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Capabilities Statement

Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans

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SUBMITTED BY:

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INTRODUCTION

The following information has been compiled to demonstrate Twin City Testing (TCT) Corporation's qualifications to provide analytical support for the determination of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs). Included is a discussion of our analytical methodologies, the quality assurance/quality control measures that we employ, and a description of our facilities.

TCT is a private corporation that is a subsidiary of Huntingdon International Holdings plc, Huntingdon, England. TCT's corporate offices and laboratories are located in St. Paul, Minnesota. The principal contacts for PCDD/PCDF related projects are Dr. Fred L. DeRoos and Mr. Charles Sueper. Dr. DeRoos can be reached at (612) 649-5502, while Mr. Sueper can be reached at (612) 649-5520.

TCT's analytical methodologies for determining PCDDs and PCDFs are based primarily on EPA Methods 8290 and 1613. These methods are similar to Method 8280, however, they include additional quantification and recovery standards and use high resolution mass spectrometry for detection. The use of additional internal standards improves the accuracy of the method, while the use of high resolution mass spectrometry provides the highest sensitivity and selectivity of currently available technology. Portions of these methods were developed by Dr. DeRoos' groups at Battelle's Columbus Laboratories and at TCT.

The following sections describe TCT's PCDD/PCDF methodology, Quality Assurance/Quality Control procedures, and facilities employed for the determination of PCDDs, PCDFs, and related compounds.

1.0 ANALYTICAL METHODOLOGY

1.1 Sample Extraction

The various sample matrices are extracted using procedures that are optimized for each particular matrix. Isotopically labeled PCDD/PCDF internal standards are added to the sample before extraction so that corrections for extraction efficiency and analyte enrichment efficiency are included in the quantification calculations. A separate ^{13}C-labeled standard is used for each of the 2,3,7,8 substituted isomers. This provides at least one internal standard for each of the chlorine congener classes. Examples of specific sample extraction methods used by TCT for some common sample matrices are described below.

1.1.1 Water Samples

Water samples are liquid-liquid extracted with methylene chloride. A 1000 ml aliquot of each sample, typically collected in a 1 L glass bottle, is transferred to a 2 L separatory funnel and spiked with the isotopically labeled internal standards. The separatory funnel is shaken for 2 minutes to assure complete mixing of the internal standards. A 60 mL portion of methylene chloride is added to the original 1 L sample bottle which is then shaken for approximately 1 minute. The methylene chloride rinse of the sample bottle is then transferred to the 2 L separatory funnel containing the sample which is then shaken for an additional 10 minutes. The methylene chloride extract is transferred to a 500 mL flask and a second 60 mL portion of methylene chloride is added to the separatory funnel. The shaking is continued for 3 minutes after which the methylene chloride extract is removed and combined with the extract from the first extraction. A third extraction with 60 mL of methylene chloride is carried out and the combined extracts are then dried with anhydrous sodium sulfate. Care is taken to prevent clumping of the sodium sulfate which can entrap the methylene chloride extract and degrade recoveries. The dried extract is transferred to a Kuderna Danish apparatus using three 25 mL methylene chloride rinses and concentrated to a final volume of 5-10 mL. The concentrated extracts are then stored at approximately 4 °C until the analyte enrichment steps.

1.1.2 Soil Samples

Soil samples are Soxhlet extracted using benzene as the extraction solvent. A Soxhlet extraction, rather than a shake extraction, is preferred due to the higher efficiency of the Soxhlet technique. Aliquots of each sample, typically 10 g, are transferred to Soxhlet thimbles and mixed with 25 g of anhydrous sodium sulfate. Alternatively, a Dean-Stark apparatus may be attached to the Soxhlet. By doing so, the extraction efficiency is enhanced for high moisture content samples. In addition, the water that is present in the sample is then collected separately so that the dry weight of the sample aliquot can be calculated. The thimbles are placed in the extractors and sufficient solvent is added to wet the sample. The internal standards are added to the thimbles and the extractors are allowed to equilibrate for 30 minutes. The extractors are then charged with benzene and the samples are extracted for approximately 18 hours. The temperatures of the heating mantles are adjusted to maintain a cycle rate of approximately 6-8 cycles per hour. Teflon tape is used to seal the joints to minimize losses of the solvent. After 18 hours, the benzene extracts are transferred to Kuderna Danish concentrators and reduced to a final volume of approximately 5-10 mL. The extraction thimble and the soxhlet apparatus are rinsed with three 25 mL portions of benzene to assure that the extract has been quantitatively transferred to the concentrator. The concentrated extracts are stored at 4 °C until the analyte enrichment steps.

1.1.3 Sediment Samples

River and lake sediment samples are Soxhlet extracted using benzene. Approximately 10-20 g of each sediment sample are transferred to Soxhlet thimbles and placed into the extraction apparatus. The Soxhlet is fitted with a Dean-Stark apparatus which is used to collect water that is present in the sample. The remainder of the extraction procedure is the same as described for the soil samples.

1.1.4 Fish Samples

Fish samples are Soxhlet extracted using a neutral extraction procedure. The samples are either filleted or the entire fish is analyzed depending upon project specifications. The selected portion of the sample is homogenized in a blender or grinder and a 10 g aliquot of the homogenate is transferred to a Soxhlet thimble and mixed with 100 g of anhydrous sodium sulfate. The dried samples are spiked with the isotopically labeled internal standards and allowed to equilibrate for 30 minutes. The extractors are then charged with methylene chloride and the samples are allowed to extract for 18 hours. The temperature of the heating mantles is adjusted to achieve a cycle rate of approximately 6-8 cycles per hour. Teflon tape is used to seal the glass joints to minimize the loss of solvent. After 18 hours, the methylene chloride extracts are transferred to Kuderna Danish concentrators. Three rinses with 25 mL portions of methylene chloride are used to assure that quantitative transfer is achieved. The extracts are then concentrated to a final volume of 5-10 mL and are stored at 4 °C until the analyte enrichment steps.

1.1.5 Adipose Tissue Samples

Due to the lipophilic nature of PCDDs and PCDFs, most human and animal analyses focus on adipose tissue. Approximately 25-50 g of adipose tissue from each sample are transferred to 100 mL beakers and heated until the adipose tissue liquefies. The liquid is filtered to remove connective tissue and the sample is poured into a glass bottle with a teflon lined cap. A 10 g aliquot of each sample is then dissolved in 100 mL of hexane and the resulting solution washed with 25 mL portions of concentrated sulfuric acid until the washes are only lightly colored. The washed hexane extracts are transferred to Kuderna Danish concentrators and reduced in volume to approximately 5-10 mL. The concentrated extracts are stored at 4 °C until the analyte enrichment steps.

1.1.6 Surface Wipes

Surface wipes from buildings that have been involved in polychlorinated biphenyl (PCB) related fires are Soxhlet extracted using benzene. The wipe pads comprising each sample, typically four pads per sample, are transferred to a soxhlet extraction thimble containing a layer of approximately 10 g of anhydrous sodium sulfate. Sufficient benzene is added to the thimble to wet the sample, followed by the addition of the internal standards. The extractor is then allowed to equilibrate for 30 minutes. Benzene is added to the flask and the extractor is allowed to extract for 16-18 hours. The temperature of the heating mantle is adjusted to maintain a cycle rate of approximately 6-8 cycles per hour. Teflon tape is used to seal the joints to minimize losses of the solvent. After the extraction, the benzene extract is transferred to a Kuderna Danish concentrator and is concentrated to approximately 5-10 ml. The extraction thimble and the Soxhlet apparatus are rinsed with three 25 ml portions of benzene to assure that all of the extract has been transferred to the concentrator. The concentrated extracts are then stored at 4 °C until the analyte enrichment steps.

1.1.7 Air Samplers

The particulate filter and adsorbent cartridge from each air sampler are transferred to a Soxhlet extraction apparatus and extracted as a single sample. Rinsates and/or condensates are extracted separately by liquid-liquid extraction using methylene chloride as described for water samples. If the filter contains a significant quantity of particulate, it is extracted in a separate Soxhlet extractor. Sufficient benzene is added to the thimble(s) to wet the sample and the internal standards are added. The extractor is then allowed to equilibrate for 30 minutes. If the filter is being extracted separately, the internal standard is divided between the two portions of the sample.

The extractor(s) is then charged with benzene and the sample is extracted for 16-18 hours. The temperature of the heating mantle is adjusted to maintain a cycle rate of approximately 6-8 cycles per hour. Teflon tape is used to seal the joints to minimize losses of the solvent. After the extraction, the benzene extract(s) and methylene chloride extract are transferred to a Kuderna Danish concentrator and reduced in volume to 5-10 ml. The extraction thimble and Soxhlet apparatus are rinsed with three 25 ml portions of benzene to assure that the extract was quantitatively transferred to the concentrator. The concentrated extract is stored at 4 °C until the analyte enrichment steps.

1.2 Analyte Enrichment

The extraction procedure often removes a variety of compounds, in addition to the PCDDs and PCDFs, from the sample matrix. Some of these compounds, for example polychlorinated biphenyls (PCBs), can directly interfere with the analyses. Other compounds can overload the capillary column causing a degradation in chromatographic resolution or suppress ionization in the mass spectrometer which results in changes in sensitivity. Ion current suppression is an especially important consideration since it can often go undetected unless it is specifically investigated. TCT checks for ion current suppression by monitoring an ion mass from a reference compound that is present in the ion source at a constant level. If ion current suppression is observed, the sample extract is subjected to additional analyte enrichment steps and is reanalyzed. The analyte enrichment procedure typically consists of three liquid chromatography columns. These columns, a multilayered silica gel column, an activated alumina column, and a carbon column, remove most of the coextracted compounds that can interfere with the detection and quantification of PCDD/PCDF isomers. They also enrich the final extract in PCDDs and PCDFs by a factor of up to 100,000 which is essential in order to achieve ultra trace detection limits.

1.2.1 Multilayered Silica Gel Column

The initial analyte enrichment column consists of alternating layers of activated silica gel, 44% concentrated sulfuric acid on silica gel, and 33% 1M sodium hydroxide on silica gel. This column removes acidic and basic compounds from the extract as well as materials that are easily oxidized. The silica gel support provides a large surface area for contact with the sample extract thereby improving the efficiency. The multilayered silica gel column is eluted with hexane and the entire eluate, including the original extract volume, is collected. The eluate is concentrated using a gentle stream of nitrogen gas to remove residual benzene or methylene chloride and then diluted to ~1 mL with hexane.

