

Analysis of Organic Tin Compounds with Capillary GC-FPD

Tributyltin (TBT) and triphenyltin (TPT) are used as antifouling coatings for ships and fishing nets. There is concern about their pollution of environmental water and fishery products, and their influence on human health. Also, they are suspected to have endocrine disruptive effects and interest in the analysis of these compounds has been growing.

The analysis of alkylated organic tin compounds was introduced in Application News M182 "Analysis of TBT and TPT by GCMS". This report discusses examples of TBT and TPT analysis by capillary GC-FPD.

As in Application News M182, deuterium-labeled compounds (d-labeled compounds) are mixed with the alkylated sample.

The quantification of tin compounds can be done by the external or internal standard method. Although the external standard method is a very simple quantification method, its quantification precision is somewhat lower than that of the internal standard method. The internal standard method features high quantification precision but involves complex operations, including the selection and addition of the internal standard material. When no particular quantification method should be designated, quantification method is based on importance of quantification precision or ease of operation.

Organic tin compounds and d-labeled compounds

can be separated on a chromatogram. However, as the retention time of the d-labeled compounds are close to those of the target compounds, quantification error may occur when resolution lowers due to column degradation or other reasons. Since the physicochemical characteristics of the d-label compounds are extremely similar to those of the target compounds, they can be used as excellent internal standard substances in GCMS analysis, where mass number data are available. However, in analyses where separation on the chromatogram is important like this GC analysis, tetrabutyltin (TeBT) and triphenyltin (TPeT) are often used as the internal standard substances.

Fig. 1 shows examples of the extraction methods for organic tin compounds. Fig. 2 shows the chromatogram of the organic tin compound standard solution (1 mg/L each). TeBT (peak No. 6) and TPeT (peak No. 8) are clearly separated from other compounds. Figs. 3 and 4 show the calibration curves for TBT and TPT (internal standard method using TPeT as IS).

Fig. 5 shows a chromatogram of the organic tin compound standard solution (0.1mg/L each). Figs. 6 and 7 show the calibration curves for TBT and TPT (external standard method). Fig. 8 shows the chromatogram for sea bass extract (0.2 to 0.4μg of standards added to 2mL sample solution).

