QUALITY ASSURANCE FOR POLYHALOGENATED DIBENZO-P-DIOXINS/DIBENZOFURANS TESTING AND REPORTING UNDER THE TOXIC SUBSTANCES CONTROL ACT (TSCA)

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ABSTRACT

The United States Environmental Protection Agency (USEPA) has promulgated a testing and reporting Rule that requires manufacturers, importers, and processors of eleven chlorinated or brominated chemicals, structurally related to dioxins and furans, to carry out testing to determine whether the chemicals are contaminated with halogenated dioxins and furans above limits of quantitation set forth in the Rule. This Rule is novel in that it requires the regulated community to develop and validate analytical methods, and to prepare sampling, analytical, and quality assurance plans. These plans are reviewed by a panel of government scientists who are considered expert in the field of dioxin analysis, and must be approved by the USEPA before testing can be conducted. Specific quality assurance provisions included in the Rule are a demonstration of limit of quantitation, and generation of data under the TSCA Good Laboratory Practices Regulations. Additional quality assurance measures are to be incorporated by the regulated community based on individual chemicals and methodologies.

KEYWORDS

Halogenated dibenzo-p-dioxins; halogenated dibenzofurans; Toxic Substances Control Act; Quality Assurance; risk assessment

INTRODUCTION

Under Section 4 of the Toxic Substances Control Act (TSCA), the USEPA may

Limit of Quantitation Demonstration

Demonstration of the sensitivity of analytical methods is specifically required by the Rule. This is accomplished by the duplicate analysis of samples fortified with mass labeled internal standards at limits of quantitation (LOQs) specified in the test Rule. These LOQ's are shown in Table 2. A successful LOQ demonstration requires recovery of the standards within 50% of the fortification level, a signal to noise ratio of at least 10:1, and precision of 20% or less, as measured by the difference between the duplicate results. This provision has been incorporated into the Rule to

Table 2: LOQs Required by the Rule

Isomer	LOQ (ppb)
Tetra-HDD	0.1
Penta-HDD	0.5
Hexa-HDD	2.5
Hepta-HDD	100
Tetra-HDF	1.0
Penta-HDF	5.0
Hexa-HDF	25
Hepta-HDF	1,000

assure that the sensitivities of the methods are adequate to accurately measure contamination at the levels of concern defined by the USEPA. Further information concerning these levels can be found in the Rule and in Johnson, et al (1989 and 1990).

Good Laboratory Practices

The test Rule requires that data be generated under TSCA Good Laboratory Practice (GLP) regulations (40 CFR 792). USEPA has conducted audits of the data generated under the Rule to determine compliance with GLP. These audits have included:

- Inspection of raw data from both sampling and analysis activities
- Verification of calibration data for measurement equipment (analytical balances, mass spectrometers, etc)
- Inspection of all data for at least one sample
- Independent recalculation and verification of selected sample data
- Comparison of the conduct of the experimental work with the sampling plan, the analytical protocol, the quality assurance plan, and with specific GLP requirements.
- Inspection of documentation, sample tracking, and archival procedures.

CONCLUSION

Specific quality assurance provisions incorporated into this novel test Rule, along with review by the expert panel, have been designed to assure that data of known and acceptable quality are generated. Additional information concerning the Rule are available in Johnson, et al (1989 and 1990).

BACKGROUND

TSCA provides that "to the extent necessary to assure that the data are reliable and adequate" a test Rule may include a prescription of:

- The manner in which data are to be developed.
- The specification of any test protocol or methodology.
- Any other requirement necessary to assure reliable and adequate data.

In previous Rules, analytical methods, which have included complete quality assurance requirements, have been provided by the USEPA. However, the USEPA recognized that a single analytical method would not be applicable to all of the chemicals subject to this testing Rule, due to their number and diversity. In addition, while analytical methods are generally available for chlorinated dioxins and furans, methods for their brominated analogs have not been fully developed. These considerations precluded specification of a specific analytical method and associated performance criteria in the Rule. Therefore, a novel approach of requiring the regulated community to develop and validate analytical methods for their chemicals, and to prepare sampling and analytical protocols and quality assurance plans for testing the chemicals has been adopted in this Rule.

Sampling, analytical, and quality assurance plans submitted to the USEPA are evaluated on a case by case basis, using the expert panel's expertise and experience and good science to determine the acceptability of these plans. The USEPA has provided guidance for the development of analytical methods, sampling and analytical protocols, and quality assurance plans, (Steele and Stanley, 1987) and for the reporting of test data (Steele and Dux, 1990).

QUALITY ASSURANCE PROVISIONS FOR TESTING UNDER THE RULE

Quality assurance measures which have been incorporated directly into the testing Rule or which have been employed based on good science are described below:

Expert Review of Protocols

Although the regulated community is responsible for development of analytical methods and protocols, EPA has provided for critical review of these plans to ensure that data of acceptable quality are generated. EPA has appointed a review panel, composed of federal government scientists who are considered expert in the field of dioxin analysis. This panel is currently chaired by Dr. Aubry Dupuy of USEPA's Environmental Chemistry Laboratory located in Bay St. Louis, Mississippi. The other members of the panel are Dr. David Firestone of the United States Food and Drug Administration in Washington, DC; Mr. Robert Harless of the USEPA's Atmospheric Research and Exposure Assessment Laboratory in Research Triangle Park, North Carolina; Dr. Doug Kuehl of USEPA's Environmental Research Laboratory in Duluth, Minnesota; and Dr. Wayne Sovocool from the USEPA's Environmental Monitoring Systems Laboratory in Las Vegas, Nevada. The panel's role is to advise the USEPA as to whether plans and protocols submitted under the Rule are likely to generate data which meet the intent of the Rule. Final approval of protocols rests with the USEPA's director of the Office of Toxic Substances.

The panel uses both specific requirements incorporated into the Rule and their expertise and experience in the field of dioxin analysis in evaluating protocols. Considerations addressed by the panel include:

- Representativeness of samples
- Sample integrity during collection and shipment
- Ability of the analytical methods to produce data which meets Rule LOQ requirements.
- Demonstration of data quality through the use of method blanks, duplicate samples, and fortified samples.

REFERENCES

Johnson, J., J.J. Breen, T.M. Murray, J.A. Glatz, D.H. Steele, and J.S. Stanley (1989). Polyhalogenated Dibenzo-p-Dioxins/Dibenzofurans Testing and Reporting Requirements under the Toxic Substances Control Act (TSCA). Chemosphere, Vol. 19, Nos. 1-6, pp. 849-852.

Johnson, J., J.J. Breen, T.M. Murray, J.A. Glatz, D.H. Steele, and J.S. Stanley (1990). Polyhalogenated Dibenzo-p-Dioxins/Dibenzofurans Testing and Reporting under the Toxic Substances Control Act (TSCA) - An Update. Chemosphere, In press.

Steele, D.H. and J.S. Stanley (1987). Guidelines for the Determination of Halogenated Dibenzo-p-dioxins and Dibenzofurans in Commercial Products. EPA Publication EPA-560/5-87-007.

Steele, D.H. and T. Dux (1990). Guidelines for Reporting Test Results of HDD and HDF Determinations in Commercial Products (40 CFR Parts 707 and 766). Midwest Research Institute, Final Report, EPA Contract No. 68-02-4252.

USEPA (1987). Polyhalogenated Dibenzo-p-dioxins/Dibenzofurans; Testing and Reporting Requirements: Final Rule. United States Environmental Protection Agency. 40 CFR Parts 707 and 766. Federal Register, 53(108), June 5, 1987.