Considerations of the Vapor Pressure of Dichlobenil Affecting Its Use and Procedure for Residue Analysis[1]

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Dichlobenil (2,6-Dichlorobenzonitrile) has proved to be an effective herbicide for use on a number of plants including woody ornamentals and nursery stock. The performance of the herbicide is affected by its relatively high volatility and attendant vapor losses (2,4). In addition to loss of activity, the release of vapors from treated soil can cause plant injury under unusual circumstances, since plants are capable of absorbing chemical from the vapor state (2).

Recently our laboratory encountered a situation where the soil floor in a plastic covered greenhouse was treated with 8 lbs/A of dichlobenil. Shortly thereafter, azalea plants

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growing in the greenhouse became chlorotic and showed symptoms of distress. It was suspected that under these circumstances the dichlobenil vapors were accumulating in the greenhouse atmosphere and were being absorbed by the plants. To show that this was the case an analytical method was devised to show the presence of dichlobenil in the azalea leaves.

Method

The volatility of the compound and the presence of two chlorine atoms indicated it should be readily detectable by gas chromatography utilizing microcoulometric or electron capture detectors. A review of the literature indicated a method was available involving an acetone extraction and purification of the extract prior to gas chromatographic analysis (1). It occurred to us that perhaps extraction and purification could be accomplished by continuous steam distillation and simultaneous extraction of the distillate. This procedure would be especially useful for microcoulometric detection since, unlike electron capture detection, it is not greatly affected by nonhalogenated compounds. Therefore, a method based on the principle was developed.

Procedure

The plant material to be assayed was macerated in a blender with water to form a slurry. In our analysis we used 24 g of azalea leaves. The slurry was placed in a 2 l boiling flask and water was added to bring the volume to 1 l. The flask was attached to a continuous steam distillation-solvent extraction apparatus described by Nickerson and Likens (3). Although this special apparatus was used, it is felt that other types continuous distillate-extraction glassware would suffice. A 50 ml flask containing hexane was attached to the solvent side of the apparatus and distillation-extraction was carried out for 6 hrs.

After the extraction period, the hexane was transferred to a sedimentation tube and carefully concentrated to 2 ml using a stream of nitrogen. A 10 µl aliquot was analyzed with a gas chromatograph equipped with a Dohrmann microcoulometric detector. The column was 4' x 1/4" packed with 5% SE-30 on Gas Chrom Q. A column temperature of 115^{o} C and a nitrogen carrier gas flow rate of 60 ml/min was used. The retention time of dichlobenil was 3.8 minutes.

Results and Discussion

The recovery of 25 µg of dichlobenil added to 25 g of plant material was 100% by this steam distillation method. Less than 6 hours was required for complete recovery, but it was decided the longer time would be preferable to give greater confidence that naturally absorbed herbicide would be removed from the tissue. The hexane extract was free from interfering compounds. At maximum usable sensitivity of the instrument 20 nanograms of dichlobenil gave 50% of full scale deflection on the recorder. Thus, with the sample size and volume of final extract employed, the sensitivity of the method was approximately 0.2 ppm.

However, since there was no interfering peak, greater sensitivity could be achieved by increasing the sample size and the size of aliquot for injection. Also the extract could be reduced to a smaller volume as long as care was exercized to avoid loss of dichlobenil.

The leaves of the azalea plants from the treated greenhouse contained 1.1 ppm of herbicide. Thus, it can be seen that these plants did absorb appreciable amounts of dichlobenil. It is not anticipated that vapor uptake will pose a problem where there is air movement. However, it can be seen that there is a potential hazard where dichlobenil is used under relatively closed conditions.

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