

REPORT OF THE COMMITTEE ON ENVIRONMENT

Chair: Dr. Gavin Meerdink, Urbana, IL
Vice Chair: Dr. Randall A. Lovell, Martinsburg, WV

Mr. L. Wayne Godwin, FL; Dr. John P. Honstead, CO; Dr. Gary D. Osweiler, IA; Dr. John C. Reagor, TX; Dr. Jane F. Robens, MD; Dr. Paul F. Ross, IA; Dr. Manuel A. Thomas, Jr., TX; Dr. Larry J. Thompson, GA; Dr. Gary M. Weber, DC.

The Committee on the Environment met on October 23, 2004 in conjunction with the American Association of Veterinary Laboratory Diagnosticians (AAVLD) Veterinary Analytical Toxicology and Mycotoxins Committee. There were forty-eight in attendance including seventeen visitors.

The mycotoxin occurrence in crops is discussed annually. Mycotoxins, particularly aflatoxin, have not been an issue for the 2004 harvest, in general. Over most of the grain belt, weather conditions have been ideal and record yields were commonplace. However, deoxynivalenol (DON) or "vomitoxin" was a problem in some upper Midwest locales in wheat grain and straw. Isolated incidences of elevated T-2 toxin and aflatoxins were reported in the Midwest and Southwest, respectively.

Dr. Emmett Braselton, Animal and Population Health Diagnostic Laboratory, Michigan State University, presented a time-specific Committee paper entitled, "Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) in the Diagnostic Lab: Then, Now and Hot off the Press." ICP-AES is an analytical instrument that is capable of analyzing for several elements at one time (including but not limited to: Ca, Cd, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, P, Pb, S and Zn). Dr. Braselton has been a pioneer in the implementation of this method for use in diagnostic medicine. Dr. Braselton reviewed the history, the strengths and weaknesses of the ICP-AES. Improvements over the years have added to the value of this instrument. The ICP-AES multi-element analysis instrument has become a useful tool in diagnostic and research laboratories. It has enabled diagnosticians to detect unexpected elemental findings and opened unanticipated avenues for research. Dr. Braselton's paper is included in its entirety elsewhere in these proceedings.

Elizabeth Tor, California Animal Health and Food Safety Laboratory System, University of California, Davis, presented "Algal toxin analyses—methodologies." Blue green algae grow in waters and produce cyanotoxins commonly throughout the world. With the appropriate combination of nutrients, light, temperature and wind direction, "blooms" that concentrate the toxin can occur and result in animal disease and

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death. For example, *Microcystis* spp., *Anabaena* spp., *Aphanizomenon* spp., *Lyngbya* spp. and *Nodularia* spp. produce toxins including, microcystins, anatoxin A, saxitoxins, and nodularin. The World Health Organization has established guidelines for some of these toxins in water, however, methods for their detection and accurate quantitation have been limited.

Bioassay, colorimetric, Enzymed Linked Immunosorbent Assay and High Performance Liquid Chromatography procedures have been used. All have time, sensitivity or specificity limitations. Also, standards for instrument calibration are expensive or not available. Method development with the addition of mass spectroscopy is in progress for use in other matrices.

Dr. Randall Lovell, Division of Animal Feeds, Center for Veterinary Medicine, Food and Drug Administration (FDA), presented a time-specific paper entitled "Dioxin Levels in Animal Feeds." This paper is included in these proceedings.

Dr. Andrew Moore, Canadian Food Inspection Agency, Food Microscopy Laboratory, Guelph, Ontario, presented "FT-Infra-red/scanning electron microscopy (IR/SEM) – a useful tool in veterinary toxicology and forensic food science." Several case examples were used to demonstrate the use of IR/SEM in the veterinary diagnostic laboratory. Food contaminants can be often readily identified with this discrete method of microscopy. Small particulate materials can be identified as insect parts, metal fragments, medications (tablets), crystals, etc. Materials reflect unique infra-red spectra and measurement of these wavelengths can lead to their identification. In one instance, the infra-red technology was useful in the identification of an insecticide that was intentionally disseminated in a city park and killed a number of dogs. The rapid determination and subsequent premises search removed the public hazard.

