Tetrachlorodibenzo-p-dioxin (TDD) Analysis in Autoclave Effluent

1. GLC Standard Preparation

This analytical procedure was developed due to the need of sensitivity in low ranges of concentration. The original analysis was performed on a thermal conductivity detector, but as lower concentrations of TDD would be expected, this procedure proved to give accurate and quantitative measurements at low levels.

This analysis is based on a normalization procedure. Internal standardization can be incorporated if needed.

a. Into a 1-liter volumetric flask is weighed 0.0000-0.1000 g. of 2,3,7,8-tetrachlorodibenzo-p-dioxin (TDD). Care should be taken in this procedure so as not to allow any TDD to contact the skin or eyes.

b. Bring the volume to 1-liter with benzene.

c. From this stock solution, several standards can be prepared. Concentration of this solution will be in the range of .10 mg/ml. or 100 ppm. It is suggested that a range of normalization standards be prepared and used to obtain a normalization curve.

d. A 0.5 µl aliquot is used for the analysis and gives full scale deflection at a moderate attenuation level.

Keep the container exposure to air to a very minimum to avoid evaporation. Standards prepared in this lab 7-8 months ago still give no evidence of change in response levels.

2. TDD Analytical Runs

The autoclave samples are diluted and transferred with water to give an approximate weight ratio of 1:1. To the aqueous sample is then added an aliquot of benzene, and the sample is placed on a Kahn shaker for 30 minutes. If the two layers do not separate on standing, the sample is centrifuged.

a. A 0.5 µl sample is withdrawn from the benzene layer and injected into the instrument. Attenuations are adjusted and duplicate runs are made. The peak heights of the TDD peaks are calculated and averaged.

b. The concentration of TDD on ppm is then calculated from the following expression:

$$TDD_{(ppm)} = \frac{(\text{Peak Hgt TDD})(\text{Conc. Std mg/µl})(\text{Total Volume Extract})}{(\text{Peak Hgt Normalization Std})(\text{Sample Wt})(\text{mg})}$$

where: attenuation adjustments are equal for standard and sample.

(See Figure 3 for an actual process sample).

3. GLC Instrument and Conditions

Instrument: P-E Model 800 with dual flame detectors.

Columns: 6' x 1/8" S.S. packed with 5% DC9F-1 (fluorosilicone oil) on 80-90 mesh Chromasorb W (HMDS treated).

Operating Temperatures: Column oven - 215°C Isothermal
Injector - 325°C
Detector - 265°C

Carrier Gas (N_2) - 40 psig. inlet pressure, 30 ml/min exit flow.

H_2 (pre-purified) - 50 psig. inlet, reference flame - 15 psig.
sample flame - 15 psig.

Air (breathing) - 5- psig. inlet, ≈500 ml/min flow.

Sample Size: 0.5 λ (0.5 ul.).