KILLING WEED SEEDS IN SOIL WITH CHLOROPICRIN (Tear Gas)

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A supply of soil comparatively free from weed seed is desirable not only for topdressing turf but also for the establishment of new seed beds. While well prepared compost is relatively free from weed seed the fact is that much of the compost used on golf courses is not well prepared. The illustration on page 64 shows that such compost may be full of weed seed. The use of soils, as for instance woods earth, which contain few seed of important turf weeds, is advisable, but such soils often are difficult to obtain. The destruction of seed by composting soil is a slow process which is not always practical.

Heat has been used to destroy weed seed in soil. Both the steaming and the baking processes have been employed for this purpose. Although these methods are effective they frequently damage the physical properties of soil and are somewhat expensive.

Agricultural workers have tested many promising chemicals in the hope of reducing costs of the partial sterlization of soil. Although good results have been obtained with some of them they generally have been unsatisfactory because of the excessive cost or due to the fact that they have remained too long in the soil and thereby have limited the growth of the plants for which this soil had been used.

In recent years chloropicrin (one of the tear gases) has been used successfully for partial sterilization of soil, chiefly for the control of nematodes. Although it had not been tested for the

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Nine samples of compost collected from golf courses showed remarkable differences in weed infestation when placed in flats and subjected to growing conditions and offer an explanation of the greater abundance of weeds in the greens of some courses as compared with others. Some of the flats are almost weed free. Tests of this kind have demonstrated that it is not unusual to plant 70,000 weed seed on a putting green in a single topdressing.

control of weed seed in soil, certain results obtained with it seemed to indicate some possibilities of useful application in turf maintenance work. Therefore numerous tests with chloropicrin were conducted at the Arlington Turf Garden. The results of some of these tests will be outlined in the pages that follow.

Chloropicrin (Trichloronitromethane—CCl_3NO_2) is an organic compound made up of carbon, chlorine, nitrogen and oxygen. It is a colorless liquid about one and three-fourths as heavy as water. It is practically non-inflammable. Although almost insoluble in water it dissolves readily in gasoline and almost all other organic solvents. It vaporizes to form a very pungent tear gas which has great penetrating power.

Chloropicrin has a pronounced odor. It is a respiratory and

lachrymatory (tear producing) irritant and, as such, was used in the World War. Prolonged exposure to relatively high concentrations of the gas causes serious detrimental effects on the respiratory system. Chloropicrin is not dangerous to work with, however, as a person can not willingly withstand even a small fraction of the concentration that would be dangerous to human life. Although it has a corrosive action on the skin there is no irritation if the liquid momentarily comes in contact with the skin.

Numerous research workers since 1907 have experimented with chloropicrin for fumigating grain and plants and for the killing of fungi. It is now extensively used as a fumigant and as an insecticide in warehouses and grain elevators. Applied at the rate of 150 pounds to the acre it has given 83 per cent control of nematodes and 52 per cent increase in the yield of pineapples. As a fumigant it has the advantage of high toxicity, practically complete freedom from fire and explosion hazards, relative non-reactivity to metals, and ability to penetrate bulk commodities. Another advantage over other common fumigants is the pronounced warning which its odor and lachrymatory effects give, even when it is present in minute quantities.

The first tests made with chloropicrin at Arlington in the spring of 1937 were concerned with the sterlization of small lots of compost in wooden boxes. The promising results which were obtained led to more extensive tests.

The clay-loam soil used for conducting the experiments described was from the surface of an area heavily infested with weeds. In some instances additional weed seed were added to the soil two or three weeks before it was treated. Supplementary tests were made on the regular compost material used at Arlington.