Michael Filigenzi and Dr. Birgit Puschner, California Animal Health and Food Safety Laboratory System, University of California, Davis discussed "Perchlorate – methodology in milk." Perchlorate (a chlorine atom with four attached oxygen atoms) is a very reactive compound. It is commonly found as a contaminant in water (i.e., rivers). Perchlorate is used in air bags (automobile), rocket fuel and in a number of other applications. There has been media attention following the recent discovery of perchlorate in milk ("milk contains rocket fuel"). Methods for the determination of perchlorate in water and food products were reviewed.

Ion chromatography (IC) has been the method of choice for this agent, although problems with selectivity and matrix effect influences are inherent, particularly in milk. A method developed using High Performance Liquid Chromatography–tandem mass spectrometry (MS/MS) appears to be an improvement over IC. A detection of limit of 0.8

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ppb has been achieved with this new method.

Milk from several sources, including organic milk, has been tested. So far, all milk samples have been found to contain traces of perchlorate. A method for forages is being developed.

No resolutions or recommendations were discussed by the Committee.

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY (ICP-AES) IN THE DIAGNOSTIC LAB: THEN, NOW AND HOT OFF THE PRESS

W. Emmett Braselton, Animal and Population Health Diagnostic Laboratory, Michigan State University, Lansing, MI

Introduction

Rapid and accurate diagnosis of animal disease problems associated with either toxic concentrations of heavy metals, or deficiencies in trace minerals is often predicated on analytical determination of a number of major, minor and trace elements. At the time the Michigan State University (MSU), Animal and Population Health Diagnostic Laboratory, Toxicology Section was being developed in the late 1970's, these determinations were being conducted in veterinary toxicology laboratories using a variety of analytical methodologies, most of which employed time consuming single element determinative steps.^{1,2} Furthermore, completely separate sample preparation procedures were often required. The laboratory established an initial objective of doing simultaneous, multielemental analysis, at the sensitivity necessary for toxicology and nutrition studies in biological tissues. Several techniques for simultaneous quantitative multielemental analysis were becoming available, including neutron activation, spark source mass spectrometry, x-ray fluorescence (XRF) and atomic emission spectroscopy.³ Emission spectroscopy had been used for years for mineral analysis, but the older arc, spark and flame emission sources were unreliable because of interferences, so people turned to atomic absorption (AA), especially in biological sciences. In 1962 Velmer Fassel (Ames, IA) and Stanley Greenfield (England) began experimenting with the inductively coupled plasma (ICP) as source, and in the mid-70s they became commercially available (see review by Fassel).⁴ Although ICP's were being utilized extensively in areas such as geochemistry, food, plants and soils analysis and clinical chemistry by the late '70s, their use in veterinary toxicology and nutrition had been relatively unexplored. With support from the Michigan State University Agricultural Experiment Station in 1979, the AHDL Toxicology Section began to investigate the capability of the ICP as a diagnostic tool for veterinary cases

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involving suspected heavy metal poisonings, mineral deficiencies, or storage disease.

Methods and Procedures

Theory: Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) is an instrumental technique that allows quantitative analysis of as many as 40 elements in solution simultaneously. The technique involves nebulization of the liquefied sample into an argon plasma that is sustained by a high frequency oscillating magnetic field. Elements are atomized and elevated to excited states in the high temperatures (5,000 to 10,000 K) of the plasma, and emit characteristic photons as they decay back to lower energy states. Wavelengths are dispersed by a diffraction grating and simultaneously measured in a direct reading polychromator or sequentially in a scanning monochromator. The advantage gained by the ICP over other methods for emission spectroscopy resides in the inductively coupled plasma itself. Plasmas are gases in which a significant fraction of their atoms or molecules are ionized. Argon plasmas generated by high frequency R.F. form an annular plasma shape, which incorporates a highly efficient introduction of sample into the hottest portion of the plasma, and provide a long residence time (2-3 milliseconds) for the mix of elements in the viewing region. The plasmas achieve a high temperature of 5,000-10,000° Kelvin resulting in excitation of atoms into a large number of energy states which often leads to a number of available emission lines for each element. The argon plasma also provides an inert atmosphere which eliminates self-reversal, leading to high sensitivity. Absence of self-reversal further leads to high linearity of emission intensity versus concentration, up to 6 orders of magnitude, achieving greater accuracy of measurement. Accuracy is also enhanced by high spectral resolution (10-20 picometers) in modern ICP spectrometers. Radial viewed geometry allows background noise separation from the desired emission line signal and therefore enhanced signal to noise and improved detection limits. Because of this unique special distribution of analyte emission and background found in the ICP discharge, the spectral background intensities are 100x smaller and 3x more stable than with other emission sources. In addition to sensitivity and accuracy, analysts are interested in maximizing precision. Direct reading polychromators or eschelle spectrometers interfaced to an ICP source utilize a minimum number of moving parts, are thermally controlled, and are usually equipped with a vacuum or purged optical bench, all of which contribute to excellent instrument stability and therefore precision. Armed with the theoretical capabilities discussed above the MSU lab set forth the following objectives to derive the maximum benefit from the multielemental analyzer: 1) develop one standardized workup for biological samples; and 2) determine 19 elements simultaneously

