	1000	N/A	1.93	127.9/85.0 amu	2007/05/03 10:47:15 AM
53 R82930 (dil 1:20000)	Unknown	CYA-2	79200	N/A	1270	N/A	☐	☐	1.00	N/A	1.94	127.9/85.0 amu	2007/05/03 10:58:22 AM
54 R82950 (dil 1:20000)	Unknown	CYA-2	24800	N/A	263	N/A	☐	☐	1.00	N/A	1.94	127.9/85.0 amu	2007/05/03 11:09:08 AM
55 R82952 (dil 1:20000)	Unknown	CYA-2	49900	N/A	738	N/A	☐	☐	1.00	N/A	1.93	127.9/85.0 amu	2007/05/03 11:19:54 AM
56 R82956 (dil 1:20000)	Unknown	CYA-2	58600	N/A	900	N/A	☐	☐	1.00	N/A	1.93	127.9/85.0 amu	2007/05/03 11:30:41 AM
57 R82971 (dil 1:20000)	Unknown	CYA-2	43500	N/A	622	N/A	☐	☐	1.00	N/A	1.93	127.9/85.0 amu	2007/05/03 11:41:28 AM
58 R82982 (dil 1:20000)	Unknown	CYA-2	46400	N/A	676	N/A	☐	☐	1.00	N/A	1.93	127.9/85.0 amu	2007/05/03 11:52:14 AM
59 R82988 (dil 1:20000)	Unknown	CYA-2	46900	N/A	685	N/A	☐	☐	1.00	N/A	1.93	127.9/85.0 amu	2007/05/03 12:03:01 PM
60 R82997 (dil 1:20000)	Unknown	CYA-2	41400	N/A	585	N/A	☐	☐	1.00	N/A	1.93	127.9/85.0 amu	2007/05/03 12:13:47 PM
61 R84548 (dil 1:20000)	Unknown	CYA-2	67300	N/A	1060	N/A	☐	☐	1.00	N/A	1.93	127.9/85.0 amu	2007/05/03 12:24:33 PM

Method Name: CYA-NRM_20070427.qmf

Results Path: D:\Analyst Data\Projects\Residue\aminopterin\Results\AptMel-dry food_070417.rdb

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-1	1280	0.00	N/A	N/A			1.00	6.62	1.81	2007/04/17 02:40:35 PM
2 Blk	Blank	MEL-1	1510	0.00	N/A	N/A			1.00	7.94	1.79	2007/04/17 02:46:10 PM
3 Std 1	Standard	MEL-1	75600	500	508	102	☒		1.00	428	1.78	2007/04/17 02:51:45 PM
4 Std 2	Standard	MEL-1	162000	1000	1040	104	☒		1.00	1860	1.78	2007/04/17 02:57:20 PM
5 Std 3	Standard	MEL-1	245000	1500	1550	103	☒		1.00	1890	1.78	2007/04/17 03:02:55 PM
6 Std 4	Standard	MEL-1	394000	2500	2460	98.6	☒		1.00	2190	1.78	2007/04/17 03:08:30 PM
7 Std 5	Standard	MEL-1	648000	4000	4020	100	☒		1.00	3590	1.78	2007/04/17 03:14:05 PM
8 Std 6	Standard	MEL-1	819000	5000	5070	101	☒		1.00	2570	1.78	2007/04/17 03:19:40 PM
9 R82930	Unknown	MEL-1	1300000	N/A	8000	N/A			1.00	4050	1.78	2007/04/17 03:25:15 PM
10 R82950	Unknown	MEL-1	641000	N/A	3980	N/A			1.00	2620	1.78	2007/04/17 03:30:50 PM
11 R82962	Unknown	MEL-1	597000	N/A	3710	N/A			1.00	3010	1.78	2007/04/17 03:36:25 PM
12 R82966	Unknown	MEL-1	1090000	N/A	6710	N/A			1.00	4630	1.77	2007/04/17 03:42:00 PM
13 Spk 1	Quality Control	MEL-1	73600	500	496	99.2	☒		1.00	222	1.78	2007/04/17 03:47:35 PM
14 R82971	Unknown	MEL-1	421000	N/A	2630	N/A			1.00	2590	1.78	2007/04/17 03:53:10 PM
15 R82982	Unknown	MEL-1	1290000	N/A	7940	N/A			1.00	5020	1.78	2007/04/17 03:58:45 PM
16 R82988	Unknown	MEL-1	823000	N/A	5100	N/A			1.00	2620	1.78	2007/04/17 04:04:19 PM
17 Spk 1A	Quality Control	MEL-1	75800	500	509	102	☒		1.00	275	1.78	2007/04/17 04:09:54 PM
18 R82997	Unknown	MEL-1	1570000	N/A	9660	N/A			1.00	7590	1.78	2007/04/17 04:15:29 PM
19 R83003	Unknown	MEL-1	1530000	N/A	9440	N/A			1.00	6910	1.78	2007/04/17 04:21:04 PM
20 R76720	Unknown	MEL-1	52600	N/A	367	N/A			1.00	165	1.78	2007/04/17 04:26:41 PM
21 Std 1	Standard	MEL-1	71600	500	484	96.8	☒		1.00	390	1.78	2007/04/17 04:32:15 PM
22 Std 3	Standard	MEL-1	229000	1500	1450	96.7	☒		1.00	1130	1.78	2007/04/17 04:37:51 PM
23 Std 6	Standard	MEL-1	787000	5000	4870	97.4	☒		1.00	3510	1.78	2007/04/17 04:43:25 PM
24 Std 6 (dil 1:5)	Quality Control	MEL-1	404000	5000	5050	101	☒		2.00	1440	1.81	2007/04/17 04:49:25 PM
25 R82930 (dil 1:5)	Unknown	MEL-1	689000	N/A	8540	N/A			2.00	2900	1.80	2007/04/17 04:54:58 PM
26 R82966 (dil 1:5)	Unknown	MEL-1	600000	N/A	7460	N/A			2.00	1670	1.80	2007/04/17 05:00:33 PM
27 R82982 (dil 1:5)	Unknown	MEL-1	682000	N/A	8460	N/A			2.00	6670	1.80	2007/04/17 05:06:09 PM
28 R82997 (dil 1:5)	Unknown	MEL-1	957000	N/A	11800	N/A			2.00	5210	1.81	2007/04/17 05:11:44 PM
29 R83003 (dil 1:5)	Unknown	MEL-1	928000	N/A	11500	N/A			2.00	6040	1.80	2007/04/17 05:17:19 PM

Method Name: APT-MEL_070409.qml

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Results Name: AptMel-dry food_070417.rdb

Results Path: D:\Analyst Data\Projects\Residue\aminopterin\Results\AptMel-dry food_070417.rdb

Method Name: APT-MEL_070409.qml

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Untitled 3 (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): $y = 163x + -7.28e+003$ ($r = 0.9992$)

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-dry food_070417.rdb

Printing Date: Tuesday, April 17, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Peak Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/17 02:40:35 PM
2 Blk	Blank	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/17 02:46:10 PM
3 Std 1	Standard	MEL-3	5640.	500.	503.	101.	☒	☐	1.00	185.	1.78	2007/04/17 02:51:45 PM
4 Std 2	Standard	MEL-3	11500.	1000.	1020.	102.	☒	☐	1.00	402.	1.78	2007/04/17 02:57:20 PM
5 Std 3	Standard	MEL-3	18300.	1500.	1620.	108.	☒	☐	1.00	440.	1.78	2007/04/17 03:02:55 PM
6 Std 4	Standard	MEL-3	27700.	2500.	2440.	97.8	☒	☐	1.00	645.	1.77	2007/04/17 03:08:30 PM
7 Std 5	Standard	MEL-3	45400.	4000.	4000.	100.	☒	☐	1.00	1520.	1.78	2007/04/17 03:14:05 PM
8 Std 6	Standard	MEL-3	57900.	5000.	5110.	102.	☒	☐	1.00	1400.	1.77	2007/04/17 03:19:40 PM
9 R82930	Unknown	MEL-3	91000.	N/A	8030.	N/A		☐	1.00	1290.	1.77	2007/04/17 03:25:15 PM
10 R82950	Unknown	MEL-3	45000.	N/A	3970.	N/A		☐	1.00	801.	1.78	2007/04/17 03:30:50 PM
11 R82962	Unknown	MEL-3	43700.	N/A	3860.	N/A		☐	1.00	965.	1.77	2007/04/17 03:36:25 PM
12 R82966	Unknown	MEL-3	75500.	N/A	6660.	N/A		☐	1.00	767.	1.77	2007/04/17 03:42:00 PM
13 Spk 1	Quality Control	MEL-3	5070.	500.	453.	90.8	☒	☐	1.00	72.8	1.77	2007/04/17 03:47:35 PM
14 R82971	Unknown	MEL-3	29600.	N/A	2620.	N/A		☐	1.00	708.	1.78	2007/04/17 03:53:10 PM
15 R82982	Unknown	MEL-3	90700.	N/A	8000.	N/A		☐	1.00	2270.	1.77	2007/04/17 03:58:45 PM
16 R82986	Unknown	MEL-3	57900.	N/A	5110.	N/A		☐	1.00	1040.	1.78	2007/04/17 04:04:19 PM
17 Spk 1A	Quality Control	MEL-3	5140.	500.	459.	91.7	☒	☐	1.00	112.	1.77	2007/04/17 04:09:54 PM
18 R82997	Unknown	MEL-3	109000.	N/A	9620.	N/A		☐	1.00	3310.	1.77	2007/04/17 04:15:29 PM
19 R83003	Unknown	MEL-3	107000.	N/A	9440.	N/A		☐	1.00	1790.	1.77	2007/04/17 04:21:04 PM
20 R76720	Unknown	MEL-3	3840.	N/A	344.	N/A		☐	1.00	54.3	1.78	2007/04/17 04:26:41 PM
21 Std 1	Standard	MEL-3	5490.	500.	490.	98.0	☒	☐	1.00	82.4	1.78	2007/04/17 04:32:15 PM
22 Std 3	Standard	MEL-3	16200.	1500.	1430.	95.4	☒	☐	1.00	204.	1.77	2007/04/17 04:37:51 PM
23 Std 6	Standard	MEL-3	54500.	5000.	4810.	96.3	☒	☐	1.00	1220.	1.78	2007/04/17 04:43:25 PM
24 Std 6 (dil 1:5)	Quality Control	MEL-3	30200.	0.00	5340.	N/A	☒	☐	2.00	881.	1.80	2007/04/17 04:49:25 PM
25 R82930 (dil 1:5)	Unknown	MEL-3	49000.	N/A	8660.	N/A		☐	2.00	1090.	1.80	2007/04/17 04:54:58 PM
26 R82966 (dil 1:5)	Unknown	MEL-3	42200.	N/A	7440.	N/A		☐	2.00	1270.	1.80	2007/04/17 05:00:33 PM
27 R82982 (dil 1:5)	Unknown	MEL-3	48400.	N/A	8550.	N/A		☐	2.00	1020.	1.80	2007/04/17 05:06:09 PM
28 R82997 (dil 1:5)	Unknown	MEL-3	68200.	N/A	12000.	N/A		☐	2.00	1390.	1.80	2007/04/17 05:11:44 PM
29 R83003 (dil 1:5)	Unknown	MEL-3	65800.	N/A	11600.	N/A		☐	2.00	1240.	1.80	2007/04/17 05:17:19 PM

Method Name: APT-MEL_070409.qmf

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Results Name: AptMel-dry food_070417.rdb

Printing Time: 05:30:20

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-dry food_070417.rdb

Printing Date: Tuesday, April 17, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Peak Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	APT-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/17 02:40:35 PM
2 Blk	Blank	APT-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/17 02:46:10 PM
3 Std 1	Standard	APT-1	104000.	100.	103.	103.	☒	☐	1.00	1490.	2.17	2007/04/17 02:51:45 PM
4 Std 2	Standard	APT-1	226000.	200.	215.	107.	☒	☐	1.00	1920.	2.18	2007/04/17 02:57:20 PM
5 Std 3	Standard	APT-1	326000.	300.	308.	103.	☒	☐	1.00	5370.	2.17	2007/04/17 03:02:55 PM
6 Std 4	Standard	APT-1	549000.	500.	513.	103.	☒	☐	1.00	8880.	2.17	2007/04/17 03:08:30 PM
7 Std 5	Standard	APT-1	850000.	800.	790.	98.8	☒	☐	1.00	5640.	2.18	2007/04/17 03:14:05 PM
8 Std 6	Standard	APT-1	1080000.	1000.	1010.	101.	☒	☐	1.00	6740.	2.17	2007/04/17 03:19:40 PM
9 R82930	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/17 03:25:15 PM
10 R82950	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/17 03:30:50 PM
11 R82962	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/17 03:36:25 PM
12 R82966	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/17 03:42:00 PM
13 Spk 1	Quality Control	APT-1	103000.	100.	102.	102.	☒	☐	1.00	763.	2.18	2007/04/17 03:47:35 PM
14 R82971	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/17 03:53:10 PM
15 R82982	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/17 03:58:45 PM
16 R82986	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/17 04:04:19 PM
17 Spk 1A	Quality Control	APT-1	98100.	100.	97.2	97.2	☒	☐	1.00	970.	2.17	2007/04/17 04:09:54 PM
18 R82997	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/17 04:15:29 PM
19 R83003	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/17 04:21:04 PM
20 R76720	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/17 04:26:41 PM
21 Std 1	Standard	APT-1	94000.	100.	93.5	93.5	☒	☐	1.00	933.	2.18	2007/04/17 04:32:15 PM
22 Std 3	Standard	APT-1	310000.	300.	293.	97.5	☒	☐	1.00	1500.	2.18	2007/04/17 04:37:51 PM
23 Std 6	Standard	APT-1	1010000.	1000.	941.	94.1	☒	☐	1.00	15500.	2.18	2007/04/17 04:43:25 PM
24 Std 6 (dil 1:5)	Quality Control	APT-1	426000.	1000.	799.	79.9	☒	☐	2.00	4860.	2.18	2007/04/17 04:49:25 PM
25 R82930 (dil 1:5)	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	2.00	N/A	0.00	2007/04/17 04:54:58 PM
26 R82966 (dil 1:5)	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	2.00	N/A	0.00	2007/04/17 05:00:33 PM
27 R82982 (dil 1:5)	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	2.00	N/A	0.00	2007/04/17 05:06:09 PM
28 R82997 (dil 1:5)	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	2.00	N/A	0.00	2007/04/17 05:11:44 PM
29 R83003 (dil 1:5)	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	2.00	N/A	0.00	2007/04/17 05:17:19 PM

Method Name: APT-MEL_070409.qmf

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Table 7.3.: Dry Food Data

Study Sample	Coded Sample Name
WFWG1	R70141
WFWG2	R79942
WFWG3	R74956
WFWG4	R74957
WFWG5	R74958
WFWG6	R85265
WFSPI1	R78180
WFSPI2	S15721
WFRPC1	S08570
WFRPC2	S08572
WFRPC3	R78179
WFRPC4	S15729

Results Name: AptMEL-wet food_070409.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMEL-wet Food_070409.rdb

Printing Time: 1:01:04 PM

Printing Date: Tuesday, April 10, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/09 11:21:38 PM
2 Blk	Blank	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/09 11:27:32 PM
3 Std 1	Standard	MEL-3	4030	500	552	110	☒	☐	1.00	109	1.67	2007/04/09 11:33:35 PM
4 Std 2	Standard	MEL-3	7000	1000	957	95.7	☒	☐	1.00	204	1.67	2007/04/09 11:39:39 PM
5 Std 3	Standard	MEL-3	11400	1500	1560	104	☒	☐	1.00	355	1.67	2007/04/09 11:45:43 PM
6 Std 4	Standard	MEL-3	18100	2500	2470	98.8	☒	☐	1.00	503	1.67	2007/04/09 11:51:46 PM
7 Std 5	Standard	MEL-3	29600	4000	4040	101	☒	☐	1.00	771	1.67	2007/04/09 11:57:50 PM
8 Std 6	Standard	MEL-3	39400	5000	5360	107	☒	☐	1.00	892	1.67	2007/04/10 12:03:25 AM
9 Spk 1	Quality Control	MEL-3	4240	500	581	116	☒	☐	1.00	69.2	1.67	2007/04/10 12:09:00 AM
10 Sample 1	Unknown	MEL-3	575000	N/A	78200	N/A		☐	1.00	9820	1.67	2007/04/10 12:14:35 AM
11 Sample 2	Unknown	MEL-3	885000	N/A	120000	N/A		☐	1.00	8470	1.67	2007/04/10 12:20:10 AM
12 Sample 3	Unknown	MEL-3	821000	N/A	112000	N/A		☐	1.00	5560	1.67	2007/04/10 12:25:45 AM
13 Sample (comp)	Unknown	MEL-3	721000	N/A	98000	N/A		☐	1.00	2750	1.67	2007/04/10 12:31:48 AM
14 Sample 1 (dup)	Unknown	MEL-3	530000	N/A	72200	N/A		☐	1.00	2660	1.67	2007/04/10 12:37:51 AM
15 Sample 2 (dup)	Unknown	MEL-3	808000	N/A	110000	N/A		☐	1.00	2500	1.67	2007/04/10 12:43:54 AM
16 Sample 3 (dup)	Unknown	MEL-3	792000	N/A	108000	N/A		☐	1.00	2640	1.67	2007/04/10 12:49:57 AM
17 Sample 1 (dil 1:100)	Unknown	MEL-3	26100	N/A	355000	N/A		☐	100	96.9	1.72	2007/04/10 12:56:00 AM
18 Sample 2 (dil 1:100)	Unknown	MEL-3	48900	N/A	666000	N/A		☐	100	387	1.72	2007/04/10 1:02:12 AM
19 Sample 3 (dil 1:100)	Unknown	MEL-3	55000	N/A	749000	N/A		☐	100	424	1.72	2007/04/10 1:08:15 AM
20 Sample 1 (dil 1:500)	Unknown	MEL-3	7380	N/A	504000	N/A		☐	500	66.0	1.73	2007/04/10 1:24:59 AM
21 Sample 2 (dil 1:500)	Unknown	MEL-3	17500	N/A	1190000	N/A		☐	500	147	1.72	2007/04/10 1:30:53 AM
22 Sample 3 (dil 1:500)	Unknown	MEL-3	16400	N/A	1110000	N/A		☐	500	136	1.73	2007/04/10 1:36:56 AM
23 Spk 1A	Quality Control	MEL-3	4210	0.00	577	N/A	☒	☐	1.00	43.4	1.68	2007/04/10 1:42:59 AM
24 Std 1	Standard	MEL-3	3350	500	460	91.9	☒	☐	1.00	32.1	1.68	2007/04/10 1:49:02 AM
25 Std 3	Standard	MEL-3	10400	1500	1420	94.6	☒	☐	1.00	124	1.68	2007/04/10 1:55:04 AM
26 Std 6	Standard	MEL-3	35400	5000	4820	96.4	☒	☐	1.00	714	1.67	2007/04/10 2:01:07 AM

AptMEL-wet food_070409.rdb (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): y = 99.8 x + 2.71e+003 (r = 0.9972)

Results Name: AptMEL-wet food_070409.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMEL-wet food_070409.rdb