For most of the tests galvanized iron garbage cans of 1 cubic-foot capacity were used. When tightly covered these cans provided relatively gas tight containers. The soil to be treated was in all cases screened before it was used. Three-fourths of a cubic foot of soil was put into each of the cans and the required amount of chemical was placed in a hole in the center of the soil mass. The rates are given in terms of actual weight of the liquid rather than in fluid ounces. The soil in these cans was left covered for several days and then placed in regular greenhouse flats, watered as needed and placed in the greenhouse or out-of-doors wherever conditions were most favorable for the germination of weed seed. Counts of the various weeds were made when new seedlings ceased to appear. Usually this was from four to six weeks after the soil was placed in the flats.

Several methods were tested for treating field plots both in turf and in cultivated soil. These consisted of sprinkling the liquid directly over the surface or in applying it in holes or narrow furrows. Various depths and spacing of holes and furrows were tested. After treatment the holes and furrows were filled with soil and the treated area immediately covered to prevent escape of the gas.

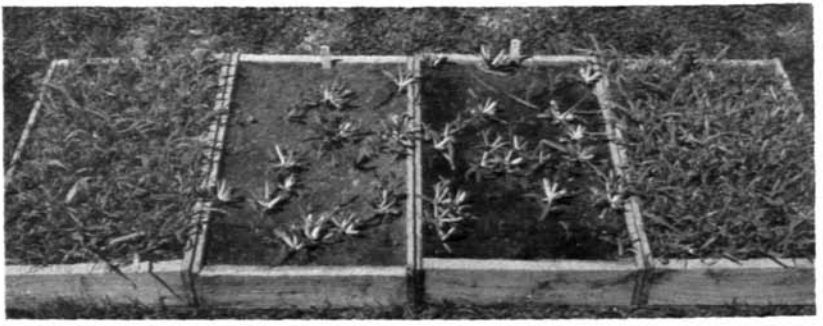
Various covers were tried, including canvas, kraft glue-coated paper, concrete water-proof paper and a layer of wet soil. The best results were obtained by using the kraft glue-coated paper. A shallow trench was dug around the plot to be treated and the edges of the paper laid in the trench and covered with soil. Four days after treatment the cover was removed and any gas remaining in the soil was permitted to escape into the air. Tests on the time necessary to keep the soil covered after treatment have indicated that no further killing is accomplished

Method of application of the liquid chloropicrin into the soil. The waterproof paper in the background was to be placed over the plot and sealed with sterilized soil as soon as the application was completed.

after four days. A shorter period than this has given reduced effectiveness.

Piles of soil were treated in several ways. Two of the methods tried seemed to be more satisfactory than the others. The most satisfactory of these consisted of building up a pile of screened soil in 6-inch layers, with the first layer somewhat less than 6 inches thick. Chloropicrin was sprinkled over each layer as the pile was built up to the desired size. A more uniform distribution of the chloropicrin was secured by first emulsifying it in water so as to obtain a larger quantity of liquid to sprinkle over the soil.

The other method consisted of building up a pile of compost and making holes to the bottom of the pile at distances of from 6 to 8 inches apart. Chloropicrin was poured into each hole, which was then filled in with compost to a depth of from 6 to 8 inches, another application made, and so on until the hole was entirely filled.



Effect of moisture on partial sterilization of compost with chloropicrin at rate of 20 ounces to a cubic yard. Left, untreated; left center, soil dry; right center, soil medium wet; right, soil saturated during treatment. Weeds are chiefly crabgrass and goosegrass.

The piles of compost were covered with kraft glue-coated paper, several widths of this paper being glued together for this purpose. When treating soil in boxes this paper was used as a cover by gluing it to the edges of the box. Other waterproof paper and even canvas which is kept moistened gave satisfactory results.

Observations at Arlington indicated that the variable results secured with chloropicrin were due probably to variations in soil moisture, temperature and species of weeds present. The effect of each of these factors was separately tested and the results are summarized in this article.

Effect of definite percentages of moisture on partial sterilization of compost with chloropicrin. Chloropicrin applied at 15 ounces to a cubic yard at soil temperature of 77° F. Back row, left to right, 40 per cent and 50 per cent moisture and untreated soil; front row, left to right, 10 per cent, 20 per cent, and 30 per cent moisture present in compost at time of application. Note most effective sterilization when soil contained 20 and 30 per cent moisture at time of application.

