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## **Background Report Reference**

**AP-42 Section Number:** 9.2.2

**Background Chapter:** 4

**Reference Number:** 9

**Title:** "Factors Affecting the Vapor Loss of EPTC from Soils" in Weeds

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mate (R-1856), and S-ethyl hexahydro-1-H-azepine-1-carbo-  
 thioate (R-4572). The soil temperature was adjusted by  
 shading to approximately the same temperature before  
 spraying with each compound. The tests were run on  
 clear, sunny, warm days. The results (Table 4) indicate

Table 4. Comparative losses by vaporization of various thiolcarbamate herbicides after application as sprays at 3 lb/A to the surfaces of moist and dry soil.

Herbicide applied at 3 lb/A	Soil temp start & final F	Water collected in trap ml	Vapor loss in first 30 min % of applied
<b>On moist soil</b>			
EPTC.....	95-116	21	27.3
PEBC.....	97-113	20	20.2
R-1607.....	93-115	20	24.5
R-1856.....	93-116	22	28.5
R-4572.....	95-113	20	14.9
<b>On dry soil</b>			
EPTC.....	102-124	18	8.3
PEBC.....	107-134	21	4.9
R-1607.....	107-143	23	4.5
R-1856.....	107-143	22	6.6
R-4572.....	106-129	18	2.6

that R-1856 and EPTC were lost by vaporization at about the same rate from moist soil. PEBC and R-1607 were somewhat less volatile than EPTC on both moist and dry soil. R-4572 was much less volatile than the other thiolcarbamates on moist and dry soil.

Recovery test.

In a test for recovery, EPTC was applied as a spray at 3 lb/A to a porcelain pan (8 x 12 inches) which rested on a pile of soil in a flat. When the soil temperature under the pan was 90 F, 49% of the applied EPTC was recovered in the three traps in one hour. When

the soil temperature was 126 F, 55% of the applied EPTC was recovered. In this latter run, all the water evaporated from the porcelain in one hour, but some of the emulsifying agent remained as a film on the porcelain. Therefore, it is not known whether the recovery by this vapor trapping method was only 55% of the total applied, or whether this was all that was lost by vaporization since some of the EPTC probably remained in the film of emulsifying agent which remained on the porcelain pan.

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Factors Affecting the Vapor Loss of EPTC from Soils<sup>1</sup>

REED A. GRAY and ANDRE J. WEIERICH<sup>2</sup>

INTRODUCTION

THE vapor trapping apparatus described in the previous report (1) was useful for identifying the vapor and showing that the main mechanism of disappearance of EPTC from soils was by vaporization. It was also helpful for determining the loss of EPTC during short periods, but it was inconvenient for determining the loss over periods of several days. Also, it appeared that the recovery of EPTC vapor using the vapor trapping apparatus was not complete. Therefore, in order to study the loss of EPTC more thoroughly, the amount of EPTC remaining in the soil was determined by steam distilling the EPTC from the soil samples and analyzing (2) the EPTC in the distillate. The factors affecting the loss of EPTC from soils that were examined included soil moisture content, soil type, depth of incorporation, sunny or cloudy skies, temperature, humidity, rainfall, sprinkler irrigation, volume of water in the spray, and certain combinations of these factors. A re-

Abstract. The disappearance of EPTC (ethyl N,N-di-n-propylthiolcarbamate) from soils was studied by steam distilling the remaining EPTC from the soil samples and analyzing the distillates. The most important factor affecting the loss of EPTC from soil was the amount of moisture in the soil. During the first 15 minutes after spraying on the soil surface, 20% of the applied EPTC disappeared from dry soil, 27% from moist soil and 44% from wet soil. The loss was 23, 49, and 69% after one day and 44, 68, and 90% after six days on dry, moist, and wet soils, respectively. No significant loss of EPTC occurred the first few hours after applying granular EPTC to dry soil or soil containing 4 to 6% moisture, but on soil at 15% moisture the loss from granules was 60% in two hours. There appeared to be a critical moisture level for each soil, above which large losses of EPTC occurred after application to the surface or after shallow incorporation. Depth of incorporation was the second most important factor in EPTC loss from soils. Incorporation to at least 2 to 3 inches was necessary to prevent large losses of EPTC when light rain or sprinkler irrigation followed treatment. EPTC was lost at a faster rate on sunny days than cloudy days. Increasing the temperature from 32 to 60 F, increased the rate of EPTC vaporization from moist soil but had no effect on the loss from dry soil.

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view of the literature pertaining to the vaporization of EPTC (1) is not repeated here.