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	APT-1	0.00	0.00	N/A	N/A			1.00	N/A	0.00	2007/04/09 11:21:38 PM
2 Blk	Blank	APT-1	0.00	0.00	N/A	N/A			1.00	N/A	0.00	2007/04/09 11:27:32 PM
3 Std 1	Standard	APT-1	132000.	100.	105.	105.	☒		1.00	5470.	2.15	2007/04/09 11:33:35 PM
4 Std 2	Standard	APT-1	265000.	200.	202.	101.	☒		1.00	5440.	2.15	2007/04/09 11:39:39 PM
5 Std 3	Standard	APT-1	385000.	300.	290.	96.6	☒		1.00	7040.	2.15	2007/04/09 11:45:43 PM
6 Std 4	Standard	APT-1	588000.	500.	437.	87.5	☒		1.00	7270.	2.15	2007/04/09 11:51:46 PM
7 Std 5	Standard	APT-1	970000.	800.	715.	89.4	☒		1.00	24400.	2.15	2007/04/09 11:57:50 PM
8 Std 6	Standard	APT-1	1580000.	1000.	1160.	116.	☒		1.00	37900.	2.15	2007/04/10 12:03:25 AM
9 Spk 1	Quality Control	APT-1	118000.	100.	95.0	95.0	☒		1.00	1620.	2.15	2007/04/10 12:09:00 AM
10 Sample 1	Unknown	APT-1	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	2007/04/10 12:14:35 AM
11 Sample 2	Unknown	APT-1	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	2007/04/10 12:20:10 AM
12 Sample 3	Unknown	APT-1	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	2007/04/10 12:25:45 AM
13 Sample (comp)	Unknown	APT-1	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	2007/04/10 12:31:48 AM
14 Sample 1 (dup)	Unknown	APT-1	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	2007/04/10 12:37:51 AM
15 Sample 2 (dup)	Unknown	APT-1	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	2007/04/10 12:43:54 AM
16 Sample 3 (dup)	Unknown	APT-1	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	2007/04/10 12:49:57 AM
17 Sample 1 (dil 1:100)	Unknown	APT-1	0.00	N/A	No Peak	N/A			100.	N/A	0.00	2007/04/10 12:56:00 AM
18 Sample 2 (dil 1:100)	Unknown	APT-1	0.00	N/A	No Peak	N/A			100.	N/A	0.00	2007/04/10 1:02:12 AM
19 Sample 3 (dil 1:100)	Unknown	APT-1	0.00	N/A	No Peak	N/A			100.	N/A	0.00	2007/04/10 1:08:15 AM
20 Sample 1 (dil 1:500)	Unknown	APT-1	0.00	N/A	No Peak	N/A			500.	N/A	0.00	2007/04/10 1:24:59 AM
21 Sample 2 (dil 1:500)	Unknown	APT-1	0.00	N/A	No Peak	N/A			500.	N/A	0.00	2007/04/10 1:30:53 AM
22 Sample 3 (dil 1:500)	Unknown	APT-1	0.00	N/A	No Peak	N/A			500.	N/A	0.00	2007/04/10 1:36:56 AM
23 Spk 1A	Quality Control	APT-1	117000.	100.	94.7	94.7	☒		1.00	6440.	2.16	2007/04/10 1:42:59 AM
24 Std 1	Standard	APT-1	123000.	100.	98.7	98.7	☒		1.00	2420.	2.16	2007/04/10 1:49:02 AM
25 Std 3	Standard	APT-1	365000.	300.	275.	91.7	☒		1.00	13200.	2.15	2007/04/10 1:55:04 AM
26 Std 6	Standard	APT-1	1540000.	1000.	1130.	113.	☒		1.00	23800.	2.14	2007/04/10 2:01:07 AM

Sample Name	Sample ID	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Calculated Concentration (ppb)	Analyte Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Acquisition Date
1 Rinse		Solvent	MEL-1	0.00	N/A	0.00	N/A	0.00	2007/05/08 1:14:48 PM
2 Blank		Blank	MEL-1	4390.	N/A	0.00	N/A	2.02	2007/05/08 1:20:22 PM
3 Std 1		Standard	MEL-1	128000.	484.	500.	96.8	2.02	2007/05/08 1:25:56 PM
4 Std 2		Standard	MEL-1	240000.	964.	1000.	96.4	2.01	2007/05/08 1:31:31 PM
5 Std 3		Standard	MEL-1	366000.	1510.	1500.	101.	2.01	2007/05/08 1:37:05 PM
6 Std 4		Standard	MEL-1	582000.	2430.	2500.	97.3	2.02	2007/05/08 1:42:39 PM
7 Std 5		Standard	MEL-1	940000.	3970.	4000.	99.3	2.02	2007/05/08 1:48:14 PM
8 Std 6		Standard	MEL-1	1120000.	4750.	5000.	95.0	2.02	2007/05/08 1:53:49 PM
9 Spk 1		Quality Control	MEL-1	117000.	437.	500.	87.5	2.02	2007/05/08 1:59:24 PM

17	SPK 1A		Quality Control	MEL-1	118000.	440.	500.	88.1	2.01			2007/05/08 2:44:02 PM
18	S15700	A743492	Unknown	MEL-1	565000.	2360.	N/A	N/A	2.04			2007/05/08 2:49:36 PM
19	S15720	A743496	Unknown	MEL-1	409000.	1690.	N/A	N/A	2.02			2007/05/08 2:55:11 PM
20	S15721	A743498	Unknown	MEL-1	622000.	2600.	N/A	N/A	2.04			2007/05/08 3:00:45 PM
21	S15728	A743500	Unknown	MEL-1	566000.	2360.	N/A	N/A	2.04			2007/05/08 3:06:19 PM
22	S15729	A743501	Unknown	MEL-1	756000.	3180.	N/A	N/A	2.03			2007/05/08 3:11:53 PM
23	S15730	A743502	Unknown	MEL-1	2900.	< 0	N/A	N/A	2.04			2007/05/08 3:17:27 PM
24	S15966	A743553	Unknown	MEL-1	29200.	59.6	N/A	N/A	2.03			2007/05/08 3:23:01 PM
25	S15967	A743553	Unknown	MEL-1	0.00	No Peak	N/A	N/A	0.00			2007/05/08 3:28:35 PM
26	S15647	A743468	Unknown	MEL-1	2750.	< 0	N/A	N/A	2.05			2007/05/08 3:34:10 PM
27	S15648	A743469	Unknown	MEL-1	0.00	No Peak	N/A	N/A	0.00			2007/05/08 3:39:44 PM

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-wet food_070508.rush.rdb

Printing Date: Wednesday, May 09, 200

Sample Name	Sample ID	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Calculated Concentration (ppb)	Analyte Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Acquisition Date
28 S15673	A743472	Unknown	MEL-1	2710.	< 0	N/A	N/A	2.04	2007/05/08 3:45:19 PM
29 S15676	A743475	Unknown	MEL-1	2280.	< 0	N/A	N/A	2.04	2007/05/08 3:50:53 PM
30 S15677	A743476	Unknown	MEL-1	2980.	< 0	N/A	N/A	2.03	2007/05/08 3:56:27 PM
31 S15680	A743480	Unknown	MEL-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 4:02:02 PM
32 S15698	A743489	Unknown	MEL-1	650000.	2720.	N/A	N/A	2.04	2007/05/08 4:07:36 PM
33 S15699	A743490	Unknown	MEL-1	520000.	2170.	N/A	N/A	2.03	2007/05/08 4:13:10 PM
34 Std 1		Standard	MEL-1	135000.	514.	500.	103.	2.01	2007/05/08 4:18:44 PM
35 Std 2		Standard	MEL-1	251000.	1010.	1000.	101.	2.01	2007/05/08 4:24:18 PM
36 Std 3		Standard	MEL-1	384000.	1580.	1500.	106.	2.02	2007/05/08 4:29:52 PM
37 Std 4		Standard	MEL-1	607000.	2540.	2500.	102.	2.02	2007/05/08 4:35:27 PM
38 Std 5		Standard	MEL-1	990000.	4180.	4000.	105.	2.01	2007/05/08 4:41:02 PM
39 Std 6		Standard	MEL-1	1170000.	4950.	5000.	99.1	2.02	2007/05/08 4:46:36 PM

Results Name: AptMel-wet food_070508.rush.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-wet food_070508.rush.rdb

Printing Time: 10:15:15 AM

Printing Date: Wednesday, May 09, 200

■ AptMel-wet food_070508.rush.rdb (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): y = 233 x + 1.53e+004 (r = 0.9988)

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-wet food_070508.rush.rdb

Printing Date: Wednesday, May 09, 2007

Results Name: AptMel-wet food_070508.rush.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-wet food_070508.rush.rdb

Printing Time: 10:16:06 AM

Printing Date: Wednesday, May 09, 2007

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMEL-wet_food_070508.rush.rdb

Results Name: AptMEL-wet food_070508.rush.rdb

Printing Time: 10:16:40 AM

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMEL-wet_food_070508.rush.rdb

Printing Date: Wednesday, May 09, 2007

Sample Name	Sample ID	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Calculated Concentration (ppb)	Analyte Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Acquisition Date
1 Rinse		Solvent	MEL-3	0.00	N/A	0.00	N/A	0.00	2007/05/08 1:14:48 PM
2 Blank		Blank	MEL-3	0.00	N/A	0.00	N/A	0.00	2007/05/08 1:20:22 PM
3 Std 1		Standard	MEL-3	7980.	454.	500.	90.8	2.01	2007/05/08 1:25:56 PM
4 Std 2		Standard	MEL-3	16400.	965.	1000.	96.5	2.01	2007/05/08 1:31:31 PM
5 Std 3		Standard	MEL-3	26000.	1550.	1500.	103.	2.01	2007/05/08 1:37:05 PM
6 Std 4		Standard	MEL-3	40200.	2410.	2500.	96.3	2.02	2007/05/08 1:42:39 PM
7 Std 5		Standard	MEL-3	65700.	3950.	4000.	98.8	2.01	2007/05/08 1:48:14 PM
8 Std 6		Standard	MEL-3	78400.	4720.	5000.	94.4	2.02	2007/05/08 1:53:49 PM
9 Spk 1		Quality Control	MEL-3	8370.	478.	500.	95.6	2.01	2007/05/08 1:59:24 PM

17 Spk 1A		Quality Control	MEL-3	7890.	449.	500.	89.8	2.01	2007/05/08 2:44:02 PM
18 S15700	A743492	Unknown	MEL-3	40200.	2410.	N/A	N/A	2.03	2007/05/08 2:49:36 PM
19 S15720	A743496	Unknown	MEL-3	27700.	1650.	N/A	N/A	2.02	2007/05/08 2:55:11 PM
20 S15721	A743498	Unknown	MEL-3	42100.	2520.	N/A	N/A	2.04	2007/05/08 3:00:45 PM
21 S15728	A743500	Unknown	MEL-3	39100.	2340.	N/A	N/A	2.03	2007/05/08 3:06:19 PM
22 S15729	A743501	Unknown	MEL-3	53200.	3190.	N/A	N/A	2.03	2007/05/08 3:11:53 PM
23 S15730	A743502	Unknown	MEL-3	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:17:27 PM
24 S15966	A743553	Unknown	MEL-3	2060.	95.5	N/A	N/A	2.02	2007/05/08 3:23:01 PM
25 S15967	A743553	Unknown	MEL-3	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:28:35 PM
26 S15647	A743468	Unknown	MEL-3	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:34:10 PM
27 S15648	A743469	Unknown	MEL-3	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:39:44 PM

Sample Name	Sample ID	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Calculated Concentration (ppb)	Analyte Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Acquisition Date
S15673	A743472	Unknown	MEL-3	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:45:19 PM
S15676	A743475	Unknown	MEL-3	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:50:53 PM
S15677	A743476	Unknown	MEL-3	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:56:27 PM
S15680	A743480	Unknown	MEL-3	0.00	No Peak	N/A	N/A	0.00	2007/05/08 4:02:02 PM
S15688	A743489	Unknown	MEL-3	45500.	2720.	N/A	N/A	2.03	2007/05/08 4:07:36 PM
S15699	A743490	Unknown	MEL-3	36600.	2190.	N/A	N/A	2.02	2007/05/08 4:13:10 PM
Std 1		Standard	MEL-3	9480.	545.	500.	109.	2.01	2007/05/08 4:18:44 PM
Std 2		Standard	MEL-3	16800.	986.	1000.	98.6	2.01	2007/05/08 4:24:18 PM
Std 3		Standard	MEL-3	27100.	1610.	1500.	107.	2.01	2007/05/08 4:29:52 PM
Std 4		Standard	MEL-3	41400.	2480.	2500.	99.3	2.01	2007/05/08 4:35:27 PM
Std 5		Standard	MEL-3	70200.	4220.	4000.	106.	2.01	2007/05/08 4:41:02 PM
Std 6		Standard	MEL-3	83300.	5020.	5000.	100.	2.01	2007/05/08 4:46:36 PM

Sample Name	Sample ID	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Calculated Concentration (ppb)	Analyte Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Acquisition Date
Rinse		Solvent	APT-1	0.00	N/A	0.00	N/A	0.00	2007/05/08 1:14:48 PM
Blank		Blank	APT-1	0.00	N/A	0.00	N/A	0.00	2007/05/08 1:20:22 PM
Std 1		Standard	APT-1	74100.	106.	100.	106.	2.37	2007/05/08 1:25:56 PM
Std 2		Standard	APT-1	131000.	189.	200.	94.3	2.37	2007/05/08 1:31:31 PM
Std 3		Standard	APT-1	201000.	289.	300.	96.4	2.36	2007/05/08 1:37:05 PM
Std 4		Standard	APT-1	322000.	464.	500.	92.9	2.37	2007/05/08 1:42:39 PM
Std 5		Standard	APT-1	556000.	804.	800.	101.	2.36	2007/05/08 1:48:14 PM
Std 6		Standard	APT-1	792000.	1150.	1000.	115.	2.36	2007/05/08 1:53:49 PM
Spk 1		Quality Control	APT-1	70500.	100.	100.	100.	2.37	2007/05/08 1:59:24 PM

Spk 1A		Quality Control	APT-1	56200.	79.6	100.	79.6	2.37	2007/05/08 2:44:02 PM
S15700	A743492	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 2:49:36 PM
S15720	A743496	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 2:55:11 PM
S15721	A743498	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:00:45 PM
S15728	A743500	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:06:19 PM
S15729	A743501	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:11:53 PM
S15730	A743502	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:17:27 PM
S15966	A743553	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:23:01 PM
S15967	A743553	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:28:35 PM
S15647	A743468	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:34:10 PM
S15648	A743469	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:39:44 PM

Sample Name	Sample ID	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Calculated Concentration (ppb)	Analyte Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Acquisition Date
28 S15673	A743472	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:45:19 PM
29 S15676	A743475	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:50:53 PM
30 S15677	A743476	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 3:56:27 PM
31 S15680	A743480	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 4:02:02 PM
32 S15698	A743489	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 4:07:36 PM
33 S15699	A743490	Unknown	APT-1	0.00	No Peak	N/A	N/A	0.00	2007/05/08 4:13:10 PM
34 Std 1		Standard	APT-1	74400.	106.	100.	106.	2.37	2007/05/08 4:18:44 PM
35 Std 2		Standard	APT-1	126000.	181.	200.	90.7	2.38	2007/05/08 4:24:18 PM
36 Std 3		Standard	APT-1	190000.	273.	300.	91.0	2.37	2007/05/08 4:29:52 PM
37 Std 4		Standard	APT-1	318000.	459.	500.	91.9	2.37	2007/05/08 4:35:27 PM
38 Std 5		Standard	APT-1	552000.	799.	800.	99.9	2.37	2007/05/08 4:41:02 PM
39 Std 6		Standard	APT-1	802000.	1160.	1000.	116.	2.37	2007/05/08 4:46:36 PM

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:00:22 PM
2 Blk 1	Blank	MEL-1	1300.	0.00	N/A	N/A		☐	1.00	13.9	1.74	2007/04/10 3:09:05 PM
3 Blk 2	Blank	MEL-1	723.	0.00	N/A	N/A		☐	1.00	9.10	1.71	2007/04/10 3:14:39 PM
4 Blk 3	Blank	MEL-1	1310.	0.00	N/A	N/A		☐	1.00	21.9	1.71	2007/04/10 3:20:14 PM
5 Std 1	Standard	MEL-1	48000.	500.	535.	107.	☒	☐	1.00	649.	1.72	2007/04/10 3:25:49 PM
6 Std 2	Standard	MEL-1	87500.	1000.	1030.	103.	☒	☐	1.00	1030.	1.72	2007/04/10 3:31:24 PM
7 Std 3	Standard	MEL-1	135000.	1500.	1620.	108.	☒	☐	1.00	915.	1.72	2007/04/10 3:36:59 PM
8 Std 4	Standard	MEL-1	211000.	2500.	2570.	103.	☒	☐	1.00	1650.	1.72	2007/04/10 3:42:34 PM
9 Std 5	Standard	MEL-1	308000.	4000.	3780.	94.5	☒	☐	1.00	2030.	1.73	2007/04/10 3:48:09 PM
10 Std 6	Standard	MEL-1	419000.	5000.	5170.	103.	☒	☐	1.00	3090.	1.73	2007/04/10 3:53:44 PM
11 LOD 1	Quality Control	MEL-1	42200.	500.	462.	92.5	☒	☐	1.00	244.	1.72	2007/04/10 3:59:19 PM
12 LOD 2	Quality Control	MEL-1	46400.	500.	515.	103.	☒	☐	1.00	458.	1.71	2007/04/10 4:04:54 PM
13 LOD 3	Quality Control	MEL-1	45200.	500.	500.	100.	☒	☐	1.00	283.	1.71	2007/04/10 4:10:29 PM
14 LOD 4	Quality Control	MEL-1	42600.	500.	468.	93.6	☒	☐	1.00	298.	1.72	2007/04/10 4:16:04 PM
15 LOD 5	Quality Control	MEL-1	42700.	500.	469.	93.8	☒	☐	1.00	429.	1.72	2007/04/10 4:21:39 PM
16 LOD 6	Quality Control	MEL-1	40900.	500.	446.	89.1	☒	☐	1.00	271.	1.72	2007/04/10 4:27:14 PM
17 LOD 7	Quality Control	MEL-1	43000.	500.	473.	94.5	☒	☐	1.00	335.	1.72	2007/04/10 4:32:49 PM
18 LOD 8	Quality Control	MEL-1	42800.	500.	470.	94.1	☒	☐	1.00	320.	1.72	2007/04/10 4:38:24 PM
19 Spk 2	Quality Control	MEL-1	181000.	2500.	2200.	88.0	☒	☐	1.00	2740.	1.72	2007/04/10 4:43:59 PM
20 Spk 2A	Quality Control	MEL-1	192000.	2500.	2330.	93.3	☒	☐	1.00	1400.	1.72	2007/04/10 4:49:34 PM
21 Spk 2B	Quality Control	MEL-1	201000.	2500.	2440.	97.8	☒	☐	1.00	1150.	1.72	2007/04/10 4:55:09 PM
22 Spk 3	Quality Control	MEL-1	282000.	4000.	3460.	86.5	☒	☐	1.00	3070.	1.72	2007/04/10 5:00:44 PM
23 Spk 3A	Quality Control	MEL-1	306000.	4000.	3760.	94.0	☒	☐	1.00	1960.	1.72	2007/04/10 5:06:19 PM
24 Spk 3B	Quality Control	MEL-1	305000.	4000.	3740.	93.5	☒	☐	1.00	1680.	1.72	2007/04/10 5:11:54 PM
25 Rinse	Solvent	MEL-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 5:17:29 PM
26 R71141	Unknown	MEL-1	10600000.	N/A	133000.	N/A		☐	1.00	68300.	1.72	2007/04/10 5:23:04 PM
27 Std 1	Standard	MEL-1	41300.	500.	452.	90.4	☒	☐	1.00	50.6	1.73	2007/04/10 5:28:39 PM
28 Std 3	Standard	MEL-1	121000.	1500.	1440.	96.3	☒	☐	1.00	269.	1.73	2007/04/10 5:34:14 PM
29 Std 6	Standard	MEL-1	383000.	5000.	4730.	94.5	☒	☐	1.00	1360.	1.72	2007/04/10 5:39:49 PM
30 R71141 (dil 1:500)	Unknown	MEL-1	160000.	N/A	986000.	N/A		☐	500.	318.	1.76	2007/04/10 5:45:24 PM
31 R71141 (dil 1:500) dup	Unknown	MEL-1	174000.	N/A	1060000.	N/A		☐	500.	549.	1.77	2007/04/10 5:50:59 PM