1.2.2 Alumina Column

The hexane solution is then fractionated using an activated alumina column. This column, containing approximately 4-5 g of activated alumina, is eluted with hexane and hexane/methylene chloride. The hexane/methylene chloride fraction, which contains the PCDD/PCDF isomers is collected and allowed to pass onto a carbon column.

1.2.3 Carbon Column

The carbon column is eluted with hexane and methylene chloride/methanol/benzene in the forward direction, and then back eluted with toluene. The toluene eluate is collected, concentrated and solvent exchanged into tridecane. The tridecane used to reconstitute the sample extracts contains a known quantity of two additional isotopically labeled PCDD/PCDF standards which are used to calculate the recoveries of the quantification standards that were added before extraction. The enriched sample extracts are stored in the dark at 4 °C until they are analyzed.

1.3 HRMS Analyses

The sample extracts are analyzed for the presence of PCDDs and PCDFs using combined capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS). The capillary column is either a 60 M DB-5 coated fused silica capillary which is used for congener class determinations and specific 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) determinations, a 60 M SP-2330 column which is used to confirm the specific level of 2,3,7,8-TCDF, or a DB-Dioxin column which is used to determine levels of 2,3,7,8-TCDD and 2,3,7,8-TCDF. The capillary column is interfaced directly into the ion source of a VG Model 70-VSE or 70-SE high resolution mass spectrometer, thus providing the highest possible sensitivity while minimizing degradation to the chromatographic resolution. The mass spectrometer is operated in the electron impact ionization mode at a mass resolution of 9000-11000 (M/ M, 10 percent valley definition). This resolution is sufficient to resolve most interferences, such as PCBs, thus providing the highest level of confidence that the detected levels of PCDD/PCDF are not false positives resulting from interferences. Typical operating parameters for the HRGC/HRMS analyses are summarized in Table 1. Two ion masses are monitored for each chlorine congener class so that chlorine isotope ratios can be calculated and used as part of the identification criteria. The two corresponding ion masses from each of the isotopically labeled standards are also monitored.

TABLE 1. HRGC/HRMS OPERATING PARAMETERS

PARAMETER	TARGET VALUE
Mass Resolution	9000-12000 (M/ M, 10 % valley)
Electron Energy	34 electron volts
Accelerating Voltage	8000 volts
Source Temperature	275 °C
Preamplifier Gain	10^{8} amp/volt
Electron Multiplier Gain	$\sim 10^{5}$
Chromatographic column	60 M DB-5, 60 M SP-2330, or 60 M DB-Dioxin
Transfer Line Temperature	300 °C
Injection Mode	Splitless
Carrier Gas	Helium
Carrier Flow Velocity	$\sim$30 cm/sec
Injection Volume	0.5 - 2.0 uL

1.3.1 Lock Mass

A lock mass, monitored during the analyses, is used to detect suppressive interferences and to assure that the mass focus of the mass spectrometer remains correct throughout the duration of the analysis. Most modern mass spectrometers are stable on a short term basis (1-10 minutes), however, they can drift from the center of the mass peak during the course of a 30-60 minute analysis. The lock mass provides real time correction for minor drift that would otherwise degrade the sensitivity and the effective mass resolution of the analyses.

1.3.2 Identification Criteria

The criteria used to judge positive responses for a PCDD/PCDF isomer include:

*Simultaneous response at both ion masses of the PCDD or PCDF

*Signal to noise ratio equal to or greater than 2.5:1.0 for both ion masses

*Chlorine isotope ratio within 15% of the theoretical value

*Chromatographic retention time within 2-3 seconds of authentic standards (when available)

*Chromatographic retention times within elution windows determined from analyses of standard mixtures of PCDD/PCDF isomers

*Absence of coeluting diphenyl ether for PCDF isomers

Particular attention is given to classes of compounds that can interfere with the identification of PCDD/PCDF isomers. These include the polychlorinated biphenylenes (PCBP) which can interfere with the PCDDs and the polychlorinated diphenyl ethers (PCDE) which can interfere with the PCDFs. Each sample is screened for the presence of PCDEs and PCBPs to assure that they are not causing false positive identifications. The M+70 ion mass is monitored for each PCDF congener class to indicate the presence of PCDEs. while the M^+ ion from each PCDD congener class is monitored to resolve PCBP interferences. A list of the exact ion masses monitored for the determination of PCDD/PCDF isomers and the PCDE interferences is presented in Table 2. Also included are the theoretical chlorine isotope ratios for the eight chlorinated congener classes.

TABLE 2. EXACT ION MASSES MONITORED
FOR THE DETERMINATION OF
PCDDs,PCDFs, AND PCDPEs

	Accurate Mass		Theoretical Ratio
Compound	Mass 1	Mass 2	Mass 1/Mass 2
Tetra-CDDs	319.8965	321.8936	0.77
Tetra-CDFs	303.9016	305.8987	0.77
Hexa-CDPEs	375.8364	377.8334	1.23
Penta-CDDs	355.8546	357.8517	1.54
Penta-CDFs	339.8597	341.8567	1.54
Hepta-CDPEs	409.7974	411.7944	1.03
Hexa-CDDs	389.8156	391.8127	1.23
Hexa-CDFs	373.8207	375.8178	1.23
Octa-CDPEs	443.7584	445.7555	0.88
Hepta-CDDs	423.7766	425.7737	1.03
Hepta-CDFs	407.7817	409.7788	1.03
Nona-CDPEs	477.7194	479.7165	0.77
Octa-CDD	457.7377	459.7347	0.88
Octa-CDF	441.7428	443.7398	0.88
Deca-CDPE	511.6805	513.6775	0.69

CDDs = Chlorinated Dibenzo-p-dioxins
CDFs = Chlorinated Dibenzofurans
CDPEs = Chlorinated Diphenyl Ethers

1.3.3 Typical Detection Limits

The single isomer detection limits that are achieved for a particular sample are dependant upon the sample matrix, the chemical interferences present in the sample, the extract volume that is injected, and the size of the sample aliquot that can be extracted. In general, analyses of matrices that are relatively clean and for which large sample aliquots can be extracted, such as water, will achieve the lowest detection limits. Samples containing high levels of soluble compounds and chemical interferences, such as sediments and sludges, will usually result in higher detection limits. Typical single isomer detection limits are listed in Table 3.

1.4 Quantification and Calculations

The PCDD/PCDF isomers are quantified by comparing their responses to the responses of the labeled internal standards. Relative response factors are calculated from multiple analyses of standard mixtures that contain the "2,3,7,8" substituted isomers from each of the PCDD/PCDF congener classes and each of the internal standards at known levels. Since labeled and unlabeled standards are not available for each of the 210 PCDD/PCDF isomers, it is necessary to assume that the "non-2,3,7,8" substituted isomers of a particular chlorine congener class have the same response factor and extract with the same efficiency as the "2,3,7,8" substituted isomers. The PCDD/PCDF response factors are calculated by comparing the sum of the responses from the two ion masses monitored for each chlorine congener class to the sum of the responses from the two ion masses from the corresponding isotopically labeled internal standard. The formula for the response factor calculation is:

TABLE 3. TYPICAL[1] DETECTION LIMITS FOR SELECTED MATRICES

MATRIX	DETECTION LIMIT
Water	0.01 - 0.05 ppt[2]
Sediment	0.5 - 50 ppt
Fish	0.5 - 10 ppt
Aquatic Life	1 - 20 ppt
Soil	0.5 - 20 ppt
Fly Ash	1 - 50 ppt
Adipose Tissue	0.5 - 5 ppt
Still Bottoms	1 - 100 ppt
Paper Sludge	0.5 - 10 ppt
Air (silica gel)	0.5 - 10 pg/M^3
Air (XAD-2 resin)	1 - 50 pg/M^3
Surface Wipes (Gauze Pads)	10 - 25 pg/M^2

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[1] Actual detection limits may be higher or lower

[2] parts-per-trillion (picograms per gram)

$$Rf = \frac{An \times Qis}{Ais \times Qn}$$

where:

Rf = Response Factor
An = Sum of integrated areas for native isomer
Qis = Quantity of labeled internal standard
Ais = Sum of integrated areas for labeled internal standard
Qn = Quantity of native isomer

The concentrations of PCDD/PCDF in the sample are calculated using the following equation:

$$C = \frac{An \times Qis}{Ais \times W \times Rf}$$

where:

C = Concentration of target isomer or congener class
An = Sum of integrated areas for the target isomer or congener class
Qis = Amount of labeled internal standard added to the sample
Ais = Sum of integrated areas for the labeled internal standard
W = Sample weight, volume, or area
Rf = Response factor

Each pair of ion mass peaks in the selected-ion-current chromatograms is evaluated manually to determine if it meets the criteria for a PCDD or PCDF isomer. By examining the peaks separately, quantitative accuracy is improved over what is obtained when all of the peaks in a selected chromatographic window are averaged. When averaged data are used, it is possible for pairs of peaks with high or low chlorine isotope ratios to produce averaged data that meet the isotope ratio criterion. For example, when two pairs of TCDD peaks of approximately the same intensity and having chlorine isotope ratios of 0.56 and 0.96 are averaged, the resultant ratio will be within the acceptable range (0.65 - 0.88). By evaluating the peaks separately, neither would be counted in the congener class total. A limit of detection (LOD) based on producing a signal that is 2.5 times the noise level is calculated for samples in which isomers of a particular chlorine congener class are not detected. The formula used for calculating the LOD is:

$$\mathrm{LOD} = \frac{\mathrm{Hn} \times \mathrm{Qis} \times 2.5}{\mathrm{His} \times \mathrm{W} \times \mathrm{Rf}}$$

where:

LOD = Single isomer limit of detection
Hn = Height of the noise at the retention time of the native isomer peak
Qis = Quantity of labeled internal standard
His = Height of labeled internal standard peak
W = Sample weight, volume, or area
Rf = Response factor

2.0 QUALITY ASSURANCE AND CONTROL

Quality assurance (QA) and quality control (QC) are an integral part of all programs carried out by TCT. They include general procedures, such as company wide Standard Operating Procedures (SOPs), staff training, and regular proficiency evaluations, as well as specific procedures designed for particular programs. A copy of the index of TCT's Quality Assurance Manual relating to analytical procedures is available upon request. Specific QA/QC procedures that are applied to PCDD/PCDF programs carried out by Twin City Testing Corporation are described in the following paragraphs.

2.1 Laboratory Glassware

Most of the laboratory glassware used during the extraction of samples, as well as the subsequent analyte enrichment steps, is disposable so as to minimize the chance of cross contamination. All reusable glassware, including Soxhlet extractors, Kuderna Danish concentrators, and separatory funnels, are permanently labeled (etched) so that their usage can be traced. The form used to record the usage of glassware is shown in Figure 1. After each use, all pieces of reusable glassware are rigorously cleaned with detergent, acid, and solvent washes. They are also periodically muffled to remove residual contaminants. In order to demonstrate freedom from contamination, solvent method blanks are generated in all reusable glassware after cleaning and prior to being reused for actual samples.

