CRYOVAC DIVISION | W.R. GRACE & CO. | DUNCAN, SOUTH CAROLINA 29334

14 December 1972

TO: Task Group for PCB Analysis in Flexible Barrier Materials - Sub-Committee F2.30

RE: Minutes of Task Group Meeting, 30 November 1972

MEMBERS: ✓Norman Bornstein - CRYOVAC
✓John Cobler - Dow
✓Gordon Patterson/John Ness - duPont
Irving Russoff/Tony Gross - NABISCO
Scott Tucker - Monsanto
✓George White - Reynolds
Jim Yeransian - General Foods

CC: R. J. Powell - duPont

The first Task Group meeting was held at the CRYOVAC headquarters in Duncan, South Carolina, on 30 November 1972. The following Task Group members were present: Gordon Patterson, John Ness, George White, John Cobler and Norman Bornstein. Lou DeMelio and LeRoy Pike of CRYOVAC also participated in the discussions. The discussions covered various aspects of PCB analysis in polymerics:

I. Polymerics to be Studied

Methodology for the following polymers appeared to be of greatest interest: polyolefins, saran, PVC, ionomers and polystyrene.

II. Extraction Procedures

Two general procedures for removing PCB were mentioned as being employed by the participants: (1) vacuum distillation and (2) extraction.

It was recognized that the solvents, the physical state of the sample and the time and temperature conditions of extraction would have to be established and that these factors might vary depending upon the polymer being analyzed.

III. Clean-up Procedures

The following procedures were mentioned:

A. Florisil column deactivated with 2% water.

B. Activated carbon column.

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C. Reaction with alcoholic KOH followed by extraction with n-hexane and an H_2SO_4 wash.

D. Pyrolysis for the elimination of interfering compounds having a lower thermal stability than the PCBs.

IV. Quantitative Analysis

It was agreed that a gas chromatographic procedure employing a ^{63}Ni electron capture detector should be employed. The represented laboratories used DC-200, OV-17 or OV-210 columns in both glass and stainless steel.

V. Calculations

The following methods for obtaining results from the chromatograms were discussed:

A. Using a single peak and referencing it against that peak in a standard Aroclor sample.

B. Integrating the total area in the chromatogram and referencing it against standard samplesof one or more Aroclors.

C. Calculating the concentrations of each component peak by peak or alternatively using only the major peaks, peak by peak.

VI. Qualitative Analysis

Methods for validating whether unknown peaks are in fact PCB were briefly discussed. Methods mentioned were substantiation by Mass Spectrometry, elemental analysis and corroboration using a hydrogen flame detector.

VII. Program

The Task Group would begin by determining the interlaboratory agreement of the results of the analysis of prepared solutions of various Aroclors in n-hexane employing a tentative GC-EC method as well as a scheme for calculating results both of which accompany this report. The tentative method employs a 10% DC-200 6 ft. x 4 mm ID, column and injection port and detector temperatures of 325°C and a column temperature of 200-225°C.

Five different samples are to be sent to each Task Group member for analysis by the tentative standard method. The round robin samples will also be analyzed by standard procedures employed by the various laboratories to determine the criticality of a uniform method. It was noted in the discussion that a given Aroclor will vary from lot to lot, the degree or variation was unknown. In order to assess the degree of variation and its affect upon the reported results, it was agreed that samples of Aroclor 1242 and 1254 employed by CRYOVAC as standards would also be sent to the Task Group members for comparison with the same materials in their possession.

N. D. Bornstein, Manager
Applied Research & Analytical Services

NDB/sbm

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