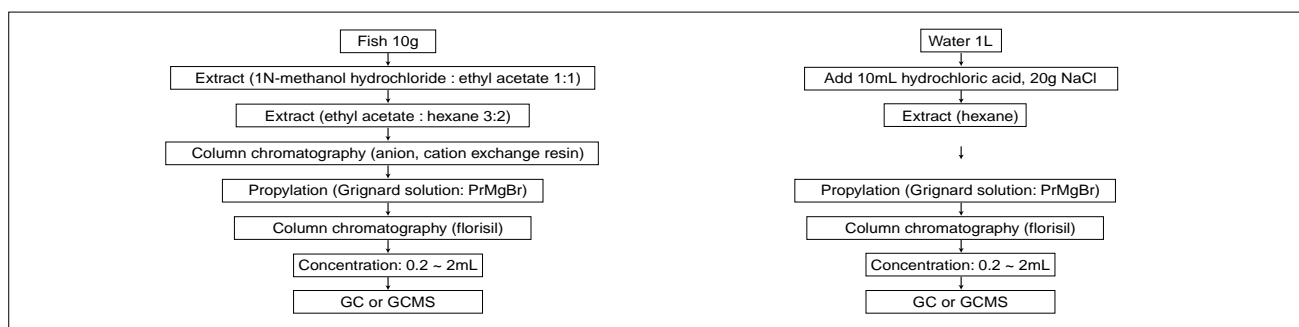


Fig.1 Examples of Extraction Methods for Fish and Water

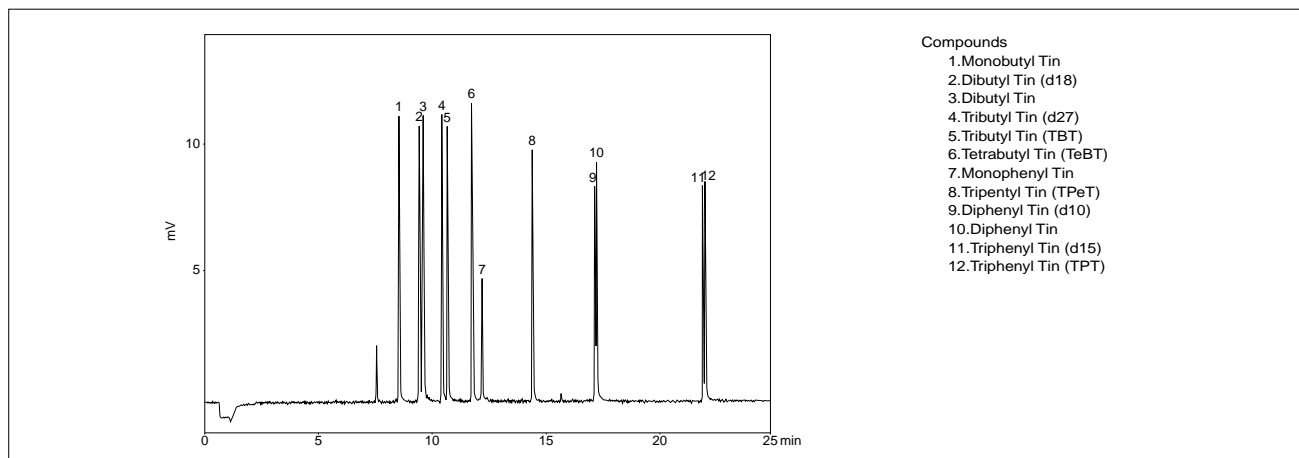


Fig.2 Chromatogram of the Standard Solution (1mg/L)

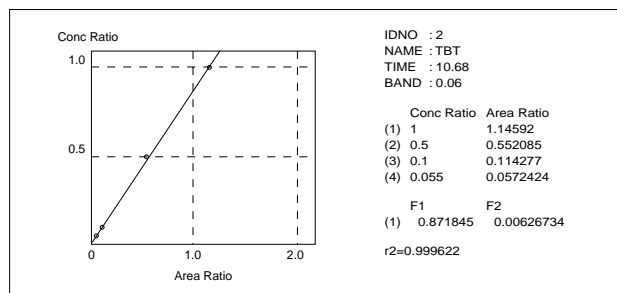


Fig.3 Calibration Curve for TBT (Internal Standard Method IS: TPeT)

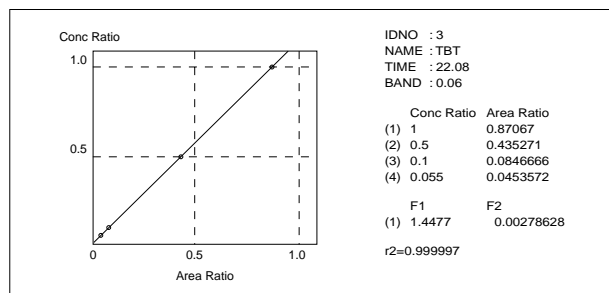


Fig.4 Calibration Curve for TPT (Internal Standard Method IS: TPeT)

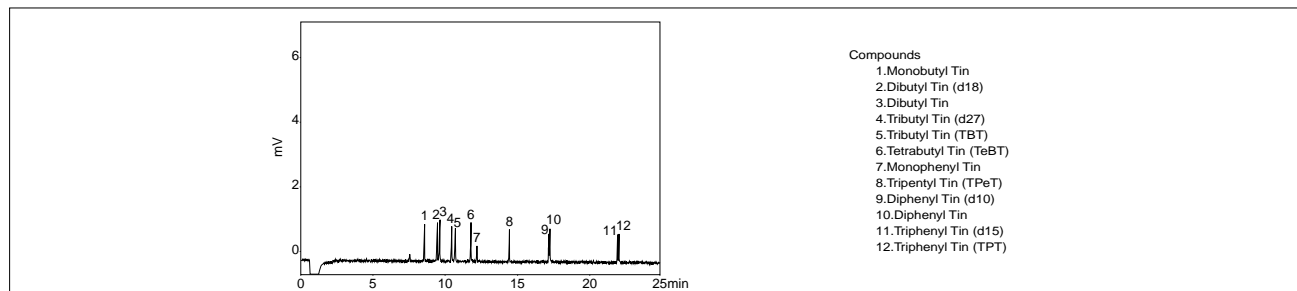


Fig.5 Chromatogram of Standard Solution (0.1mg/L)