2.2 Solvents and Reagents

All solvents and reagents used during the extraction and analyte enrichment steps are screened for PCDD/PCDF contamination by means of laboratory method blanks. At least one laboratory method blank sample is prepared for each group of 8-10 samples of a particular matrix. Additional laboratory method blanks are prepared after unusually high concentration samples are processed.

FIGURE 1

GLASSWARE RECORD SHEET

CLIENT

INVOICE #:

DATE:

SAMPLE #	ROUND BOTTOM	SOXHLET TOP	SEPARATORY FUNNEL	Na_2SO_4 COLUMN	KUDERNA DANISH	AMPULE	SNYDER	DEAN STARK	SILICA CLEANUP BEAKER	250 ml BEAKER	BUCHNER FLASK

COMMENTS

2.3 HRGC/HRMS SYSTEMS

The mass resolution and mass focus accuracies of the HRGC/HRMS systems are evaluated before the analysis of each sample or standard. The mass resolution is evaluated by introducing a reference compound, typically perfluorokerosene (PFK), into the ion source and demonstrating that a mass resolution of 10,000-12,000 (M/ M, 10 % valley definition) is achieved over the mass range of interest. The mass focus accuracy is verified by observing known ion masses from PFK. The mass resolution and focus are reestablished during the analytical period if there is an indication that they have changed. Typical indicators include the appearance of interfering ion mass peaks and chlorine isotope ratios that deviate by greater than 15 % from the theoretical values.

The operation of the HRGC/HRMS is evaluated every 12 hours by analyzing standard solutions containing PCDD/PCDF isomers. These solutions are used to demonstrate quantitative accuracy, chromatographic resolution, and the assignment and stability of the chromatographic elution windows. These solutions are also analyzed during the twelve hour analytical period if there is an indication that the sensitivity or chromatographic characteristics of the HRGC/HRMS system have changed. Typical indicators include a decrease in the area response of one or both of the isotopically labeled recovery standards, 1,2,3,4-TCDD and 1,2,3,7,8,9-HxCDD, or an increase in the width of the chromatographic peaks. Typically the area/height ratio of the chromatographic peaks must be less than 50.

The standard solution used to assure the quantitative accuracy of the analyses is one of the solutions used to generate the standard curve. The relative response factors for each chlorine congener class from this analysis must agree within 20 % of the relative response factor from the standard curve. If the relative response factors differ by greater than 20 %, corrective measures are initiated. Typical corrective measures include retuning the instrument and/or cleaning the ion source. If the relative response factors do not return to within 20 % of the values from the standard curve, a new standard curve is the prepared.

The standard solution used to evaluate the chromatographic resolution contains the specific PCDD/PCDF isomer of interest, for example 2,3,7,8-TCDD, and several of the closely eluting TCDD isomers. Adequate chromatographic resolution is obtained when 2,3,7,8-TCDD is resolved from the nearest eluting TCDD isomer with a valley that is no greater than 25 %. If the chromatographic resolution is insufficient, a portion of the column is removed or the column is replaced. The specific TCDD and TCDF isomers used to evaluate the isomer specificity are:

Column	2,3,7,8-TCDD	2,3,7,8-TCDF
60 M DB-5	1,2,3,7-TCDD 1,2,3,8-TCDD 1,2,3,9-TCDD	Not isomer specific
60 M SP-2330	Not isomer specific	2,3,4,7-TCDF 2,3,7,8-TCDF 2,3,4,8-TCDF
DB-Dioxin	1,2,3,7-TCDD 1,2,3,8-TCDD 1,2,3,9-TCDD	2,3,4,7-TCDF 2,3,7,8-TCDF 2,3,4,8-TCDF

The standard solution used to determine the chromatographic elution windows and to validate their accuracy contains the first and last eluting PCDD/PCDF isomer of each chlorine congener class. Correct elution window assignments require that both isomers for a particular congener class be contained within the elution window. If one or both of the isomers is not observed, corrective action is taken. The corrective action consists of verifying the carrier gas flow velocity and the temperature program of the gas chromatograph. Adjustments are made to the carrier gas flow velocity and/or the time windows as required. If the original elution windows cannot be reproduced, new windows are established.

2.4 Field and Method Blanks

Field and laboratory method blank analyses are routinely carried out to demonstrate that the sample shipping/storage containers, solvents, reagents, and glassware are free from contamination. The analyses of shipping/storage containers demonstrate that they are free from contamination and have not contributed to any detected levels of PCDD or PCDF isomers. The laboratory method blanks are used to demonstrate that the laboratory glassware, solvents, and reagents are free from contamination. If contamination is detected, all analyses are suspended until the origin of the contamination is determined.

2.5 Solvent Injections

Tridecane is injected into the HRGC/HRMS to demonstrate that PCDD/PCDF isomers are not being retained in the system and subsequently released during the injection of the sample extracts. It is also injected to verify that PCDD and PCDF isomers are not adsorbed onto the syringe from previous high level standards or sample extracts. The solvent injections are carried out periodically to screen for retention, especially after injections of sample extracts found to contain high levels of PCDDs and/or PCDFs.

If retention is observed, analyses are suspended and the origin of the contamination is determined. Typically the syringe is recleaned and the solvent injection is repeated. If retention is still observed, the syringe is replaced and the solvent injection repeated to demonstrate that the problem has been corrected.

2.6 Matrix Spikes

Whenever possible, samples of the matrix which do not contain PCDDs and PCDFs are spiked with known quantities of selected PCDD/PCDF isomers and analyzed to determine the quantitative accuracy of the analyses. The expected accuracy is a function of the matrix and the concentration, however, it is usually required that the measured value be within 20 % of the true value. Matrix spikes are also carried out to demonstrate the ability to achieve specific detection limits in given matrices.

2.7 Duplicate Analyses

Approximately one sample from each batch of 8-10 samples may be prepared and analyzed in duplicate. The expected agreement between duplicate analyses will depend upon the concentrations of the PCDDs and PCDFs, the levels of interferences, and the homogeneity of the samples. Agreement between duplicate analyses is typically within 30-50% for PCDD/PCDF levels that are > 50 ppt.

2.8 Chain-of-Custody

All samples are logged into the laboratory and stored following procedures defined by the National Enforcement Investigations Center (NEIC) for Contract Laboratory Program (CLP) Laboratories. The samples are delivered to TCT's St. Paul Laboratory and are immediately placed under the custody of Ms. Paula Krause, the Chemistry Division's Sample Custodian. Ms. Krause signs the custody sheet(s) (when provided) and logs the samples into the TCT Sample Manager Data System. The Sample Manager assigns a unique identification number to each sample which can be traced to the information supplied with the sample on the chain-of-custody form(s). After the samples are logged into the laboratory, they are stored in a locked facility until they are transported to the extraction laboratory. At this time they are placed in the custody of Mr. Wayne Askew or a designee. Mr. Askew maintains the samples and sample extracts in his custody until they are transferred to Mr. Charles Sueper or a designee. Mr. Sueper maintains custody of the sample extracts until they have been analyzed and the data are entered into the computer for data reduction. Upon completion of the program, the sample extracts and any remaining portions of the samples are either held or returned to the client. The typical holding time is a minimum of 60 days or as directed by the client.

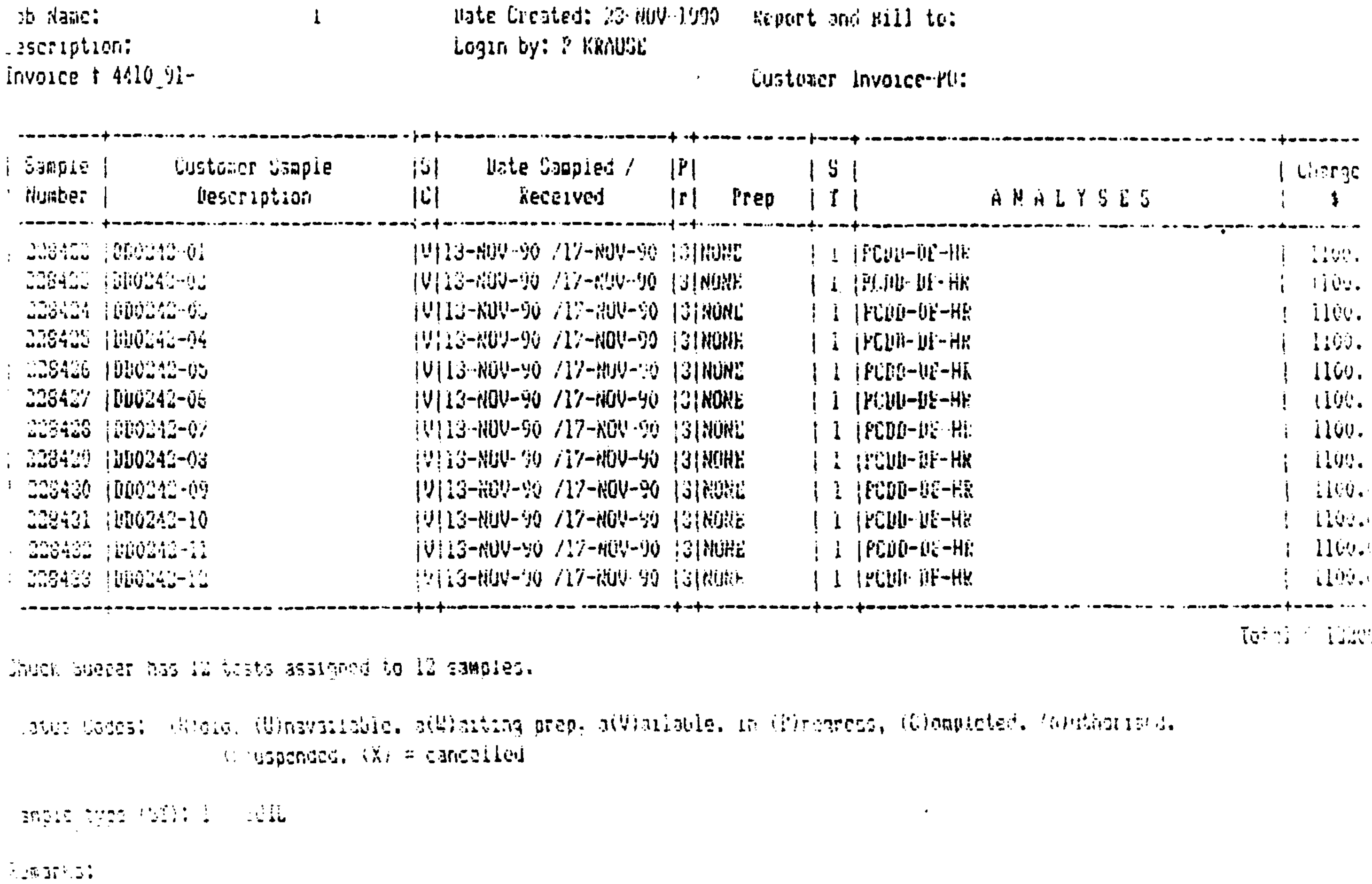
3.0 SAMPLE LOG-IN AND DATA TRACKING

Sample log-in and data tracking are an important part of the analysis process. Due to the ramifications of PCDD/PCDF analytical data, it is essential that samples can be traced during the analyses. TCT uses a VG Sample Manager to assign a unique log-in number to each sample and to track the status of the sample from the time it arrives at the laboratory until the final analytical results are reported to the client. The VG Sample Manager is based on a Digital Equipment Model 6310 VAX computer and has approximately 10 nodes for sample log-in. The system also includes three printers, a magnetic tape drive for data archival, and a data link to the chromatography and mass spectrometry data systems. Full chain-of-custody procedures are followed whenever documentation is supplied with the samples.