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under a single set of operating conditions.

Operation: There are certain limitations on the samples to be introduced into the ICP: 1) They must be in liquid or gaseous form; 2) dissolved solids must be low; and 3) viscosity should be <20% mineral acid which limits the ability to analyze a concentrated digest. The lab investigated several alternatives to sample preparation, including dilute and shoot, dry ashing (muffle furnace), hydride generation (selected elements) and wet ashing. A wet ashing procedure which utilized sealed heavy walled Teflon containers was developed from the method described for mercury analysis by H.M. Stahr.⁵ Biological materials including tissue, feeds and serum were digested with conc. HNO₃ overnight at 90°C. Nearly complete mineralization was achieved under these conditions, and viscosity differences were corrected for by use of yttrium as internal standard.

Validation of Results: Having worked out problems with samples themselves, it was necessary to determine the *accuracy* of the results obtained. Accuracy was assessed in several ways, including: 1) dynamic profiling of each spectral emission line about the peak to determine if the peak was present at the proper wavelength and if there was evidence of spectral overlap or background interferences; 2) confirm concentration by the standard additions method; 3) compare with certified standards such as NIST Bovine Liver SRM 1577a and other NIST certified reference materials^a appropriate to the matrix being analyzed; 4) compare results with an independent method such as AA in another laboratory;⁶ and 5) participate in national or international check sample reference programs.^{b,c}

Selected Studies:

Then: By early 1981 the MSU laboratory had developed an argon plasma ICP atomic emission spectroscopy method for determination of 8 elements in serum and determination or monitoring of 19 elements in tissues and feeds. It was clear that the method provided a diagnostic advantage with its rapid screening ability, and at that early stage had confirmed suspected metal poisonings, confirmed mineral deficiencies and storage disease, and identified unsuspected etiologic factors in other cases.⁷

Biopsy mineral analysis: ICP-AES became the routine method for determination of serum elements in live animals and for liver and kidney elements in post-mortem cases^{8,9} but, it was clear that for certain elements liver concentration would be a more useful measure of nutritional status in live animals. This is particularly true of bovine copper. However, because liver biopsies of sufficient size for copper analysis were inconvenient to obtain, serum or plasma coppers were most often used as monitors of bovine copper status. The commercial availability of the ultrasonic nebulizer (USN) made possible the analysis of

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very small samples such as Tru-Cut tissue biopsies. The USN provides a much smaller and more uniform liquid droplet size than conventional nebulizers, and allows a greater concentration of analyte to be introduced, resulting in approximately a 10-fold enhancement of sensitivity. To demonstrate the feasibility of the method, biopsies were obtained from a single post mortem bovine liver, dried, digested overnight in conc. HNO_3 and diluted to volume. It was determined that Cu, Ca, Fe, Mg, Mn, Mo, P, Zn, Na, S and K were present in normal bovine liver in concentrations that could be quantified on samples as small as 5 mg dry weight. Element concentrations in biopsy samples taken in triplicate from the five lobes of the bovine liver were compared to those from triplicate wedge sections (1 gram) taken adjacent to the biopsies and analyzed by conventional ICP-AES. Precision between biopsies was equal to or better than precision between wedge samples. Cu concentrations determined by the biopsy procedure differed statistically from those determined by the wedge procedure, but differences were not sufficient to influence clinical interpretation of data. The element concentration frequency distribution profile of the 11 elements above plus Cd was compared to profiles of the elements in fat, muscle, vena cava, kidney, and clotted blood. The profiles could be used to confirm the authenticity of blind liver biopsy samples from live animals.¹⁰