#### MATERIALS AND METHODS

Santa Cruz loamy sand was used in most of the tests, and unless indicated otherwise, this was the soil used. The pH of this soil was 5.1; it contained 5% clay and 4.6% organic matter. The soil used in the field tests was Sorrento loam, which had a pH of 7.2 and contained 18% clay and 2.8% organic matter.

In the first group of tests, one pint of soil was used for each sample. The soil was spread out  $\frac{3}{4}$  inch deep in aluminum flats  $7.5 \times 5$  inches by 2 inches deep. Different soil moisture levels were prepared by mixing eleven pints of dry soil in a 5-gallon cement mixer with 1000 ml of water and repeating this procedure with lesser amounts of water. The moisture content of each mixture was determined by drying one pint of it in an oven for 4 hours at 110 C. The remaining ten pints of soil at the same moisture level were measured into ten individual flats. EPTC in water solution was applied immediately to each of the ten flats of soil at a rate of 3 lb/A using a spray volume of 80 gal/A. The sprays were applied with a 2 ml volumetric pipette using a fast zigzagging motion in order to cover the entire soil area. A pipette was used to avoid spray drift or loss of EPTC during application. For soil incorporation tests, the same amount of EPTC was applied while mixing the soil in a 5-gallon cement mixer and then the soil was spread out evenly in the pans. Soil temperatures were measured at a depth  $\frac{1}{4}$  inch below the soil surface in all tests.

Granular applications were made at the same rate by weighing out 163 mg of a 5% EPTC granular formulation on 24/48 mesh Attaclay and sprinkling it evenly over the soil surface. As before, ten flats of soil of the same moisture level were treated at the same time. The flats of soil were steam distilled after various periods to determine the amount of EPTC remaining in the soil.

For the field tests, holes were dug which were exactly 12 inches square and 3.5 inches deep. The moist soil was removed from six holes and mixed in a wheelbarrow. A sample was removed for moisture determination. A certain volume of soil was measured out so that when it was replaced in the hole the loose soil was 3 inches deep. When  $\frac{1}{3}$  this amount was mixed with EPTC in a cement mixer and then placed in the hole on top of the other  $\frac{2}{3}$  of the untreated soil which was placed in the hole first and leveled, this gave uniform incorporation of EPTC to a depth of 1 inch. Treating other portions of the soil in a similar manner, EPTC at 6 lb/A was incorporated at depths of  $\frac{1}{2}$ , 1, 2, and 3 inches. A surface treatment was applied with a pipette for comparison. The rain that fell on the plots and the amount of water applied by sprinkling were measured using small cans for catching the water. Two soil samples were taken from each plot 3 days after treatment and again 6 days after treatment using a sharp edged can to remove a core of soil 4 inches deep and 3.87 inches in diameter. The soil in each core sample was steam-distilled to determine the EPTC content, and

the average for each treatment was determined. If the soil sample could not be steam distilled at once it was placed in a glass jar, sealed and kept in a freezer until it could be steam-distilled the following day or later.

The apparatus used for steam-distilling the EPTC from the soil samples is shown in Figure 1. One pint of

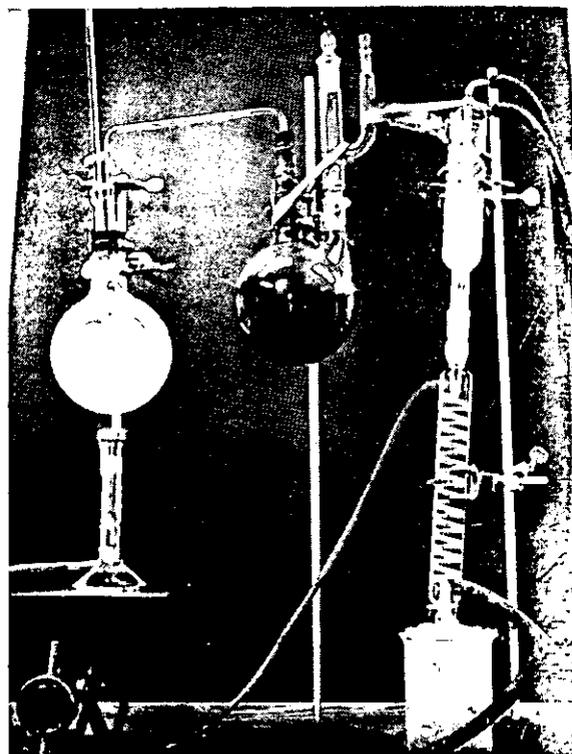


Figure 1. Steam distillation apparatus used for recovering the EPTC remaining in the soil after various treatments.