An example of the Sample Manager Log-in Sheet is shown in Figure 2. It includes the dates when the sample was collected, received at the laboratory, logged in, analyzed, and when the results are required. It also includes the specific analyses assigned to the sample, the name of the person responsible for the sample, and various administrative information.

The Extraction Worksheet is shown in Figure 3. It includes the sample log-in numbers, the date that the samples were processed, the specific reusable glassware that was used, the solvent lot numbers, and the analyte enrichment procedure reference. This information is also recorded in a bound laboratory notebook.

FIGURE 2



```
ob Name:                    1            Date Created: 23-NOV-1990   Report and Bill to:
escription:                              Login by: P KRAUSE
Invoice # 4410_91-                                                   Customer Invoice-PO:

 --------+---------------------------------+-+-----------------------+-+--------------+---+----------------------------------------------+-------
| Sample |      Customer Sample            |S|   Date Sampled /      |P|              | S |                                              | Charge
 Number  |        Description              |C|      Received         |r|    Prep      | T |                 A N A L Y S E S              |   $
 --------+---------------------------------+-+-----------------------+-+--------------+---+----------------------------------------------+-------
  228422 |DD0242-01                        |V|13-NOV-90 /17-NOV-90   |3|NONE          | 1 |PCDD-DF-HR                                    |  1100.
  228423 |DD0242-02                        |V|13-NOV-90 /17-NOV-90   |3|NONE          | 1 |PCDD-DF-HR                                    |  1100.
  228424 |DD0242-03                        |V|13-NOV-90 /17-NOV-90   |3|NONE          | 1 |PCDD-DF-HR                                    |  1100.
  228425 |DD0242-04                        |V|13-NOV-90 /17-NOV-90   |3|NONE          | 1 |PCDD-DF-HR                                    |  1100.
  228426 |DD0242-05                        |V|13-NOV-90 /17-NOV-90   |3|NONE          | 1 |PCDD-DF-HR                                    |  1100.
  228427 |DD0242-06                        |V|13-NOV-90 /17-NOV-90   |3|NONE          | 1 |PCDD-DF-HR                                    |  1100.
  228428 |DD0242-07                        |V|13-NOV-90 /17-NOV-90   |3|NONE          | 1 |PCDD-DF-HR                                    |  1100.
  228429 |DD0242-08                        |V|13-NOV-90 /17-NOV-90   |3|NONE          | 1 |PCDD-DF-HR                                    |  1100.
  228430 |DD0242-09                        |V|13-NOV-90 /17-NOV-90   |3|NONE          | 1 |PCDD-DF-HR                                    |  1100.
  228431 |DD0242-10                        |V|13-NOV-90 /17-NOV-90   |3|NONE          | 1 |PCDD-DF-HR                                    |  1100.
  228432 |DD0242-11                        |V|13-NOV-90 /17-NOV-90   |3|NONE          | 1 |PCDD-DF-HR                                    |  1100.
  228433 |DD0242-12                        |V|13-NOV-90 /17-NOV-90   |3|NONE          | 1 |PCDD-DF-HR                                    |  1100.
 ------------------------------------------+-+-----------------------+-+--------------+---+----------------------------------------------+-------
                                                                                                                                    Total $ 1320
Chuck Sueper has 12 tests assigned to 12 samples.

tatus Codes:  (H)old, (U)navailable, a(W)aiting prep, a(V)ailable, in (P)rogress, (C)ompleted, (A)uthorised,
              (S)uspended, (X) = cancelled

ample type (ST): 1 = SOIL

Remarks:
```

FIGURE 3

DIOXIN EXTRACTION WORKSHEET

CLIENT:

INVOICE #:

DATE EXTRACTION BEGAN:

DATE SUBMITTED TO GC/MS:

SAMPLE #	L OR g EXTRACTED	ACID BASE	SILICA	ALUMINA	CARBON	INT STD	CL-37 STD	REC STD
			Date: ______ Init:	Date: ______ Init:	Date: ______ Init:			
			Hexane Lot #:	2% MeCL₂/Hexane Batch #:	50% MeCL₂/Cyclo Batch #:			
				60% MeCL₂/Hexane Batch #:	75% MeCL₂ Batch #:			
					Benzene Lot #:			

EXTRACTION SOLVENTS: BENZENE LOT #________

HEXANE LOT #________

METHYLENE CHLORIDE LOT #________

ACID/BASE: SULFURIC ACID LOT #________

BASE BATCH #________

WATER LOT #:________

4.0 DIOXIN FACILITIES

4.1 Personnel

TCT has a qualified and experienced staff of professional and technical support personnel who are available to carry out PCDD/PCDF related programs. The principal technical resource is Dr. Fred L. DeRoos, an internationally recognized expert in the areas of high resolution mass spectrometry and PCDD/PCDF analyses. Prior to joining TCT, Dr. DeRoos was employed at Battelle's Columbus Laboratories where he established and managed their Mass Spectrometry and PCDD/PCDF Facilities. During the 10 years that he was at Battelle, he directed over 250 PCDD/PCDF programs for government and industrial clients. Sample matrices that have been analyzed under his direction at Battelle and at TCT include:

Soil
Wood
River Sediment
XAD-2 Resin (MM-5 Samplers)
Grapes
Fish
Milk
Pharmaceutical Products
Pesticides
Industrial Wastes
Askarel Fluids
Transformer Laminations
Fly Ash
Surface Wipes
Ink
Florisil
Pigments
Polyurethane Foam
Water
Paper
Lake Sediment
Carbon
Walnuts
Pork
Human Adipose
Pentachlorophenol
Herbicides
Sewage Sludge
Mineral Oils
Automotive Exhaust
Bottom Ash
Lubricating Oils
Solvents
Silica Gel
Polymer Resins

Dr. DeRoos is assisted in the conduct of PCDD/PCDF related programs by several task leaders. The sample extraction and analyte enrichment steps are coordinated by Ms. Chris Bremer and Ms. Jean Osterheim. Ms. Bremer has eight years of experience in sample extraction and analyte enrichment procedures and is presently the Manager of Laboratory Operations. Ms. Osterheim has over two years of laboratory experience and is the

Supervisor of the Organic Extraction Laboratory. The extractions and analyte enrichment steps are carried out by Mr. Wayne Askew, Mr. Steve McKelvey, Mr. Matt Hogenson, Mr. Bill Morrison, and Ms. Eileen Skok.

The HRGC/HRMS analyses are coordinated by Mr. Charles Sueper. Mr. Sueper has over eight years of mass spectrometry experience, most involving the determination of PCDDs and PCDFs. He is assisted by Mr. Steve Hannan, Mr. Dennis Daigle, and Mr. Mike Kurkowski. Mr. Hannan has over five years of experience in mass spectrometry, three of which were at Battelle. Mr. Daigle has approximately two years of experience in mass spectrometry and gas chromatography, while Mr. Kurkowski has approximately 1 year of experience.

Data reduction and report coordination are carried out under the direction of Dr. DeRoos and Mr. Sueper. All reports are subjected to a minimum of three reviews before being released to the client. The first review is carried out by Mr. Hannan. The results are then reviewed by Mr. Sueper and finally by Dr. DeRoos. Calculations are also spot checked by Mr. Wallace Nosek, Jr., the QA/QC Manager of the Chemistry Division. Mr. Nosek's responsibilities are described in the following section.

Mr. Wallace J. Nosek, Jr., the QA/QC Manager for the Chemistry Division, serves as the Quality Assurance Manager for all PCDD/PCDF programs carried out by TCT. Mr. Nosek has worked in the QA/QC area for the past 10 years and his experience helps to assure that the data are valid and of high quality. Mr. Nosek is responsible for evaluating the results from internal quality control samples and for reviewing all laboratory records pertaining to PCDD/PCDF programs. He also conducts periodic QA/QC checks by submitting blind duplicate and blank samples to the analytical team.

4.2 Extraction Laboratory

Twin City Testing Corporation has a dedicated dioxin laboratory for the preparation of samples. This laboratory includes hoods for sample extraction and column cleanup and refrigerated storage for samples, sample extracts, and standard solutions. It also includes adequate storage space for dedicated glassware, solvents, and reagents used for sample preparation. The TCT Chemistry/Environmental Building is a limited access facility that provides safety for the technicians as well as security for the samples. Due to the range of PCDD/PCDF levels that are encountered, TCT pays close attention to preventing cross contamination of samples. The majority of the glassware is disposable, however, records are maintained for each piece of reusable glassware so that its use can be documented: In addition, separate glassware are used for samples that are known to contain or suspected of containing high levels of PCDDs or PCDFs.

Most sample extracts can be processed using only conventional column chromatography cleanup steps. Typically two or three columns are used for analyte enrichment. Some extracts, however, contain interferences that cannot be removed by column chromatography. For these extracts, TCT applies either high performance liquid chromatography (HPLC) or gel permeation chromatography (GPC). The HPLC system consists of a Waters Model 600 pump, a Model 712 autosampler and a Model 990 photodiode array detector. The GPC system is an Analytical Bio-Chemistry (ABC) Laboratories, Inc. Model 1002B automated GPC. This unit can automatically provide analyte enrichment for batches of up to 23 crude extracts.