Ethylene glycol/cholecalciferol: Early on it was found that the ICP mineral screen revealed unexpected etiologies⁷ and one of these was that high kidney Ca (>5,000 parts per million (ppm) wet weight) indicated possible ethylene glycol toxicity. The question then arose "in that case what about cholecalciferol toxicity?" The AHDL ICP data base of canine kidney Ca and P (1985-1998) was compared to known cases of ethylene glycol poisoning and cholecalciferol poisonings. It revealed that canine kidney Ca in unaffected animals was generally less than 800 ppm, Ca in cholecalciferol poisoned animals was between 2,000 and 3,000, and in ethylene glycol poisoning, greater than 5,000 ppm; P in unaffected and ethylene glycol poisonings from 100 to 3,500 ppm, while in cholecalciferol toxicity, P was greater 4,000 ppm. The Ca/P ratio was between 0.4-0.8 in cholecalciferol toxicity, whereas it was greater than 2.5 in ethylene glycol toxicity.¹¹ The Ca/P ratio has become a useful addition to diagnosis of these toxicities.

Mercury by ICP: One of the "needs" identified in the initial development of the ICP procedure was for a rapid, multielemental screen for heavy metals, including Hg. Conventional wisdom indicated that Hg was not a good element to determine by ICP-AES, but the laboratory set up a series of experiments to validate the ICP-AES determination of Hg by comparison with the established procedure of cold vapor-AAS. Feathers, livers, kidneys, and brains were obtained from wild loons, and kidneys obtained from rats treated with mercuric chloride. Hg was determined by cold-vapor AAS and by ICP-AES and results

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compared using regression analysis and Bland and Altman 'limits of agreement' tests.¹² Regression analysis of loon feathers with Hg concentrations of 1.7-48.6 ppm showed $[Hg_{ICP}] = 0.924[Hg_{CVAAS}] + 0.641$, $r = 0.996$. Bland/Altman 'limits of agreement on the $[Hg_{CVAAS}]/[Hg_{ICP}]$ ratio against the mean gave a mean ratio of 1.002, with limits from 0.886-1.118. Analysis of rat and loon tissues with Hg concentrations of 2.2-73 ppm showed $[Hg_{ICP}] = 0.553[Hg_{CVAAS}] + 0.423$, $r = 0.9998$, and Bland/Altman 'limits of agreement on the $[Hg_{CVAAS}]/[Hg_{ICP}]$ ratio against the mean gave a mean ratio of 1.019 with limits from 0.915-1.122. It was clear that ICP-AES was an acceptable alternative for determination of Hg in feathers and tissues at a concentration of 2 ppm and above.

Iohexol clearance: In addition to multielemental screening procedures, the ICP-AES has proven useful as a tool for element specific methods. A method for measurement of iodine in serum to enable the clinical determination of glomerular filtration rate by iohexol clearance in small animals was needed to replace the less sensitive XRF method used in human medicine, where larger volumes of blood could be safely taken over time. Initial experiments indicated that the most sensitive I emission line, at 178.276 nanometer (nm), was seriously overlapped by the P line at 178.287. The ICP was set up to measure I in the P channel by offsetting 0.011nm, and correcting for the P interference by measuring P at a second emission line, 214.914 nm. Iohexol could be measured in serum at 15-600 ppm I, regression line $y = 0.3217 + 0.9951x$, with $r^2 = 0.9995$. The ability to measure down to 15 ppm (XRF detection limit was 40 ppm) allowed measurement of glomerular filtration rate in dogs and cats by using a single compartment model for plasma clearance of iohexol with three samples drawn 3 to 7 hr after treatment.¹³ The method has been validated by comparison with the conventional procedure of urinary exogenous creatinine clearance¹⁴ and has proven useful in clinical studies¹⁵ as well as a routine clinical diagnostic procedure.

Now, Wild Animal Data Base: Extensive use of the ICP-AES method utilizing serum in diagnostic cases involving live animals and tissues, particularly liver and kidney, post mortem, allowed the development of a data base of expected serum and tissue concentrations in a variety of domestic and wild species. This data is presently being collated into useable tables of tissue and serum mineral concentrations, and may be used as an estimate of expected ranges for species with little or no literature values available.