the soil sample to be analyzed was placed in the 2-liter two-necked round bottom flask shown in the center and the soil was covered with 400 ml of distilled water. The steam generator on the left was a 2-liter round bottom flask containing water and fitted with a 3-foot length of glass tubing for a safety valve, and a steam outlet tube. Steam was passed through the soil slurry through an inlet tube which almost touched the bottom of the flask containing the soil. Two condensers were attached to the flask containing the soil, and the distillate was collected in a 500 ml Erlenmeyer flask cooled in crushed ice. For each sample, 400 ml of distillate was collected and this was acidified to remove any interfering amines and extracted twice with 30 ml of isoöctane each time. The EPTC in the combined isoöctane extract was determined quantitatively following the procedure of Batchelder and Patchett (2), except that no clean-up solvent extraction was needed to remove background color, as is used for residue determinations of plant tissues.

To determine the recovery at zero time for each test, the same amount of EPTC spray solution that was applied to each flat or core sample area was also added to the same amount of soil in the flask and steam dis-

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tilled. The recovery of EPTC averaged 94% for 10 of these zero time standards run in five types of soil where 8.17 mg of EPTC was mixed in the flask with one pint of soil and steam distilled immediately. Recoveries were just as efficient with the granular EPTC as with the liquid, and soil type and depth of incorporation did not affect the recovery. The amount of EPTC lost was obtained by subtracting the amount found after the treatment in the greenhouse or field from the amount found at zero time in the flask.

### RESULTS AND DISCUSSION

*EPTC loss from dry, moist, and wet soil after spraying on-the soil surface.* On three clear, sunny days at 10 AM, EPTC was sprayed on the surface of dry, moist, or wet loamy sand contained in small aluminum flats in the greenhouse. The soil temperature at the start of each test was 77 F and the air temperature was 78 F. The relative humidity was near 21%. The rate of loss of EPTC was greatly increased as soil moisture was increased (Figure 2). The greatest rate of loss on dry,

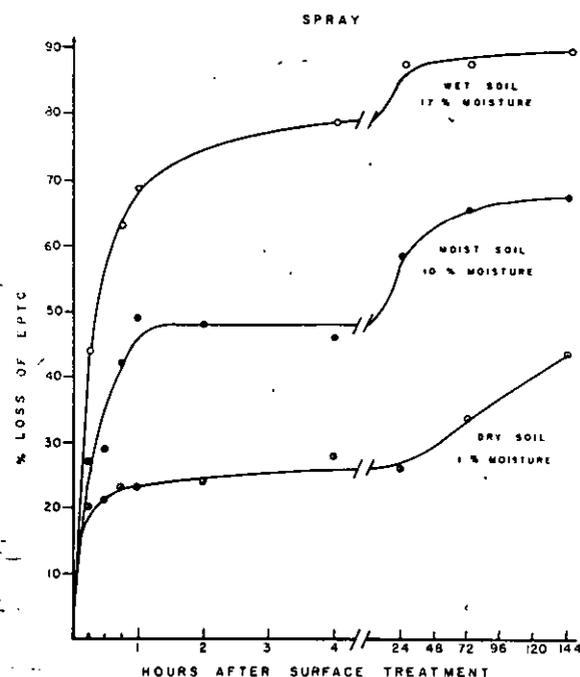


Figure 2. Loss of EPTC from dry, moist, and wet soil after application as a spray at 3 lb/A to the soil surface on sunny days.

moist, and wet soils occurred during the first 15 minutes after spraying EPTC on the soil. On dry soil, the rate of loss of EPTC slowed down after the first 15 minutes so that little more loss occurred the first few hours, or even in 24 hours. This indicated that most of the vaporization of EPTC from dry soil occurred while the spray was drying which usually required about 10 minutes. After the spray had dried, EPTC appeared to be adsorbed rather strongly to the dry soil.

The slower loss from dry soil suggests that one might spray one extra pound of EPTC (4 lb/A instead of 3)

on dry soil, then incorporate one day later and get the same results as incorporating immediately. If there needs to be a delay of 15 to 30 minutes before incorporating EPTC on moist soil, using an additional 1 lb/A should help to make up for the loss. The loss was so rapid from wet soil in 15 minutes that application of EPTC to wet soil should be avoided.