4.3 Gas Chromatography/Mass Spectrometry

TCT has two capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) instruments dedicated to PCDD/PCDF analyses. These instruments, a VG 70-VSE and a VG 70-SE, are interfaced to separate VG Model 11-250 data systems which provide instrument control and data acquisition. The instruments also include Hewlett Packard Model 5890 gas chromatographs which are fully controllable by the mass spectrometry data systems. The HRGC/HRMS mass spectrometers are operated in the electron impact ionization mode (34 ev) at a mass resolution of 9000-12000 (M/ M, 10% valley definition). This mass resolution is sufficient to resolve most of the common environmental compounds that can interfere with PCDD/PCDF analyses.

TCT also has three VG Trio 2 Quadrupole Mass Spectrometers which are available for PCDD/PCDF screening and for programs that do not require the ultratrace capabilities of the HRMS equipment. Each of the Trio 2 instruments includes a Hewlett Packard Model 5890A gas chromatograph and a dedicated VG Model 11-250 Data System. An example of the type of program that can be carried out using this instrumentation is the determination of 2,3,7,8-tetrachloro-dibenzo-p-dioxin (2,3,7,8-TCDD) in soil that has been previously characterized by other methods. The majority of the EPA analyses from the Times Beach, MO sites were carried out by low resolution quadrupole mass spectrometry in order to minimize the cost of the analyses and to achieve the required sample analysis capacity.

The quadrupole mass spectrometers are controlled and the data are acquired and processed using dedicated data systems. Each of the quadrupole mass spectrometers is interfaced to a VG Model 11-250J Data System which is linked to the Sample Manager via DECNET. The DECNET system is also linked to several personal computers which are used for reporting and for data transfer. Data from each of the mass spectrometry data systems can also be processed off line so that the maximum utilization of the mass spectrometers is assured. All of the raw data from the analyses are archived on magnetic tape for a period of one year or as directed by the client.

4.4 Capillary Columns

Several types of capillary columns are used by TCT for PCDD/PCDF analyses. The specific type of column that is selected for an analysis is dependent upon the particular requirements of the analyses. For example, a 60 M DB-5 fused silica capillary column is used for the determination of the congener class concentrations. This column offers good separation of the congener classes and provides isomer specific analyses for 2,3,7,8-TCDD, as well as the other "2,3,7,8" substituted PCDD isomers. A 60 M SP-2330 fused silica capillary column is used to provide confirmation of the specific levels of 2,3,7,8-TCDF. A 60 M DB-Dioxin column is used to provide specific levels of both 2,3,7,8-TCDD and 2,3,7,8-TCDF. Other capillary columns, such as CP Sil-88, CP Sil-43, and DB-225 are also available when required.

4.5 Sample Capacity

The approximate number of samples of representative matrices that can be processed each week is listed below in Table 4. The analysis capacity assumes that each sample extract will be analyzed for the specific "2,3,7,8" substituted PCDDs and PCDFs as well as the chlorine congener concentrations of the Cl_4 through Cl_8 classes. Analysis capacity for limited analyses, eg. only 2,3,7,8-TCDD, is higher. In addition, rapid turn around analyses can also be provided by split shift operation. The analytical and extraction/cleanup capacities listed in Table 4 are for actual samples. The required analytical standards, method blanks, native spiked samples, and duplicates, are also analyzed during the run sequences.

TABLE 4. SAMPLE CAPACITY PER WEEK BY MATRIX TYPE

Matrix	Extraction/Cleanup	Analysis
Water	50	60
Sediment (Soxhlet Extraction)	40	60
Fish	30	60
Aquatic Life	30	60
Soil (Soxhlet Extraction)	40	60
Soil (Shake Extraction)	60	60
Ash	50	60
Leachate	30	60
XAD-2 Resin	40	60
Surface Wipes	40	60

The capacities listed in Table 4 are not cumulative.

5.0 REPORTING

The Final Report can be customized to contain whatever level of documentation that the client requires. The standard Final Report includes a brief description of the analytical methodology and tabular summaries of the analytical results. The table(s) contain the chlorine congener class concentrations and specific isomer concentrations requested in the scope of work, as well as single isomer detection limits for classes or isomers which were not detected. The results from QA/QC analyses, including duplicates, method blanks, and laboratory spikes, and the initial calibration data are also included.

Floppy disk copies of the Reports, including tables, are available upon request. The format for the disk copies can be ASCII, Word Perfect V 4.2 or V 5.0, or standard Data Interchange Format as used on IBM PCs and IBM Clones. If extremely rapid analytical turnaround is required, electronic data transfer (modem-to-modem) is also available.

Hard copies of the single-ion-current chromatograms are normally not included in the Final Report, however, these can be made available upon request. There is no additional charge for a single copy, but multiple copies are charged at $0.25/page.

APPENDIX A

SELECTED RESUMES

RESUME

NAME:	Fred L. DeRoos
DATE OF EMPLOYMENT:	1987
EDUCATION:	Bachelor of Science - Chemistry University of South Dakota - Vermillion, SD, 1969 Doctor of Philosophy - Analytical Chemistry University of Nebraska - Lincoln, NE, 1976

EXPERIENCE:

Present:
VICE PRESIDENT, Analytical Chemistry Department, Twin City Testing Corporation, St. Paul, MN.
Responsibilities include business planning and financial/technical management of the Chemistry Department. This department provides organic and inorganic analytical services to industrial and government clients.

1987 to 1990:
MANAGER, Organic Chemistry Department, Twin City Testing Corporation, St. Paul, MN.
Responsibilities include business planning and financial/technical management of the Organic Chemistry Department. This department, which includes gas chromatography, liquid chromatography, mass spectrometry, and infrared spectrometry, performs a wide range of routine EPA analyses as well as state-of-the-art analyses using methods developed in-house. Specialized areas include ultratrace analyses for polyaromatic hydrocarbons, polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans.

1977 to 1987:
SENIOR RESEARCH SCIENTIST & MANAGER, Mass Spectrometry, Battelle Columbus Laboratories, Columbus, OH.
Responsibilities included financial and technical management of the mass spectrometry group comprised of approximately twenty technical staff members and nine mass spectrometers. Responsible for proposal preparation, methods development, program management, and reporting on numerous industrial and government projects. Also established high resolution mass spectrometry as an analytical tool at Battelle and was responsible for designing and setting up a dedicated dioxin/furan laboratory.

1976 to 1977:
ASSISTANT PROFESSOR & MANAGER, Mass Spectrometry, University of Pennsylvania and Children's Hospital, Philadelphia, PA.
Responsibilities included the financial and technical management of mass spectrometry facility comprised of two staff members and four mass spectrometers. Worked with the University's "Laboratory for Research on the Structure of Matter" and the Chemistry Department to help solve original research problems. Also worked on projects involving metabolic defects with medical staff of Children's Hospital. Taught graduate and undergraduate level courses covering the principles and applications of mass spectrometry.

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PROFESSIONAL MEMBERSHIPS:

American Chemical Society

American Society for Mass Spectrometry

International Association of Environmental Analytical Chemistry

Air & Waste Management Association

American Water Works Association

PUBLICATIONS:

Gross, M.L., E. Chiu, D. Pokorny, and F.L. DeRoos, 1976, Regiospecificity for Water Elimination, A Mass Spectral Study of 1-Tetralol and 2-Tetralol, Org. Mass Spectrom., 12:55.

Gross, M.L. and F.L. Deroos, 1976, Ionic Cycloreversion Reactions in Tetralin Derivatives, Structure of C_8H_{80} Ions from 1-Tetralol. J. Am. Chem. Soc., 98:7128.

Gross, M.L., F.L. DeRoos, and M.K. Hoffman, 1977, Ionic Dehydration Reactions: The Effect of Location of the Activated Complex on Kinetic Energy Release, Org. Mass Spectrom., 12:258.

DeRoos, F.L., 1980, Compilation of Analytical Methods to Detect Dioxins, Report to U.S. Environmental Protection Agency on Task 2 on Contract 68-03-2659.

Shafer, K.H., W.M. Cooke, F.L. DeRoos, R.J. Jakobsen, O. Rosario, and J.D. Mulik. 1982. WCOT Capillary Column GC/FT-IR and GC/MS for Identifying Toxic Organic Pollutants, Journal of Applied Spectroscopy.

Carraher, C.E., A.L. Gasper, M.L. Trombley, F.L. DeRoos, D.J. Giron, G.G. Hess, and K.M. Casberg. 1984. Polymer Synthesis, Edited by W. Culbertson and C.V. Pittman, Plenum.

Cooke, W.M., F.L. DeRoos, and B. Rising, 1984, Dioxin Collection from Hot Stack Gas Using Source Assessment Sampling System and Modified Method 5 Trains--An Evaluation. Incineration and Treatment of Hazardous Waste, EPA-60019-84-015.

Rouse, T.O., R.E. Koch, and F.L. DeRoos, Formation of PCDF and PCDD in Electrical Discharges. IEEE Transactions on Electrical Insulation, 1183, 28 May, 1988.

DeRoos, F.L., S.C. Watson, S.E. Miller, J.E. Tabor, J.E. Hatchel, and R.G. Lewis, Evaluation of a High-Volume Air Sampler for Collection and Retention of Polychlorinatd Dibenzo-p-dioxins and Polychlorinated Dibenzo Furans. Submitted to Analytical Chemistry, August 1987.

PRESENTATIONS:

DeRoos, F.L. and R.L. Foltz, 1979, An HPLC/MS Study of Some Polycyclic Aromatic Hydrocarbons, Carbamates and Hydrazines. National Meeting of American Society for Mass Spectrometry, June 6, Seattle, WA.

DeRoos, F.L. and L.E. Slivon, 1980, Improved Signal Averaging System for High-Resolution Selected Ion Monitoring Detection of Tetrachlorodibenzo-p-dioxins (TCDDs). National Meeting of American Society for Mass Spectrometry, May 29, New York, NY.

Roy, T.A., F.L. DeRoos, B.J. Hidy, and C.C. Howard, 1980, Application of Combined HPLC/MS to the Analysis of Selected Cephalosporins and Penicillins. National Meeting of the American Society for Mass Spectrometry, May 29, New York, NY.

DeRoos, F.L. and D.G. Aichele, 1980, Ultratrace Analyses for Specific Tetrachlorodibenzo-p-dioxin Isomers (TCDDS). 24th Conference on Analytical Chemistry in Energy Technology, October 7-9, Gatlinburg, TN.

Nishioka, M.G., F.L. DeRoos, and B.A. Petersen, 1982, Mass Spectral Characterization of Diesel Engine Particulate Emission Extracts. National Meeting of the American Society for Mass Spectrometry, Honolulu, HI.

