

GEOCHEMICAL & ENVIRONMENTAL RESEARCH GROUP

College of Geosciences Texas A&M University

Friends of Merrymeeting Bay Pesticides, PCB, dioxin/furans and Trace Metal Analyses GERG SDG F1184

Background

Five (5) eel samples were received in one sample delivery group (SDG F118) on January 6, 2006. Samples were logged in under custody and stored at -20°C. Samples were homogenized. Samples for organic analyses were extracted in QC batch T1469 on February 7, 2005. Extracts were cleaned up and then analyzed for pesticide, PCB, dioxin and furans. In addition sample aliquots were freeze dried and then digested for trace metal analyses as sample batch HG-1155 for mercury and TS-308 for the remaining trace metals. The data for all of these analyses are reported here.

Standard Operating Procedures

The samples were extracted and analyzed following the protocol contained in the following SOPs used at GERG:

SOP-9807

Extracting Biological Tissues for the Analyses of Organochlorine Pesticides, Polychlorinated Biphenyls, and Aromatic Hydrocarbons. Rev. 1 as of October 27, 1998.

SOP-9810

Quantitative Determination of Chlorinated Hydrocarbons by Gas Chromatography/Electron Capture Detection. Rev. 3 November 9, 2000.

SOP-9811

Charcoal: Silica Gel Column Chromatography for Separation of Planar Polychlorinated Biphenyls in Sample Extracts Prior to Gas Chromatography/Electron Capture Detector Analyses. Rev. 1 November 12, 1998.

SOP-9722

Quantitative Determination of Tetra- through Octa-Polychlorinated Dibenzo-p-dioxins (PCDDs) and Dibenzofuranf (PCDFs) by Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS). Rev. 1 May 2, 1997.

The samples were digested and analyzed for mercury following the protocol contained in the following SOPs used at GERG:

SOP-0006

Digestion of Tissue Samples Prior to Mercury Analysis using Cold Vapor Atomic Absorption Technique. Rev1 as of March 1, 2000.

SOP-0202

Determination of mercury by Cold Vapor Atomic Absorption Spectroscopy. Rev 1 as of March 22, 2002.

The samples were digested and analyzed for the other trace metals following the protocol contained in the following SOPs used at GERG:

SOP-9408

Digestion of Biological Materials for Trace metal Analysis. Rev 1 as of June 23, 1994.

SOP-9802

Standard Operating Procedures for Trace Metal Analysis Using Inductively Coupled Plasma (ICP) Emission Spectroscopy. Rev 1 as of June 3, 1998.

SOP 0201

Determination of Trace Metals by Graphite Furnace Atomic Absorption Spectroscopy. Rev 1 as of February 6, 2002.

Laboratory Qualifiers

All of the analytical data have been qualified based on the lower calibration limits (LCL). Concentrations that were less than the LCL adjusted for sample size and dilution, are qualified "L" and those analytes not detected are qualified "ND". Concentrations that exceeded the calibration limits are qualified "EC". The concentrations that are determined by analyses of a diluted aliquot are qualified "D". If the quantification of an analyte is interfered with by another analyte due to its high concentration the data will be left blank and qualified "I" to denote this interference.

Analytical Difficulties

Pesticide/ PCBs

The procedural blank contained four analytes above three times the MDL. Concentrations for all analytes in 4 of 5 samples were several orders of magnitude higher than blank concentrations. Analytes (PCB 135 and PCB 207) in sample C45376 were labeled with a "B" to indicate possible blank contamination for that sample. No further action was taken. The percent recovery for all surrogates was acceptable. No action was required. The relative percent difference for duplicate sample had acceptable RPDs. No further action was taken. The matrix spike recovery had one analyte (4,4'-DDE) recovery that exceeded the QC criteria. 4,4'-DDE was an invalid spike due to high native concentration in the sample. The data is qualified "Q" to denote the variance. No further action was taken. The recoveries of all analytes in the laboratory spike blank were acceptable. No further action was taken. Concentrations of all analytes in the SRM were acceptable. No further action was taken.

Dioxin/Furans

The procedural blank contained no analytes above three times the MDL. No further action was taken. The percent recovery for most surrogates was acceptable. Selected surrogates in sample C45377 and C45379 were lower that the acceptance criteria. None of the analytes represented by these surrogates was detected. No further action was taken. The relative percent differences for duplicate sample were not calculated as all analytes were below the LCL. No further action was taken. The matrix spike recovery had three of 17 analyte recovery that exceeded the QC criteria. Only one of these analytes (OCDD) was detected and in only one of 5 samples (just 2 times the LCL). The data is qualified "Q" to denote the variance. No further action was taken. The recoveries of all analytes in the laboratory spike blank except OCDF were acceptable. OCDF was not detected in any of the samples. No further action was taken. Concentrations of most analytes in the SRM were acceptable compared to the non-certified values. This SRM always has high concentrations for the 1,2,3,7,8-PeCDF non-certified concentration. No further action was taken.

Trace Metals

The negative concentrations are the results of the calibration curve. They can be interpreted as zeros. The procedural blank contained no analytes that were above the MDL. The recoveries for the spiked blank were within the QC criteria for all analytes. No further action was taken. The relative percent difference for the duplicate sample met the QC criteria. The percent recovery for matrix spike met the QC criteria. No further action was required. The recovery of analytes certified for SRMs NRCC Dolt-2, NRCC Dorm-2 and NIST 2977 (GERG IDs I2334, I2335 and I2336) met the QC criteria. Several certified analytes are below the detection limit and were not considered. No further action was taken. No further variances or difficulties were observed.

Conclusions

Samples concentrations are all reported on a dry weight bases. Care should be taken when comparing to consumption advisories which are normally on a wet weight bases. Four of the five samples required dilutions for 4,4-DDE, 4,4'-DDD and selected PCB congeners due to high concentrations. Generally dioxin, furan and trace elements concentrations were at background concentrations. No TCDD or TCDF were detected.

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Reviewed and Approved:

Terry L. Wade, Ph.D.

Date

Deputy Director for Environmental Sciences