*EPTC loss when applied as a granular formulation to the surface of dry, moist, and wet soil.* Small flats of loamy sand  $\frac{3}{4}$  inch deep were sprinkled with granular EPTC at 3 lb/A. The material was applied at 10 AM to dry, moist, or wet soil in the greenhouse on three cloudy days. The soil temperatures at the start ranged from 77-79 F, the air temperatures ranged from 77-79 F, and the relative humidities varied from 15 to 32%. The results (Figure 3) indicate that very little

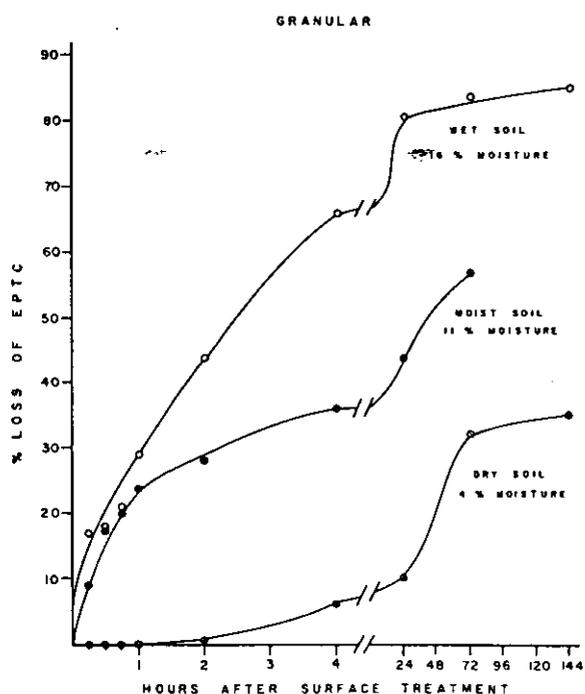


Figure 3. Loss of EPTC from dry, moist, and wet soil after application as a granular formulation at 3 lb/A to the soil surface on cloudy days.

EPTC was lost the first few hours after application of granular EPTC to the surface of dry soil, but a considerable amount was lost in 3 days. On the other hand, EPTC was lost quite rapidly the first two hours after applying the granules to moist and wet soil, even though the sky was overcast. The results indicate that incorporation may be delayed several hours after application of granular EPTC to dry soil but not after application to moist soil.

Another test with granular EPTC was run in the small aluminum flats in the same way as in the previous experiment, except that more moisture levels were used, and the experiment was run on a clear, sunny day. The temperature of the dry soil ranged from 79-86 F, the air temperature was 81-82 F and the relative humidity was 9-13%. The results (Figure 4) show again

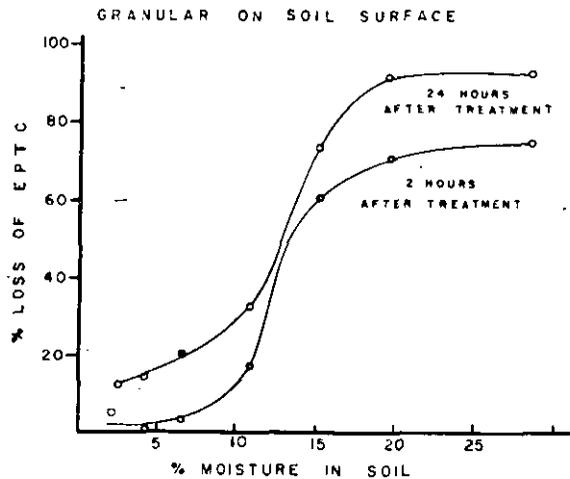


Figure 4. Effect of soil moisture content on the loss of EPTC from granules applied to the soil surface at 3 lb/A.

that the loss of EPTC was negligible the first 2 hours after applying granular EPTC to air dry soil (2.2% moisture) or from soil low in moisture content (4.4 to 6.6% moisture). When the soil moisture content was increased to 11% there was a considerable increase in loss (17%), while increasing the moisture content to 15.4% caused a tremendous increase in EPTC loss (60%) in 2 hours. The advantage of granular formulations over sprays of EPTC appears to be on dry soils or soils low in moisture content when incorporation cannot be carried out immediately.

**Sunny day compared to a cloudy day.** A test was run on a clear sunny day and on a cloudy day by applying granular EPTC on the surface of wet soil (16.1% moisture). The relative humidity was near 5% during the sunny day and 23% during the cloudy day. The air and soil temperatures varied between 77 and 79 F in both tests. EPTC vaporized much faster during the first 15, 30, 45, and 60 minutes on a sunny day than on a cloudy day (Table 1). However, after 6 hours the loss

Table 1. Effect of sunny and cloudy days on the loss of EPTC from granules on wet soil.

Hours after treatment	Loss of EPTC from granules, percent of applied	
	Sunny day	Cloudy day
1/4	24	17
1/2	32	17
1	41	21
2	47	29
4	67	44
6	72	66
24	75	71

on the cloudy day almost caught up to the loss on a sunny day. This may be explained by the soil drying out faster on the sunny day, so that the rate of loss slowed down considerably after two hours. Because of the slower drying of the soil on the cloudy day, the rate of EPTC loss didn't slow down until after 4 hours.