Influence of Soil Moisture

The effect of varying amounts of moisture in the soil at the time of the application of chloropicrin on the percentage of weed seed killed has been studied. Varying quantities of water were added to air-dried soil, ¾-cubic foot of soil was placed in each iron garbage can, chloropicrin was added at rates varying from 10 to 30 ounces to a cubic yard, and the cans were immediately covered. After four days the lids were removed and the different lots of soil placed in flats to permit germination of the viable weed seed.

Results from numerous such experiments showed that the best kill of weed seed was obtained when the soil was medium wet. As the soil became more nearly saturated with water the

rate of kill fell off rapidly. In the case of all weeds except crabgrass there was not as much kill in the extremely dry soil as in the medium wet soil.

In the experiment for which the data are given in the following table chloropicrin was applied at the rate of 20 ounces to a cubic yard with the soil temperature at 77° F. Approximately 90 per cent of the plants that developed in the untreated area were crabgrass. The figures show that about the same percentage of crabgrass seed was killed in the dry as in the medium wet soil but that when the soil was very wet the percentage of kill was less. For other weeds the percentage of kill was better in the medium wet than in the dry or the wet soil.

EFFECT OF SOIL MOISTURE ON PERCENTAGE KILL OF WEED SEED BY CHLOROPICRIN AT THE 20-OUNCE RATE AT 77° F.

Moisture content of soil	Percentage kill of seed	
	Crabgrass	All weeds
Air-dry (0-10 per cent)	97	74
Medium-wet (20-30 per cent)	97	91
Very wet (40-50 per cent)	25	26

Other series of tests made with soil containing different proportions of moisture consistently indicated that best results may be expected when chloropicrin is applied to this type of soil when it contains from 20 to 30 per cent of moisture.

EFFECT OF TEMPERATURE

Experiments on the effect of temperature on the power of chloropicrin to kill weed seed were set up in a manner similar to those used in the investigations on the effect of moisture. Rates varying from 5 to 20 ounces to a cubic yard were applied to ¾-cubic-foot lots of soil at temperatures ranging from 32° F. to 104° F. After four days the covers were removed and the

Effect of temperature on killing of weed seeds in compost with chloropicrin at various rates. Temperatures from left to right, 32°, 50°, 68°, 86°, and 104° F. Rate of treatment from front to back, untreated, 5, 10, 15, and 20 ounces to a cubic yard. Note stimulation of crabgrass in soil treated at 5-ounce rate and also at the 10- and 15-ounce rates at 32° F. Note also the most complete sterilization at highest temperatures and highest rates.

soil was placed in flats to allow the germination of remaining viable weed seed. At the same time equal volumes of untreated soil were held at similar temperatures for the same period of time and then placed in flats.

The number of seedlings appearing in treated soil at each temperature was compared with the number in the untreated soil at the same temperature. The untreated lots of soil held at different temperatures were also used to ascertain whether the short exposure to these different temperatures might in any way affect germination of the seed.

The following table shows that, with a few minor exceptions, percentages of crabgrass seed and of weed seed as a whole killed with applications of chloropicrin at rates of 10, 15 and 20 ounces to the cubic yard increased as the temperature rose from 32° F. to 104° F. At the highest temperatures with the heavier rates there was practically a complete kill of the weed seed.

At the 5-ounce rate with temperatures of 32°, 50°, 68° and 86° F., and at the 10-ounce rate at 32° F. there was actually an increase in the number of crabgrass plants. An increase of all weeds will also be noted in the table in the 5-ounce series at 32°, 50° and 68° F. The numbers of most weeds, however, were not increased. The increase in number of all weeds reported is due to the large proportion of crabgrass.