Note Sample ID is R 70141 2007/04/10/11 08
CE 2007/04/11 11 48

■ Untitled 2 (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): y = 80.1 x + 5.18e+003 (r = 0.9958)

Results Name: AptMel-Val wet_070410.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-Val wet_070410.rdb

Printing Time: 10:58:11 AM

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-Val wet_070410.rdb

Results Name: AptMel-Val wet_070410.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-Val wet_070410.rdb

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:00:22 PM
2 Blk 1	Blank	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:09:05 PM
3 Blk 2	Blank	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:14:39 PM
4 Blk 3	Blank	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:20:14 PM
5 Std 1	Standard	MEL-3	3780.	500.	553.	111.	☒	☐	1.00	77.3	1.73	2007/04/10 3:25:49 PM
6 Std 2	Standard	MEL-3	6380.	1000.	995.	99.5	☒	☐	1.00	282.	1.71	2007/04/10 3:31:24 PM
7 Std 3	Standard	MEL-3	10100.	1500.	1630.	109.	☒	☐	1.00	222.	1.72	2007/04/10 3:36:59 PM
8 Std 4	Standard	MEL-3	15300.	2500.	2520.	101.	☒	☐	1.00	397.	1.71	2007/04/10 3:42:34 PM
9 Std 5	Standard	MEL-3	22300.	4000.	3720.	92.9	☒	☐	1.00	517.	1.72	2007/04/10 3:48:09 PM
10 Std 6	Standard	MEL-3	31200.	5000.	5230.	105.	☒	☐	1.00	578.	1.72	2007/04/10 3:53:44 PM
11 LOD 1	Quality Control	MEL-3	2950.	500.	412.	82.4	☒	☐	1.00	52.4	1.72	2007/04/10 3:59:19 PM
12 LOD 2	Quality Control	MEL-3	3630.	500.	527.	105.	☒	☐	1.00	72.2	1.70	2007/04/10 4:04:54 PM
13 LOD 3	Quality Control	MEL-3	3090.	500.	434.	86.9	☒	☐	1.00	66.4	1.71	2007/04/10 4:10:29 PM
14 LOD 4	Quality Control	MEL-3	3740.	500.	546.	109.	☒	☐	1.00	99.5	1.71	2007/04/10 4:16:04 PM
15 LOD 5	Quality Control	MEL-3	3270.	500.	465.	93.1	☒	☐	1.00	39.6	1.71	2007/04/10 4:21:39 PM
16 LOD 6	Quality Control	MEL-3	3210.	500.	456.	91.2	☒	☐	1.00	70.8	1.72	2007/04/10 4:27:14 PM
17 LOD 7	Quality Control	MEL-3	3240.	500.	461.	92.1	☒	☐	1.00	76.5	1.72	2007/04/10 4:32:49 PM
18 LOD 8	Quality Control	MEL-3	3070.	500.	432.	86.5	☒	☐	1.00	68.3	1.71	2007/04/10 4:38:24 PM
19 Spk 2	Quality Control	MEL-3	13700.	2500.	2240.	89.5	☒	☐	1.00	519.	1.71	2007/04/10 4:43:59 PM
20 Spk 2A	Quality Control	MEL-3	14400.	2500.	2350.	94.2	☒	☐	1.00	364.	1.71	2007/04/10 4:49:34 PM
21 Spk 2B	Quality Control	MEL-3	15800.	2500.	2600.	104.	☒	☐	1.00	428.	1.72	2007/04/10 4:55:09 PM
22 Spk 3	Quality Control	MEL-3	19800.	4000.	3290.	82.3	☒	☐	1.00	433.	1.72	2007/04/10 5:00:44 PM
23 Spk 3A	Quality Control	MEL-3	23100.	4000.	3840.	96.0	☒	☐	1.00	438.	1.71	2007/04/10 5:06:19 PM
24 Spk 3B	Quality Control	MEL-3	22100.	4000.	3670.	91.7	☒	☐	1.00	655.	1.72	2007/04/10 5:11:54 PM
25 Rinse	Solvent	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 5:17:29 PM
26 R71141	Unknown	MEL-3	799000.	N/A	136000.	N/A		☐	1.00	12900.	1.72	2007/04/10 5:23:04 PM
27 Std 1	Standard	MEL-3	3110.	500.	438.	87.6	☒	☐	1.00	28.7	1.72	2007/04/10 5:28:39 PM
28 Std 3	Standard	MEL-3	9300.	1500.	1490.	99.6	☒	☐	1.00	108.	1.72	2007/04/10 5:34:14 PM
29 Std 6	Standard	MEL-3	28600.	5000.	4780.	95.7	☒	☐	1.00	362.	1.71	2007/04/10 5:39:49 PM
30 R71141 (dil 1:500)	Unknown	MEL-3	11800.	N/A	963000.	N/A		☐	500.	225.	1.76	2007/04/10 5:45:24 PM
31 R71141 (dil 1:500) dup	Unknown	MEL-3	13100.	N/A	1070000.	N/A		☐	500.	180.	1.70	2007/04/10 5:50:59 PM

Note Sample ID is R77+ 70141 2007/04/11 ~~DR~~
CE 2007/04/11 ~~DR~~

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-Val wet_070410.rdb

Printing Date: Wednesday, April 11, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	APT-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:00:22 PM
2 Blk 1	Blank	APT-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:09:05 PM
3 Blk 2	Blank	APT-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:14:39 PM
4 Blk 3	Blank	APT-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:20:14 PM
5 Std 1	Standard	APT-1	140000	100	108	108	☒	☐	1.00	3860	2.21	2007/04/10 3:25:49 PM
6 Std 2	Standard	APT-1	218000	200	182	91.2	☒	☐	1.00	3380	2.21	2007/04/10 3:31:24 PM
7 Std 3	Standard	APT-1	308000	300	268	89.2	☒	☐	1.00	5390	2.22	2007/04/10 3:36:59 PM
8 Std 4	Standard	APT-1	550000	500	498	99.7	☒	☐	1.00	8650	2.21	2007/04/10 3:42:34 PM
9 Std 5	Standard	APT-1	890000	800	821	103	☒	☐	1.00	11700	2.21	2007/04/10 3:48:09 PM
10 Std 6	Standard	APT-1	1220000	1000	1140	114	☒	☐	1.00	17800	2.22	2007/04/10 3:53:44 PM
11 LOD 1	Quality Control	APT-1	92600	100	63.0	63.0	☒	☐	1.00	1390	2.21	2007/04/10 3:59:19 PM
12 LOD 2	Quality Control	APT-1	138000	100	106	106	☒	☐	1.00	3600	2.21	2007/04/10 4:04:54 PM
13 LOD 3	Quality Control	APT-1	104000	100	74.0	74.0	☒	☐	1.00	1640	2.21	2007/04/10 4:10:29 PM
14 LOD 4	Quality Control	APT-1	120000	100	88.7	88.7	☒	☐	1.00	2780	2.21	2007/04/10 4:16:04 PM
15 LOD 5	Quality Control	APT-1	132000	100	101	101	☒	☐	1.00	1630	2.21	2007/04/10 4:21:39 PM
16 LOD 6	Quality Control	APT-1	128000	100	96.9	96.9	☒	☐	1.00	2420	2.22	2007/04/10 4:27:14 PM
17 LOD 7	Quality Control	APT-1	132000	100	101	101	☒	☐	1.00	2720	2.21	2007/04/10 4:32:49 PM
18 LOD 8	Quality Control	APT-1	123000	100	91.9	91.9	☒	☐	1.00	2740	2.22	2007/04/10 4:38:24 PM
19 Spk 2	Quality Control	APT-1	602000	500	547	109	☒	☐	1.00	12500	2.21	2007/04/10 4:43:59 PM
20 Spk 2A	Quality Control	APT-1	662000	500	604	121	☒	☐	1.00	7570	2.21	2007/04/10 4:49:34 PM
21 Spk 2B	Quality Control	APT-1	521000	500	471	94.1	☒	☐	1.00	4200	2.21	2007/04/10 4:55:09 PM
22 Spk 3	Quality Control	APT-1	1040000	800	960	120	☒	☐	1.00	15100	2.21	2007/04/10 5:00:44 PM
23 Spk 3A	Quality Control	APT-1	1030000	800	954	119	☒	☐	1.00	9520	2.21	2007/04/10 5:06:19 PM
24 Spk 3B	Quality Control	APT-1	1050000	800	977	122	☒	☐	1.00	10400	2.21	2007/04/10 5:11:54 PM
25 Rinse	Solvent	APT-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 5:17:29 PM
26 R71141	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/10 5:23:04 PM
27 Std 1	Standard	APT-1	134000	100	103	103	☒	☐	1.00	2360	2.21	2007/04/10 5:28:39 PM
28 Std 3	Standard	APT-1	291000	300	252	83.8	☒	☐	1.00	9460	2.22	2007/04/10 5:34:14 PM
29 Std 6	Standard	APT-1	1170000	1000	1090	109	☒	☐	1.00	25600	2.20	2007/04/10 5:39:49 PM
30 R71141 (dil 1:500)	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	500	N/A	0.00	2007/04/10 5:45:24 PM
31 R71141 (dil 1:500) dup	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	500	N/A	0.00	2007/04/10 5:50:59 PM

note Sample ID is R70141
2007/04/11/08A

Results Name: AptMel-Val wet_070410.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-Val wet_070410.rdb

Printing Time: 11:05:34 AM

Printing Date: Wednesday, April 11, 2007

■ AptMel-Val wet_070410.rdb (APT-1): "Linear" Regression ("1 / (x * x)" weighting): y = 1.05e+003 x + 2.64e+004 (r = 0.9891)

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-Vol wet_070410.rdb

Printing Date: Wednesday, April 11, 2006

Results Name: AptMel-Vol wet_070410.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-Vol wet_070410.rdb

Printing Date: Wednesday, April 11, 2006

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-Val wet_070410.rdb

Printing Date: Wednesday, April 11, 2008

Results Name: AptMel-Val wet_070410.rdb

Printing Time: 11:06:20 AM

Results Path: R:\Projects\Residue\Aminopterin\Results\AptMel-Val wet_070410.rdb

Printing Date: Wednesday, April 11, 2008

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	APT-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:00:22 PM
2 Blk 1	Blank	APT-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:09:05 PM
3 Blk 2	Blank	APT-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:14:39 PM
4 Blk 3	Blank	APT-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 3:20:14 PM
5 Std 1	Standard	APT-3	43500.	100.	104.	104.	☒	☐	1.00	976.	2.21	2007/04/10 3:25:49 PM
6 Std 2	Standard	APT-3	70500.	200.	183.	91.3	☒	☐	1.00	1050.	2.21	2007/04/10 3:31:24 PM
7 Std 3	Standard	APT-3	100000.	300.	269.	89.7	☒	☐	1.00	2640.	2.21	2007/04/10 3:36:59 PM
8 Std 4	Standard	APT-3	181000.	500.	504.	101.	☒	☐	1.00	3130.	2.21	2007/04/10 3:42:34 PM
9 Std 5	Standard	APT-3	292000.	800.	826.	103.	☒	☐	1.00	4280.	2.21	2007/04/10 3:48:09 PM
10 Std 6	Standard	APT-3	394000.	1000.	1120.	112.	☒	☐	1.00	7210.	2.21	2007/04/10 3:53:44 PM
11 LOD 1	Quality Control	APT-3	30200.	100.	65.5	65.5	☒	☐	1.00	333.	2.20	2007/04/10 3:59:19 PM
12 LOD 2	Quality Control	APT-3	42400.	100.	101.	101.	☒	☐	1.00	855.	2.20	2007/04/10 4:04:54 PM
13 LOD 3	Quality Control	APT-3	29500.	100.	63.5	63.5	☒	☐	1.00	446.	2.20	2007/04/10 4:10:29 PM
14 LOD 4	Quality Control	APT-3	39200.	100.	91.7	91.7	☒	☐	1.00	881.	2.21	2007/04/10 4:16:04 PM
15 LOD 5	Quality Control	APT-3	42100.	100.	100.	100.	☒	☐	1.00	836.	2.21	2007/04/10 4:21:39 PM
16 LOD 6	Quality Control	APT-3	42500.	100.	101.	101.	☒	☐	1.00	748.	2.22	2007/04/10 4:27:14 PM
17 LOD 7	Quality Control	APT-3	40100.	100.	94.2	94.2	☒	☐	1.00	620.	2.20	2007/04/10 4:32:49 PM
18 LOD 8	Quality Control	APT-3	41100.	100.	97.1	97.1	☒	☐	1.00	1030.	2.21	2007/04/10 4:38:24 PM
19 Spk 2	Quality Control	APT-3	197000.	500.	549.	110.	☒	☐	1.00	2890.	2.21	2007/04/10 4:43:59 PM
20 Spk 2A	Quality Control	APT-3	206000.	500.	574.	115.	☒	☐	1.00	4820.	2.21	2007/04/10 4:49:34 PM
21 Spk 2B	Quality Control	APT-3	173000.	500.	481.	96.2	☒	☐	1.00	3150.	2.21	2007/04/10 4:55:09 PM
22 Spk 3	Quality Control	APT-3	334000.	800.	947.	118.	☒	☐	1.00	5420.	2.20	2007/04/10 5:00:44 PM
23 Spk 3A	Quality Control	APT-3	335000.	800.	950.	119.	☒	☐	1.00	3430.	2.21	2007/04/10 5:06:19 PM
24 Spk 3B	Quality Control	APT-3	349000.	800.	989.	124.	☒	☐	1.00	4420.	2.21	2007/04/10 5:11:54 PM
25 Rinse	Solvent	APT-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/10 5:17:29 PM
26 R71141	Unknown	APT-3	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/10 5:23:04 PM
27 Std 1	Standard	APT-3	44400.	100.	107.	107.	☒	☐	1.00	917.	2.21	2007/04/10 5:28:39 PM
28 Std 3	Standard	APT-3	92900.	300.	248.	82.5	☒	☐	1.00	1450.	2.21	2007/04/10 5:34:14 PM
29 Std 6	Standard	APT-3	386000.	1000.	1100.	110.	☒	☐	1.00	9040.	2.20	2007/04/10 5:39:49 PM
30 R71141 (dil 1:500)	Unknown	APT-3	0.00	N/A	No Peak	N/A		☐	500.	N/A	0.00	2007/04/10 5:45:24 PM
31 R71141 (dil 1:500) dup	Unknown	APT-3	0.00	N/A	No Peak	N/A		☐	500.	N/A	0.00	2007/04/10 5:50:59 PM

Note: Sample ID is R70141
2007/04/11/08

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Blk	Blank	MEL-1	1150.	0.00	N/A	N/A		☐	1.00	17.6	1.79	2007/04/11 01:43:17 PM
2 Std 1	Standard	MEL-1	35800.	500.	458.	91.5	☒	☐	1.00	505.	1.78	2007/04/11 01:49:04 PM
3 Std 2	Standard	MEL-1	63700.	1000.	908.	90.8	☒	☐	1.00	1370.	1.77	2007/04/11 01:54:51 PM
4 Std 3	Standard	MEL-1	89400.	1500.	1320.	88.2	☒	☐	1.00	1190.	1.77	2007/04/11 02:00:38 PM
5 Std 4	Standard	MEL-1	149000.	2500.	2290.	91.5	☒	☐	1.00	1220.	1.77	2007/04/11 02:06:25 PM
6 Std 5	Standard	MEL-1	255000.	4000.	4000.	100.	☒	☐	1.00	2950.	1.77	2007/04/11 02:12:12 PM
7 Std 6	Standard	MEL-1	332000.	5000.	5250.	105.	☒	☐	1.00	3150.	1.77	2007/04/11 02:17:59 PM
8 R71483A (1-5)	Unknown	MEL-1	5720.	N/A	< 0	N/A		☐	1.00	45.2	1.79	2007/04/11 02:23:46 PM
9 R71483B (6-10)	Unknown	MEL-1	6070.	N/A	< 0	N/A		☐	1.00	53.6	1.79	2007/04/11 02:29:33 PM
10 R71483A (1:500)	Unknown	MEL-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 02:35:20 PM
11 R71483B (1:500)	Unknown	MEL-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 02:41:07 PM
12 Rinse	Solvent	MEL-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/11 01:28:44 PM
13 DQC (dil 1:500)	Quality Control	MEL-1	239000.	400000.	1870000.	469.	☒	☐	500.	1910.	1.82	2007/04/11 02:46:54 PM
14 DQC (dil 1:500)	Quality Control	MEL-1	252000.	400000.	1980000.	495.	☒	☐	500.	2310.	1.83	2007/04/11 02:52:41 PM
15 R73435	Unknown	MEL-1	2150.	N/A	< 0	N/A		☐	1.00	19.8	1.80	2007/04/11 02:58:28 PM
16 R73435 (1:500)	Unknown	MEL-1	300.	N/A	< 0	N/A		☐	1.00	2.56	1.91	2007/04/11 03:04:15 PM
17 R73442	Unknown	MEL-1	98900.	N/A	1480.	N/A		☐	1.00	932.	1.79	2007/04/11 03:10:02 PM
18 R73442 (1:500)	Unknown	MEL-1	1290.	N/A	< 0	N/A		☐	1.00	14.9	1.83	2007/04/11 03:15:49 PM
19 Std 1	Standard	MEL-1	43000.	500.	574.	115.	☒	☐	1.00	965.	1.78	2007/04/11 03:21:36 PM
20 Std 3	Standard	MEL-1	107000.	1500.	1610.	107.	☒	☐	1.00	1380.	1.77	2007/04/11 03:27:23 PM
21 Std 6	Standard	MEL-1	351000.	5000.	5550.	111.	☒	☐	1.00	3820.	1.78	2007/04/11 03:33:10 PM

* R71727 CE 2007/04/11/08

AptMel-wet food_070411.rdb (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): y = 61.9 x + 7.54e+003 (r = 0.9895)

Method Name: APT-MEL_070409.qmf

Page 1 of 1

Results Name: AptMel-wet food_070411.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070411.rdb