Table 1 is an outline of the number of species with data available in various class, order and family groupings throughout the animal king-

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Class	Order/ Suborder	Family/ Subfamily	Genus/Species/ Breed
Fish	5	6	9
Amphibia	2	3	4
Reptilia	4	13	16
Aves	18	28	46
Mammalia ¹	2	17	18
¹ Not completed			

dom.

Table 2 gives an example of the type of data available and its arrangement in the database. Since many elements show a skewed distribution the median and the 5% to 95 % ranges are given to indicate where the expected range might fall.

Hot off the Press: New technologies available in the past 10-15 years have made possible enhanced sensitivity (axial plasma viewing) and enhanced resolution (eschelle polychromators coupled with semiconductor chip detector arrays). The MSU laboratory has been working with an axial plasma instrument with eschelle polychromator/semiconductor chip array interfaced with an ultrasonic nebulizer to obtain maximum sensitivity. The goal was to develop a method to determine marginal, normal and elevated concentrations of serum Mn and Mo, with quantification limits of 5 parts per billion (ppb) Mn and 10 ppb Mo. Results indicated that the instrument detection limits (3xSTD of the noise) were 0.006 ppb and 0.048 ppb for Mn and Mo respectively. The theoretical limits of quantification (LOQ, 3.3x detection limits) in serum were 0.20 ppb and 1.6 ppb for Mn and Mo respectively. These were well below the original goal of 5 and 10 ppb respectively. Since a certified reference material for Mn and Mo in serum is not available, confirmation of accuracy was conducted by standard addition experiments on a pool of commercially available bovine serum. Results indicated that Mn in the serum determined without standard addition, 6.65 ppb (STD DEV 0.78), was very close to the number confirmed by standard addition , 6.47 ppb (STD DEV 0.74). Likewise, Mo in the serum determined without standard addition, 14.1 ppb (STD DEV 0.75), was very close to the number confirmed by standard addition , 13.0 (STD DEV 0.95). Mn and Mo are now quantified with this method in clinical cases on a routine basis.

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Table 2. Raptors Liver.														
Total data base, including Bald eagles (33), Golden eagles (2), Osprey (3), Hawks (40), Falcons (6), Vulture (1)														
	Ca	Cu	Fe	Mg	P	Zn	K	Na	Mn	Mo	Cd*	Pb*	Hg*	Se*
N	85	85	85	85	85	85	85	85	85	69	20	19	13	15
MEAN	91.1	8.3	853	189	3064	51.4	2362	1441	3.5	0.45	0.24	11.8	3.28	3.26
STDEV	74.2	9.72	756	38.4	449	40.9	627	401	1.4	0.41	0.14	12.8	1.06	1.48
MIN	33.5	2.8	77.7	121	1710	13.4	923	764	0.94	<0.1	<0.1	<0.5	<2.0	<2.0
5%	45.7	3.04	153	139	2318	18.5	1512	1028	1.77	<0.1	0.1	0.61	2.21	2.05
MEDIAN	71	5.53	627	190	3130	32.8	2330	1330	3.1	0.41	0.21	8.65	3.02	2.79
95%	165	21.3	2100	250	3826	145	3304	2054	6.2	1.02	0.43	36.1	5.08	6.39
MAX	609	68.1	3680	318	3930	200	4550	3390	8.17	2.6	0.65	43.5	5.36	7.01

*Values were below detections limits (0.1, 0.5, 2.0, 2.0 ppm for Cd, Pb, Hg, Se respectively) for the majority of animals tested.

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In summary, ICP-AES has provided a useful multielemental screen for diagnostic purposes, has served as a valuable tool for research collaborators, and opened unexpected avenues for investigation of animal health.

