Bull. Environm. Contam. Toxicol. 25, 55-58 (1980)

# An Efficient Device for Collecting Soil Samples for Pesticide Residue Analysis[1]

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Soil samples for residue analysis are often collected when granular formulations of pesticides are tested for efficacy. With such pesticide formulations, it would appear that collection of a small volume of soil from many sites rather than a large volume of soil from a few sites would yield more reliable measurements of residues because of the aggregated distribution that usually occurs when the granules are broadcast.

Recently, a sampling device for taking small plugs of soil for witchweed seed analysis was designed by the USDA Witchweed Methods Development Laboratory in Whiteville, North Carolina. This study was conducted to compare this sampler against one that collects a much larger volume of soil.

## METHODS AND MATERIALS

Soil sampling devices. The plug sampler[2] was modified by placing a lead collar (9.1 kg) around the axle inside of the collection drum (Fig. 1). The lead weight was required to assure that the plug cutters would fully penetrate densely packed soils. A digital counter (Sperry New Holland No. 26995) was fixed to a metal bar (0.3 x 3.8 x 10 cm) that had been welded to the axle support above the collection drum (Fig. 1). A 5 cm length of flexible steel accelerator cable housing was attached to the trip arm of the counter so that each plug cutter would register on the counter as the sampling device was pushed over the surface of the soil. Each plug cutter collected a 3.9 $cm^3$ volume (1 cm dia. x 5 cm length) of soil.

A device for collecting a large volume of soil was constructed by removing the bottom from a 10 gauge aluminum

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[1]Paper No. 6374 of the Journal Series of the North Carolina Agricultural Research Service, Raleigh, N. C.

[2]Plans for the soil plug sampling device can be obtained by contacting R. Eplee.

0007-4861/80/0025-0055 $01.00

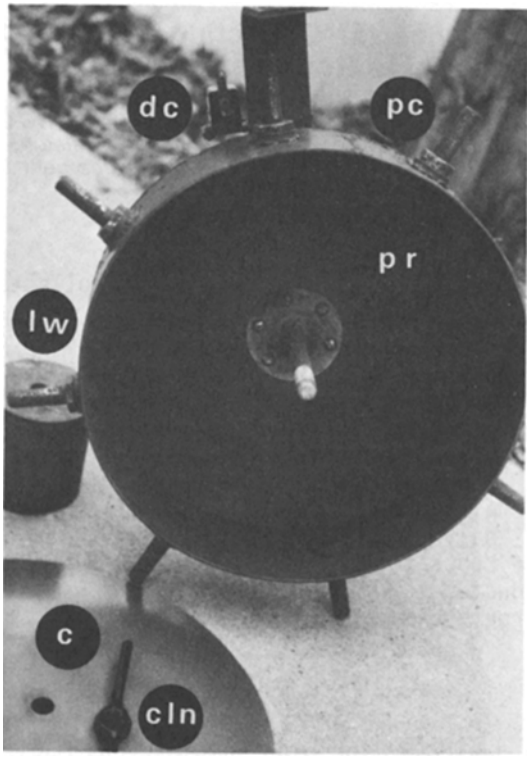


Fig. 1. Photograph of plug sampling device showing the plug reservoir (pr); plug cutters (pc); digital counter (dc); lead weight (lw), the cover (c) and cover lock nut (CLN).

pot (43 cm dia.). Teeth were cut into the bottom rim and handles attached to the sides to facilitate operation of the sampler in densely packed soils. The sampler collected 7,258 $cm^3$ volume (1451.5 $cm^2$ area x 5 cm depth) of soil.

Pesticide treatment and sample collection. Mirex 10:5 imported fire ant bait was broadcast at a rate of 2.8 kg/ha over a fescue grass pasture near Tabor City in Brunswick County, North Carolina, on June 24, 1978.

Stratified random transect and grid sampling designs were used for the plug and large area samplers, respectively. Immediately following the application of mirex bait, plug samples were taken from 10 contiguous 3x30 m transects while large area samples were taken from a system of 3x3 m grids (total of 100 grids). Random numbers were drawn to determine the transect or grid to be sampled. A sampling regimen of 400, 800, and 1200 plugs was chosen for comparison to the large area samples; and 5 samples were collected for each treatment. The samples

were placed individually in paper bags, transported to the laboratory and stored at -20 C until analyzed.

Pesticide residue and statistical analysis. Samples were thawed, dried at 25 C for 48 h and sifted to remove plant matter. For each sample, duplicate 100 g and 200 g subsamples were removed and analyzed according to the methods of MARKIN et al. (1972). Samples were shaken with 300 ml of a 50% benzene: 25% ethyl acetate: 25% toluene solution for 6 h. Two hundred ml were decanted, concentrated, and washed 3 times with concentrated sulfuric acid. After neutralization, interferences were removed on a nonactivated Florisil column and measured by GLC.