Printing Time: 04:18:16 PM

Printing Date: Wednesday, April 11, 2006

Method Name: APT-MEL_070409.qmf

Page 1 of 3

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070411.rdb

Printing Date: Wednesday, April 11, 2000

Method Name: APT-MEL_070409.qmf

Page 2 of 3

Results Name: AptMel-wet food_070411.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070411.rdb

Printing Time: 04:18:16 PM

Printing Date: Wednesday, April 11, 2000

Method Name: APT-MEL_070409.qmf

Page 3 of 3

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070411.rdb

Printing Date: Wednesday, April 11, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Blk	Blank	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/11 01:43:17 PM
2 Std 1	Standard	MEL-3	2970.	500.	471.	94.1	☒	☐	1.00	148.	1.77	2007/04/11 01:49:04 PM
3 Std 2	Standard	MEL-3	4840.	1000.	884.	88.4	☒	☐	1.00	154.	1.76	2007/04/11 01:54:51 PM
4 Std 3	Standard	MEL-3	6450.	1500.	1240.	82.8	☒	☐	1.00	261.	1.77	2007/04/11 02:00:38 PM
5 Std 4	Standard	MEL-3	11700.	2500.	2410.	96.4	☒	☐	1.00	468.	1.76	2007/04/11 02:06:25 PM
6 Std 5	Standard	MEL-3	19000.	4000.	4020.	101.	☒	☐	1.00	484.	1.77	2007/04/11 02:12:12 PM
7 Std 6	Standard	MEL-3	24400.	5000.	5210.	104.	☒	☐	1.00	721.	1.77	2007/04/11 02:17:59 PM
8 R71483A (1-5)	Unknown	MEL-3	548.	N/A	< 0	N/A		☐	1.00	14.8	1.78	2007/04/11 02:23:46 PM
9 R71483B (6-10)	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 02:29:33 PM
10 R71483A (1:500)	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 02:35:20 PM
11 R71483B (1:500)	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 02:41:07 PM
12 Rinse	Solvent	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/11 01:28:44 PM
13 DQC (dil 1:500)	Quality Control	MEL-3	17500.	400000.	1850000.	461.	☒	☐	500.	645.	1.82	2007/04/11 02:46:54 PM
14 DQC (dil 1:500)	Quality Control	MEL-3	19000.	400000.	2010000.	501.	☒	☐	500.	573.	1.82	2007/04/11 02:52:41 PM
15 R73435	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 02:58:28 PM
16 R73435 (1:500)	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 03:04:15 PM
17 R73442	Unknown	MEL-3	7310.	N/A	1430.	N/A		☐	1.00	318.	1.79	2007/04/11 03:10:02 PM
18 R73442 (1:500)	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 03:15:49 PM
19 Std 1	Standard	MEL-3	3450.	500.	577.	115.	☒	☐	1.00	160.	1.77	2007/04/11 03:21:36 PM
20 Std 3	Standard	MEL-3	7730.	1500.	1530.	102.	☒	☐	1.00	269.	1.77	2007/04/11 03:27:23 PM
21 Std 6	Standard	MEL-3	27100.	5000.	5820.	116.	☒	☐	1.00	933.	1.77	2007/04/11 03:33:10 PM

Method Name: APT-MEL_070409.qmf

Page 1 of 1

Results Name: AptMel-wet food_070411.rdb

Printing Time: 04:19:43

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070411.rdb

Printing Date: Wednesday, April 11, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Blk	Blank	APT-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/11 01:43:17 PM
2 Std 1	Standard	APT-1	112000.	100.	96.9	96.9	☒	☐	1.00	4980.	2.27	2007/04/11 01:49:04 PM
3 Std 2	Standard	APT-1	190000.	200.	184.	92.0	☒	☐	1.00	4370.	2.27	2007/04/11 01:54:51 PM
4 Std 3	Standard	APT-1	284000.	300.	289.	96.3	☒	☐	1.00	4740.	2.27	2007/04/11 02:00:38 PM
5 Std 4	Standard	APT-1	463000.	500.	488.	97.7	☒	☐	1.00	8530.	2.27	2007/04/11 02:06:25 PM
6 Std 5	Standard	APT-1	797000.	800.	860.	107.	☒	☐	1.00	14200.	2.27	2007/04/11 02:12:12 PM
7 Std 6	Standard	APT-1	912000.	1000.	987.	98.7	☒	☐	1.00	11400.	2.27	2007/04/11 02:17:59 PM
8 R71483A (1-5)	Unknown	APT-1	431.	N/A	< 0	N/A		☐	1.00	10.1	2.23	2007/04/11 02:23:46 PM
9 R71483B (6-10)	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 02:29:33 PM
10 R71483A (1:500)	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 02:35:20 PM
11 R71483B (1:500)	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 02:41:07 PM
12 Rinse	Solvent	APT-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/11 01:28:44 PM
13 DQC (dil 1:500)	Quality Control	APT-1	573000.	80000.	305000.	382.	☒	☐	500.	19800.	2.26	2007/04/11 02:46:54 PM
14 DQC (dil 1:500)	Quality Control	APT-1	619000.	80000.	331000.	413.	☒	☐	500.	21700.	2.27	2007/04/11 02:52:41 PM
15 R73435	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 02:58:28 PM
16 R73435 (1:500)	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 03:04:15 PM
17 R73442	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 03:10:02 PM
18 R73442 (1:500)	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/11 03:15:49 PM
19 Std 1	Standard	APT-1	122000.	100.	108.	106.	☒	☐	1.00	7090.	2.28	2007/04/11 03:21:36 PM
20 Std 3	Standard	APT-1	290000.	300.	295.	98.4	☒	☐	1.00	5840.	2.27	2007/04/11 03:27:23 PM
21 Std 6	Standard	APT-1	963000.	1000.	1040.	104.	☒	☐	1.00	9490.	2.27	2007/04/11 03:33:10 PM

Method Name: APT-MEL_070409.qmf

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Sample Name	Sample Type	Analyte Peak Name	Analyte Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-1	0.00	0.00	N/A	N/A			1.00	N/A	0.00	2007/04/18 01:18:05 PM
2 Blk	Blank	MEL-1	2910.	0.00	N/A	N/A			1.00	33.3	1.75	2007/04/18 01:24:14 PM
3 Std 1	Standard	MEL-1	67100.	500.	486.	97.3			1.00	427.	1.75	2007/04/18 01:29:49 PM
4 Std 2	Standard	MEL-1	169000.	1000.	1040.	104.			1.00	1520.	1.75	2007/04/18 01:35:24 PM
5 Std 3	Standard	MEL-1	237000.	1500.	1410.	94.0			1.00	2590.	1.75	2007/04/18 01:40:58 PM
6 Std 4	Standard	MEL-1	445000.	2500.	2540.	102.			1.00	3110.	1.75	2007/04/18 01:45:33 PM
7 Std 5	Standard	MEL-1	742000.	4000.	4160.	104.			1.00	7040.	1.75	2007/04/18 01:52:08 PM
8 Std 6	Standard	MEL-1	881000.	5000.	4920.	98.3			1.00	5710.	1.75	2007/04/18 01:57:42 PM
9 Spk 1	Quality Control	MEL-1	94400.	500.	695.	127.			1.00	865.	1.75	2007/04/18 02:03:17 PM
10 R85281	Unknown	MEL-1	3900000.	N/A	21300.	N/A			1.00	14900.	1.75	2007/04/18 02:08:51 PM
11 R84603	Unknown	MEL-1	9630.	N/A	174.	N/A			1.00	16.4	1.77	2007/04/18 02:14:26 PM
12 R84606	Unknown	MEL-1	12600.	N/A	190.	N/A			1.00	101.	1.77	2007/04/18 02:20:01 PM
13 Spk 1A	Quality Control	MEL-1	86700.	500.	593.	119.			1.00	426.	1.76	2007/04/18 02:25:36 PM
14 Std 1	Standard	MEL-1	72000.	500.	513.	103.			1.00	406.	1.74	2007/04/18 02:31:11 PM
15 Std 3	Standard	MEL-1	249000.	1500.	1470.	98.3			1.00	1360.	1.75	2007/04/18 02:36:48 PM
16 Std 6	Standard	MEL-1	895000.	5000.	4990.	99.8			1.00	4420.	1.75	2007/04/18 02:42:23 PM
17 Std 6 (dil 1:50)	Quality Control	MEL-1	133000.	5000.	848.	17.0			1.00	779.	1.79	2007/04/18 03:06:07 PM
18 R85281 (dil 1:50)	Unknown	MEL-1	721000.	N/A	4040.	N/A			1.00	5650.	1.79	2007/04/18 03:14:15 PM

1:50 dilution = 848 x 50 = 42400 ppb

% accuracy = $\frac{42400}{5000} \times 100 = 848\%$

Note
R 85281
is R 85281
% enhancement = dilution = $\frac{50}{848/100} = 5.90$
% acc of dilution

∴ result is 5.90x higher than it should be
actual concn in sample as = 4040 ppb x 5.90

= 23800 ppb
2007/04/19/2007

■ AptMEL-wet food_070418.rdb (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): y = 184 x + -2.24e+004 (r = 0.9988)

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070418.rdb

Printing Date: Thursday, April 19, 200

Method Name: APT-MEL_070409.qmf

Results Name: AptMel-wet Food_070418.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070418.rdb

Printing Time: 10:08:20 A

Printing Date: Thursday, April 19, 20

Method Name: APT-MEL_070409.qmf

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070418.rdb

Printing Date: Thursday, April 19, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/18 01:18:05 PM
2 Blk	Blank	MEL-3	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/18 01:24:14 PM
3 Std 1	Standard	MEL-3	4620	500	484	96.8	☒	☐	1.00	106	1.74	2007/04/18 01:29:49 PM
4 Std 2	Standard	MEL-3	11500	1000	1020	102	☒	☐	1.00	527	1.74	2007/04/18 01:35:24 PM
5 Std 3	Standard	MEL-3	16600	1500	1410	94.1	☒	☐	1.00	260	1.74	2007/04/18 01:40:58 PM
6 Std 4	Standard	MEL-3	31500	2500	2570	103	☒	☐	1.00	835	1.74	2007/04/18 01:46:33 PM
7 Std 5	Standard	MEL-3	51800	4000	4150	104	☒	☐	1.00	1390	1.74	2007/04/18 01:52:08 PM
8 Std 6	Standard	MEL-3	61600	5000	4910	98.2	☒	☐	1.00	1000	1.74	2007/04/18 01:57:42 PM
9 Spk 1	Quality Control	MEL-3	6300	500	615	123	☒	☐	1.00	144	1.74	2007/04/18 02:03:17 PM
10 R85261	Unknown	MEL-3	273000	N/A	21300	N/A		☐	1.00	4620	1.75	2007/04/18 02:08:51 PM
11 R84603	Unknown	MEL-3	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/18 02:14:26 PM
12 R84606	Unknown	MEL-3	718	500	614	123	☒	☐	1.00	16.4	1.76	2007/04/18 02:20:01 PM
13 Spk 1A	Quality Control	MEL-3	6290	500	520	104	☒	☐	1.00	149	1.75	2007/04/18 02:25:36 PM
14 Std 1	Standard	MEL-3	5080	500	520	104	☒	☐	1.00	110	1.75	2007/04/18 02:31:11 PM
15 Std 3	Standard	MEL-3	17300	1500	1470	98.0	☒	☐	1.00	362	1.75	2007/04/18 02:36:48 PM
16 Std 6	Standard	MEL-3	62900	5000	5010	100	☒	☐	1.00	921	1.74	2007/04/18 02:42:23 PM
17 Std 6 (dil 1:50)	Quality Control	MEL-3	9460	0.00	860	N/A	☒	☐	1.00	389	1.79	2007/04/18 03:06:07 PM
18 R85261 (dil 1:50)	Unknown	MEL-3	51300	N/A	4110	N/A		☐	1.00	1500	1.79	2007/04/18 03:14:15 PM

Method Name: APT-MEL_070409.qmf

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Results Name: AptMel-wet food_070418.rdb

Printing Time: 10:09:21

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070418.rdb

Printing Date: Thursday, April 19, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Acquisition Date
1 Rinse	Solvent	APT-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/18 01:16:05 PM
2 Blk	Blank	APT-1	0.00	0.00	N/A	N/A		☐	1.00	N/A	0.00	2007/04/18 01:24:14 PM
3 Std 1	Standard	APT-1	118000	100	103	103	☒	☐	1.00	2270	2.20	2007/04/18 01:29:49 PM
4 Std 2	Standard	APT-1	207000	200	180	90.1	☒	☐	1.00	3810	2.20	2007/04/18 01:35:24 PM
5 Std 3	Standard	APT-1	356000	300	310	103	☒	☐	1.00	6180	2.19	2007/04/18 01:40:58 PM
6 Std 4	Standard	APT-1	625000	500	545	109	☒	☐	1.00	8660	2.20	2007/04/18 01:46:33 PM
7 Std 5	Standard	APT-1	766000	800	668	83.5	☒	☐	1.00	10000	2.19	2007/04/18 01:52:08 PM
8 Std 6	Standard	APT-1	1220000	1000	1060	106	☒	☐	1.00	16500	2.19	2007/04/18 01:57:42 PM
9 Spk 1	Quality Control	APT-1	135000	100	118	118	☒	☐	1.00	1380	2.20	2007/04/18 02:03:17 PM
10 R85261	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/18 02:08:51 PM
11 R84603	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/18 02:14:26 PM
12 R84606	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/18 02:20:01 PM
13 Spk 1A	Quality Control	APT-1	110000	100	95.7	95.7	☒	☐	1.00	1440	2.20	2007/04/18 02:25:36 PM
14 Std 1	Standard	APT-1	114000	100	99.9	99.9	☒	☐	1.00	2100	2.19	2007/04/18 02:31:11 PM
15 Std 3	Standard	APT-1	345000	300	301	100	☒	☐	1.00	4220	2.19	2007/04/18 02:36:48 PM
16 Std 6	Standard	APT-1	1200000	1000	1050	105	☒	☐	1.00	19500	2.19	2007/04/18 02:42:23 PM
17 Std 6 (dil 1:50)	Quality Control	APT-1	96100	0.00	83.9	N/A	☒	☐	1.00	3200	2.20	2007/04/18 03:06:07 PM
18 R85261 (dil 1:50)	Unknown	APT-1	0.00	N/A	No Peak	N/A		☐	1.00	N/A	0.00	2007/04/18 03:14:15 PM

Method Name: APT-MEL_070409.qmf

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Sample Name	Sample ID	Use Record	Sample Type	Analyte Peak Name	Analyte Mass Ranges (amu)	Analyte Peak Area (count)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Record Modified	File Name	Acquisition Method	Acquisition Date
1 Rinse			Solvent	MEL-1	126.8/65.2 amu	289	0.00	N/A	N/A	1.74		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:18:16 PM
2 Rinse			Solvent	MEL-2	126.8/68.0 amu	371	0.00	N/A	N/A	1.73		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:18:16 PM
3 Rinse			Solvent	MEL-3	126.8/68.0 amu	0	0.00	N/A	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:16:16 PM
4 Blank			Blank	MEL-1	126.8/65.2 amu	3780	0.00	N/A	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:22:10 PM
5 Blank			Blank	MEL-2	126.8/68.0 amu	328	0.00	N/A	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:22:10 PM
6 Blank			Blank	MEL-3	126.8/68.0 amu	0.00	0.00	N/A	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:22:10 PM
7 Std 1			Standard	MEL-1	126.8/65.2 amu	106000	500	500	100	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:27:45 PM
8 Std 1			Standard	MEL-2	126.8/68.0 amu	32400	500	499	99.9	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:27:45 PM
9 Std 2			Standard	MEL-3	126.8/65.2 amu	7290	500	504	101	1.76		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:27:45 PM
10 Std 2			Standard	MEL-1	126.8/65.2 amu	204000	1000	1040	104	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:33:19 PM
11 Std 2			Standard	MEL-2	126.8/68.0 amu	63300	1000	1060	106	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:33:19 PM
12 Std 2			Standard	MEL-3	126.8/65.2 amu	13800	1000	998	99.8	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:33:19 PM
13 Std 3			Standard	MEL-1	126.8/65.2 amu	282000	1500	1470	98.0	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:38:55 PM
14 Std 3			Standard	MEL-2	126.8/68.0 amu	89300	1500	1460	97.4	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:38:55 PM
15 Std 3			Standard	MEL-3	126.8/68.0 amu	19400	1500	1440	96.0	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:38:55 PM
16 Std 4			Standard	MEL-1	126.8/65.2 amu	505000	2500	2700	108	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:44:29 PM
17 Std 4			Standard	MEL-2	126.8/68.0 amu	167000	2500	2880	112	1.76		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:44:29 PM
18 Std 4			Standard	MEL-3	126.8/65.2 amu	35500	2500	2680	107	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:44:29 PM
19 Std 5			Standard	MEL-1	126.8/65.2 amu	756000	4000	4090	102	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:50:03 PM
20 Std 5			Standard	MEL-2	126.8/68.0 amu	248000	4000	4180	105	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:50:03 PM
21 Std 5			Standard	MEL-3	126.8/65.2 amu	53500	4000	4070	102	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:50:03 PM
22 Std 6			Standard	MEL-1	126.8/65.2 amu	890000	5000	4820	96.5	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:55:39 PM
23 Std 6			Standard	MEL-2	126.8/68.0 amu	281000	5000	4750	94.9	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:55:39 PM
24 Std 6			Standard	MEL-3	126.8/65.2 amu	84300	5000	4910	98.2	1.76		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:55:39 PM
25 Rinse			Solvent	MEL-1	126.8/65.2 amu	552	0.00	N/A	N/A	1.87		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:01:14 PM
26 Rinse			Solvent	MEL-2	126.8/68.0 amu	287	0.00	N/A	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:01:14 PM
27 Rinse			Solvent	MEL-3	126.8/68.0 amu	0.00	0.00	N/A	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:01:14 PM
28 R77715 A734413			Unknown	MEL-1	126.8/65.2 amu	107000	N/A	< 0	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:06:48 PM
29 R77715 A734413			Unknown	MEL-2	126.8/68.0 amu	2970	N/A	< 0	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:06:48 PM
30 R77715 A734413			Unknown	MEL-3	126.8/65.2 amu	575	N/A	< 0	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:06:48 PM
31 R77716 A734413			Unknown	MEL-1	126.8/65.2 amu	2480	N/A	< 0	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:12:23 PM
32 R77716 A734413			Unknown	MEL-2	126.8/68.0 amu	508	N/A	< 0	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:12:23 PM
33 R77716 A734413			Unknown	MEL-3	126.8/65.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:12:23 PM
34 R77717 A734413			Unknown	MEL-1	126.8/65.2 amu	5840	N/A	< 0	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:17:58 PM
35 R77717 A734413			Unknown	MEL-2	126.8/68.0 amu	1510	N/A	< 0	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:17:58 PM
36 R77722 A734415			Unknown	MEL-1	126.8/65.2 amu	343000	N/A	1810	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:23:33 PM
37 R77722 A734415			Unknown	MEL-2	126.8/68.0 amu	107000	N/A	1770	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:23:33 PM
38 R77736 A734418			Unknown	MEL-1	126.8/65.2 amu	24000	N/A	1790	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:23:33 PM
39 R77736 A734418			Unknown	MEL-2	126.8/68.0 amu	341000	N/A	1800	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:23:33 PM
40 R77736 A734418			Unknown	MEL-3	126.8/65.2 amu	108000	N/A	1790	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:23:33 PM
41 R77736 A734418			Unknown	MEL-1	126.8/65.2 amu	24000	N/A	< 0	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:23:33 PM
42 R77736 A734418			Unknown	MEL-2	126.8/68.0 amu	945	N/A	< 0	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:23:33 PM
43 R78150 A734548			Unknown	MEL-1	126.8/65.2 amu	282	N/A	< 0	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:34:43 PM
44 R78150 A734548			Unknown	MEL-2	126.8/68.0 amu	282	N/A	< 0	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:34:43 PM
45 R78150 A734548			Unknown	MEL-3	126.8/65.2 amu	391000	N/A	2070	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:34:43 PM
46 R78150 A734548			Unknown	MEL-1	126.8/65.2 amu	129000	N/A	2140	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:40:18 PM
47 R78150 A734548			Unknown	MEL-2	126.8/68.0 amu	279000	N/A	2100	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:40:18 PM
48 R78150 A734548			Unknown	MEL-3	126.8/65.2 amu	1490	N/A	< 0	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:40:18 PM
49 R78152 A734548			Unknown	MEL-1	126.8/65.2 amu	307	N/A	< 0	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:45:52 PM
50 R78152 A734548			Unknown	MEL-2	126.8/68.0 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:45:52 PM
51 R78152 A734548			Unknown	MEL-3	126.8/65.2 amu	252000	N/A	1300	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:45:52 PM
52 R78216 A734580			Unknown	MEL-1	126.8/65.2 amu	77400	N/A	1270	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:51:27 PM
53 R78216 A734580			Unknown	MEL-2	126.8/68.0 amu	18500	N/A	1370	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:51:27 PM
54 R78216 A734580			Unknown	MEL-3	126.8/65.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:51:27 PM