DeRoos, F.L., D.G. Aichele, J.E. Tabor, and M.E. Larson, 1982, High Pressure Liquid Carbon Dioxide Extraction of Tetrachlorodibenzo- p-dioxins (TCDDs). National Meeting of the American Society for Mass Spectrometry, Honolulu, HI.

Cooke, W.M., F.L. DeRoos, B. Risking, M. Jackson, L. Johnson, and R. Merrill, 1983, Dioxin Sampling from Hot Stack Gas Using Source Assessment Sampling System (SASS) and Modified Method Five (MM5) Trains-An Evaluation. Ninth Annual Research Symposium on Land Disposal, Incineration and Treatment of Hazardous Waste, March, Ft. Mitchell, KY.

DeRoos, F.L., B. Rising, W.M. Cooke, D.G. Aichele, R. Heffelfinger, M.D. Jackson, R.G. Merrill, and L.D. Johnson, 1983, TCDD Recovery from Hot Flue Gas Streams. 1983 Federation of Analytical Chemistry and Spectroscopy Societies Meeting, Philadelphia, PA.

Aichele, D.G., F.L. DeRoos, J.E. Tabor, and M.E. Larson. 1983 Federation of Analytical Chemistry and Spectroscopy Societies Meeting, Philadelphia, PA.

Cook, W.M., J.E. Gebhart, P.J. Mondron, F.L. DeRoos, and L.E. Slivon, 1983. An Update on Analytical Methods for Polychlorinated Biphenyls (PCB), Polychlorinated Dibenzofurans (PCDF), and Polychlorinated Dibenzo-p-dioxins (PCDD). Electric Power Research Institute PCB Seminar, Atlanta, GA.

DeRoos, F.L., D.L. Miller, and J.E. Tabor, 1983, Modification of an AEI MS-9 Mass Spectrometer into a Hydrbrid MS/MS. National Meeting of the American Society for Mass Spectrometry, Boston, MA.

DeRoos, F.L., W.M. Cooke, G. Addis, R. Komai, and J. Guertin, 1984, Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans in Dielectric Fluids. EPRI PCB Seminar, Palo Alto, CA.

DeRoos, F.L. and D.G. Aichele, 1985, Detection and Quantification of Pyridostigmine Using Fast Atom Bombardment Mass Spectrometry. Scientific Conference on Chemical Defense Research, Aberdeen Proving Ground, MA.

Miller, D.L., F.L. DeRoos, S.C. Watson, C.V. Sueper, J.E. Tabor, and J.A. Hatchel, 1985, Analysis of Halogenated Dibenzo-p-dioxins and Dibenzofurans. National Meeting of the American Society for Mass Spectrometry, San Diego, CA.

DeRoos, F.L., W.M. Cooke, B. Rising, and W.H. Martin, 1985, Simulated Building Fire Study Using Retrofilled Transformer Dielectric Coolant. 5th International Meeting of Polychlorinated Dibenzo-p-dioxins and Related Compounds, Bayreuth, West Germany.

Rouse, T.O., R.E. Koch, and F.L. DeRoos, 1985, Formation of PCDFs and PCDDs in Electrical Discharges, EPRI PCB Seminar, Seattle, WA.

Gordon, S.M., M. Miller, F.L. DeRoos, and J. Guertin, 1985, Measurement of PCDF/PCDD in Utility Equipment, EPRI PCB Seminar, Seattle, WA.

Cooke, W.M., F.L. DeRoos, B. Risking, and W.H. Martin, 1985, Safety of Non-PCB Reclassified Transformers in Fire Incidents - A Building Scale Engineering Study, EPRI PCB Seminar, Seattle, WA.

DeRoos, F.L., S.C. Watson, J.A. Hatchel, and J.R. Kominsky, 1985, Polychlorinated Dibenzofuran Contamination of the State Highway Department Office Building in Santa Fe, New Mexico as a Result of a PCB Transformer Malfunction, EPRI PCB Seminar, Seattle, WA.

DeRoos, F.L., S.C. Watson, and D.G. Aichele, 1986, Artifact Problems in PCDD/PCDF Analyses, National ACS Meeting, Anaheim, California.

DeRoos, F.L., S.E. Miller, J.E. Tabor, R.G. Lewis, and N.K. Wilson, 1986, Evaluation of EPA High Volume Air Sampler for Collection and Retention of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans. EPA/APCA Symposium Measurement of Toxic Air Pollutants, Raleigh, NC.

DeRoos, F.L., 1987, Screening for Polychlorinated Biphenyl Ethers and Polychlorinated Biphenylenes while Quantifying Polychlorinated Dioxins and Furans. 35th ASMS Conference on Mass Spectrometry and Allied Topics, Denver, CO.

DeRoos, F.L., 1987, Analysis of Fog Samples for PCDD and PCDF. Seventh International Symposium of Chlorinated Dioxins and Related Compounds, Las Vegas, NV.

DeRoos, F.L., 1987, Acquisition and Interpretation of Environmental Data. The Center for Professional Advancement in East Brunswick, NJ.

DeRoos, F.L, 1987, Ambient Air Monitoring for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibensofurans. The Ninth Annual Midwest Conference on Environmental Laboratory Technology, St. Paul, MN.

DeRoos, F.L., 1988, Acquisition and Interpretation of Environmental Data. The Center for Professional Advancement in East Brunswick, NJ.

DeRoos, F.L., R. Houck and M.K.L. Bicking, 1989, Evaluation of Supercritical Fluid Extraction for Determination of PCDDs and PCDFs in Selected Matrices. Twelfth Annual EPA Conference on Analysis of Pollutants in the Environment, Norfolk, VA.

Bicking, M.K.L. and F.L. DeRoos, 1989, Novel Analyte Enrichment Method for the Determination of PCDDs and PCDFs in Complex Matrices. Twelfth Annual EPA Conference on Analysis of Pollutants in the Environment, Norfolk, VA.

DeRoos, F.L., 1989, Description of an Optimum Method for the Determination of PCDF/PCDD in Utility Fluids. 1989 Electric Power Research Institute PCB Conference, San Diego, CA.

DeRoos, F.L. and M.K.L. Bicking, 1989, Supercritical Fluid Extraction for Determination of PCDDs and PCDFs in Selected Matrices. Ninth International Symposium on Chlorinated Dioxins and Related Compounds, Toronto, Canada.

DeRoos, F.L., 1989, Acquisition and Interpretation of Environmental Data. The Center for Professional Advancement in East Brunswick, NJ.

RESUME

NAME: Susan D. Max

DATE OF EMPLOYMENT: 1990

EDUCATION:

Bachelor of Arts, Biology/Chemistry - 1974
St. Olaf College, Northfield, MN

Medical Technology, ASCP - 1975
Metro Medical Center, Minneapolis, MN

Master of Public Health Candidate, Environmental Health/Toxicology
University of Minnesota, Minneapolis, MN

EXPERIENCE:

June 1990 to Present:
MANAGER, PROJECT COORDINATION AND DEVELOPMENT, Twin City Testing Corporation, St. Paul, MN.
Lead member of a team of TCT chemists whose primary responsibilities are customer service and project management. Responsible for the coordination of large multi-faceted laboratory projects with special requirements for scheduling, method development, and/or reporting. Also coordinates all laboratory proposal activity and project plan development.

1989 to 1990:
ORGANIC LABORATORY MANAGER, PACE, Inc., Minneapolis, MN.
Responsible for 32 chemists and technicians involved in analysis of environmental and industrial hygiene projects by GC, GC/MS, and HPLC using EPA and NIOSH methodologies. Coordinated analysis of samples for the EPA Contract Laboratory Program (CLP) involving strict QA/QC measures to produce legally defensible data for Superfund project sites.

1988 to 1989:
ASSISTANT DIRECTOR OF ANALYTICAL SERVICES, PACE, Inc., Minneapolis, MN.
Responsible for development of laboratory Quality Assurance and Client Services Departments.

1987 to 1988:
SUPERVISOR, GC/MS LABORATORY, PACE, Inc., Minneapolis, MN.
Supervised GC/MS laboratory performing analysis of environmental samples for BNA and volatile compounds using EPA methodologies. Responsible for the successful completion of projects for the EPA Contract Laboratory Program (CLP).

1984 to 1987:
CHEMIST, TOXICOLOGY DEPARTMENT, Metro Medical Center, Minneapolis, MN.
Responsible for qualitative and quantitative drug testing using GC and GC/MS instrumentation. Performed rush analysis of biological fluids for drug overdose screening.

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1974 to 1984:
MEDICAL TECHNOLOGIST, Metro Medical Center, Minneapolis, MN. Performed clinical laboratory analyses of biological samples using chemistry, hematology, microbiology, and serology methods. Seven years of supervisory responsibilities for a staff of 22 professionals. Coordinated the activities of several work groups on a multi-departmental level.

PROFESSIONAL MEMBERSHIPS:

Minnesota Chromatography Forum

Minnesota Mass Spectrometry Discussion Group

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RESUME

NAME: R. Alan Doughty

DATE OF EMPLOYMENT: 1991

EDUCATION:

Bachelor of Science - Chemistry
Purdue University - 1961

Master of Science - Organic Chemistry
University of Minnesota - 1963

Ph.D. - Physical Organic Chemistry
University of Miami - 1967

Master of Business Administration
Roosevelt University - 1978

EXPERIENCE:

Present:
DIRECTOR-LABORATORY OPERATIONS, Twin City Testing Corporation, St. Paul, MN.

1988 to 1991:
LABORATORY DIRECTOR, RMT, Inc.
Managed the laboratory and supervised a staff of 50 employees engaged in environmental analyses; developed a new marketing plan; wrote and implemented a new quality assurance plan; designed and oversaw construction of a new laboratory; and wrote and implemented a new laboratory health and safety plan.

1984 to 1988:
DIRECTOR, International Technology Corporation
As Director, Field Services, supervised 50 employees in New Jersey, Tennessee, and California offices. As Laboratory Director, supervised a staff of 50 employees analyzing hazardous wastes, ground water samples, and plant discharges. Implemented a marketing program, computerized data reduction from laboratory instrumentation, implemented a computerized sample tracking system, increased instrument utilization, and designed and installed a quality assurance program.

1984:
PRINCIPAL AND LABORATORY DIRECTOR, Western Technologies, Inc.
Investigated opportunities to add environmental engineering and analysis as a new business for the firm; managed the laboratory and consulted with clients on environmental concerns. Wrote and implemented a laboratory quality assurance program; developed a marketing program.

