Monsanto INDUSTRIAL CHEMICALS COMPANY

FROM (NAME & LOCATION): E. M. Emery - T3E

DATE: November 7, 1972

SUBJECT: PCB PROCEDURES ASTM

REFERENCE:

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This is in answer to the comments submitted by Dow Corning on the proposed method for "Gas Chromatographic Determination of Higher Boiling Homologs in ASTM D2233 Type D and Type A (Aroclor 1016 and Aroclor 1242)". As you recall I wrote the original draft of this procedure for Earl L. Morrison. I have not seen the final draft that he submitted to Dow Corning. A copy of my original draft is attached for reference along with a copy of the Dow Corning comments.

I certainly agree with all of the comments made by Dow Corning as representing a more scientific approach to this analysis. We would have preferred using this more scientific approach in our work. However, several difficulties described below have caused us to adopt the simpler empirical approach.

As a former chairman of ASTM Committee E-19 on Chromatography, I am quite familiar with ASTM E 355-68 and E 260-69 referred to by Dow Corning. These two procedures were developed by Committee E-19.

Comment 1 - related to division of the chromatogram

I agree that the retention index is the preferred retention parameter. We chose the simpler approach of providing reference samples with corresponding chromatograms to any laboratory attempting to setup the procedure. The separation will only work properly if the column and total chromatographic system are in good condition and operated properly. We felt that another laboratory could most easily tell if they were properly carrying out the analysis by running sequence samples.

It may well be that ASTM will not allow the use of reference samples. In that case certainly the use of retention indices to describe the division of the chromatogram and the range over which the compounds are measured is the best approach.

ASTM Committee E-19 has been recently studying the reproducibility of retention indices between laboratories which were all supplied the same batch of column packing. We found a surprising variability in the reported retention indices. The retention index is of course temperature dependent. It was felt that this variability was due to the fact that the thermocouple/pyrometer readout systems on most commercial chromatographs are quite inaccurate. Certainly if retention indices are to be used in this procedure, then it will be necessary to specify that the column temperature be measured by some more accurate means.

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Comment 2 - on use of pure known calibration materials

We have found by GC/MS that even when we slow the analysis down using a longer column, there is still overlap of the tetra- and penta-chlorobiphenyls. In addition, most of the peaks contain more than one component (sometimes of differring chlorine level) and the ratio of these components in some peaks can vary depending upon the source and method of manufacture of the Aroclor 1242. Therefore, to apply individual response factors even in the case where one has pure known compounds would require a GC/MS run on each sample being analyzed. We felt that this was not practical for the majority of laboratories. Therefore we have adopted the more rapid empirical procedure for plant control use, recognizing that it does not give the true absolute level of penta- and hexa-chlorobiphenyls.

Comment 3 - on sensitivity and minimum detectable amounts

I agree and such information can be obtained experimentally if the need for it is accepted.

Comment 4 - on precision

This relates to something that must have been added to a later draft. Five measurements would certainly give better precision than two, but of course only in the ratio of the square roots of the number of measurements.

I am sending along extra copies of this memo that can be forwarded to Dow Corning and other interested parties.

E. M. Emery

att.

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