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Printing Time: 08:16:08

Printing Date: Monday, April 16, 2006

Sample Name	Sample ID	Use Record	Sample Type	Analyte Peak Name	Analyte Mass Ranges (amu)	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Record Modified	File Name	Acquisition Method	Acquisition Date
55 R78248 A734588			Unknown	MEL-1	126.8/65.2 amu	1310	N/A	< 0	N/A	1.75		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
56 R78248 A734588			Unknown	MEL-2	126.8/68.0 amu	455	N/A	< 0	N/A	1.55		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
57 R78248 A734588			Unknown	MEL-3	126.8/68.0 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
58 R78155 A734551			Unknown	MEL-1	126.8/65.2 amu	3460	N/A	< 0	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
59 R78155 A734551			Unknown	MEL-2	126.8/68.0 amu	383	N/A	< 0	N/A	1.81		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
60 R78155 A734551			Unknown	MEL-3	126.8/68.0 amu	271	N/A	< 0	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
61 R78186 A734564			Unknown	MEL-1	126.8/65.2 amu	282000	N/A	1470	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
62 R78186 A734564			Unknown	MEL-2	126.8/68.0 amu	89900	N/A	1480	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
63 R78186 A734564			Unknown	MEL-3	126.8/68.0 amu	20100	N/A	1490	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
64 R78179 A734559			Unknown	MEL-1	126.8/65.2 amu	504000	N/A	2690	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
65 R78179 A734559			Unknown	MEL-2	126.8/68.0 amu	16000	N/A	2780	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
66 R78179 A734559			Unknown	MEL-3	126.8/68.0 amu	35900	N/A	2710	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
67 R78233 A734582			Unknown	MEL-1	126.8/65.2 amu	248000	N/A	1280	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
68 R78233 A734582			Unknown	MEL-2	126.8/68.0 amu	78200	N/A	1280	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
69 R78233 A734582			Unknown	MEL-3	126.8/68.0 amu	17600	N/A	1300	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
70 R78247 A734587			Unknown	MEL-1	126.8/65.2 amu	217000	N/A	1110	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
71 R78247 A734587			Unknown	MEL-2	126.8/68.0 amu	69300	N/A	1130	N/A	1.76		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
72 R78247 A734587			Unknown	MEL-3	126.8/68.0 amu	19100	N/A	1110	N/A	1.76		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
73 Spk 1			Quality Control	MEL-1	126.8/65.2 amu	102000	500	477	95.5	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
74 Spk 1			Quality Control	MEL-2	126.8/68.0 amu	31800	500	488	97.8	1.76		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
75 Spk 1			Quality Control	MEL-3	126.8/68.0 amu	7720	500	537	107	1.76		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
76 R78158 A734554			Unknown	MEL-1	126.8/65.2 amu	400000	N/A	2400	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
77 R78158 A734554			Unknown	MEL-2	126.8/68.0 amu	139000	N/A	2320	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
78 R78158 A734554			Unknown	MEL-3	126.8/68.0 amu	31100	N/A	2240	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
79 R78138 A734536			Unknown	MEL-1	126.8/65.2 amu	1110	N/A	< 0	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
80 R78138 A734536			Unknown	MEL-2	126.8/68.0 amu	300	N/A	< 0	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
81 R78138 A734536			Unknown	MEL-3	126.8/68.0 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
82 R78193 A734566			Unknown	MEL-1	126.8/65.2 amu	255000	N/A	1360	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
83 R78193 A734566			Unknown	MEL-2	126.8/68.0 amu	84300	N/A	1390	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
84 R78193 A734566			Unknown	MEL-3	126.8/68.0 amu	19100	N/A	1410	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
85 R78268 A734595			Unknown	MEL-1	126.8/65.2 amu	1750	N/A	< 0	N/A	1.76		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
86 R78268 A734595			Unknown	MEL-2	126.8/68.0 amu	517	N/A	< 0	N/A	1.56		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
87 R78268 A734595			Unknown	MEL-3	126.8/68.0 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
88 R78215 A734579			Unknown	MEL-1	126.8/65.2 amu	277000	N/A	1440	N/A	1.76		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
89 R78215 A734579			Unknown	MEL-2	126.8/68.0 amu	87000	N/A	1430	N/A	1.76		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
90 R78215 A734579			Unknown	MEL-3	126.8/68.0 amu	20100	N/A	1500	N/A	1.76		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
91 R78133 A734534			Unknown	MEL-1	126.8/65.2 amu	504000	N/A	2690	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
92 R78133 A734534			Unknown	MEL-2	126.8/68.0 amu	154000	N/A	2570	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
93 R78133 A734534			Unknown	MEL-3	126.8/68.0 amu	34000	N/A	2570	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
94 R78214 A734578			Unknown	MEL-1	126.8/65.2 amu	208000	N/A	1060	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
95 R78214 A734578			Unknown	MEL-2	126.8/68.0 amu	67100	N/A	1090	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
96 R78214 A734578			Unknown	MEL-3	126.8/68.0 amu	14600	N/A	1070	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
97 R78180 A734559			Unknown	MEL-1	126.8/65.2 amu	482000	N/A	2580	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
98 R78180 A734559			Unknown	MEL-2	126.8/68.0 amu	161000	N/A	2700	N/A	1.79		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
99 R78180 A734559			Unknown	MEL-3	126.8/68.0 amu	34200	N/A	2590	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
100 R78139 A734537			Unknown	MEL-1	126.8/65.2 amu	1620	N/A	< 0	N/A	1.80		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
101 R78139 A734537			Unknown	MEL-2	126.8/68.0 amu	236	N/A	< 0	N/A	1.82		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
102 R78139 A734537			Unknown	MEL-3	126.8/68.0 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
103 R78131 A734532			Unknown	MEL-1	126.8/65.2 amu	191000	N/A	969	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
104 R78131 A734532			Unknown	MEL-2	126.8/68.0 amu	56800	N/A	916	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
105 R78131 A734532			Unknown	MEL-3	126.8/68.0 amu	13300	N/A	970	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
106 R78146 A734542			Unknown	MEL-1	126.8/65.2 amu	1840	N/A	< 0	N/A	1.77		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
107 R78146 A734542			Unknown	MEL-2	126.8/68.0 amu	311	N/A	< 0	N/A	1.78		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
108 R78146 A734542			Unknown	MEL-3	126.8/68.0 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM

Sample Name	Sample ID	Use Record	Sample Type	Analyte Peak Name	Analyte Mass Ranges (amu)	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Record Modified	File Name	Acquisition Method	Acquisition Date
109 Spk 1A		☒	Quality Control	MEL-1	126.8/65.2 amu	928000	500	426	95.2	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:37:32 PM
110 Spk 1A		☒	Quality Control	MEL-2	126.8/68.0 amu	304000	500	466	93.1	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:37:32 PM
111 Spk 1A		☒	Quality Control	MEL-3	126.8/68.0 amu	69200	500	475	95.0	1.76	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:37:32 PM
112 R78106	A734523	☒	Unknown	MEL-1	126.8/65.2 amu	176000	N/A	12.3	N/A	1.80	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:43:07 PM
113 R78106	A734523	☒	Unknown	MEL-2	126.8/68.0 amu	51500	N/A	35.1	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:43:07 PM
114 R78106	A734523	☒	Unknown	MEL-3	126.8/68.0 amu	13700	N/A	46.8	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:43:07 PM
115 R78144	A734540	☒	Unknown	MEL-1	126.8/68.0 amu	11200	N/A	< 0	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:48:42 PM
116 R78144	A734540	☒	Unknown	MEL-2	126.8/68.0 amu	500	N/A	< 0	N/A	2.01	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:48:42 PM
117 R78144	A734540	☒	Unknown	MEL-3	126.8/68.0 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:48:42 PM
118 R78235	A734584	☒	Unknown	MEL-1	126.8/65.2 amu	283000	N/A	1510	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:54:17 PM
119 R78235	A734584	☒	Unknown	MEL-2	126.8/68.0 amu	88600	N/A	1460	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:54:17 PM
120 R78235	A734584	☒	Unknown	MEL-3	126.8/68.0 amu	201000	N/A	1480	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:54:17 PM
121 R78125	A734529	☒	Unknown	MEL-1	126.8/65.2 amu	428000	N/A	2260	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:59:52 PM
122 R78125	A734529	☒	Unknown	MEL-2	126.8/68.0 amu	307000	N/A	2310	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:59:52 PM
123 R78125	A734529	☒	Unknown	MEL-3	126.8/68.0 amu	248000	N/A	1280	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:05:27 PM
124 R78183	A734551	☒	Unknown	MEL-1	126.8/65.2 amu	768000	N/A	1260	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:05:27 PM
125 R78183	A734551	☒	Unknown	MEL-2	126.8/68.0 amu	175000	N/A	13000	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:05:27 PM
126 R78183	A734551	☒	Unknown	MEL-3	126.8/68.0 amu	417	N/A	< 0	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:11:02 PM
127 R78156	A734552	☒	Unknown	MEL-1	126.8/68.0 amu	284000	N/A	< 0	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:11:02 PM
128 R78156	A734552	☒	Unknown	MEL-2	126.8/68.0 amu	417	N/A	< 0	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:11:02 PM
129 R78156	A734552	☒	Unknown	MEL-3	126.8/68.0 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:11:02 PM
130 R78208	A734572	☒	Unknown	MEL-1	126.8/65.2 amu	428000	N/A	2270	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:16:37 PM
131 R78208	A734572	☒	Unknown	MEL-2	126.8/68.0 amu	1390000	N/A	2310	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:16:37 PM
132 R78208	A734572	☒	Unknown	MEL-3	126.8/68.0 amu	297000	N/A	2190	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:16:37 PM
133 R78234	A734583	☒	Unknown	MEL-1	126.8/65.2 amu	397000	N/A	2100	N/A	1.80	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:22:12 PM
134 R78234	A734583	☒	Unknown	MEL-2	126.8/68.0 amu	1330000	N/A	2210	N/A	1.80	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:22:12 PM
135 R78234	A734583	☒	Unknown	MEL-3	126.8/68.0 amu	284000	N/A	2130	N/A	1.80	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:22:12 PM
136 R78267	A734596	☒	Unknown	MEL-1	126.8/65.2 amu	363000	N/A	1920	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:27:47 PM
137 R78267	A734596	☒	Unknown	MEL-2	126.8/68.0 amu	1180000	N/A	1960	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:27:47 PM
138 R78267	A734596	☒	Unknown	MEL-3	126.8/68.0 amu	259000	N/A	1940	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:27:47 PM
139 R78246	A734586	☒	Unknown	MEL-1	126.8/65.2 amu	297000	N/A	1610	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:33:22 PM
140 R78246	A734586	☒	Unknown	MEL-2	126.8/68.0 amu	973000	N/A	1610	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:33:22 PM
141 R78246	A734586	☒	Unknown	MEL-3	126.8/68.0 amu	218000	N/A	1620	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:33:22 PM
142 R78207	A734571	☒	Unknown	MEL-1	126.8/65.2 amu	627000	N/A	3370	N/A	1.80	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:38:57 PM
143 R78207	A734571	☒	Unknown	MEL-2	126.8/68.0 amu	2010000	N/A	3370	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:38:57 PM
144 R78207	A734571	☒	Unknown	MEL-3	126.8/68.0 amu	446000	N/A	3380	N/A	1.79	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:38:57 PM
145 R78213	A734577	☒	Unknown	MEL-1	126.8/65.2 amu	4290000	N/A	2280	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:44:32 PM
146 R78213	A734577	☒	Unknown	MEL-2	126.8/68.0 amu	300000	N/A	2280	N/A	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:44:32 PM
147 R78213	A734577	☒	Unknown	MEL-3	126.8/68.0 amu	1040000	N/A	488	N/A	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:50:07 PM
148 Std 1		☒	Standard	MEL-1	126.8/65.2 amu	31500	500	485	97.0	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:50:07 PM
149 Std 1		☒	Standard	MEL-2	126.8/68.0 amu	6970	500	480	95.9	1.78	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:50:07 PM
150 Std 1		☒	Standard	MEL-3	126.8/68.0 amu	2000000	1000	1020	102	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:55:42 PM
151 Std 2		☒	Standard	MEL-1	126.8/65.2 amu	628000	1000	1010	101	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:55:42 PM
152 Std 2		☒	Standard	MEL-2	126.8/68.0 amu	150000	1000	1100	110	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:55:42 PM
153 Std 2		☒	Standard	MEL-3	126.8/68.0 amu	2740000	1500	1430	95.0	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:01:16 AM
154 Std 3		☒	Standard	MEL-1	126.8/65.2 amu	869000	1500	1430	95.2	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:01:16 AM
155 Std 3		☒	Standard	MEL-2	126.8/68.0 amu	196000	1500	1450	97.0	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:01:16 AM
156 Std 3		☒	Standard	MEL-3	126.8/68.0 amu	4920000	2500	2690	105	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:06:51 AM
157 Std 4		☒	Standard	MEL-1	126.8/65.2 amu	1530000	2500	2590	103	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:06:51 AM
158 Std 4		☒	Standard	MEL-2	126.8/68.0 amu	345000	2500	2610	104	1.76	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:06:51 AM
159 Std 4		☒	Standard	MEL-3	126.8/68.0 amu	732000	4000	3950	98.8	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:12:26 AM
160 Std 5		☒	Standard	MEL-1	126.8/65.2 amu	2340000	4000	3940	98.5	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:12:26 AM
161 Std 5		☒	Standard	MEL-2	126.8/68.0 amu	523000	4000	3980	98.5	1.76	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:12:26 AM
162 Std 5		☒	Standard	MEL-3	126.8/68.0 amu	523000	4000	3980	98.5	1.76	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:12:26 AM

Method Name: APT-MEL_070409.qmf

Sample Name	Sample ID	Use Record	Sample Type	Analyte Peak Name	Analyte Mass Ranges (amu)	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Record Modified	File Name	Acquisition Method	Acquisition Date
163 Std 6		☒	Standard	MEL-1	126.8/65.2 amu	857000	5000	4640	92.9	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/14 12:18:01 AM
164 Std 6		☒	Standard	MEL-2	126.8/68.0 amu	2690000	5000	4530	90.6	1.77	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/14 12:18:01 AM
165 Std 6		☒	Standard	MEL-3	126.8/68.0 amu	594000	5000	4530	90.6	1.76	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/14 12:18:01 AM

Method Name: APT-MEL_070409.qmf

■ AptMel-wet food_070413.rdb (MEL-1): "Linear" Regression ("1 / (x * x)" weighting): $y = 181x + 1.57e+004$ ($r = 0.9978$) ■ AptMel-wet food_070413.rdb (MEL-2): "Linear" Regression ("1 / (x * x)" weighting): $y = 58.7x + 3.09e+003$ ($r = 0.9962$)

■ AptMel-wet food_070413.rdb (MEL-3): "Linear" Regression ("1 / (x * x)" weighting): y = 12.9 x + 766 (r = 0.9969)

Method Name: APT-MEL_070409.qm2

Page 1 of 1

Results Name: AptMel-wet food_070413.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070413.rdb

Printing Time: 08:17:04 AM

Printing Date: Monday, April 16, 2006

Method Name: APT-MEL_070409.qm2

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Results Path: D:\Analysis Data\Projects\Residue\Amrop\amr\results\Apixelwet food_00413.rdb

Method Name: APT-MEL_070409.qtd

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Results Name: APMEI-wel food_070413.rdb

Results Path: C:\Analysis\Data\Projects\Residue\Aminoprop\In\results\Apmel-wet food_070413.rdb

Comment: " Annotation: " pbb
 Sample Index: 9
 Sample Type: Solvent
 Concentration: 0.00
 Calculated Conc: N/A
 Acq. Date: 2007/04/13
 Acq. Time: 09:01:14 PM
 Modified: NO

Nation Name: APF-NEL 076409.qm

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Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070413.rdb

Method Name: APT-MEL_070409.qmf

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Results Name: AptMel-wet food_070413.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070413.rdb

Method Name: APT-MEL_070409.qmf

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Method Name: APT-MEL_070402.qmz

Method Name: APT-MEL_070409.qm1

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070413.rdb

Method Name: APT-MEL_070409.qmf

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Results Name: AptMel-wet food_070413.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070413.rdb

Method Name: APT-MEL_070409.qmf

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Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070413.rdb

Method Name: APT-MEL_070409.qm

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Results Name: AptMel-wet food_070413.rdb

