ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF ENFORCEMENT
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
BUILDING 53, BOX 25227, DENVER FEDERAL CENTER
DENVER, COLORADO 80225

DATE: February 26, 1976

Dr. James Mieure
Monsanto Industrial Chemicals Co.
800 N. Lindbergh Boulevard
St. Louis, Missouri 63166

Dear Dr. Mieure:

Following completion of the round robin testing of ASTM Method D-3304 for polychlorinated biphenyls, we would like to suggest consideration of some modifications to the procedure which may help to eliminate some of the problems encountered during the round robin study. These suggestions result in part from procedures that we are presently using for the analysis of PCB's and chlorinated hydrocarbon insecticides in environmental materials. A copy of our procedure is attached for your perusal.

First we'd like to suggest that during alumina preparation, the alumina be heated to 200°C for 2-1/2 hours and then deactivated with approximately 3% water. This will eliminate the need for a muffle furnace and provide a suitable degree of activation.

Secondly, we would like to suggest the use of the 1 liter sample which should be extracted with 100 ml of hexane followed by 50 ml. The larger sample will increase sensitivity, and allow extraction in one separatory funnel saving glassware and confusion.

We have found that extracts can be concentrated to 10 ml on a hot plate under a gentle stream of purified air without significant loss of the PCB's. This approach is much simpler than the use of Kuderna Danish concentrators. The saponification procedure seemed to increase interference peaks in the river and distilled water samples and, we think, should be avoided in all but the most grossly contaminated samples. In the latter situation, an acetonitrile partition would be preferable.

During the alumina column cleanup we normally apply a 10 ml sample to the column and elute with 50 ml of ethyl ether in hexane. This procedure eliminates most of the common chlorinated hydrocarbon pesticide interferences which can then be eluted from the column and analyzed separately.

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We hope these suggestions will be helpful to your activities on ASTM Committee D-27. Should you have further questions, please give us a call at 303/234-4661.

Sincerely,

Theodore O. Meiggs, Ph.D.
Chief, Chemistry Branch

Attachment

TOM:ct

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