

Environmental Protection Agency

§ 766.18

(7408), Office of Pollution Prevention and Toxics, U.S. Environmental Protection Agency, Room E-543B, 1200 Pennsylvania Ave., NW., Washington, DC 20460, Telephone: (202) 554-1404, TDD: (202) 544-0551. Copies are also located in the public docket for this part (Docket No. OPPTS-83002) and are available for inspection in the Non-Confidential Information Center (NCIC) (7407), Office of Pollution Prevention and Toxics, U.S. Environmental Protection Agency, Room B-607 NEM, 401 M St., SW., Washington, DC 20460, between the hours of 12 p.m. and 4 p.m. weekdays excluding legal holidays.

[60 FR 34466, July 3, 1995]

§ 766.14 Contents of protocols.

Protocols should include all parts of the *Quality Assurance Plan for Measurement of Brominated or Chlorinated Dibenzofurans and Dibenzodioxins*, as stated in the Guidelines. For each chemical substance and each process, the manufacturer must submit a statement of how many grades of the chemical substance it produces, a justification for selection of the specific grade of chemical substance for testing, specific plans for collection of samples from the process stream, naming the point of collection, the method of collecting the sample, and an estimate of how well the samples will represent the material to be characterized; a description of how control samples (blanks) and HDD/HDF-reinforced control samples, or isotopically labeled compounds (standards) and duplicate samples will be handled; a description of the chemical extraction and clean up procedures to be used; how extraction efficiency and measurement efficiency will be established; and a description of instrument hardware and operating conditions, including type and source of columns, carrier gas and flow rate, operating temperature range, and ion source temperature.

§ 766.16 Developing the analytical test method.

Because of the matrix differences of the chemicals listed for testing, no one method for sample selection, preparation, extraction and clean up is prescribed. For analysis, High Resolution Gas Chromatography (HRGC) with

High Resolution Mass Spectrometry (HRMS) is the method of choice, but other methods may be used if they can be demonstrated to reach the target LOQs as well as HRGC/HRMS.

(a) *Sample selection.* The chemical product to be tested should be sampled so that the specimens collected for analysis are representative of the whole. Additional guidance for sample selection is provided under § 766.12.

(b) *Sample preparation.* The sample must be mechanically homogenized and subsampled as necessary. Subsamples must be spiked or reinforced with surrogate compounds or with standard stock solutions, and the surrogates or standards must be thoroughly incorporated by mechanical agitation. Additional guidance is provided under § 766.12.

(c) *Sample extraction and cleanup.* The spiked samples must be treated to separate the HDDs/HDFs from the sample matrix. Methods are reviewed in the Guidelines under § 766.12, but the final method or methods are left to the discretion of the analyst, provided the instrumental response of the surrogates meets the criteria listed in the *Quality Assurance Plan for Measurement of Brominated or Chlorinated Dibenzofurans and Dibenzodioxins*, Appendixes B and C of the Guidelines. Cleanup techniques are described in the Guidelines. These are chosen at the discretion of the analyst to meet the requirements of the chemical matrix.

(d) *Analysis.* The method of choice is High Resolution Gas Chromatographic/High Resolution Mass Spectrometric Determination, (HRGC/HRMS) but alternate methods may be used if the manufacturer can demonstrate that the method will reach the target LOQs as well as HRGC/HRMS. Specific operating requirements are found in the Guidelines.

§ 766.18 Method sensitivity.

The target level of quantitation required under § 766.27 for each HDD/HDF congener is the level which must be attempted for each resolved HRGC peak for that congener. For at least one product sample, at least two analyses of the same isotopically labeled HDD/HDF internal calibration standards spiked to a final product concentration