

Colorimetric Determination of Lindane in Soils and Crops

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A procedure based on the method of Schechter and Hornstein was used to determine lindane directly in soils and crops.

THE ANALYTICAL METHOD used here for the determination of lindane (γ -1,2,3,4,5,6-hexachlorocyclohexane) (1, 2) is based on the dehydrochlorination of hexachlorocyclohexane "to benzene by means of zinc in acetic acid. The dechlorination and the subsequent nitration are carried out in a specially designed all-glass apparatus. The benzene is absorbed in a nitrating mixture and converted into *m*-dinitrobenzene. After extraction, it reacts with methyl ethyl ketone in the presence of strong alkali and the violet-red color produced is measured photometrically."

Initially the soil samples were extracted with petroleum ether, and it was necessary to remove interfering aromatics present in petroleum ether from the extracts before proceeding with the analysis. The following procedure, which gave a low blank—2 γ of apparent lindane in 50 ml. of petroleum ether—was found satisfactory.

The amount of petroleum ether solution to be analyzed was added to 125 ml. of acetic acid in a 250-ml. Erlenmeyer flask fitted with a reflux condenser, in order to minimize losses due to splashing. The acetic acid was then concentrated on a hot plate to 50 ml. To this volume 125 ml. of acetic acid was added and boiled down to 100 ml.

Known amounts of lindane added to acetic acid in a petroleum ether solution were recovered by this procedure to an extent of 94 to 95%. However, it was found that the analysis can be run directly on the material under investigation, thus eliminating the extraction and clean-up procedure.

Direct Determination of Lindane

In Soil Low in Organic Content (Loam and Sandy Loam). After the soil had been screened and thoroughly mixed, 100 grams was dried for 24 hours at 50° C. in order to determine the dry weight of the soil under investigation.

For analysis, 50 grams or less of wet soil—depending on the estimated amount of lindane present—was placed in a 250-ml. Erlenmeyer flask. To flasks that contained less than 50 grams of soil, lindane-free soil was added, in order to bring the amount of soil in all the flasks to 50 grams. To this 130 ml. of acetic acid was added, and 30 ml. was boiled off, using a reflux condenser, on a hot plate. After cooling, 10 grams of zinc and 20 grams of malonic acid were added and the procedure described by Schechter and Hornstein was run. (The use of 5 or 10 grams of malonic acid did not give consistent results.) Nearly 100% of known amounts of lindane added to soil in an acetic acid solution was recovered.

In Soil High in Organic Content (Muck). After the wet soil had been added to the Erlenmeyer flask, 150 ml. of acetic acid and 5 grams of malonic acid were added. Malonic acid develops carbon dioxide, which accelerates distillation of aromatics present in the material under investigation. The acetic acid was concentrated to 100 ml., and, after cooling, the regular analysis was run. Blanks of muck soil ranged between 0.25 and 0.35 p.p.m. of apparent lindane. Nearly 100% of known amounts of lindane added to muck soil in an acetic acid solution was recovered.

In Crops. The fresh plant parts were washed with warm water, then with acetic acid in order to remove adherent lindane, and finally the acetic acid was rinsed off with water. After the crop had been cleaned, it was diced and mixed in a Waring Blendor. Forty grams of this material was placed in a 250-ml. Erlenmeyer flask and 130 ml. of acetic acid and 5 grams of malonic acid were added. The acetic acid was boiled down to 50 ml. Then 80 ml. of acetic acid was added to the Erlenmeyer flask and the acetic acid was boiled down to 100 ml.

After cooling, 10 grams of zinc and 20

grams of malonic acid were added and the regular analysis was run. Nearly 100% of known amounts of lindane added in an acetic acid solution to the crops under investigation was recovered. Blanks obtained from plants grown on lindane-free plots varied according to the plant and the soil type on which the plants grew. The crops analyzed were carrots, potatoes, peas, cabbage, cucumbers, beans, and tomatoes. Potato tubers grown on a sandy loam gave an apparent lindane content of 0.125 p.p.m.; on muck soil, 0.175 p.p.m. Tomato fruit grown on loam gave an apparent lindane content of 0.2 p.p.m.; on muck, 0.3 p.p.m.

Analytical Setup and Calculation

Each analysis was run in duplicate, using a soil blank for determination of apparent lindane. In addition, known amounts of lindane were added to two lindane-free soil samples. The unknowns, after the value for apparent lindane had been subtracted, were calculated on the basis of the value obtained for the known amounts. Results are expressed finally in parts per million, based on dry weight for soils and fresh weight for plants.

Literature Cited

- (1) Schechter, M. S. and Hornstein, Irwin, *Anal. Chem.* **24**, 544, (1952).
- (2) *Ibid.*, pp. 1036-7.

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