Printing Time: 08:17:04

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070413.rdb

Method Name: APT-MEL_070409.qm

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Sample Name	Sample ID	Use Record	Sample Type	Analyte Peak Name	Analyte Mass Ranges (amu)	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Record Modified	File Name	Acquisition Method	Acquisition Date
1	Rise		Solvent	APT-1	441.3/294.1 amu	0.00	0.00	N/A	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:18:16 PM
2	Rise		Solvent	APT-2	441.3/120.2 amu	0.00	0.00	N/A	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:18:16 PM
3	Rise		Solvent	APT-3	441.3/175.2 amu	0.00	0.00	N/A	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:18:16 PM
4	Blank		Blank	APT-1	441.3/294.1 amu	510	0.00	N/A	N/A	2.15		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:22:10 PM
5	Blank		Blank	APT-2	441.3/120.2 amu	441	0.00	N/A	N/A	2.14		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:22:10 PM
6	Blank		Blank	APT-3	441.3/175.2 amu	0.00	0.00	N/A	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:22:10 PM
7	Sid 1		Standard	APT-1	441.3/294.1 amu	226000	100	107	107	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:27:45 PM
8	Sid 1		Standard	APT-2	441.3/120.2 amu	60200	100	103	103	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:27:45 PM
9	Sid 1		Standard	APT-3	441.3/175.2 amu	32800	100	106	106	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:27:45 PM
10	Sid 2		Standard	APT-1	441.3/294.1 amu	431000	200	204	102	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:33:19 PM
11	Sid 2		Standard	APT-2	441.3/120.2 amu	122000	200	211	106	2.18		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:33:19 PM
12	Sid 2		Standard	APT-3	441.3/175.2 amu	160000	200	202	101	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:33:19 PM
13	Sid 3		Standard	APT-1	441.3/294.1 amu	618000	300	293	97.4	2.18		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:38:55 PM
14	Sid 3		Standard	APT-2	441.3/120.2 amu	168000	300	293	97.5	2.18		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:38:55 PM
15	Sid 3		Standard	APT-3	441.3/175.2 amu	236000	300	297	98.1	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:38:55 PM
16	Sid 4		Standard	APT-1	441.3/294.1 amu	1120000	500	534	107	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:44:29 PM
17	Sid 4		Standard	APT-2	441.3/120.2 amu	300000	500	523	105	2.18		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:44:29 PM
18	Sid 4		Standard	APT-3	441.3/175.2 amu	419000	500	526	103	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:44:29 PM
19	Sid 5		Standard	APT-1	441.3/294.1 amu	1730000	800	826	105	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:50:03 PM
20	Sid 5		Standard	APT-2	441.3/120.2 amu	468000	800	819	102	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:50:03 PM
21	Sid 5		Standard	APT-3	441.3/175.2 amu	655000	800	819	102	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:50:03 PM
22	Sid 6		Standard	APT-1	441.3/294.1 amu	1610000	1000	768	76.8	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:55:39 PM
23	Sid 6		Standard	APT-2	441.3/120.2 amu	428000	1000	748	74.8	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 07:55:39 PM
24	Sid 6		Standard	APT-3	441.3/175.2 amu	592000	1000	741	74.1	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:01:14 PM
25	Rise		Solvent	APT-1	441.3/294.1 amu	1040	0.00	N/A	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:01:14 PM
26	Rise		Solvent	APT-2	441.3/120.2 amu	290	0.00	N/A	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:01:14 PM
27	Rise		Solvent	APT-3	441.3/175.2 amu	290	0.00	N/A	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:01:14 PM
28	R7715		Unknown	APT-1	441.3/294.1 amu	1310	N/A	< 0	N/A	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:06:48 PM
29	R7715		Unknown	APT-2	441.3/120.2 amu	904	N/A	< 0	N/A	2.14		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:06:48 PM
30	R7715		Unknown	APT-3	441.3/175.2 amu	459	N/A	3.70	N/A	2.17		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:06:48 PM
31	R7716		Unknown	APT-1	441.3/294.1 amu	841	N/A	< 0	N/A	2.18		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:12:23 PM
32	R7716		Unknown	APT-2	441.3/120.2 amu	845	N/A	< 0	N/A	2.16		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:12:23 PM
33	R7716		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:12:23 PM
34	R7717		Unknown	APT-1	441.3/294.1 amu	4750	N/A	0.931	N/A	2.17		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:17:59 PM
35	R7717		Unknown	APT-2	441.3/120.2 amu	1240	N/A	< 0	N/A	2.14		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:17:59 PM
36	R7717		Unknown	APT-3	441.3/175.2 amu	511	N/A	3.75	N/A	2.16		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:17:59 PM
37	R7722		Unknown	APT-1	441.3/294.1 amu	0.00	N/A	< 0	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:23:33 PM
38	R7722		Unknown	APT-2	441.3/120.2 amu	452	N/A	< 0	N/A	2.14		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:23:33 PM
39	R7722		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:23:33 PM
40	R7736		Unknown	APT-1	441.3/294.1 amu	372	N/A	< 0	N/A	2.17		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:29:08 PM
41	R7736		Unknown	APT-2	441.3/120.2 amu	171	N/A	< 0	N/A	2.14		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:29:08 PM
42	R7736		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:29:08 PM
43	R78150		Unknown	APT-1	441.3/294.1 amu	261	N/A	< 0	N/A	2.10		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:34:43 PM
44	R78150		Unknown	APT-2	441.3/120.2 amu	382	N/A	< 0	N/A	2.10		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:34:43 PM
45	R78150		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:34:43 PM
46	R78184		Unknown	APT-1	441.3/294.1 amu	121	N/A	< 0	N/A	2.37		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:40:18 PM
47	R78184		Unknown	APT-2	441.3/120.2 amu	446	N/A	< 0	N/A	2.13		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:40:18 PM
48	R78184		Unknown	APT-3	441.3/175.2 amu	720	N/A	4.03	N/A	2.11		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:40:18 PM
49	R78152		Unknown	APT-1	441.3/294.1 amu	281	N/A	< 0	N/A	2.39		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:45:52 PM
50	R78152		Unknown	APT-2	441.3/120.2 amu	656	N/A	< 0	N/A	2.14		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:45:52 PM
51	R78152		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:45:52 PM
52	R78216		Unknown	APT-1	441.3/294.1 amu	225	N/A	< 0	N/A	2.12		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:51:27 PM
53	R78216		Unknown	APT-2	441.3/120.2 amu	587	N/A	< 0	N/A	2.18		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:51:27 PM
54	R78216		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:51:27 PM

Method Name: APT-MEL_070409.qmf

Printing Date: Monday, April 16, 2024

Sample Name	Sample ID	Use Record	Sample Type	Analyte Peak Name	Analyte Mass Ranges (amu)	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Record Modified	File Name	Acquisition Method	Acquisition Date
55	R78248		Unknown	APT-1	441.3/294.1 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
56	R78248		Unknown	APT-2	441.3/120.2 amu	196	N/A	< 0	N/A	2.22		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
57	R78248		Unknown	APT-3	441.3/175.2 amu	268	N/A	3.46	N/A	2.18		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
58	R78155		Unknown	APT-1	441.3/294.1 amu	6740	N/A	1.88	N/A	2.18		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
59	R78155		Unknown	APT-2	441.3/120.2 amu	1490	N/A	0.406	N/A	2.16		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
60	R78155		Unknown	APT-3	441.3/175.2 amu	1260	N/A	4.70	N/A	2.20		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
61	R78186		Unknown	APT-1	441.3/294.1 amu	282	N/A	< 0	N/A	2.02		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
62	R78186		Unknown	APT-2	441.3/120.2 amu	859	N/A	< 0	N/A	2.13		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
63	R78186		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
64	R78179		Unknown	APT-1	441.3/294.1 amu	0.00	N/A	< 0	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
65	R78179		Unknown	APT-2	441.3/120.2 amu	900	N/A	< 0	N/A	2.17		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
66	R78179		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
67	R78233		Unknown	APT-1	441.3/294.1 amu	0.00	N/A	< 0	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
68	R78233		Unknown	APT-2	441.3/120.2 amu	182	N/A	< 0	N/A	2.13		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
69	R78233		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
70	R78247		Unknown	APT-1	441.3/294.1 amu	350	N/A	< 0	N/A	2.03		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
71	R78247		Unknown	APT-2	441.3/120.2 amu	482	N/A	< 0	N/A	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
72	R78247		Unknown	APT-3	441.3/175.2 amu	182	N/A	3.38	N/A	2.14		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
73	Spk 1		Quality Control	APT-1	441.3/294.1 amu	233000	100	110	110	2.19		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
74	Spk 1		Quality Control	APT-2	441.3/120.2 amu	64700	100	111	111	2.18		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
75	Spk 1		Quality Control	APT-3	441.3/175.2 amu	87500	100	112	112	2.18		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
76	R78158		Unknown	APT-1	441.3/294.1 amu	196	N/A	< 0	N/A	2.15		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
77	R78158		Unknown	APT-2	441.3/120.2 amu	444	N/A	< 0	N/A	2.09		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
78	R78158		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
79	R78136		Unknown	APT-1	441.3/294.1 amu	586	N/A	< 0	N/A	2.34		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
80	R78136		Unknown	APT-2	441.3/120.2 amu	330	N/A	< 0	N/A	2.32		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
81	R78136		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
82	R78193		Unknown	APT-1	441.3/294.1 amu	0.00	N/A	< 0	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
83	R78193		Unknown	APT-2	441.3/120.2 amu	354	N/A	< 0	N/A	2.10		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
84	R78193		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
85	R78266		Unknown	APT-1	441.3/294.1 amu	0.00	N/A	< 0	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
86	R78266		Unknown	APT-2	441.3/120.2 amu	758	N/A	< 0	N/A	2.33		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
87	R78266		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
88	R78215		Unknown	APT-1	441.3/294.1 amu	344	N/A	< 0	N/A	2.10		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
89	R78215		Unknown	APT-2	441.3/120.2 amu	344	N/A	< 0	N/A	2.10		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
90	R78215		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
91	R78133		Unknown	APT-1	441.3/294.1 amu	883	N/A	< 0	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
92	R78133		Unknown	APT-2	441.3/120.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
93	R78133		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	< 0	N/A	2.15		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
94	R78214		Unknown	APT-1	441.3/294.1 amu	684	N/A	< 0	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
95	R78214		Unknown	APT-2	441.3/120.2 amu	684	N/A	< 0	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
96	R78214		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	2.21		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
97	R78180		Unknown	APT-1	441.3/294.1 amu	220	N/A	< 0	N/A	2.06		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
98	R78180		Unknown	APT-2	441.3/120.2 amu	729	N/A	< 0	N/A	2.14		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
99	R78180		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
100	R78139		Unknown	APT-1	441.3/294.1 amu	347	N/A	< 0	N/A	2.21		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
101	R78139		Unknown	APT-2	441.3/120.2 amu	234	N/A	< 0	N/A	2.16		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
102	R78139		Unknown	APT-3	441.3/175.2 amu	394	N/A	3.62	N/A	2.09		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
103	R78131		Unknown	APT-1	441.3/294.1 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
104	R78131		Unknown	APT-2	441.3/120.2 amu	481	N/A	< 0	N/A	2.13		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
105	R78131		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
106	R78146		Unknown	APT-1	441.3/294.1 amu	0.00	N/A	< 0	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
107	R78146		Unknown	APT-2	441.3/120.2 amu	589	N/A	< 0	N/A	2.11		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM
108	R78146		Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00		APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 08:57:03 PM

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070413.rdb

Printing Date: Monday, April 16, 2007

Sample Name	Sample ID	Use Record	Sample Type	Sample Peak Name	Analyte Mass Ranges (amu)	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Record Modified	File Name	Acquisition Method	Acquisition Date
109 Spk 1A		☒	Quality Control	APT-1	441.3/294.1 amu	235000	100	111	111	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:37:32 PM
110 Spk 1A		☒	Quality Control	APT-2	441.3/120.2 amu	62700	100	108	108	2.18	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:37:32 PM
111 Spk 1A		☒	Quality Control	APT-3	441.3/175.2 amu	87500	100	112	112	2.18	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:37:32 PM
112 R78106	A734523	☒	Unknown	APT-1	441.3/294.1 amu	291	N/A	< 0	N/A	2.09	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:43:07 PM
113 R78106	A734523	☒	Unknown	APT-2	441.3/120.2 amu	172	N/A	< 0	N/A	2.05	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:43:07 PM
114 R78106	A734523	☒	Unknown	APT-3	441.3/175.2 amu	411	N/A	3.64	N/A	2.32	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:43:07 PM
115 R78144	A734540	☒	Unknown	APT-1	441.3/294.1 amu	174	N/A	< 0	N/A	2.30	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:48:42 PM
116 R78144	A734540	☒	Unknown	APT-2	441.3/120.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:48:42 PM
117 R78144	A734540	☒	Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:48:42 PM
118 R78235	A734584	☒	Unknown	APT-1	441.3/294.1 amu	657	N/A	< 0	N/A	2.08	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:54:17 PM
119 R78235	A734584	☒	Unknown	APT-2	441.3/120.2 amu	932	N/A	< 0	N/A	2.16	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:54:17 PM
120 R78235	A734584	☒	Unknown	APT-3	441.3/175.2 amu	499	N/A	3.75	N/A	2.28	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:59:52 PM
121 R78125	A734529	☒	Unknown	APT-1	441.3/294.1 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:59:52 PM
122 R78125	A734529	☒	Unknown	APT-2	441.3/120.2 amu	950	N/A	< 0	N/A	2.13	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:59:52 PM
123 R78125	A734529	☒	Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 10:59:52 PM
124 R78183	A734581	☒	Unknown	APT-1	441.3/294.1 amu	247	N/A	< 0	N/A	2.11	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:05:27 PM
125 R78183	A734581	☒	Unknown	APT-2	441.3/120.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:05:27 PM
126 R78183	A734581	☒	Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:05:27 PM
127 R78156	A734552	☒	Unknown	APT-1	441.3/294.1 amu	178	N/A	< 0	N/A	1.98	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:11:02 PM
128 R78156	A734552	☒	Unknown	APT-2	441.3/120.2 amu	414	N/A	< 0	N/A	2.12	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:11:02 PM
129 R78207	A734571	☒	Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:11:02 PM
130 R78207	A734571	☒	Unknown	APT-1	441.3/294.1 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:16:37 PM
131 R78207	A734571	☒	Unknown	APT-2	441.3/120.2 amu	562	N/A	< 0	N/A	2.16	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:16:37 PM
132 R78207	A734571	☒	Unknown	APT-3	441.3/175.2 amu	116	N/A	3.31	N/A	2.09	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:22:12 PM
133 R78234	A734583	☒	Unknown	APT-1	441.3/294.1 amu	117	N/A	< 0	N/A	1.97	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:22:12 PM
134 R78234	A734583	☒	Unknown	APT-2	441.3/120.2 amu	321	N/A	< 0	N/A	2.10	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:22:12 PM
135 R78234	A734583	☒	Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:27:47 PM
136 R78267	A734596	☒	Unknown	APT-1	441.3/294.1 amu	1680	N/A	0.738	N/A	2.17	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:27:47 PM
137 R78267	A734596	☒	Unknown	APT-2	441.3/120.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:27:47 PM
138 R78246	A734586	☒	Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:33:22 PM
139 R78246	A734586	☒	Unknown	APT-1	441.3/294.1 amu	399	N/A	< 0	N/A	2.18	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:33:22 PM
140 R78246	A734586	☒	Unknown	APT-2	441.3/120.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:33:22 PM
141 R78207	A734571	☒	Unknown	APT-3	441.3/175.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:38:57 PM
142 R78207	A734571	☒	Unknown	APT-1	441.3/294.1 amu	298	N/A	< 0	N/A	2.11	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:38:57 PM
143 R78207	A734571	☒	Unknown	APT-2	441.3/120.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:38:57 PM
144 R78213	A734577	☒	Unknown	APT-3	441.3/175.2 amu	1110	N/A	< 0	N/A	2.11	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:44:32 PM
145 R78213	A734577	☒	Unknown	APT-1	441.3/294.1 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:44:32 PM
146 R78213	A734577	☒	Unknown	APT-2	441.3/120.2 amu	0.00	N/A	No Peak	N/A	0.00	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:50:07 PM
147 R78213	A734577	☒	Unknown	APT-3	441.3/175.2 amu	204000	100	96.0	96.0	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:50:07 PM
148 Std 1		☒	Standard	APT-1	441.3/294.1 amu	56600	100	97.0	97.0	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:50:07 PM
149 Std 1		☒	Standard	APT-2	441.3/120.2 amu	74400	100	95.9	95.9	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:50:07 PM
150 Std 1		☒	Standard	APT-3	441.3/175.2 amu	406000	200	192	192	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:55:42 PM
151 Std 2		☒	Standard	APT-1	441.3/294.1 amu	112000	200	194	194	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:55:42 PM
152 Std 2		☒	Standard	APT-2	441.3/120.2 amu	191000	200	191	191	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 11:55:42 PM
153 Std 2		☒	Standard	APT-3	441.3/175.2 amu	596000	300	278	278	2.20	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:01:16 AM
154 Std 3		☒	Standard	APT-1	441.3/294.1 amu	153000	300	284	284	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:01:16 AM
155 Std 3		☒	Standard	APT-2	441.3/120.2 amu	228000	300	287	287	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:01:16 AM
156 Std 3		☒	Standard	APT-3	441.3/175.2 amu	1050000	500	502	502	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:06:51 AM
157 Std 4		☒	Standard	APT-1	441.3/294.1 amu	287000	500	502	502	2.18	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:06:51 AM
158 Std 4		☒	Standard	APT-2	441.3/120.2 amu	403000	500	506	506	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:06:51 AM
159 Std 4		☒	Standard	APT-3	441.3/175.2 amu	1680000	800	790	790	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:12:26 AM
160 Std 5		☒	Standard	APT-1	441.3/294.1 amu	446000	800	760	760	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:12:26 AM
161 Std 5		☒	Standard	APT-2	441.3/120.2 amu	625000	800	763	763	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:12:26 AM
162 Std 5		☒	Standard	APT-3	441.3/175.2 amu	625000	800	763	763	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/13 12:12:26 AM

Method Name: APT-MEL_070409.qmf

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Results Name: AptMel-wet food_070413.rdb

Printing Time: 08:14:35

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070413.rdb

Printing Date: Monday, April 16, 2007

Sample Name	Sample ID	Use Record	Sample Type	Sample Peak Name	Analyte Mass Ranges (amu)	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Analyte Retention Time (min)	Record Modified	File Name	Acquisition Method	Acquisition Date
163 Std 6		☒	Standard	APT-1	441.3/294.1 amu	1480000	1000	707	70.7	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/14 12:18:01 AM
164 Std 6		☒	Standard	APT-2	441.3/120.2 amu	405000	1000	708	70.8	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/14 12:18:01 AM
165 Std 6		☒	Standard	APT-3	441.3/175.2 amu	575000	1000	720	72.0	2.19	☐	APT-MEL_wet_070413Wet Food.wif	AptMel-MRM_20070409.dam	2007/04/14 12:18:01 AM

Method Name: APT-MEL_070409.qmf

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■ AptMel-wet food_070413.rdb (APT-1): "Linear" Regression ("1 / (x * x)" weighting): $y = 2.1e+003 x + 2.8e+003$ ($r = 0.9971$)

Method Name: APT-MEL_070409.qmf

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■ AptMel-wet food_070413.rdb (APT-2): "Linear" Regression ("1 / (x * x)" weighting): $y = 570 x + 1.26e+003$ ($r = 0.9981$)

Method Name: APT-MEL_070409.qmf

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AptMel-wet food_070413.rdb (APT-3): "Linear" Regression ("1 / (x * x)" weighting): y = 802 x + -2.51e+003 (r = 0.9981)

Method Name: APT-MEL_070409.qmf

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Results Name: AptMel-wet food_070413.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070413.rdb

Printing Time: 08:15:31

Printing Date: Monday, April 16, 2006

Method Name: APT-MEL_070409.qmf

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Printing Date: Monday, April 15, 2007

67 30 8 2563

Printing Time: 08:15:31

Printing Date: Monday, April 16, 2012

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070413.rdb

Printing Date: Monday, April 16, 2006

Method Name: APT-MEL_070409.qmf

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Results Name: AptMel-wet food_070413.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\AptMel-wet food_070413.rdb