The results showed that weed seed are most easily killed when treatments are made at high temperatures. This fact probably accounts for the greater effectiveness of chloropicrin in compost or turf in mid-summer than in fall. In June and July 98 per cent of the weed seed was killed by the treatments when made out-doors. In late September and in October under similar conditions except for the temperature the kill was 76 per cent.

PERCENTAGE CONTROL OF WEED SEED AS INFLUENCED BY TEMPERATURE

Temperature	Number in check	Percentage control			
		5-oz.	10-oz.	15-oz.	20-oz.
Crabgrass					
32° F.	203	+ 76	+ 30	23	71
50° F.	183	+ 93	47	52	95
68° F.	134	+ 188	25	86	93
86° F.	151	+ 29	89	97	95
104° F.	148	63	90	99	100
All Weeds					
32° F.	360	+ 42	1	24	45
50° F.	304	+ 30	44	45	43
68° F.	261	+ 29	14	81	58
86° F.	300	30	74	83	87
104° F.	333	50	69	98	99

The + indicates the percentage of increase in weeds.

EFFECT ON DIFFERENT WEEDS

The seed of different turf weeds vary in their susceptibility to chloropicrin. The percentages of weed seed killed at a 20-

ounce rate at both favorable temperatures and soil moisture have been taken from numerous experiments. These weeds have been listed here in the order of the decreasing susceptibility of the seed to chloropicrin in the soil.

Seed of chickweed, witchgrass, quackgrass and sedge have proved to be easily killed. Clover seed was generally resistant to chloropicrin, although the results were somewhat inconsistent. Other tests indicate that the seed of plantain, *Poa annua,* foxtail, Jimson-weed and *Muhlenbergia* also are easily killed.

The percentage of goosegrass seed killed by a treatment at the 20-ounce rate varied under similar environmental conditions. In some instances 50 per cent of the seed was killed while in others the chloropicrin appeared actually to stimulate germination so that there were 95 per cent more goosegrass seedlings in the treated than in the untreated soil. In this case, however, the soil temperature during germination was too low for the normal germination of goosegrass seed.

AVERAGE PERCENTAGE KILL OF SEED OF VARIOUS WEEDS BY CHLOROPICRIN AT 20-25 OUNCE RATE

Weed	Percentage kill
Chickweed	100
Witchgrass	100
Quackgrass	98
Sedge	95
Crabgrass	83
Clover	23

CHLOROPICRIN IN COMPOST PILES

Excellent results have been secured by treating 5 cubic yards of compost at a time at the rate of 30 ounces to the cubic yard. In this experiment a pro-rated quantity of chloropicrin was sprinkled over each 6-inch layer of compost as the pile was

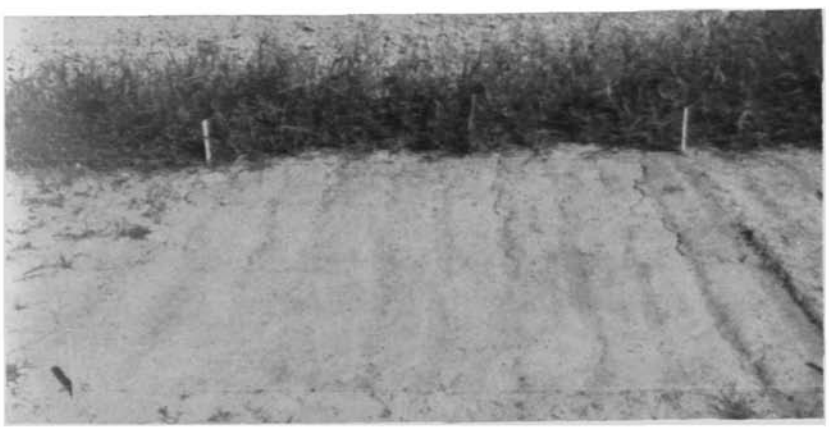
built up. The pile was then sealed with kraft glue-coated paper. Several flats of the untreated compost taken before treatment and flats of compost taken after treatment were placed in a favorable place for the germination of any viable weed seed. After two months counts were made of the seedlings in both sets of flats. Apparently the crabgrass, chickweed and *Poa annua* seed had been killed 100 per cent. On the other hand the resulting stand of clover was reduced only 15 per cent and that of goosegrass was increased 40 per cent. This experiment was set up in October, but similar results were obtained from like experiments conducted in June, July and August.