1982 to 1984:
PROJECT MANAGER, The Bionetics Corporation
Responsible for a US EPA contract which involved managing chemists and marine biologists to collect and analyze fish, water, and sediments from the Great Lakes utilizing the facilities and equipment of the US EPA Central Regional Laboratory in Chicago. Wrote a quality assurance plan to meet EPA requirements; developed methods to measure productivity; and wrote proposals for government projects.

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1977 to 1982:
DIRECTOR, Scientific Resources, Quaker Oats Company
Administrative and technical responsibility for analytical chemistry, microbiology, biochemistry, nutrition, flavor chemistry, computer/statistical services, and library services. Developed computerized project cost accounting system; developed program to document computer programs; and developed GLP program.

1974 to 1977:
MANAGER, Analytical and Stability Services, Abbott Laboratories
Provided production and R & D with support in analytical chemistry, sterility testing, bioassay, and product stability.

1972 to 1974:
LABORATORY MANAGER, Kitchens of Sara Lee
Supervised 15 people in chemistry and microbiology in support of production, R & D, and quality assurance. Initiated a program to obtain data on all products for nutrition labeling.

1967 to 1972:
CHEMIST, A.E. Staley Manufacturing Company
Developed natural and synthetic polymers for wet-end applications, size-press coatings, and paper coatings. Explored characteristics of carbohydrate complexes and soybean oil derivatives.

RESUME

NAME: Charles V. Sueper

DATE OF EMPLOYMENT: 1988

EDUCATION: Bachelor of Science - Chemistry
University of Nebraska, Lincoln, NE - 1983

EXPERIENCE:

April 1990 to Present:
SUPERVISOR-HIGH RESOLUTION MASS SPECTROMETRY, Twin City Testing Corporation, St. Paul, MN.
Responsibilities include supervision of GCMS staff, development and updating of analytical methods, scheduling of sample priority, proposal preparation, report review, project management, and dioxin method troubleshooting. Also includes training new staff members, financial projections, and general problem solving.

May 1989 to April 1990:
SCIENTIST V, Twin City Testing Corporation, St. Paul, MN.
Responsibilities include operation and maintenance of high resolution mass spectrometry systems, project management, proposal preparation, report preparation, and data review. Also includes data reduction, instrument scheduling, and dioxin troubleshooting.

June 1988 to May 1989:
SCIENTIST III, Twin City Testing Corporation, St. Paul, MN.
Responsibility included operation and maintenance of high resolution mass spectrometer systems (VG 70-E and VG 70-SE) for the determination of ultratrace levels of PCDDs and PCDFs. Also includes evaluation of mass spectrometry data, reduction of data, and preparation of summary reports.

1984 to 1988:
RESEARCHER, Battelle Memorial Institute, Columbus, OH.
Operated and maintained a VG 70-E high resolution mass spectrometer for the determination of PCDDs and PCDFs. Managed projects, reduced mass spectral data, and prepared reports for clients. Operated and maintained a VG 70-SEQ mass spectrometer for research purposes including amino acid sequencing and development of continuous flow FAB probe. Also maintained stock of spare parts for the mass spectrometry facility and conducted training sessions in the operation of high resolution mass spectrometers.

1982 to 1984:
MASS SPECTROMETRIST, University of Nebraska, Department of Chemistry, Lincoln, NE.
Operated and performed basic maintenance of high resolution mass spectrometer

PROFESSIONAL MEMBERSHIPS:
American Society for Mass Spectrometry
Phi Lambda Upsilon Chemistry Honorary
American Chemical Society

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PUBLICATIONS AND PRESENTATIONS: DeRoos, F.L., Sueper, C.V., and McCarty, H.B., 1989, Evaluation and Optimization of EPA Method 1613, Seattle, WA.

Miller, D.L., DeRoos, F.L., Watson, S.C., Sueper, C.V., Tabor, J.E., and Hatchel, J.A., 1985, Analysis of Halogenated Dibenzo-p-dioxins and Dibenzofurans, National Meeting of the American Society for Mass Spectrometry, San Diego, CA.

RESUME

NAME: Steven W. Hannan

DATE OF EMPLOYMENT: June 1988

EDUCATION:

Bachelor of Science with Distinction - Medical Technology
The Ohio State University, College of Medicine, Columbus, Ohio - 1986

Bachelor of Music - Theory and Composition
The Ohio State University, College of the Arts, Columbus, Ohio - 1982

EXPERIENCE:

<u>1988 to Present</u>:
SCIENTIST, Analytical Chemistry Department, Twin City Testing Corporation, St. Paul, MN.
Analysis of environmental samples by GC/MS. Preparation of reports and project organization for projects related to PCDD/PCDF and other environmental samples.

<u>1984 to 1988</u>:
RESEARCH TECHNICIAN, Analytical and Structural Chemistry, Battelle Memorial Institute, Columbus, OH.
Experience in processing environmental samples for PAH analysis, including extraction, concentration, open column chromatography, and preparation of standards. Analysis experience with Finnigan 3200 and 4500 GC/MS systems and INCOS software.

<u>1979 to 1981</u>:
OPERATOR, UVONICS Co., Inc.
Electrochemical plating facility for production of printed circuit boards. Responsibilities included titrations, maintenance, and QC of electroplating solutions.

PRESENTATIONS AND SELECTED STUDIES:

Chuang, J.C.; Hannan, S.W.; and Wilson, N.K., "Field Comparison of Polyurethane Foam and XAD-2 Resin for Air Sampling for Polynuclear Aromatic Hydrocarbons." Environ. Sci. Technol., Vol. 21, No. 8, 1987.

Hannan, S.W., and Roberts, W.H., "Digestion of Lyophilized Tissue by Nitric Acid and Hydrogen Peroxide for the Fluorometric Determination of Selenium." Honors Research Final Report presented to the OSU School of Allied Medical Professions Honors Committee, May 1986.

Chuang, J.C.; Hannan, S.W.; and Koetz, J.R., "Stability of Polynuclear Aromatic Compounds Collected from Air on Quartz Fiber Filters and XAD-2 Resin." EPA Contract 68-02-4127, 1986.

Chuang, J.C.; Hannan, S.W.; and Cooke, W.M., "Chemical Characterization of Indoor Air Particles and Semi-volatile Organic Matter from Coal and Wood Combustion in Xuan Wei, China." EPA Contract 68-02-3745, 1985.

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RESUME

NAME: Wayne Askew

DATE OF EMPLOYMENT: 1988

EDUCATION: Northeast Metro Technical Institute
White Bear Lake, MN, 1986-87

EXPERIENCE:

1990 to Present:
CHEMICAL TECHNICIAN III, Twin City Testing Corporation, St. Paul, MN.
Responsibilities include sample preparation for dioxin analysis in soil, water, sludge, filters, and resin; all health and safety issues and concerns in the dioxin laboratory; training of new technicians; and light laboratory maintenance.

1988 to 1990:
CHEMICAL TECHNICIAN II, Organic Chemistry Department, Twin City Testing Corporation, St. Paul, MN.
Duties included sample preparation for dioxin analysis in soil, water, sludge, filters, and resin; all health and safety issues and concerns in the dioxin laboratory; and light laboratory maintenance.

1986 to 1987:
LABORATORY TECHNICIAN, Northeast Metro Technical Institute, White Bear Lake, MN.
Responsibilities included analyzing student experiments for accuracy, mixing solutions for student use, designing programs for monthly student progress reports, and data input.

1981 to 1986:
INVENTORY CONTROLLER, Diversified Products, Inc., Wyoming, MN.
Responsibilities included purchasing and disbursing 300,000 parts and accessories for fiberglass boat manufacturer, monitoring warranty requirements, and performing customer and client service.

1975 to 1980:
WORKING FOREMAN, Trailer Electric Services, North St. Paul, MN.
Responsibilities included wiring trucks and automobiles for electrical accessories, purchasing and maintaining inventory, and supervising and training five employees.

1971 to 1975:
WORKING FOREMAN, Midwest Mobile Home Supply & Snowmobile Specialties, St. Paul, MN.
Responsibilities included shipping and receiving $250,000 to $750,000 a month in parts and accessories, and supervising ten employees.

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RESUME

NAME: Patricia M. McClintock

DATE OF EMPLOYMENT: September 1991

EDUCATION: B.A. - Chemistry
Monmouth College, Monmouth, IL - 1985

EXPERIENCE:

Present:
QUALITY CONTROL SUPERVISOR, Twin City Testing Corporation, St. Paul, MN

January to September 1991:
ASST. QUALITY CONTROL COORDINATOR, RMT, Inc., Madison, WI
Statistical analysis of laboratory control sample, spike, and duplicate recoveries for establishing control limits; inorganic and organic contract laboratory program (CLP) data review; statistical analysis of non-conformance reports; tracking and maintaining standard operating procedures (SOP) and analytical method files.

1989 to 1991:
INORGANIC DOCUMENTS OFFICER, RMT, Inc., Madison, WI
CLP reportables and data packages; formatting and putting into place SOPs and analytical methods; secondary analyst for metals section; familiar with EPA 600 series, SW-846 3rd Edition, and CLP protocol. Software used: Telecation Associates, SMARTLOG and CLP MODULE, WordPerfect 5.1.

1988 to 1989:
ANALYST, RMT, Inc., Madison, WI
Performed trace metal analysis of environmental samples using Graphite Furnace AA and Plasma II ICP. Instruments operated: Perkin Elmer 5100 graphite furnace/Zeeman, Perkin Elmer 5000 graphite furnace, Perkin Elmer plasma II ICP.

1987 to 1988:
CHEMIST, Sipi Metals Corp., Chicago, IL
Analyzed samples for precious metals (gold, palladium, platinum) in foundry samples using a Perkin Elmer 5000 flame AA. Duties included sample preparation and analysis.

1985 to 1987:
CHEMIST, I-Chem Research, Inc., Hayward, CA
Performed trace metal analysis (flame AA and ICP) and various wet chemistry procedures of waste waters (TSS, COD, TOC, oil and grease, phenolics, cyanide). Duties included training and supervising laboratory technicians.

PROFESSIONAL MEMBERSHIPS: American Chemical Society

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APPENDIX B

RELATED EXPERIENCE

RELATED EXPERIENCE

1. Determination of PCDDs and PCDFs Produced by PCB Fires

Directed approximately 25 government and industrial programs involving the determination of PCDDs, PCDFs, and PCBs produced as a result of catastrophic failures of electrical utility equipment. These programs involved the analysis of dielectric fluids, construction materials, soot and ash, surface wipes, and ambient air samples. The detection limits were at the parts-per-trillion (pg/g) level for the solid and liquid samples and at the parts-per-quadrillion (fg/g) level for the air samples. Programs have included the Binghamton State Office Building in Albany, NY., the One Market Plaza Building in San Francisco, CA., the State Highway Department Building in Santa Fe, NM., as well as others in Florida, Oklahoma, Massachusetts, Tennessee, Canada, and France.

