

Note on Removal of Sulfur Interferences from Sediment Extracts for Pesticide Analysis

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Extracts of bottom muds from lakes, streams, and estuaries are quite often very difficult to analyze for pesticides because of interfering co-extractives. This is especially true whenever the samples are taken from an anaerobic zone. Ordinary cleanup techniques such as the Mills procedure (1), that detailed by Law and Goerlitz (2) or partition techniques (3) improve the situation only slightly. It is not unusual, in gas chromatographic analysis of these extracts, for the insecticides eluting between the solvent and o,p'-DDT peak on a DC-200 column to be obscured when electron capture detection is employed. Until now, the usual alternative to circumvent this problem was microcoulometric detection.

Analysis of the interfering coextractives by flame-photometric gas chromatography revealed the presence of a number of sulfur containing compounds. Elemental sulfur, often used as a fungicide and miticide, has appeared in hexane extracts of water samples taken from streams in cotton growing areas. It has been noted that gas chromatographic tests for aldrin are particularly susceptible to interferences by elemental sulfur (4, 5, 6).

Shaking of a small drop of metallic mercury with the concentrated extract apparently removes sulfur and sulfur-organic compounds by precipitation. Elemental sulfur is removed very rapidly, within 1 or 2 minutes whereas other sulfur compounds react more slowly with the metallic mercury. Since the initial findings, 11 samples containing interfering sulfur compounds have been analysed by electron capture gas chromatography after passing the extracts through cleanup columns. The addition of mercury has been successful in removing the interfering co-extractives each time. Removal is usually complete 10 minutes after shaking the mixture.

Preliminary findings indicate that aldrin, dieldrin, heptachlor, lindane, and p,p'-DDT are not affected by the addition of mercury to hexane solutions of the insecticides, even after 5 days in contact with the metal. Malathion, methyl parathion and parathion do react with mercury and removal of these compounds is noticeable after about 10 minutes. About 1 mg/l of mercury dissolves in hexane at room temperature and this may help to explain the rapidity of the reaction (7).

Recovery studies are now underway on bottom muds from various locations and these findings will be published later.

References

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