Printing Time: 08:15:31

Printing Date: Monday, April 16, 2006

Method Name: APT-MEL_070409.qmf

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Printing Date: Monday, April 16, 20

Printing Time: 08:15:31
Printing Date: Monday, April 16, 20

Results Path: D:\Analysis Data\Projects\Residue/Aminopterin/Results/ASpec1-wet food 070913.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Val_Wet_070502.rdb

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Rinse	Solvent	CYA-1	0.00	0.00	N/A	N/A			1.00	N/A	0.00	127.9/42.0 amu	2007/05/02 04:52:42 PM
2 Blk 1	Blank	CYA-1	0.00	0.00	N/A	N/A			1.00	N/A	0.00	127.9/42.0 amu	2007/05/02 05:11:40 PM
3 Blk 2	Blank	CYA-1	0.00	0.00	N/A	N/A			1.00	N/A	0.00	127.9/42.0 amu	2007/05/02 05:22:26 PM
4 Std 1	Standard	CYA-1	76900	500	503	101	☒		1.00	1200	1.91	127.9/42.0 amu	2007/05/02 05:33:12 PM
5 Std 2	Standard	CYA-1	132000	1000	927	92.7	☒		1.00	1990	1.92	127.9/42.0 amu	2007/05/02 05:43:58 PM
6 Std 3	Standard	CYA-1	195000	1500	1420	94.6	☒		1.00	1730	1.91	127.9/42.0 amu	2007/05/02 05:54:44 PM
7 Std 4	Standard	CYA-1	317000	2500	2360	94.4	☒		1.00	3470	1.91	127.9/42.0 amu	2007/05/02 06:05:31 PM
8 Std 5	Standard	CYA-1	328000	4000	4000	99.9	☒		1.00	8230	1.91	127.9/42.0 amu	2007/05/02 06:16:17 PM
9 Std 6	Standard	CYA-1	684000	5000	5210	104	☒		1.00	5630	1.91	127.9/42.0 amu	2007/05/02 06:27:03 PM
10 LOD 1	Quality Control	CYA-1	66400	500	421	84.2	☒		1.00	721	1.92	127.9/42.0 amu	2007/05/02 06:37:49 PM
11 LOD 2	Quality Control	CYA-1	68200	500	436	87.1	☒		1.00	997	1.92	127.9/42.0 amu	2007/05/02 06:48:35 PM
12 LOD 3	Quality Control	CYA-1	66200	500	420	84.0	☒		1.00	834	1.92	127.9/42.0 amu	2007/05/02 06:59:21 PM
13 LOD 4	Quality Control	CYA-1	66500	500	422	84.4	☒		1.00	961	1.92	127.9/42.0 amu	2007/05/02 07:10:07 PM
14 LOD 5	Quality Control	CYA-1	67000	500	426	85.3	☒		1.00	1190	1.92	127.9/42.0 amu	2007/05/02 07:20:54 PM
15 LOD 6	Quality Control	CYA-1	72500	500	469	93.8	☒		1.00	737	1.92	127.9/42.0 amu	2007/05/02 07:31:40 PM
16 LOD 7	Quality Control	CYA-1	67900	500	433	86.6	☒		1.00	1020	1.92	127.9/42.0 amu	2007/05/02 07:42:27 PM
17 LOD 8	Quality Control	CYA-1	73500	500	476	95.2	☒		1.00	1180	1.92	127.9/42.0 amu	2007/05/02 07:53:13 PM
18 Spk 2	Quality Control	CYA-1	194000	1500	1410	94.2	☒		1.00	2730	1.92	127.9/42.0 amu	2007/05/02 08:03:59 PM
19 Spk 2A	Quality Control	CYA-1	203000	1500	1480	98.5	☒		1.00	2500	1.92	127.9/42.0 amu	2007/05/02 08:14:45 PM
20 Spk 2B	Quality Control	CYA-1	206000	1500	1500	100	☒		1.00	1950	1.92	127.9/42.0 amu	2007/05/02 08:25:32 PM
21 Spk 3	Quality Control	CYA-1	561000	4000	4250	106	☒		1.00	5900	1.92	127.9/42.0 amu	2007/05/02 08:36:18 PM
22 Spk 3A	Quality Control	CYA-1	519000	4000	3930	98.3	☒		1.00	7230	1.92	127.9/42.0 amu	2007/05/02 08:47:04 PM
23 Spk 3B	Quality Control	CYA-1	487000	4000	3680	92.0	☒		1.00	8620	1.92	127.9/42.0 amu	2007/05/02 08:57:51 PM
24 R78125	Unknown	CYA-1	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	127.9/42.0 amu	2007/05/02 09:08:37 PM
25 R78133	Unknown	CYA-1	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	127.9/42.0 amu	2007/05/02 09:19:23 PM
26 R78158	Unknown	CYA-1	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	127.9/42.0 amu	2007/05/02 09:30:09 PM
27 R78179	Unknown	CYA-1	1150	N/A	< 0	N/A			1.00	16.0	1.92	127.9/42.0 amu	2007/05/02 09:40:56 PM
28 R78180	Unknown	CYA-1	2220	N/A	< 0	N/A			1.00	35.6	1.92	127.9/42.0 amu	2007/05/02 09:51:42 PM
29 R78183	Unknown	CYA-1	4920	N/A	< 0	N/A			1.00	78.0	1.92	127.9/42.0 amu	2007/05/02 10:02:28 PM
30 R78184	Unknown	CYA-1	2860	N/A	< 0	N/A			1.00	36.6	2.07	127.9/42.0 amu	2007/05/02 10:13:14 PM
31 R78186	Unknown	CYA-1	3250	N/A	< 0	N/A			1.00	70.5	1.93	127.9/42.0 amu	2007/05/02 10:24:00 PM
32 S08572	Unknown	CYA-1	179000	N/A	1300	N/A			1.00	2110	1.92	127.9/42.0 amu	2007/05/02 10:34:46 PM
33 S08570	Unknown	CYA-1	233000	N/A	1710	N/A			1.00	4810	1.92	127.9/42.0 amu	2007/05/02 10:45:32 PM
34 S08589	Unknown	CYA-1	115000	N/A	796	N/A			1.00	1500	1.93	127.9/42.0 amu	2007/05/02 10:56:18 PM
35 S08554	Unknown	CYA-1	137000	N/A	971	N/A			1.00	1580	1.93	127.9/42.0 amu	2007/05/02 11:07:04 PM
36 S08549	Unknown	CYA-1	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	127.9/42.0 amu	2007/05/02 11:17:51 PM
37 S08553	Unknown	CYA-1	132000	N/A	932	N/A			1.00	2530	1.93	127.9/42.0 amu	2007/05/02 11:28:37 PM
38 S08573	Unknown	CYA-1	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	127.9/42.0 amu	2007/05/02 11:39:23 PM
39													
40													
41													
42													
43 Std 1	Standard	CYA-1	81300	500	537	107	☒		1.00	1000	1.94	127.9/42.0 amu	2007/05/03 12:33:16 AM

Method Name: CYA-MRM_20070427.qmf

Results Name: CYA-Val_Wet_070502.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Val_Wet_070502.rdb

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
44 Std 2	Standard	CYA-1	134000	1000	947	94.7	☒		1.00	1910	1.94	127.9/42.0 amu	2007/05/03 12:44:03 AM
45 Std 3	Standard	CYA-1	204000	1500	1480	99.0	☒		1.00	3230	1.94	127.9/42.0 amu	2007/05/03 12:54:49 AM
46 Std 4	Standard	CYA-1	328000	2500	2450	97.8	☒		1.00	3960	1.94	127.9/42.0 amu	2007/05/03 01:05:35 AM
47 Std 5	Standard	CYA-1	569000	4000	4320	106	☒		1.00	8730	1.94	127.9/42.0 amu	2007/05/03 01:16:21 AM
48 Std 6	Standard	CYA-1	702000	5000	5340	107	☒		1.00	8000	1.94	127.9/42.0 amu	2007/05/03 01:27:07 AM
49 Spk 1	Quality Control	CYA-1	78600	500	517	103	☒		1.00	144	1.93	127.9/42.0 amu	2007/05/03 12:35:19 PM
50 Std 7 (dil 1.50)	Quality Control	CYA-1	880000	50000	336000	672	☒		50.0	1640	1.92	127.9/42.0 amu	2007/05/03 12:46:09 PM
52 Std 6 (dil 1.50)	Quality Control	CYA-1	103000	5000	703	14.1	☒		1.00	249	1.92	127.9/42.0 amu	2007/05/03 02:04:05 PM

Method Name: CYA-MRM_20070427.qmf

■ CYA-WetFood-Val_20070502.rdb (CYA-1): "Linear" Regression ("1 / (x * x)" weighting): y = 129 x + 1.2e+004 (r = 0.9966)

Method Name: CYA-MRM_20070427.qmf

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Results Name: CYA-Val_Net_070502.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Val_Net_070502.rdb

Printing Time: 02:15:15

Method Name: CYA-MRM_20070427.qmf

Page 1 of 5

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-val_Wet_070502.rdb

Method Name: CYA-NRM_20070427.qm1

Page 2 of 5

Results Name: CYA-val_Wet_070502.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-val_Wet_070502.rdb

Method Name: CYA-NRM_20070427.qm1

Page 3 of 5

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Val_Wet_070502.rdb

Method Name: CYA-MRM_20070427.qmf

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Results Name: CYA-Val_Wet_070502.rdb

Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Val_Wet_070502.rdb

Printing Time: 02:15:15
Printing Date: Thursday, May 03,

Method Name: CYA-MRM_20070427.qmf

Page 4 of 5

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
Std 2	Standard	CYA-2	79500.	1000.	953.	95.3	☒	☐	1.00	808.	1.94	127 9/85.0 amu	2007/05/03 12:44:03 AM
Std 3	Standard	CYA-2	120000.	1500.	1490.	99.1	☒	☐	1.00	1440.	1.94	127 9/85.0 amu	2007/05/03 12:54:49 AM
Std 4	Standard	CYA-2	193000.	2500.	2460.	98.5	☒	☐	1.00	2460.	1.94	127 9/85.0 amu	2007/05/03 01:05:35 AM
Std 5	Standard	CYA-2	328000.	4000.	4260.	107.	☒	☐	1.00	2800.	1.95	127 9/85.0 amu	2007/05/03 01:16:21 AM
Std 6	Standard	CYA-2	410000.	5000.	5350.	107.	☒	☐	1.00	4780.	1.95	127 9/85.0 amu	2007/05/03 01:27:07 AM
Spk 1	Quality Control	CYA-2	48100.	500.	555.	107.	☒	☐	1.00	154.	1.93	127 9/85.0 amu	2007/05/03 12:35:19 PM
Std 7 (dil 1:50)	Quality Control	CYA-2	518000.	50000.	340000.	680.	☒	☐	50.0	2670.	1.93	127 9/85.0 amu	2007/05/03 12:46:09 PM
Std 6 (dil 1:50)	Quality Control	CYA-2	65600.	5000.	702.	14.0	☒	☐	1.00	327.	1.93	127 9/85.0 amu	2007/05/03 02:04:05 PM

Printing Time: 02:17:04
Printing Date: Thursday, May 03, 20

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Results Name: C:\Val_Met_070502.rdb
Results Path: D:\Analyst Data\Projects\Residue\Aminopterin\Results\CYA-Val_Met_070502.rdb

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Printing Time: 02:17:04 PM

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Dilution Factor	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date	
1 Rinse	Solvent	CYA-2	0.00	0.00	N/A	N/A			1.00	N/A	0.00	127.9/85.0 amu	2007/05/02 04:52:42 PM	
2 Bk 1	Blank	CYA-2	0.00	0.00	N/A	N/A			1.00	N/A	0.00	127.9/85.0 amu	2007/05/02 05:11:40 PM	
3 Bk 2	Blank	CYA-2	0.00	0.00	N/A	N/A			1.00	N/A	0.00	127.9/85.0 amu	2007/05/02 05:22:26 PM	
4 Std 1	Standard	CYA-2	45600.	500.	501.	100.	☒	☐	1.00	777.	1.92	127.9/85.0 amu	2007/05/02 05:33:12 PM	
5 Std 2	Standard	CYA-2	76000.	1000.	806.	90.6	☒	☐	1.00	1410	1.92	127.9/85.0 amu	2007/05/02 05:43:58 PM	
6 Std 3	Standard	CYA-2	116000.	1500.	1440.	96.3	☒	☐	1.00	1620.	1.92	127.9/85.0 amu	2007/05/02 05:54:44 PM	
7 Std 4	Standard	CYA-2	185000.	2500.	2360.	94.2	☒	☐	1.00	2350.	1.92	127.9/85.0 amu	2007/05/02 06:05:31 PM	
8 Std 5	Standard	CYA-2	313000.	4000.	4060.	102.	☒	☐	1.00	5510.	1.92	127.9/85.0 amu	2007/05/02 06:16:17 PM	
9 Std 6	Standard	CYA-2	394000.	5000.	5140.	103.	☒	☐	1.00	4850.	1.92	127.9/85.0 amu	2007/05/02 06:27:03 PM	
10 LOD 1	Quality Control	CYA-2	39400.	500.	418.	83.8	☒	☐	1.00	360.	1.92	127.9/85.0 amu	2007/05/02 06:37:49 PM	
11 LOD 2	Quality Control	CYA-2	41700.	500.	450.	90.0	☒	☐	1.00	603.	1.92	127.9/85.0 amu	2007/05/02 06:48:35 PM	
12 LOD 3	Quality Control	CYA-2	39000.	500.	414.	82.8	☒	☐	1.00	497.	1.92	127.9/85.0 amu	2007/05/02 06:59:21 PM	
13 LOD 4	Quality Control	CYA-2	40200.	500.	430.	85.9	☒	☐	1.00	423.	1.92	127.9/85.0 amu	2007/05/02 07:10:07 PM	
14 LOD 5	Quality Control	CYA-2	39100.	500.	416.	83.2	☒	☐	1.00	664.	1.92	127.9/85.0 amu	2007/05/02 07:20:54 PM	
15 LOD 6	Quality Control	CYA-2	43700.	500.	477.	95.4	☒	☐	1.00	480.	1.93	127.9/85.0 amu	2007/05/02 07:31:40 PM	
16 LOD 7	Quality Control	CYA-2	40500.	500.	433.	86.7	☒	☐	1.00	362.	1.92	127.9/85.0 amu	2007/05/02 07:42:27 PM	
17 LOD 8	Quality Control	CYA-2	44100.	500.	482.	96.3	☒	☐	1.00	480.	1.93	127.9/85.0 amu	2007/05/02 07:53:13 PM	
18 Spk 2	Quality Control	CYA-2	116000.	1500.	1430.	95.6	☒	☐	1.00	657.	1.92	127.9/85.0 amu	2007/05/02 08:03:59 PM	
19 Spk 2A	Quality Control	CYA-2	121000.	1500.	1500.	100.	☒	☐	1.00	1280.	1.92	127.9/85.0 amu	2007/05/02 08:14:45 PM	
20 Spk 2B	Quality Control	CYA-2	121000.	1500.	1510.	100.	☒	☐	1.00	940.	1.93	127.9/85.0 amu	2007/05/02 08:25:32 PM	
21 Spk 3	Quality Control	CYA-2	329000.	4000.	4280.	107.	☒	☐	1.00	5090.	1.92	127.9/85.0 amu	2007/05/02 08:36:18 PM	
22 Spk 3A	Quality Control	CYA-2	304000.	4000.	3940.	98.6	☒	☐	1.00	3940.	1.92	127.9/85.0 amu	2007/05/02 08:47:04 PM	
23 Spk 3B	Quality Control	CYA-2	290000.	4000.	3760.	93.9	☒	☐	1.00	1930.	1.92	127.9/85.0 amu	2007/05/02 08:57:51 PM	
24 R/8125	Unknown	CYA-2	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	127.9/85.0 amu	2007/05/02 09:08:37 PM	
25 R/8133	Unknown	CYA-2	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	127.9/85.0 amu	2007/05/02 09:19:23 PM	
26 R/8158	Unknown	CYA-2	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	127.9/85.0 amu	2007/05/02 09:30:09 PM	
27 R/8179	Unknown	CYA-2	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	127.9/85.0 amu	2007/05/02 09:40:56 PM	
28 R/8180	Unknown	CYA-2	1860.	N/A	< 0	N/A			1.00	10.6	1.92	127.9/85.0 amu	2007/05/02 09:51:42 PM	
29 R/8183	Unknown	CYA-2	1690.	N/A	< 0	N/A			1.00	14.2	1.93	127.9/85.0 amu	2007/05/02 10:02:28 PM	
30 R/8184	Unknown	CYA-2	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	127.9/85.0 amu	2007/05/02 10:13:14 PM	
31 R/8186	Unknown	CYA-2	1180.	N/A	< 0	N/A			1.00	17.7	1.93	127.9/85.0 amu	2007/05/02 10:24:00 PM	
32 S08572	Unknown	CYA-2	106000.	N/A	1300.	N/A			1.00	951.	1.93	127.9/85.0 amu	2007/05/02 10:34:46 PM	
33 S08570	Unknown	CYA-2	133000.	N/A	1670.	N/A			1.00	1250.	1.93	127.9/85.0 amu	2007/05/02 10:45:32 PM	
34 S08569	Unknown	CYA-2	65600.	N/A	768.	N/A			1.00	874.	1.93	127.9/85.0 amu	2007/05/02 10:56:18 PM	
35 S08554	Unknown	CYA-2	75300.	N/A	837.	N/A			1.00	1160.	1.93	127.9/85.0 amu	2007/05/02 11:07:04 PM	
36 S08549	Unknown	CYA-2	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	127.9/85.0 amu	2007/05/02 11:17:51 PM	
37 S08553	Unknown	CYA-2	77000.	N/A	920.	N/A			1.00	610.	1.94	127.9/85.0 amu	2007/05/02 11:28:37 PM	
38 S08573	Unknown	CYA-2	0.00	N/A	No Peak	N/A			1.00	N/A	0.00	127.9/85.0 amu	2007/05/02 11:39:23 PM	
39														
40														
41														
42														
43	Std 1	Standard	CYA-2	48400.	500.	539.	103.	☒	☐	1.00	297.	1.94	127.9/85.0 amu	2007/05/03 12:33:16 PM