2. Validation of the Modified Method 5 Train

Directed the analytical portion of three U.S. EPA programs to evaluate the Modified Method 5 (MM5) sampling train for the collection of PCDDs and PCDFs. Studies were carried out at a pilot plant and at an actual operating incinerator. As part of the study, designed a dynamic gas phase spiking system which was used to introduce known levels of PCDD/PCDF isomers into the train during sampling.

3. Validation of the ASTM/ASME Sampling and Analytical Protocol

Participated on a panel to develop the ASTM/ASME analytical protocol for the determination of PCDD/PCDF isomers. Also directed the analytical portion of a U.S. EPA study to evaluate the protocol at a working waste incinerator.

4. Evaluation of Ocean Incineration of Hazardous Waste

Directed the analytical portion of a U.S. EPA program involving the determination of PCDDs and PCDFs produced from the ocean incineration of hazardous wastes.

5. PCB Trial Burn

Participated in a program involving the design of a TSCA test burn of a mobile hazardous waste incinerator. Directed the analytical portion of the program involving the analysis of Method 5 and Modified Method 5 samples for PCDDs and PCDFs. The results of the study were included in the permit application.

RELATED EXPERIENCE (Continued)

6. Determination of PCDD and PCDF Levels in Effluent Streams From a Carbon Reactivation System

Directed and participated in several programs to determine the levels of PCDD/PCDF that were emitted from a fluidized bed carbon regeneration system. Also evaluated the effectiveness of an afterburner to decrease the emitted levels. Study involved the analyses of virgin carbon, spent carbon, reactivated carbon, scrubber water, cyclone fines, and drinking water. Stack gas samples were also collected and analyzed using the Modified Method 5 sampling train.

7. Determination of PCDD and PCDF in Soil Samples

Directed approximately 50 programs in support of the U.S. EPA's Special Analytical Services (SAS) program to determine the levels of PCDD/PCDF in soil samples from contaminated areas in various parts of the United States. Also developed much of the High Resolution Mass Spectrometry protocol used for these analyses.

8. Evaluation of a High Volume Air Sampler for the Collection of PCDDs and PCDFs

Directed 5 U.S. EPA programs to evaluate a high-volume air sampler consisting of a fiber glass filter and a polyurethane foam adsorbent for the collection of PCDDs and PCDFs in ambient air. The study used a dynamic gas phase spiking system to introduce known levels of PCDDs and PCDFs into the sampler during a 24 hour sampling period. Also determined the feasibility of using other adsorbents for collecting the PCDD/PCDF.

9. Determination of PCDDs and PCDFs in Ambient Air

Prepared ambient air sampling equipment and analyzed samples for PCDD and PCDF levels in ambient air. Samplers were spiked with known levels of selected labeled PCDD/PCDF isomers prior to sampling which were used as a quality assurance measure to verify the performance of the sampling equipment.

10. Determination of PCDD and PCDF in Pentachlorophenol

Developed an analytical method to determine the levels of PCDD and PCDF in samples of commercial grade pentachlorophenol (PCP). Also evaluated an extraction process to prepare PCDD/PCDF free PCP. Have carried out several programs for industrial clients to determine levels of PCDDs and PCDFs in commercial PCP products.

RELATED EXPERIENCE (continued)

11. Determination of 2,3,7,8-TCDD in Pharmaceutical Products

Developed an analytical method to determine the level of 2,3,7,8-tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD) in a topical fungicide. The limit-of-detection for the method was approximately 1 part-per-trillion.

12. Determination of PCDDs and PCDFs in Food Products

Developed analytical methods to measure the levels of PCDDs and PCDFs in food products. Programs involved determining if PCDDs and PCDFs were transferred into food crops exposed to contaminated sludge. Products included grapes, walnuts, and corn. The detection limits were in the low parts-per-trillion.

13. Determination of PCDDs and PCDFs in Fish

Directed two U.S. EPA programs and several industrial programs involving the measurement of PCDD and PCDF levels in fish. Programs involved method development resulting in parts-per-trillion detection limits. Also involved the development of techniques to identify environmental contaminants that interfere with PCDF analyses by producing false positives.

14. Determination of PCDDs and PCDFs in Combustion Related Products

Directed approximately 10 programs involving the characterization of combustion related products for levels of PCDDs and PCDFs. Matrices included bottom ash, fly ash, leachate, and stack gases.

15. Measurement of PCDDs and PCDFs in Automobile Exhaust Emissions

Carried out a program to determine the levels of PCDDs and PCDFs produced by automobile engines. Study involved automobiles burning leaded gasoline, unleaded gasoline, and diesel fuel.

16. Survey of Dielectric Fluids Used in the Electrical Utility Industry for the Presence of PCDDs and PCDFs

Directed the analytical portion of a multi-year study to determine the levels of PCDDs and PCDFs in utility dielectric fluids. Study involved distribution transformers, load transformers, and capacitors. As part of the program, the formation of PCDDs and PCDFs from arcing and corona discharge was also investigated.

RELATED EXPERIENCE (Continued)

17. Comparison of Analytical Techniques for the Determination of Polychlorinated Biphenyls in Electrical Utility Fluids

Directed a program which compared packed column gas chromatography with electron capture detection, capillary column gas chromatography with electron capture detection and capillary column gas chromatography/mass spectrometry for the determination of polychlorinated biphenyls in Askarel fluids.

18. Interlaboratory Study to Evaluate the Accuracy of Commercially Available PCDD/PCDF Standard Solutions

Participated in an interlaboratory study, involving ten laboratories, which evaluated the quantitative accuracy of "2,3,7,8" substituted PCDD/PCDF isomer solutions. Primary standard solutions were prepared from neat standards and compared to the commercial solutions. The results of this study were reported at the Seventh International Symposium on Chlorinated Dioxins and Related Compounds.

19. Preparation of Quality Control/Quality Assurance Samples for Evaluating the Performance of Contract Laboratories

Prepared spiked human adipose samples which were used as QA/QC samples to evaluate the performance of laboratories participating in an EPA program. Also analyzed replicates of the samples to demonstrate the accuracy of the spikes.

20. Evaluation of EPA Method 8290 - A High Resolution Mass Spectrometry Method for the Determination of PCDDs and PCDFs

Participated in an interlaboratory study to evaluate the performance of EPA Method 8290. Method 8290 is revision and modification of Method 8280 which uses additional labeled internal standards and high resolution mass spectrometry.

21. Characterization of Ash Samples from a Hazardous Waste Incinerator

TCT is providing analytical support to the Department of Energy to help characterize toxic emissions from a hazardous waste incinerator. The target analytes include polychlorinated dibenzo-p-dioxins, polychlorinated dibenzofurans, polynuclear aromatic hydrocarbons, pesticides, herbicides, and metals.

RELATED EXPERIENCE (Continued)

22. Measurement of PCDD and PCDF Levels in Surface Wipes and Air Sampling Equipment Collected From a Contaminated Office Building

TCT is currently conducting a program involving the measurement of PCBs, PCDDs, and PCDFs in surface wipe and air samples collected from a building contaminated by a PCB related fire. The program involves both the preparation of the sampling equipment and the analyses of the samples.

23. Development of a Method for the Determination of PCDDs and PCDFs in Electrical Utility Fluids

TCT recently completed a program for a utility institute for the development of an optimized method for the determination of PCDDs and PCDFs in both mineral oil and askarel oil based dielectric fluids. The program involved the evaluation of methods that were used during a round robin study. A combined, optimized method was then developed and validated.

24. Determination of Polynuclear Aromatic Hydrocarbons (PAHs), PCDDs and PCDFs in Waste Products from a Refuse Derived Fuel (RDF) Facility

TCT is currently conducting a program for a major electrical utility company that involves the determination of polynuclear aromatic hydrocarbons (PAHs), PCDDs, and PCDFs in ash and leachate samples from several refuse derived fuel (RDF) facilities. This ongoing program is in support of State EPA requirements.

25. Determination of PCDDs and PCDFs in Well Water Samples

TCT is currently providing analytical services to a private engineering firm for the determination of PCDDs and PCDFs in well water samples. The samples, collected from private drinking water wells, are used to verify that PCDDs and PCDFs are not migrating from land fill areas adjacent to the wells.

26. Determination of PCDDs and PCDFs in Municipal Sewage Sludge

TCT is currently under contract with the U.S. EPA to analyze sewage sludge samples for the presence of PCDDs and PCDFs. As part of this program, TCT is evaluating EPA Method 1613 and providing the EPA with suggestions for improvements. To date, more than 150 samples have been analyzed.

Related Experience (Continued)

27. Determination of PCDDs and PCDFs in Samples From a Power Plant Fire

TCT is providing analytical services to a major electrical utility to assess the levels of contamination in a power plant that experienced a catastrophic failure. The samples consist of ash, oils, surface wipes, and air sampler cartridges. The target analytes include PCBs, PCDDs, and PCDFs. Approximately 15 samples were analyzed during the first ten days of the program.

28. Determination of PCDDs and PCDFs in Fish Samples

TCT was recently under contract with the U.S. EPA to determine the levels of PCDDs and PCDFs in fish samples. Other target parameters included the levels of PCBs and the lipid percentages. This program included the analysis of approximately 25 samples.

29. Determination of PCDDs and PCDFs in Environmental Samples Surrounding an RDF Power Plant

TCT is currently under contract with a major electrical utility to provide analytical assistance for assessing background levels of PCDDs and PCDFs in environmental samples collected from an area surrounding several Refuse Derived Fuel (RDF) power plant. The matrices of interest include soil, water, milk, vegetation, and fish. During this one year program, which started in July, approximately 125 samples will be analyzed.

30. Determination of PCDDs and PCDFs in Sludge From a Metropolitan Sewerage District

TCT is providing analytical support to a city sewerage district. As part of this program, samples are being collected under several different operating conditions and then being analyzed to determine the levels of PCDDs and PCDFs.

31. Determination of PCDDs and PCDFs in Effluents From Oil Refineries

TCT is currently under contract with a major oil company to analyze effluent samples collected during the regeneration of a catalytic cracking unit. This project, which is part of a U.S. EPA study, will involve the analysis of both water and sludge samples.

32. Analytical Support to a Program Involving the Development of a PCB Destruction Process

TCT is providing analytical support to a major contract research and development organization involving the development of a PCB destruction process. The samples consist of filters and XAD-2 resins modules from Modified Method 5 (MM-5) trains.