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^aStandard Reference Materials Program, Bldg. 202, Room 204, National Institute of Standards and Technology, Gaithersburg, MD 20899

^bVeterinary Laboratory Association Quality Assurance Program, Diagnostic Chemicals Limited, West Royalty Industrial Park, Charlottetown, PE, Canada C1E 1B0

^cAssociation of American Feed Control Officials Check Sample Program, 4760 Hammermill Road-Suite 104, Tucker, GA 30084

DIOXIN LEVELS IN ANIMAL FEEDS

Dr. Randall Lovell, Veterinary Medical Officer, Division of Animal Feeds, Center for Veterinary Medicine, Food and Drug Administration (FDA)

GRAINS

	ppt TEQ [ND=0]* (17 D/F congeners) (mean ± stdev)	ppt TEQ [ND=0]* (3 PCB congeners) (mean ± stdev)
CORN	0.0032 ± 0.0081 (n=21) range = 0.0 – 0.034	0.015 ± 0.026 (n=10) range = 0.0 – 0.080
COTTON SEED	0.0026 ± 0.0052 (n=15) range = 0.0 – 0.034	0.0055 (n=2) range = 0.0 – 0.011
BARLEY	0.0068 ± 0.0090 (n=13) range = 0.0 – 0.026	0.047 (n=2) range = 0.037 – 0.057
RYE	0.0052 ± 0.0062 (n=11) range = 0.0 – 0.018	0.0073 ± 0.0012 (n=5) range = .0057 – .0090

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OATS	0.0059 ± 0.0081 (n=10) range = 0.0 – 0.020	0.034 (n=2) range = .00013 – .068
MILO	0.0011 ± 0.0030 (n=9) range = 0.0 – 0.0090	0.0 (n=1)
WHEAT	0.0022 ± 0.0036 (n=5) range = 0.0 – 0.0014	no data
PEANUTS	0.00026 ± 0.0058 (n=5) range = 0.0 – 0.0013	no data
RICE	0.00071 ± 0.00069 (n=4) range = .00011 – .0014	0.0053 ± 0.0061 (n=4) range = 0.0 – 0.011

*preliminary data that has not been verified

GRAIN BY-PRODUCTS

	ppt TEQ [ND=0]* (17 D/F congeners) (mean ± stdev)	ppt TEQ [ND=0]* (3 PCB congeners) (mean ± stdev)
	_____	_____
SOYBEAN MEAL	0.017 ± 0.027 (n=10)	0.0 (n=2) range = 0.0 – 0.075
CANOLA MEAL	0.00058 ± 0.00105 (n=10) range = 0.0 – 0.0030	0.023 ± 0.045 (n=9) range = 0.0 – 0.13
COTTON SEED MEAL	0.025 ± 0.028 (n=9) range = 0.0 – 0.087	0.00065 (n=2) range = 0.0 – 0.0013
RICE BRAN	0.0083 ± 0.0131 (n=8) range = 0.00062 – 0.039	0.031 ± 0.040 (n=8) range = 0.0 – 0.12
WHEAT MIDDS	0.044 ± 0.047 (n=5) range = 0.004 – 0.122	no data
Corn Meal	0.004 (n=1)	no data
Grain Screening Pellet	0.121 (n=1)	no data
Peanut Meal & Hulls	0.0 (n=1)	no data
Rice Hulls	0.074 (n=1)	0.054 (n=1)
Rice Mill Feed	0.004 (n=1)	no data
Safflower Meal	0.008 (n=1)	no data
Sunflower Meal	0.025 (n=1)	no data

*preliminary data that has not been verified

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FISH MEAL

	ppt TEQ [ND=0]* (17 D/F congeners) (mean ± stdev)	ppt TEQ [ND=0]* (3 PCB congeners) (mean ± stdev)
MENHADEN	1.36 ± 0.44 (n=22) range = 0.89 – 3.0· ·2 nd highest value = 1.8	0.58 ± 0.31 (n=22) range = 0.011 – 1.1
PERUVIAN/ S. AMERICA	0.091 (n=3) range = 0.018 – 0.24	0.22 (n=3) range = 0.20 – 0.23
HERRING CANADA	0.58 (n=4) range = 0.20 – 1.1	0.37 (n=4) range = 0.24 – 0.42
SARDINE MEXICO	0.02 (n=3) range = 0.0 – 0.06	0.021 (n=3) range = 0.0 – 0.063
MIXED SPECIES PACIFIC COAST	0.43 (n=3) range = 0.29 – 0.70	0.36 (n=3) range = 0.26 – 0.45
COD (Alaska)	0.0032 (n=1)	0.061 (n=1)
KRILL (Alaska)	0.00038 (n=1)	0.0018 (n=1)

