Table III. Recovery of EPTC Added to Macerated Crops

Crop	EPTC Added, p.p.m.	EPTC Recovered, %
Hexane Extraction Method		
Beans, dried	0.20	90
Carrots	0.50	78
Snap bean plants	0.10	100
Sugar beets	0.20	105
Tops	0.20	90
Direct Steam Distillation Extraction Method		
Blackberries	1.0	101
	0.1	100
Cantaloupes	1.0	90
	0.1	80
Onions, dried	1.0	86
Peas	1.0	79
Soil		
Sandy loam	6.0	92
Clay	2.0	99

absorbance at 440 mμ within 10 minutes after the color is formed.

During color formation, solution temperature should be 20° to 25° C. for optimum results. The reaction may not be completed at lower temperatures and side reactions can occur at higher temperatures. After addition of reagents, a delay in starting the 4-minute shaking period also can result in side reactions. The color formed in absence of sample extractives is normally stable after drying, but slow fading has been observed in the presence of crop extractives.

Determine the amount of EPTC in the sample by referring the net absorbance (the difference between absorbance before and after color formation) to a standard curve prepared by processing aliquots of the EPTC standard solution in a similar manner. Typical absorbance values are listed in Table I.

The colorimetry is not significantly affected by the small quantities of sample extractives introduced into the concentrated sulfuric acid. Typical recovery values are presented in Table II for EPTC added to a variety of crop extracts.

Discussion

Thirty consecutive colorimetric determinations of 400 γ of EPTC over a 7-week period of routine analyses by a single operator have resulted in a relative average deviation of ±3.4%.

EPTC is miscible in all proportions with most organic solvents but is only slightly soluble in water (375 ± 15 p.p.m. at 20° C.). It is readily extracted with hexane from a variety of crops (Table III). The hexane extraction procedure has been employed for most crop residue analyses to date. However, the recently adapted direct steam distillation method of extraction is being used currently for all soil and most crop samples. The latter method has the significant advantages of combining the extraction and cleanup steps, and of eliminating the handling of large quantities of hexane. Crops with a high starch content, such as potatoes and dried beans, cannot be extracted conveniently by this method because of excessive foaming and scorching. Some typical recoveries of EPTC added to macerated crops, employing both extraction methods, are given in Table III.

Acknowledgment

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HERBICIDE RESIDUES

Improved Extraction Procedure for the Determination of EPTC Residues in Potatoes

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An improved method of extracting the herbicide, EPTC, from potatoes is described. Potatoes are blended with isopropyl alcohol and Skellysolve B (*n*-hexane). After filtering, hexane separates and is collected. The aqueous isopropyl alcohol is extracted with hexane. The combined hexane extracts are evaporated and EPTC is determined. Stable emulsions do not form when isopropyl alcohol is used. Recovery of 0.1 p.p.m. of EPTC from potatoes ranged from 93.5 to 99.0%. Analysis of potatoes treated with EPTC for weed control showed no residue.

Ethyl *N*,*N*-di-*n*-propylthiolcarbamate (EPTC, Eptam) is a selective pre-emergence herbicide which will control grassy and certain broadleaf weeds. A method by Batchelder and Patchett (*1*) for determining EPTC residues has been developed. It involves extraction of the herbicide from crop material with Skellysolve B (*n*-hexane) and separation from plant waxes and pigments by steam distillation. EPTC is then hydrolyzed to di-*n*-propylamine. The amine is determined colorimetrically after reaction with carbon disulfide in the presence of cupric ion and ammonia to form the yellow cupric dithiocarbamate.

Extraction of EPTC from potatoes by blending with hexane produces emulsions which are difficult to break. Recovery of EPTC using the above method after hexane extraction is erratic. Extraction with hexane–isopropyl alcohol eliminates the formation of stable emulsions and good recovery of EPTC is obtained.