**Comparison of surface and incorporated treatments at different moisture levels.** In the small aluminum flats, EPTC was applied at 3 lb/A as a surface spray and by

incorporation at the same time, using soil at seven different moisture levels. The amount of EPTC remaining in the soil after 24 hours was determined. The results which are summarized in Table 2 show that incorpora-

Table 2. Effect of soil incorporation and soil moisture on the loss of EPTC after application as a spray at 3 lb/A.

Moisture in soil, percent	Loss of EPTC in 24 hours, percent of applied	
	Surface	Incorp 3/4 in
1.4	12	0
3.6	17	0
5.8	21	2
10.2	34	6
14.6	69	14
19.0	79	23
27.8	73	27

tion 3/4 inch deep greatly reduced the loss of EPTC at all soil moisture levels. However, at moisture levels of 14.6% and greater, considerable amounts of EPTC were lost in just 2 hours after incorporating. In soils containing 10% moisture or less, very little EPTC vapor loss occurred after incorporation and the soils behaved about the same as dry soil. With the surface sprays, the decrease in loss of EPTC going from 19% moisture to 28% moisture was probably due to oversaturation of the soil as shown by the slight puddling of the water on top of the soil at the higher moisture level.

**Effect of soil type on EPTC loss.** An experiment similar to that just described was carried out using five different types of soil in the small aluminum pans in the greenhouse and determining the EPTC remaining after 24 hours. The different soil moisture levels were prepared by mixing pints of each air-dried soil with 150, 100, 75, 50, 25, and 12.5 ml of water. All treatments were applied at 10 AM on sunny days or partially cloudy days. The air temperatures varied from 78-84 F, the soil temperatures ranged from 80-84 F and the relative humidity ranged from 5-20%. In all soil types tested, incorporation 3/4 inch deep reduced the EPTC loss greatly at all moisture levels (Figure 5). There was very little if any loss of EPTC from the five dry soils or soils low in moisture content in 24 hours after incorporating the EPTC immediately to a depth of 3/4

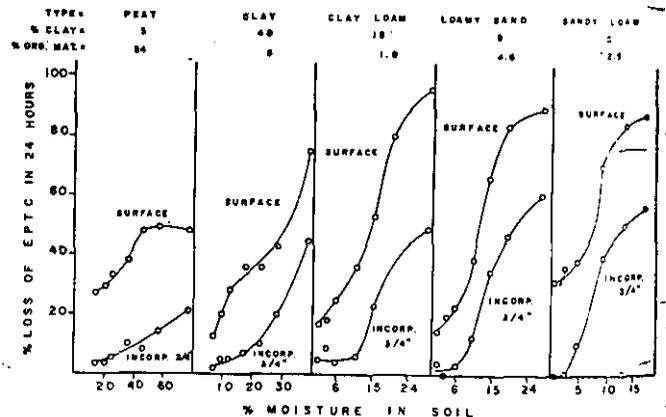


Figure 5. Effect of soil type, incorporation and soil moisture content on the loss of EPTC from soils after application as a spray at 3 lb/A.

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inch. However in the wetter soils, considerable loss of EPTC occurred in the incorporated treatments. There appeared to be a critical moisture level for most soils, above which the rate of loss of EPTC increased greatly. This critical moisture level was about 5-6% for the sandy soils, 11% for the light clay loam, 23% for heavy clay and around 40% for the peat soil. When not incorporated, about 90% of the applied EPTC was lost in 24 hours from the wettest sandy and light clay soils, but the loss was less (75%) on the wettest heavy clay soil, and considerably less (48%) on the wettest peat soil.

*Effect of temperature on the loss of EPTC.* A small aluminum pan containing one pint of air-dried (1% moisture) Santa Cruz loamy sand and a pan of the same soil containing 14% moisture were placed in an environmental growth chamber at 32 F and EPTC was applied as a surface spray. After 24 hours, the soil was steam distilled. This procedure was repeated at growth chamber temperatures of 40, 60, 80, and 100 F. The soil temperature was very near the air temperature in these tests. The experiment was repeated and the average losses are shown in Table 3. Increasing the temperature

Table 3. Effect of temperature on the loss of EPTC after application as a spray at 3 lb/A to the soil surface.

Air temp F	Loss of EPTC in 24 hours, percent of applied*	
	Moist soil 14% moisture	Dry soil 1% moisture
32.....	62.4	12.0
40.....	67.0	12.2
60.....	81.0	9.2
80.....	80.8	12.2
100.....	75.3	15.7

\*Average of 2 tests.

from 32 to 60 F caused an increase in the loss of EPTC from the moist soil but not from the dry soil. The loss at 80 F from the moist soil was about the same as at 60 F and increasing the temperature from 80 to 100 F appeared to decrease the loss slightly. This may be explained by more rapid drying of the soil at 100 F, thus allowing soil adsorption of the EPTC sooner than at 80 F.

