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MONSANTO COMMENTS ON THE ANALYTICAL METHOD SPECIFIED IN THE PROPOSED TOXIC POLLUTANT EFFLUENT STANDARDS FOR POLYCHLORINATED BIPHENYLS

The analytical method specified in the Proposed Standards is unsatisfactory for measuring PCBs at water concentrations approaching 1 µg/l. At higher concentrations the method may have adequate specificity and accuracy for monitoring effluents. It has the inherent sensitivity to detect and measure PCB levels below 1 µg/l. However, at PCB concentrations of about 1 µg/l the precision and accuracy of the method, when applied to actual industrial effluents, has not been demonstrated. The method has not been subjected to adequate multi-laboratory testing to define the accuracy, precision or limits of applicability. Multi-laboratory round robin testing of the precision of a similar method, American Society for Testing Materials Method D-3304, "Analysis of Environmental Materials for Polychlorinated Biphenyls," has been carried out. This test by twelve laboratories showed that the 95% confidence level for precision agreement between any two participating laboratories was greater than a factor of two. Using the ASTM method, the only method subjected to government and industrial multi-laboratory testing, if one laboratory measures PCB in a sample containing only PCBs and water and reports a value of 1.0 µg/l, a second laboratory could expect to measure a

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value in the range of about 0.5 to 2.0 µg/l. This precision variability very likely applies also to the method specified in the Toxic Pollutant Effluent Standards. However, if you are trying to measure PCBs in an industrial effluent you would not be able to achieve the sensitivity or reproducibility reported in ASTM D 3304. The accuracy of neither method has been adequately tested in industrial effluents.

Since the precision and accuracy of the recommended method for real effluents are unknown, this uncertainty should be reflected in the Proposed Standards.

An abbreviated procedure is needed for routine monitoring of PCBs at the sources covered by the Proposed Standards, namely PCB manufacturers and capacitor and transformer manufacturers. Effluents from these facilities typically have a very low ratio of pesticides to PCBs. Potential interferences likely to be in these effluents are not pesticides and are not materials classified as toxic pollutants. Therefore, we propose that these facilities should be allowed to use a straightforward procedure designed to measure only PCBs.

The method specified in the Proposed Standards is not such a procedure. It contains elaborate and time-consuming separation schemes to isolate each potential interference and permit its measurement. However, as pointed out above, this is beyond the scope of the Toxic Pollutant Effluent Standards and represents an undue hardship for those monitoring source effluents.

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We propose ASTM Method D-3304 as an alternate procedure for these source effluents. This method was developed specifically for PCBs and omits the elaborate separation procedures for organochlorine pesticide interferences. Instead, pesticides and many other potential interferences are eliminated by a simple chemical treatment. Hence, the method is more direct, less prone to handling errors and less time-consuming.

The following specific comments apply to the EPA method:

1. The method title should be changed to reflect the intended dual application of this method to PCBs and organochlorine pesticides.
2. When the observed PCB level is significantly above laboratory and reagent background, background subtraction should be permitted.
3. In Section 4.1, reference to a glass lined injection port should be rephrased to insure that it includes on-column injection into glass columns.
4. Acetonitrile partitioning similar to that described in Section 10.2 leads to incomplete recovery of PCBs as well as pesticides. Recoveries of various PCB product mixtures during this partitioning should be carefully examined.
5. Confirmation of PCBs using the second GC liquid phase referenced in Section 4.4.4.2 should be clarified in the experimental portion of the method.
6. Total peak areas should be used for quantitation of each PCB product mixture. This technique is equally accurate and much more rapid than using peak weight factors for calculating effluent concentrations.

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7. Chromatograms of Aroclor 1016 should be included with the reference chromatograms of the other PCB products.

Sampling and sample storage procedures are even more important than the analytical method for establishing accurate effluent concentrations. Random "grab" samples, for example, are inadequate for determining daily or monthly average concentrations. Improper sampling or storage can introduce order of magnitude errors in measured concentration values. Proposed Standards and the analytical method specified therein completely ignore sampling and storage. It is important that samples be representative of the average effluent and be properly preserved. Accordingly the Standard must specify precisely how samples are to be obtained. It must also impose limitations on sample storage conditions, including containers.

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