Results in Turf and Cultivated Areas

Chloropicrin has also been used on a turf badly infested with weeds in order to destroy weed seed in the soil and avoid removing the upper layers of soil before reseeding. In September, 1937, chloropicrin at the rate of 8 pounds to 1,000 square feet was applied to turf in furrows 2 inches deep. The treated areas were covered with canvas. Ten days later the treated and untreated areas were seeded to Kentucky bluegrass and two months after treatment there was a better stand of grass on the treated than on the untreated plots. The weeds were completely controlled. The color of the grass was also better and the favorable result of the treatment was clearly evident nearly a year later.

In recent experiments on turfed areas it has been shown that the application of chloropicrin at the rate of 14 pounds to 1,000 square feet applied to a depth of 3 inches will destroy weed seed in the surface soil. In several series of experiments chloropicrin was applied to 4 by 4 foot plots of turf at rates of 12, 14, 27.5 and 55 pounds to 1,000 square feet, and the areas covered with kraft glue-coated paper.

A week following the application of the chloropicrin a 2 by 2 foot section in each of the experimental plots treated at 27.5 and 55-pound rates was scraped to remove the dead vegetation. These sections as well as untreated plots were then seeded to redtop.



Sterilization of soil in the field with one application of chloropicrin to a depth of 2 inches at the rate of 4.6 pints (8 pounds) to the 1,000 square feet. Check untreated strip in the background.

After 10 days the redtop had come up to good stands on the untreated areas and on the plots treated at the 27.5-pound rate but to an irregular stand on the plot treated at the 55-pound rate. After six weeks the stand of grass on the plots treated at the 27.5-pound rate was twice as high and twice as dense as that on the untreated plots.

These results indicated a decided stimulation of the grass with the chloropicrin when applied to a depth of 3 inches at the rate of 27.5 pounds to 1,000 square feet. A toxic effect on the grass seedlings was evident only when chloropicrin was used at the high rate of 55 pounds to 1,000 square feet. Allow-

ing a longer period of time for aeration of the soil between treating and seeding probably would have eliminated this toxic effect.

These experiments were conducted on both Kentucky bluegrass and Bermuda grass turf. All vegetation, even the tough, resistant rhizomes of Bermuda grass and the bulbs of wild garlic which are usually difficult to destroy, was killed at the 14-pound rate. On the plots treated at the 12-pound rate, *Poa annua* began to appear after one month, and other weeds came in at the edge of the plots two weeks afterwards.

Soil from the treated and the untreated plots was placed in flats and taken into the greenhouse to check the germination of the weed seed under more favorable conditions. In the soil treated at the 14-pound rate there were 75 per cent fewer crabgrass seedlings and 87 per cent fewer weed seedlings of all kinds than in the untreated soil.

PERCENTAGE KILL OF WEED SEED IN TURF WITH CHLOROPICRIN APPLIED BY SEVERAL METHODS AT THE 14-POUND RATE

	Crabgrass	*Poa annua*	All weeds
Applied in holes	76	28	46
Applied in furrows	75	100	87
Applied by sprinkling can	+7	21	+7

The + indicates the percentage increase over the check.

Experiments were set up to compare the efficiency of the several methods of application. In a series of experiments chloropicrin was applied to 4 by 4 foot plots at rates of 12, 14, 27.5 and 55 pounds to 1,000 square feet. In one set it was put into narrow furrows 3 inches deep and spaced 8 inches apart; in another, in holes made 3 inches deep and 8 inches apart; and in a third set surface applications at the three heaviest rates were made with a sprinkling can. In all cases

the treated areas were promptly covered with glue-coated kraft paper. All vegetation apparently was killed by all three of the methods. However on plots treated by the sprinkling can method a medium stand of weeds was reestablished within a month and a half, even when treated at the 27.5-pound rate. On the other hand, no vegetation appeared two months later on plots on which the chloropicrin was applied either in furrows or in holes to a depth of 3 inches at the 14, 27.5 or 55-pound rates.