Table I. Recovery of 0.1 P.P.M. EPTC from Potatoes

Code	Check Value, γ	Recovery, % (Corrected for Check)
A	0.3	100.0
B	0.7	96.0
C	0.8	93.5
D	0.3	94.0
E	0.2	99.0
F	0.7	99.0

Extraction Procedure

Thoroughly macerate about a dozen potatoes in their own liquid in a 1-gallon blender. Weigh a 200-gram portion of the blended tissue, transfer to a 1-quart blender, and blend with 200 ml. of isopropyl alcohol (reagent grade) for 30 seconds. Add 300 ml. of Skellysolve B (technical) to the mixture and blend for 2 minutes. Pour the contents into a large, coarse porosity, sintered-glass funnel, and filter by suction. Use 25 ml. of hexane (used hereafter to designate technical Skellysolve B) to rinse the blender and funnel. Then transfer the contents of the suction flask to a 500-ml. separatory funnel and allow the layers to separate. Drain the aqueous isopropyl alcohol (lower) layer into a beaker and the upper hexane layer into a 500-ml. Erlenmeyer flask with a 24/40 standard-taper joint. Re-extract the isopropyl alcohol–water mixture twice with 50-ml. portions of hexane, using the latter to rinse the suction flask and beaker each time. Combine the hexane extracts, and distill off the solvent as in step 2 of the hexane extraction method (*1*). Then use the subsequent steps of the method (*1*) for cleanup and determination of the herbicide.

Results and Discussion

Recovery studies with potatoes were made using the isopropyl alcohol–hexane extraction procedure. Twenty micrograms (0.1 p.p.m.) of EPTC in hexane was added to each of six 200-gram portions of blended potato tissue. The herbicide was then blended in, 200 ml. of isopropyl alcohol was added, and the procedure was completed as described. Check samples were also carried through the procedure. Table I shows the recoveries and check values obtained.

The average of the six recoveries was 96.9%. The average of the six check values was 0.5 γ of EPTC.

Potato plants, 8 to 10 inches tall, were treated with EPTC in 1958 for weed control. Using a randomized block design, a comparison was made between two granular formulations, clay and vermiculite, and an emulsifiable concentrate. One application of each formulation was made on July 23 using a hand sprayer. The granular clay formulation was applied at a rate of 3 pounds per acre. The granular vermiculite and emulsion formulations were applied at both 3 and 4.5 pounds per acre. The potato plants were also treated with DDT, parathion, Maneb, and sodium arsenite. The tubers were harvested on October 11. Analyses of the tubers for EPTC from each of the treatments using the method described showed no residue.

Acknowledgment

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SEED DISINFECTION

Fungicide and Dye Distribution in Liquid Seed Treatment

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The mercurial distribution produced in liquid seed treatment (Panogen process) was determined by a new chemical method which permits mercury assay of individual kernels of seed. Distribution statistics based on whole kernel assay differs but slightly from the statistics with the radioactive method, where only part of the kernel surface was examined. Because of vapor action, here illustrated by simultaneous treatment with nonvolatile tracer and labeled mercurial, a reasonably uniform mercurial distribution is obtained in laboratory and commercial treatment, irrespective of treatment conditions and liquid dosage. More or less uniformly colored treated seed is of no value in judging the mercurial distribution.

The mechanism of liquid seed treatment (Panogen process) was recently studied by radioactive tracer methods with particular attention to distribution (*3*). This method accounts only for the amount of fungicide present on that part of the kernel facing the window of the Geiger tube, roughly one sixth of the total kernel surface. Since then methods have been developed for the assay of the total mercury content of single kernels of treated seed. Gamma counting is performed at near 4π geometry by scintillation techniques (*2*). Irradiation of treated seed in a nuclear reactor produces by neutron activation the radioactive isotope mercury-197, which is determined by radioactive techniques (*5*). Finally, a new chemical method with spectrophotometric mercury determination has been developed for the mercury assay of single kernels of treated seed (*2*). This method, called flame analysis, is used in the present investigation.

The effects of important variables in seed treatment were studied, particularly the liquid dosage and treatment conditions. Commercially treated seeds were included. The relationships between distribution statistics based on beta counting and whole kernel assay are illuminated. Regression analysis is used to get the relation between fungicide uptake and kernel size. The importance of vapor action is demonstrated in an experiment involving simultaneous treatment with a nonvolatile tracer and the labeled mercurial fungi-