*preliminary data that has not been verified

FISH OIL

	ppt TEQ [ND=0]* (17 D/F congeners) (mean ± stdev)	ppt TEQ [ND=0]* (3 PCB congeners) (mean ± stdev)
MENHADEN	12.2 ± 6.8 (n=7) range = 7.8 – 27· ·2 nd highest value = 13	6.9 ± 2.0 (n=7) range = 4.4 – 10
CATFISH	12.7 (n=3) range = 12 – 14	0.85 (n=3) range = 0.52 – 1.3
WHITING	1.5 (n=2) range = 1.0 – 1.9	2.6 (n=2) range = 2.3 – 2.9
FISH NOS (Norway)	0.013 (n=1)	0.28 (n=1)

*preliminary data that has not been verified

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FORAGES

	ppt TEQ [ND=0]* (17 D/F congeners) (mean ± stdev)	ppt TEQ [ND=0]* (3 PCB congeners) (mean ± stdev)
ALFALFA HAY (CA, KS, NY, PA & OR)	0.033 ± 0.046 (n=5) range = 0.0 – 0.103	no data
CORN SILAGE (IA, IL, NY & OH)	0.016 ± 0.010 (n=4) range = 0.0069 – 0.031	no data
Sweet Corn Cannery Waste (corn husks)	0.0 (n=1)	no data

Proposed Fiscal Year 2005 Survey

Dioxins in Rendered Mammalian/Poultry Fats, in Yellow Grease, and in Filtering/Bleaching Agents – Nationwide Survey

Sample Description	No. of Samples
rendered fat, poultry	10
rendered fat, swine	10
rendered fat, cattle	10
rendered fat, mixed animal species	10
yellow grease	10
filtering/bleaching agents	10

Follow-up Cattle Investigations to a Recent USDA Survey

	ppt TEQ [ND=0]* (17 D/F congeners) (mean ± stdev)	ppt TEQ [ND=0]* (3 PCB congeners) (mean ± stdev)
MIXED CATTLE RATION	0.057 ± 0.151 (n=11) range = 0.00071 – 0.51 -2 nd highest value only 0.040	0.018 ± 0.033 (n=9) range = 0.0 – 0.078
MINERAL/ SUPPLEMENT	0.14 ± 0.26 (n=15) range = 0.0 – 0.74	0.0011 ± 0.0017 (n=6) range = 0.0 – 0.0041
WATER	.00025 ± .00057 (n=12) range = 0.0 – 0.0019	.00059 ± .00183 (n=11) range = 0.0 – 0.0061°

°2nd highest value = 0.00031

ENVIRONMENT

FAT	0.41 ± 0.29 (n=6) range = 0.048 – 0.75	0.15 (n=3) range = 0.058 – 0.34
HAY/ SILAGE	0.049 ± 0.031 (n=6) range = 0.0 – 0.0030	0.031 ± 0.024 (n=4) range = 0.0 – 0.058
Corn	0.0 – 0.041 (n=2)	0.0 (n=1)
Corn Steepwater	0.054 (n=1)	0.00053 (n=1)
Molasses	0.0068 (n=1)	no data
Bedding	2.9 (n=1)	0.010 (n=1)

*preliminary data that has not been verified

Follow-up Investigations to a Recent USDA Dioxin Survey

	ppt TEQ [ND=0]* (17 D/F congeners)	ppt TEQ [ND=0]* (3 PCB congeners)
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Utility Pole (barn support)	44,432	0.0
Feeder (home made; gnawed)	3,197	0.0
Feeder (home made)-	1,117	0.0045
Corral Post	852	no data
Security Light Pole	318	0.0
Corral Post	44	no data
Utility Pole (barn support)	7.3	0.0019
Corral Post	0.93	no data
Treated Lumber	0.42	0.0049
Feeder (built in 1980s)	0.22	0.077
Treated Lumber	0.10	0.0
Fence Post	0.0047	0.0
Green Treated Lumber	0.0	0.0
Treated Lumber	0.0	0.0

- mixed cattle ration containing 0.51 ppt TEQ was collected from the bottom of a feeder similar to this one. The dioxin/furan congener pattern in the mixed ration was similar to that in this feeder.

*preliminary data that has not been verified