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ABSTRACT

A dimethylsulfoxide-based cleanup was used for the determination of PCBs in waste oil. The procedure was found to be effective in eliminating the matrix interferences while maintaining good overall recovery of PCB congeners. The recovery of individual congeners was found to be between 85-95%.

INTRODUCTION

Determination of contaminants at trace levels often requires extensive cleanup procedures to remove interferences contributed by the matrix. Low level analysis of PCBs in waste oil can suffer from interferences when ECD or MS are used for detection.

Dimethyl sulfoxide (DMSO) has been used for selective extraction of PCDD and PCDF from waste oil (1). The present study reports a DMSO-based cleanup procedure for congener specific analyses of PCBs in waste oil. The six congeners incorporated in the European legislation were analyzed together with five other congeners which, due to their high toxicity and bioaccumulation, have recently been designated as priority PCBs (2).

EXPERIMENTAL

Aliquots of waste oil (0.5 ml) were diluted with iso-octane and partitioned with DMSO. The DMSO phase was back-extracted with hexane after addition of distilled water. The hexane phase containing PCBs was fractionated on a multilayer column containing activated silica, potassium silicate, and sulfuric acid-impregnated silica. The PCB fraction was solvent exchanged to iso-octane and the volume brought down to 1 ml. The chromatographic separation was achieved on a 50 m x 0.25 mm i.d. fused silica capillary column with a 5% phenyl 95% methylpolysiloxane stationary phase and a 60 m x 0.25 mm i.d. column with a cyanopropyl/phenyl polysiloxane stationary phase. The efficacy of the DMSO based cleanup procedure was compared with more traditional adsorbent column based cleanup procedures.

RESULTS AND DISCUSSION

The oil matrix caused severe interference problems in the mass spectral determination of PCBs. The ECD detection was much less affected by the presence of an oil matrix. However, concentrations of the matrix higher than 50% were

significantly reduced the GC-ECD response of PCBs, probably due to insufficient transfer from the splitless injector to the column. The DMSO based cleanup was found to be effective in eliminating the matrix interferences while maintaining good overall recovery of PCB congeners. The recovery of individual congeners was found to be between 85-95%. The relative percent difference of 5 replicates was 5-10%. The procedure was evaluated over a concentration range of 100-1500 ppb. However, the procedure should be applicable down to sub-ppb determinations.

REFERENCES

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