*EPTC loss during rainfall.* In one experiment, EPTC was sprayed on the surface of moist (11% moisture) loamy sand contained in small flats outside while it was raining. The soil and air temperatures were both 57 F and the relative humidity was 84% and rising. At the same time, some flats of the same soil were sprayed in the greenhouse where the soil temperature was 74 F, the air temperature was 75 F, and the relative humidity was 35%. The loss of EPTC in 15 minutes was 20% outside in the rain and 21% in the greenhouse. After one hour the losses were 48% in the rain and 44% in the greenhouse, and after 4 hours the losses were 57% in the rain and 59% in the greenhouse. This experiment showed that EPTC was lost very rapidly after application to a moist soil surface under high humidity and raining conditions.

*Effect of depth of soil incorporation on EPTC loss under field conditions.* In small field plots, EPTC was incorporated at 6 lb/A at depths of 1/2, 1, 2, and 3 inches

in moist (15.7% moisture) Sorrento loam, and also applied as a surface spray. The plots were treated at 3 PM on a clear sunny day when the air temperature was 71 F and the soil temperature was 73 F. During the two days following treatment, 0.9 inch of rain fell on the plots, so the test was repeated except that on the second day after treatment the plots were covered with a clear plastic sheet which was supported 4 inches above the soil to prevent rain from wetting the treated soil. The soil moisture content was 14.5%, the air temperature was 63 F and the soil temperature was 64 F when treated. The data from these two tests (Table 4) show that the

Table 4. Effect of depth of incorporation and rainfall on the loss of EPTC after application to moist loam in field plots at 6 lb/A.

Depth of incorporation, inches	Loss of EPTC, percent of applied*			
	0.9 Inch rain		No rain	
	3 days	6 days	3 days	6 days
0.....	85	89	33	36
1/2.....	80	88	18	43
1.....	46	45	12	14
2.....	35	40	8	19
3.....	23	25	13	15

\*Average of two samples for each sampling date.

deeper the EPTC was incorporated into moist soil, the better it was retained. Incorporation only 1/2 inch deep was not much better than the surface treatment in the plots moistened by rain. Much less EPTC loss occurred when the soil dried out and stayed dry than where it was kept moist by rain.

This test was repeated under the same conditions except that no cover was needed to shield the plots from rain, and the soil was drier (11% moisture) when treated. Using the drier soil, the 1/2 inch depth of incorporation gave about the same loss (17%) in 3 days as deeper incorporation of 1, 2, or 3 inches (9-15% loss). In one treatment the soil was compressed by applying 1 lb/sq inch pressure on the soil after incorporating 2 inches deep to see if packing the soil would influence the loss. The results showed approximately the same loss in 3 days from the compressed soil (15%) as from the loose soil (14%).

*Effect of sprinkler irrigation and depth of incorporation on EPTC loss under field conditions.* Two sets of five holes were prepared several feet apart in the field and filled with Sorrento loam (17.6% moisture) which contained EPTC incorporated at different depths at 6 lb/A. One set of plots was sprinkled with a sprinkler set to give intermittent sprinkling for about 8 seconds out of 100 seconds needed for the sprinkler to make one revolution. By this method of irrigation, the plots received 0.24 inch of water from the time of treatment at 10 AM until the end of the experiment at 4 PM. During this period the other set of five plots received no irrigation. The test was run in June on a clear sunny day when the air temperature at the start was 65 F and the soil temperature six hours after treatment was 65 F in the sprinkled plots and 86 F in the non-irrigated plots. Two core samples were taken from each plot six hours after treatment and the EPTC determined. The average losses (Figure 6) show that when EPTC

unidentified fractions representing about 17% of the total C<sup>14</sup> activity were observed in the extract.