Soil from each treated plot and from untreated plots was taken into the greenhouse to obtain the germination of viable weed seed. The following data taken from the record of plots treated at the 14-pound rate shows that the best kill of weed seed follows when chloropicrin is put into furrows.

Control of Microorganisms

As the experiments on the killing of weed seed by chloropicrin were in progress numerous samples of the treated and untreated lots of soil were used by Nathan R. Smith, of the Bureau of Plant Industry, United States Department of Agriculture, to determine the power of chloropicrin to kill fungi, bacteria and protozoa. The results of these tests, together with others conducted by Mr. Smith, will be published soon in Soil Science Society of America, Proceedings, Vol. 3.

Mr. Smith reports that the effect of chloropicrin on the microorganisms of the soil varies in much the same manner as it does on weed seed. Temperature was found to have a definite influence on the efficiency of chloropicrin. At 32° and 50° F. the killing was not as great as at temperatures of 68°, 86° and 104° F. At the high temperature of 104° with the 15 and 20-ounce rates "the number of fungi

was reduced from 130,000 per gram to 5 per gram; actinomycetes from 28,000,000 to 6,000 and 8,000, respectively; and the total viable organisms from 180,000,000 to around 3,000,000 per gram."

Growth of grass in compost treated with chloropicrin. Red top seed was sown in a 4-inch strip of each flat eight days after the application of chloropicrin. Flats in back row contained untreated soil. Coming forward, the rates used were 5, 10, 15, and 20 ounces to a cubic yard. Temperatures at time of treatment from left to right were 104°, 86°, 68°, 50°, and 32° F. There was no retardation in germination of red top seed even in the soil receiving the heaviest rate. On the contrary, growth was stimulated in the treated flats.

Soil moisture was also found to have a decided influence on the efficiency of chloropicrin in destroying microorganisms. The fungi and actinomycetes were killed best when the soil contained from 10 to 15 per cent moisture. The bacteria and protozoa were killed at the same moisture content and also when the soil contained more moisture. The conclusion reached was that from the standpoint of controlling soil microorganisms "a temperature above 20° C. (68° F.) and a soil moisture content of 10 to 15 per cent are optimum conditions for treatment with chloropicrin and that these conditions seem as important as the rate of application."

Tests were also made to determine the effect of the treatment on the later development of soil organisms. At the lower concentrations of chloropicrin fungi and bacteria were found

to develop rapidly within 10 days. In the following 10 days the soil that had received the higher applications also showed some increase in microorganisms. Nitrates decreased slightly and ammonia increased slightly in the treated soil within 20 days after treatment.

Some Advantages and Disadvantages of Chloropicrin

Advantages

The chloropicrin method of partially sterilizing soil has certain advantages over the common methods now in use. It requires no expensive equipment, which may be a distinct advantage in treating small lots of soil. Unlike the heat process of sterilization, chloropicrin has no harmful effect on the physical properties of the soil. The soil can be used soon after it has been treated with chloropicrin as then it will not be toxic but actually may be stimulating to grass. In one case when weeds were controlled completely grass seed germinated satisfactorily when planted only two days after the chemical was applied. Soil can be treated successfully with chloropicrin in piles or in turf without incurring the cost of moving the soil in and out of containers for treatments.

Disadvantages

It requires careful covering of the treated soil to prevent escape of gas. As its effectiveness is decidedly reduced at low temperatures, it can not be used to advantage at all seasons. Heavy rates must be used to accomplish complete control of some of the most troublesome turf weeds. Where seed of the more resistant weeds are abundant in soil the cost of the treatment is high.