The chromatograph was a Tracor Model 222 equipped with a $^{63}$Ni detector. The column was U-shaped glass (122 x 0.2 cm i.d.) packed with 5% QF-1 on Gas Chrom Q (80/100). The nitrogen carrier was supplied at a flow rate of 70 ml/min. to the column and a purge flow of 20 ml/min. Temperatures were as follows: oven, 175 C; detector 290 C; inlet, 225 C. Mirex concentrations were determined using the peak-height method calibrated against standards of known concentration. Recovery studies, using mirex in concentrations varying from 0.25 to 5.0 ppb were done to determine the efficiency of the analytical method. Two fortified samples were analyzed with each set of experimental samples. Recoveries averaged 85% with a range of 70 to 90%.

The data were subjected to an analysis of variance and the duplicate 100 and 200 g subsample measurements for each of the 5 replicate samples were averaged and the mean, standard deviation and coefficient of variation (C) calculated for each sampling treatment.

## RESULTS AND DISCUSSION

Significant differences ($p = .05$) were not found between or among subsample measurements, which indicates that the plug and large area samples were equally reliable; however, more consistent results were obtained with the plug sampler (Fig. 2). Coefficients of variation for the 800 and 1200 plug sampling treatments were 5-17% lower than the C values for the large area sampler for the 100 and 200 g subsamples. The plug sampler in the 800 and 1200 plug sampling treatments collected 42 and 64%, respectively, of the volume of soil collected by the large area sampler. The plug sampler required ca. 2/3 the amount of time for sample collection and was also found to be a more versatile sampling device relative to the large area sampler because sample size could be varied considerably. The capacity of the collection drum was approximately 2000 plugs, which is equivalent to a surface area of 1580 $cm^2$ and a volume of 7800 $cm^3$ of soil.

Because of its ease of operation, versatility and consistency, we feel that the plug sampler will be a useful collection tool in soil-pesticide residue investigations.

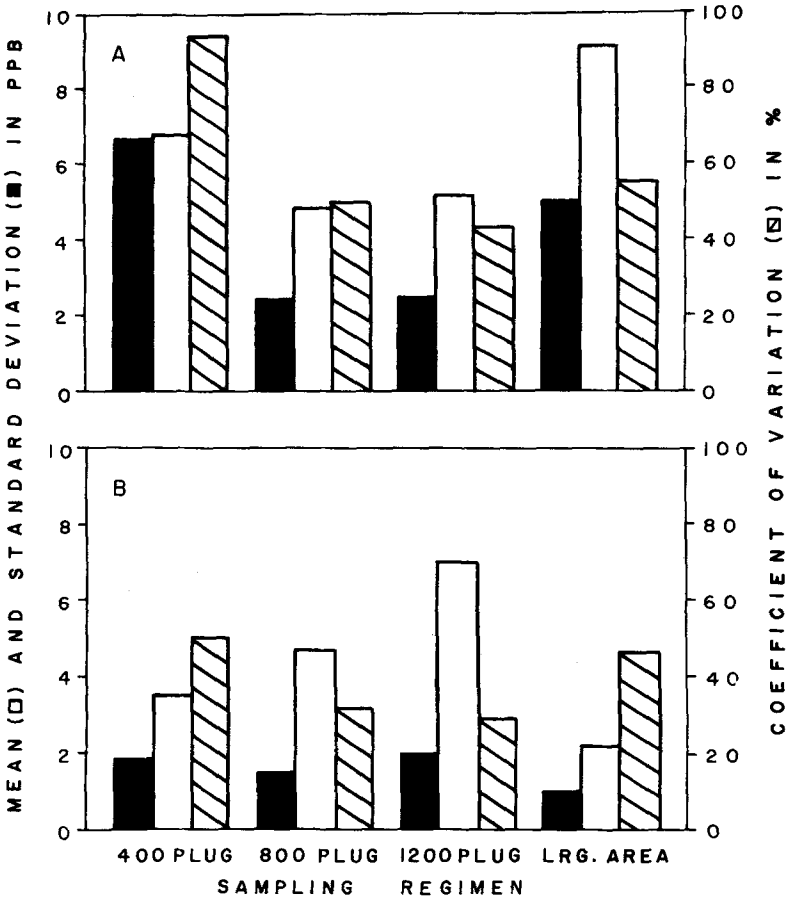


Fig. 2. Statistical analysis of mirex residues found in 100g (A) and 200g (B) subsamples from pasture soil samples collected with plug and large area samplers.

## ACKNOWLEDGMENTS

Thanks are extended to W. L. Jones for assisting in the residue analyses and to Gene Powell and John Murray for collecting the soil samples.

## REFERENCES

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