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## A Vapor Trapping Apparatus for Determining the Loss of EPTC and Other Herbicides from Soils<sup>1</sup>

REED A. GRAY<sup>2</sup>

**Abstract.** An apparatus for trapping the vapors of ethyl N,N-di-propylthiolcarbamate (EPTC) and other herbicides from soils under field conditions is described. The herbicide vapors were collected in cooled traps along with ice crystals, identified by gas chromatography and determined quantitatively. The amount of EPTC lost and trapped in 30 minutes ranged from 22 to 38 % of that applied to the surface of six different types of wet soils. The amount lost from dry soils was much less than from moist soils. ~~Letting the surface of six different types of wet soils. The amount lost from dry soils was much less than from moist soils.~~ Letting the surface of freshly worked moist soil dry out to depth of 1/2 inch before spraying with EPTC, greatly reduced the loss by vaporization. Immediate incorporation prevented any loss of EPTC from dry soil and greatly reduced the loss from moist soils. After spraying EPTC on dry soil, sprinkling with small amounts of water increased the loss. Comparison of several thiolcarbamate herbicides showed that n-propyl N,N-di-n-propylthiolcarbamate (R-1607) and n-propyl ethyl-n-butylthiolcarbamate (PEBC) were lost by vaporization at a slower rate than EPTC, and S-ethyl hexahydro-1-H-azepine-1-carbothioate (R-4572) was lost at a much slower rate than the other herbicides tested.

INTRODUCTION

HERBICIDE losses from soil are sometimes reported as volatility losses, without giving proof of vaporization from the soil. An herbicide may also disappear by other mechanisms, including catalytic decomposition on contact with the soil, photoinactivation, irreversible adsorption on the soil, microbial decomposition, leaching, and plant uptake. Sodium N-methyldithiocarbamate is not volatile, but on contact with moist soil it decomposes completely in one hour producing methyl isothiocyanate which is volatile (8). Since other herbicides may also decompose to some extent on contact with soil, volatility losses determined by bioassay methods in closed chambers or by radioactive measurements of the amount remaining in planchets may be misleading as well as having other disadvantages. Therefore, losses attributed to vaporization should be confirmed by trapping and identifying the material arising from the soil. An apparatus for trapping the vapors after treating the soil with EPTC and other herbicides under field conditions is described in this report.

The loss of EPTC from soils has been reported by a number of workers who used smaller soil samples and

different methods than those employed here. Ashton and Sheets (2) reported that the loss of EPTC from a free liquid surface was 57 micrograms per sq cm per hour at 30 C, but it was less volatile from a soil surface. Radioactive measurements on 2-gram soil samples which were enclosed in a chamber with a dish containing EPTC-S<sup>35</sup> showed that the EPTC was adsorbed more on dry soil than on moist soil. Fang and Theisen (6) found the EPTC was completely lost within one hour when a small amount was applied to a glass surface or a stainless steel planchet, but it was partially adsorbed on filter paper. Vernetti and Freed (9) reported that EPTC and PEBC when mixed in sandy soil gave vaporization losses of 18.3 and 16.6%, respectively, in dry soil, and 33.4 and 24.8% in moist soil in 24 hours. Antognin et al. (1) reported that the activity of EPTC was much greater when it was incorporated into the soil than when it was applied to the soil surface. This finding has been confirmed by many workers in field tests conducted in many different areas. Fang et al. (7) measured the disappearance of S<sup>35</sup> labeled EPTC from 1-gram soil samples in planchets and found that the loss of EPTC during drying, increased with the soil moisture content. EPTC was very persistent in dry soil.

Danielson et al. (5) found by bioassay methods that the persistence of EPTC in soil was strongly influenced by the solvent carrier used for applying the EPTC. EPTC dissipated much more rapidly when applied in kerosene than in water or acetone. Danielson and Gerner (4) found that air movement was also an important factor in the persistence of surface-applied EPTC. In addition to describing the vapor trapping apparatus in this report is also concerned with the amount of EPTC lost and recovered by the vapor trapping technique during short time periods after different soil treatments.

MATERIALS AND METHODS

Vapor trapping apparatus.

A clear plastic box 24 inches long, 12 inches wide and 6 inches deep was constructed from 3/8 inch Plexiglas glass sheeting. Two holes 3/8 inch in diameter were drilled in one end of the chamber to serve as air intake. A hole 3/4 inch in diameter was drilled in the opposite end and it was fitted with a one-hole rubber stopper.

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<sup>1</sup>Received for publication July 11, 1964.  
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## GRAY : VAPOR TRAPPING APPARATUS

containing a short piece of glass tubing as the air outlet. The outlet tube was connected with a short piece of rubber tubing to another "Z" shaped glass tube which was connected at the upper end to an empty Pyrex glass trap 1.2 inches in diameter and 15 inches long. It contained an inner glass tube 0.35 inch in inside diameter which extended to within 4 inches of the bottom of the outer tube. The first trap was connected to two more traps by means of short pieces of rubber tubing. The three traps were immersed in dry ice-acetone baths contained in Dewar flasks. A vacuum pump was connected to the third trap as shown in the complete apparatus (Figure 1).