Results Path: R:\Projects\Residue\Aminopterin\Results\CYA-Wet_070509.rdb

Printing Date: Thursday, May 10, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Rinse	Solvent	CYA-1	0.00	0.00	N/A	N/A		☐	N/A	0.00	127.9/42.0 amu	2007/05/09 3:57:34 P
2 Blk	Blank	CYA-1	1940.	0.00	N/A	N/A		☐	14.8	2.04	127.9/42.0 amu	2007/05/09 4:41:44 P
3 Std 1	Standard	CYA-1	99500.	500.	487.	97.4	☒	☐	946.	2.06	127.9/42.0 amu	2007/05/09 4:52:31 P
4 Std 2	Standard	CYA-1	199000.	1000.	1070.	107.	☒	☐	1220.	2.06	127.9/42.0 amu	2007/05/09 5:03:18 P
5 Std 3	Standard	CYA-1	295000.	1500.	1630.	109.	☒	☐	1440.	2.06	127.9/42.0 amu	2007/05/09 5:14:04 P
6 Std 4	Standard	CYA-1	453000.	2500.	2560.	103.	☒	☐	3300.	2.06	127.9/42.0 amu	2007/05/09 5:24:50 P
7 Std 5	Standard	CYA-1	677000.	4000.	3880.	97.1	☒	☐	5520.	2.06	127.9/42.0 amu	2007/05/09 5:35:36 P
8 Std 6	Standard	CYA-1	868000.	5000.	5010.	100.	☒	☐	5470.	2.06	127.9/42.0 amu	2007/05/09 5:46:22 P
9 S15647	Unknown	CYA-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	127.9/42.0 amu	2007/05/09 5:57:09 P
10 S15648	Unknown	CYA-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	127.9/42.0 amu	2007/05/09 6:07:55 P
11 S15673	Unknown	CYA-1	788.	N/A	< 0	N/A		☐	5.39	2.06	127.9/42.0 amu	2007/05/09 6:18:41 P
12 S15676	Unknown	CYA-1	650.	N/A	< 0	N/A		☐	4.58	2.06	127.9/42.0 amu	2007/05/09 6:29:27 P
13 S15677	Unknown	CYA-1	12900.	N/A	< 0	N/A		☐	110.	2.06	127.9/42.0 amu	2007/05/09 6:40:14 P
14 S15680	Unknown	CYA-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	127.9/42.0 amu	2007/05/09 6:51:00 P
15 S15698	Unknown	CYA-1	1280.	N/A	< 0	N/A		☐	9.68	2.06	127.9/42.0 amu	2007/05/09 7:01:47 P
16 Spk 1	Quality Control	CYA-1	93400.	500.	451.	90.3	☒	☐	693.	2.06	127.9/42.0 amu	2007/05/09 7:12:33 P
17 S15699	Unknown	CYA-1	1040.	N/A	< 0	N/A		☐	10.2	2.06	127.9/42.0 amu	2007/05/09 7:23:19 P
18 S15700	Unknown	CYA-1	1300.	N/A	< 0	N/A		☐	9.01	2.07	127.9/42.0 amu	2007/05/09 7:34:05 P
19 S15720	Unknown	CYA-1	2790.	N/A	< 0	N/A		☐	16.4	2.06	127.9/42.0 amu	2007/05/09 7:44:51 P
20 S15721	Unknown	CYA-1	4300.	N/A	< 0	N/A		☐	31.5	2.06	127.9/42.0 amu	2007/05/09 7:55:37 P
21 S15728	Unknown	CYA-1	943.	N/A	< 0	N/A		☐	11.9	2.06	127.9/42.0 amu	2007/05/09 8:06:24 P
22 S15729	Unknown	CYA-1	13800.	N/A	< 0	N/A		☐	168.	2.06	127.9/42.0 amu	2007/05/09 8:17:11 P
23 S15730	Unknown	CYA-1	1110.	N/A	< 0	N/A		☐	9.52	2.06	127.9/42.0 amu	2007/05/09 8:27:57 P
24 S15966	Unknown	CYA-1	570.	N/A	< 0	N/A		☐	4.51	1.86	127.9/42.0 amu	2007/05/09 8:38:43 P
25 Spk 1A	Quality Control	CYA-1	101000.	500.	496.	99.2	☒	☐	546.	2.06	127.9/42.0 amu	2007/05/09 8:49:29 P
26 S15967	Unknown	CYA-1	0.00	N/A	No Peak	N/A		☐	N/A	0.00	127.9/42.0 amu	2007/05/09 9:00:15 P

Results Path: R:\Projects\Residue\Aminopterin\Results\CYA-Wet_070509.rdb

Printing Date: Thursday, May 10, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
39 Std 6	Standard	CYA-1	824000.	5000.	4750.	94.9	☒	☐	6100.	2.06	127.9/42.0 amu	2007/05/09 11:20:17 P

Untitled 17 (CVA-1): "Linear" Regression ("1 / (x * x)" weighting); y = 170 x + 1.66e+004 (r = 0.9976)

Results Name: CVA-Wet_070509.rdb

Results Path: R:\Projects\Residue\Aminopterin\Results\CVA-Wet_070509.rdb

Printing Date: Thursday, May 10, 20

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Printing Date: Thursday, May 10, 20

Sample Name: 312701	Sample ID: 127 942.0	File: Vial.Pat.Food.war
<p>Peak Name: "CVA1" Mass(es): 127.942.0 amu</p> <p>Comment: "A74348" Annotation: 23</p> <p>Sample Index: 23</p> <p>Sample Type: Unknown</p> <p>Concentration: N/A</p> <p>Calculated Conc: No Intercept</p> <p>Acq. Date: 2007/05/03</p> <p>Acq. Time: 8:27:15 PM</p>		
<p>Modified: No</p> <p>Proc. Algorithm: Analyst Classic</p> <p>Smoothing Factor: 1.00 cps</p> <p>Noise Threshold: 100.00 cps</p> <p>Area Threshold: 100.00 cps</p> <p>Num. Smoother: 2</p> <p>Sep. Width: 0.00</p> <p>Exp. Peak Ratio: 1.0004</p> <p>Exp. Adj. Ratio: 1.0004</p> <p>Exp. Val. Ratio: 1.0004</p> <p>Exp. Min. Ratio: 1.0004</p> <p>Exp. Max. Ratio: 1.0004</p> <p>Expected RT: 2.03 min</p> <p>Use Relative RT: No</p> <p>Int. Type: Base To Base</p> <p>Retention Time: 2.06 min</p> <p>Area: 370.49 counts</p> <p>Height: 5.75e+02 cps</p> <p>Start Time: 2.01 min</p> <p>End Time: 2.11 min</p>		
<p>Sample Name: 312701 </p>		

Sample Name: 312701	Sample ID: 127 942.0	File: Vial.Pat.Food.war
<p>Peak Name: "CVA1" Mass(es): 127.942.0 amu</p> <p>Comment: "A74348" Annotation: 23</p> <p>Sample Index: 23</p> <p>Sample Type: Unknown</p> <p>Concentration: N/A</p> <p>Calculated Conc: No Intercept</p> <p>Acq. Date: 2007/05/03</p> <p>Acq. Time: 8:27:15 PM</p>		
<p>Modified: No</p> <p>Proc. Algorithm: Analyst Classic</p> <p>Smoothing Factor: 1.00 cps</p> <p>Noise Threshold: 100.00 cps</p> <p>Area Threshold: 100.00 cps</p> <p>Num. Smoother: 2</p> <p>Sep. Width: 0.00</p> <p>Exp. Peak Ratio: 1.0004</p> <p>Exp. Adj. Ratio: 1.0004</p> <p>Exp. Val. Ratio: 1.0004</p> <p>Exp. Min. Ratio: 1.0004</p> <p>Exp. Max. Ratio: 1.0004</p> <p>Expected RT: 2.03 min</p> <p>Use Relative RT: No</p> <p>Int. Type: Base To Base</p> <p>Retention Time: 2.06 min</p> <p>Area: 370.49 counts</p> <p>Height: 5.75e+02 cps</p> <p>Start Time: 2.01 min</p> <p>End Time: 2.11 min</p>		
<p>Sample Name: 312701 </p>		

Sample Name: 312701	Sample ID: 127 942.0	File: Vial.Pat.Food.war
<p>Peak Name: "CVA1" Mass(es): 127.942.0 amu</p> <p>Comment: "A74348" Annotation: 23</p> <p>Sample Index: 23</p> <p>Sample Type: Unknown</p> <p>Concentration: N/A</p> <p>Calculated Conc: No Intercept</p> <p>Acq. Date: 2007/05/03</p> <p>Acq. Time: 8:27:15 PM</p>		
<p>Modified: No</p> <p>Proc. Algorithm: Analyst Classic</p> <p>Smoothing Factor: 1.00 cps</p> <p>Noise Threshold: 100.00 cps</p> <p>Area Threshold: 100.00 cps</p> <p>Num. Smoother: 2</p> <p>Sep. Width: 0.00</p> <p>Exp. Peak Ratio: 1.0004</p> <p>Exp. Adj. Ratio: 1.0004</p> <p>Exp. Val. Ratio: 1.0004</p> <p>Exp. Min. Ratio: 1.0004</p> <p>Exp. Max. Ratio: 1.0004</p> <p>Expected RT: 2.03 min</p> <p>Use Relative RT: No</p> <p>Int. Type: Base To Base</p> <p>Retention Time: 2.06 min</p> <p>Area: 370.49 counts</p> <p>Height: 5.75e+02 cps</p> <p>Start Time: 2.01 min</p> <p>End Time: 2.11 min</p>		
<p>Sample Name: 312701 </p>		

Sample Name: 312701	Sample ID: 127 942.0	File: Vial.Pat.Food.war
<p>Peak Name: "CVA1" Mass(es): 127.942.0 amu</p> <p>Comment: "A74348" Annotation: 23</p> <p>Sample Index: 23</p> <p>Sample Type: Unknown</p> <p>Concentration: N/A</p> <p>Calculated Conc: No Intercept</p> <p>Acq. Date: 2007/05/03</p> <p>Acq. Time: 8:27:15 PM</p>		
<p>Modified: No</p> <p>Proc. Algorithm: Analyst Classic</p> <p>Smoothing Factor: 1.00 cps</p> <p>Noise Threshold: 100.00 cps</p> <p>Area Threshold: 100.00 cps</p> <p>Num. Smoother: 2</p> <p>Sep. Width: 0.00</p> <p>Exp. Peak Ratio: 1.0004</p> <p>Exp. Adj. Ratio: 1.0004</p> <p>Exp. Val. Ratio: 1.0004</p> <p>Exp. Min. Ratio: 1.0004</p> <p>Exp. Max. Ratio: 1.0004</p> <p>Expected RT: 2.03 min</p> <p>Use Relative RT: No</p> <p>Int. Type: Base To Base</p> <p>Retention Time: 2.06 min</p> <p>Area: 370.49 counts</p> <p>Height: 5.75e+02 cps</p> <p>Start Time: 2.01 min</p> <p>End Time: 2.11 min</p>		
<p>Sample Name: 312701 </p>		

Sample Name: 312701	Sample ID: 127 942.0	File: Vial.Pat.Food.war
<p>Peak Name: "CVA1" Mass(es): 127.942.0 amu</p> <p>Comment: "A74348" Annotation: 23</p> <p>Sample Index: 23</p> <p>Sample Type: Unknown</p> <p>Concentration: N/A</p> <p>Calculated Conc: No Intercept</p> <p>Acq. Date: 2007/05/03</p> <p>Acq. Time: 8:27:15 PM</p>		
<p>Modified: No</p> <p>Proc. Algorithm: Analyst Classic</p> <p>Smoothing Factor: 1.00 cps</p> <p>Noise Threshold: 100.00 cps</p> <p>Area Threshold: 100.00 cps</p> <p>Num. Smoother: 2</p> <p>Sep. Width: 0.00</p> <p>Exp. Peak Ratio: 1.0004</p> <p>Exp. Adj. Ratio: 1.0004</p> <p>Exp. Val. Ratio: 1.0004</p> <p>Exp. Min. Ratio: 1.0004</p> <p>Exp. Max. Ratio: 1.0004</p> <p>Expected RT: 2.03 min</p> <p>Use Relative RT: No</p> <p>Int. Type: Base To Base</p> <p>Retention Time: 2.06 min</p> <p>Area: 370.49 counts</p> <p>Height: 5.75e+02 cps</p> <p>Start Time: 2.01 min</p> <p>End Time: 2.11 min</p>		
<p>Sample Name: 312701 </p>		

Sample Name: 312701	Sample ID: 127 942.0	File: Vial.Pat.Food.war
<p>Peak Name: "CVA1" Mass(es): 127.942.0 amu</p> <p>Comment: "A74348" Annotation: 23</p> <p>Sample Index: 23</p> <p>Sample Type: Unknown</p> <p>Concentration: N/A</p> <p>Calculated Conc: No Intercept</p> <p>Acq. Date: 2007/05/03</p> <p>Acq. Time: 8:27:15 PM</p>		
<p>Modified: No</p> <p>Proc. Algorithm: Analyst Classic</p> <p>Smoothing Factor: 1.00 cps</p> <p>Noise Threshold: 100.00 cps</p> <p>Area Threshold: 100.00 cps</p> <p>Num. Smoother: 2</p> <p>Sep. Width: 0.00</p> <p>Exp. Peak Ratio: 1.0004</p> <p>Exp. Adj. Ratio: 1.0004</p> <p>Exp. Val. Ratio: 1.0004</p> <p>Exp. Min. Ratio: 1.0004</p> <p>Exp. Max. Ratio: 1.0004</p> <p>Expected RT: 2.03 min</p> <p>Use Relative RT: No</p> <p>Int. Type: Base To Base</p> <p>Retention Time: 2.06 min</p> <p>Area: 370.49 counts</p> <p>Height: 5.75e+02 cps</p> <p>Start Time: 2.01 min</p> <p>End Time: 2.11 min</p>		
<p>Sample Name: 312701 </p>		

Sample Name: 312701	Sample ID:
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Results Path: R:\\Projects\\Residue\\Aminopterin\\Results\\CYA-Wet_070509.rdb

Printing Date: Thursday, May 10, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
1 Rinse	Solvent	CYA-2	282.	0.00	N/A	N/A	☐	☐	3.75	2.07	127.9/85.0 amu	2007/05/09 3:57:34 PM
2 Blk	Blank	CYA-2	513.	0.00	N/A	N/A	☐	☐	4.35	2.05	127.9/85.0 amu	2007/05/09 4:41:44 PM
3 Std 1	Standard	CYA-2	55600.	500.	501.	100.	☒	☐	569.	2.06	127.9/85.0 amu	2007/05/09 4:52:31 PM
4 Std 2	Standard	CYA-2	109000.	1000.	1040.	104.	☒	☐	1250.	2.06	127.9/85.0 amu	2007/05/09 5:03:18 PM
5 Std 3	Standard	CYA-2	167000.	1500.	1620.	108.	☒	☐	1560.	2.07	127.9/85.0 amu	2007/05/09 5:14:04 PM
6 Std 4	Standard	CYA-2	256000.	2500.	2510.	101.	☒	☐	4140.	2.07	127.9/85.0 amu	2007/05/09 5:24:50 PM
7 Std 5	Standard	CYA-2	399000.	4000.	3950.	98.8	☒	☐	5180.	2.07	127.9/85.0 amu	2007/05/09 5:35:36 PM
8 Std 6	Standard	CYA-2	507000.	5000.	5040.	101.	☒	☐	5940.	2.06	127.9/85.0 amu	2007/05/09 5:46:22 PM
9 S15647	Unknown	CYA-2	0.00	N/A	No Peak	N/A	☐	☐	N/A	0.00	127.9/85.0 amu	2007/05/09 5:57:09 PM
10 S15648	Unknown	CYA-2	0.00	N/A	No Peak	N/A	☐	☐	N/A	0.00	127.9/85.0 amu	2007/05/09 6:07:55 PM
11 S15673	Unknown	CYA-2	0.00	N/A	No Peak	N/A	☐	☐	N/A	0.00	127.9/85.0 amu	2007/05/09 6:18:41 PM
12 S15676	Unknown	CYA-2	372.	N/A	< 0	N/A	☐	☐	3.91	1.93	127.9/85.0 amu	2007/05/09 6:29:27 PM
13 S15677	Unknown	CYA-2	7280.	N/A	14.5	N/A	☐	☐	81.6	2.07	127.9/85.0 amu	2007/05/09 6:40:14 PM
14 S15680	Unknown	CYA-2	672.	N/A	< 0	N/A	☐	☐	6.48	2.07	127.9/85.0 amu	2007/05/09 6:51:00 PM
15 S15698	Unknown	CYA-2	495.	N/A	< 0	N/A	☐	☐	9.64	2.07	127.9/85.0 amu	2007/05/09 7:01:47 PM
16 Spk 1	Quality Control	CYA-2	51200.	500.	457.	91.3	☒	☐	399.	2.07	127.9/85.0 amu	2007/05/09 7:12:33 PM
17 S15699	Unknown	CYA-2	0.00	N/A	No Peak	N/A	☐	☐	N/A	0.00	127.9/85.0 amu	2007/05/09 7:23:19 PM
18 S15700	Unknown	CYA-2	241.	N/A	< 0	N/A	☐	☐	6.45	2.07	127.9/85.0 amu	2007/05/09 7:34:05 PM
19 S15720	Unknown	CYA-2	1410.	N/A	< 0	N/A	☐	☐	10.8	2.06	127.9/85.0 amu	2007/05/09 7:44:51 PM
20 S15721	Unknown	CYA-2	2110.	N/A	< 0	N/A	☐	☐	23.8	2.07	127.9/85.0 amu	2007/05/09 7:55:37 PM
21 S15728	Unknown	CYA-2	742.	N/A	< 0	N/A	☐	☐	3.06	2.05	127.9/85.0 amu	2007/05/09 8:06:24 PM
22 S15729	Unknown	CYA-2	7420.	N/A	15.9	N/A	☐	☐	122.	2.07	127.9/85.0 amu	2007/05/09 8:17:11 PM
23 S15730	Unknown	CYA-2	592.	N/A	< 0	N/A	☐	☐	5.77	2.06	127.9/85.0 amu	2007/05/09 8:27:57 PM
24 S15966	Unknown	CYA-2	939.	N/A	< 0	N/A	☐	☐	3.82	2.17	127.9/85.0 amu	2007/05/09 8:38:43 PM
25 Spk 1A	Quality Control	CYA-2	53900.	500.	484.	96.8	☒	☐	561.	2.07	127.9/85.0 amu	2007/05/09 8:49:29 PM
26 S15967	Unknown	CYA-2	0.00	N/A	No Peak	N/A	☐	☐	N/A	0.00	127.9/85.0 amu	2007/05/09 9:00:15 PM
34 Std 1	Standard	CYA-2	53400.	500.	478.	95.7	☒	☐	442.	2.07	127.9/85.0 amu	2007/05/09 10:26:24 PM
35 Std 2	Standard	CYA-2	106000.	1000.	1000.	100.	☒	☐	1070.	2.07	127.9/85.0 amu	2007/05/09 10:37:11 PM
36 Std 3	Standard	CYA-2	158000.	1500.	1530.	102.	☒	☐	1300.	2.07	127.9/85.0 amu	2007/05/09 10:47:58 PM
37 Std 4	Standard	CYA-2	249000.	2500.	2440.	97.7	☒	☐	2580.	2.07	127.9/85.0 amu	2007/05/09 10:58:44 PM
38 Std 5	Standard	CYA-2	389000.	4000.	3950.	96.3	☒	☐	3690.	2.07	127.9/85.0 amu	2007/05/09 11:09:30 PM

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Printing Time: 9:33:23

Printing Date: Thursday, May 10, 2007

Sample Name	Sample Type	Analyte Peak Name	Analyte Peak Area (counts)	Analyte Concentration (ppb)	Calculated Concentration (ppb)	Accuracy (%)	Use Record	Record Modified	Analyte Signal To Noise	Analyte Retention Time (min)	Analyte Mass Ranges (amu)	Acquisition Date
39 Std 6	Standard	CYA-2	482000.	5000.	4790.	95.8	☒	☐	2820.	2.07	127.9/85.0 amu	2007/05/09 11:20:17 P