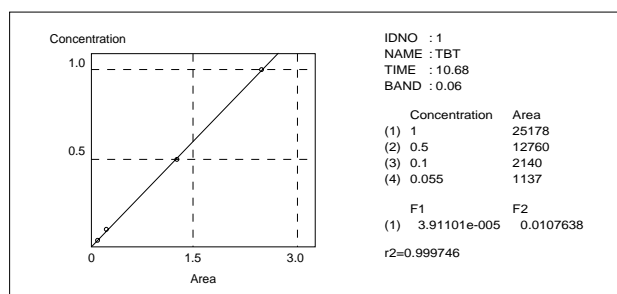


Fig.6 Calibration Curve for TBT (External Standard Method)

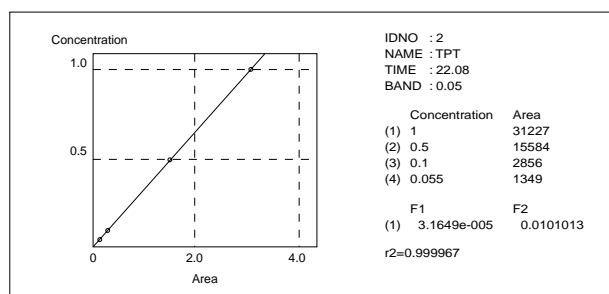


Fig.7 Calibration Curve for TPT (External Standard Method)

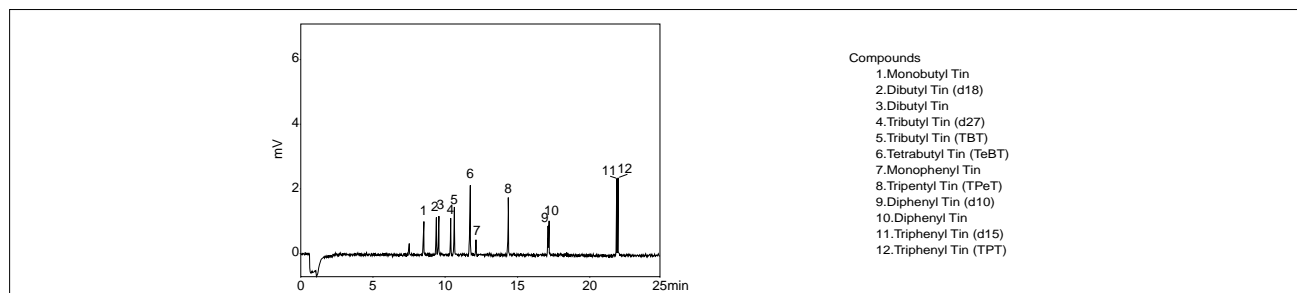


Fig.8 Chromatogram of Fish Extract (0.2 ~ 0.4µg standards added to 2mL of sea bass extract solution)

References

Standard Methods of Analysis for Hygienic Chemists; With 1990 Annotations and Supplements (1995): Edited by the Pharmaceutical Society of Japan, Published by Kanehara Co. Ltd., 1995.
Environmental Endocrine Disrupting Chemicals Investigation Provisional Manual (Water Quality, Sediment, Aquatic Life): Water Quality Management Division, Water Quality Bureau, Environmental Agency (October 1998)

Table 1 Analytical Conditions

Model	: GC-17A AFw ver.3, FPD-17c, AOC-20i	Inj.Temp.	: 290°C
Column	: DB-5 30m × 0.25mm I.D. df=0.25µm	Det.Temp.	: 300°C
Column Temp	: 60°C (1min) -20°C/min-140°C-7°C/min-280°C (5min)	Injection Method	: High Pressure Splitless (1min)
Carrier Gas	: He, 350kPa (1min) -150kPa (2.4mL/min)	Injection Volume	: 3µL
Det.	: FPD-17c (Sn Filter)		



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