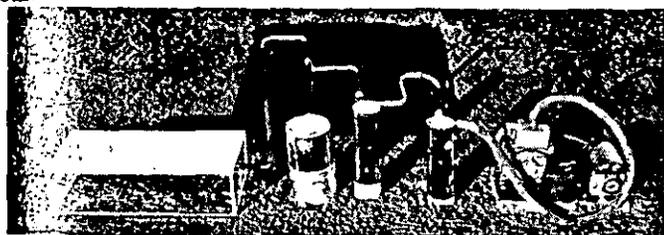


Figure 1. Vapor trapping apparatus for recovering vapors lost from the soil after application of EPTC and other herbicides under field conditions.

In operation, the plastic chamber was placed over the herbicide-treated soil, and soil was pushed up around the edges of the box to seal the sides from air movement. The vacuum pump was turned on and air was drawn through the chamber and through the traps at a rate of 25 liters per minute. The volatile EPTC vapor was picked up in the air stream and trapped in the cold traps along with the water vapor which came off the soil and collected as ice crystals in the traps. In several experiments the vapors were trapped from soil that was treated in the field. In other tests, metal flats 12 inches long and 8 inches wide containing soil 3 inches deep were treated with the herbicide and the flats were placed under the chamber immediately to collect the vapors. In all tests, emulsifiable EPTC was applied at 3 lb/A using 80 gallons of aqueous spray per acre. A volumetric pipette was used to apply the herbicides as a simulated spray. By moving the pipette back and forth with a rapid zigzagging motion over the entire soil area, good coverage was obtained. When dry soil was used in the flat, another small aluminum flat of the same area containing wet soil was placed in the chamber in front of the treated flat of dry soil to provide the same amount of moisture for condensing in the traps as when wet soil was treated. This was done to eliminate differences in recovery that might result when different amounts of water were trapped. Soil temperatures inside the chamber were taken with a thermometer placed  $\frac{1}{4}$  inch below the soil surface in a horizontal position.

### Determination of the amount of EPTC trapped.

The ice which collected in the three traps was melted, yielding a turbid solution having a strong odor of EPTC. The solutions from the three traps were combined and the traps were rinsed once with 15 ml of distilled water

which was added to the combined sample. The EPTC in the combined sample was extracted twice with iso-octane and determined quantitatively using the method reported by Batchelder and Patchett (3). As little as 0.02 ppm EPTC could be detected by this method. The step of extracting the benzene-soluble background color was omitted since no interfering materials were present in the clear aqueous condensate. A standard curve was prepared for each thiolcarbamate tested.

## RESULTS AND DISCUSSION

### EPTC vaporization loss from soil in the field.

In a field of freshly cultivated moist Sorrento loam on a clear warm day, EPTC was sprayed on the soil surface over a 2 sq ft area. The treated area was covered immediately with the plastic chamber and the vacuum pump was turned on. Only two dry ice traps were used in this first experiment and the traps were replaced with clean traps at 15 minute intervals. During the test, the air temperature inside the chamber rose slightly so that it was 6-10 degrees above the outside air temperature. The soil moisture content sampled to a depth of 3 inches was 16.1% at the start. The results, which are summarized in Table 1, show

Table 1. EPTC lost as vapor and recovered with the vapor trapping apparatus during short time intervals after spraying on the surface of moist soil in the field.

Collection period, min	Water trapped, ml	Air temp in chamber, F	EPTC trapped as vapor	
			Mg	Pct of applied
0-15	10.6	95-99	6.35	10.5
15-30	11.8	99-103	6.13	10.2
30-45	12.0	103-104	1.45	2.4
45-60	12.7	104-104	1.20	2.0
60-90	25.3	104-104	1.04	1.7
90-120	24.3	104-104	0.44	0.7

that over 20% of the EPTC that was applied to a moist soil surface was lost by vaporization and recovered in the traps in 30 minutes. The rate of loss of EPTC was greatest during the first 30 minutes after application and then the rate of loss decreased considerably with time as the soil dried out.

Another test was conducted in the field the same day as the previous test and under the same conditions except the surface of the soil was allowed to dry out for two hours after cultivating the moist soil. The soil moisture content was 13.1% in the soil sampled three inches deep at the start, but the soil surface was dry about  $\frac{1}{2}$  inch deep. After application to this dry soil surface, only 3% of the applied EPTC was lost by vaporization and recovered in the traps along with 30 ml of water during the first hour. In later experiments greater recoveries were obtained using three traps instead of two.

The colorimetric method used in these determinations indicated that the material coming off the soil was the intact thiolcarbamate. The odor of the trapped material also indicated that it was EPTC. The identity was confirmed by running another field test and subjecting a portion of the trapped aqueous solution to gas chromatographic analysis. Two peaks were obtained with a hydrogen flame detector which corresponded to