§ 766.14 Contents of protocols.

Protocols should include all parts of the Quality Assurance Plan for Measurement of Brominated or Chlorinated Dibenzo-p-dioxins and Dibenzo-furans, 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 cleanup 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. 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 equal to the LOQ for that congener must be reproducibly extracted, cleaned up, and quantified to within ±20 percent of each other. For each spiked product sample, the signal to noise ratio for the calibration standard peaks after complete extraction and cleanup must be 10:1 or greater. The recovery of the internal calibration standards in the extracted and cleaned up product samples must be within 50 to 150 percent of the amount spiked, and the results must be corrected for recovery.