TB 427 (1934) USDA TECHNICAL BULLETINS UPDATA
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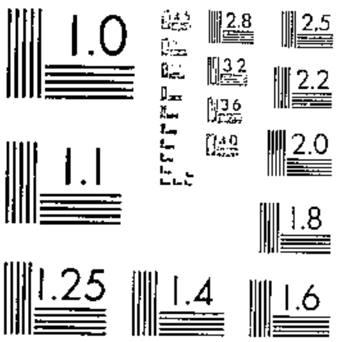

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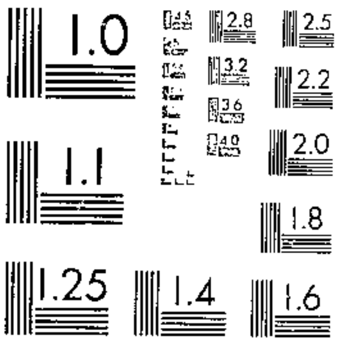

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Technical Bulletin No. 427 August 1934

UNITED STATES DEPARTMENT OF AGRICULTURE
WASHINGTON, D.C.

THE USE OF NAPHTHALENE AGAINST THE JAPANESE BEETLE

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INTRODUCTION

Naphthalene has been investigated extensively by entomologists, both as an insecticide and as a repellent for controlling different insect pests that inhabit soil. During the past 10 years it has been tested against the Japanese beetle (*Popillia japonica* Newm.). Practically all of these experiments with naphthalene were made with the resublimed compound, the crude naphthalene sometimes recommended for soil treatment being too variable in composition to be satisfactory. In view of the widespread interest of entomologists and the public at large in the control of the Japanese beetle, the authors have attempted to bring together experimental data and pertinent facts relating to the use of naphthalene against this insect. The research on this problem has led to a better understanding of the insecticidal value of the compound, and, as a result of these investigations, a quarantine procedure (*54*)[2] for destroying the immature stages of the beetle was adopted in March 1929 and has been in operation ever since.

[1] The writers wish to acknowledge the assistance rendered by R. Wagner, K. B. Rogers, and H. W. Coward, formerly on the staff of the Japanese Beetle Laboratory, in conducting the analytical work for this investigation.

[2] Italic numbers in parentheses refer to Literature Cited, p. 25.

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PROPERTIES AND ESTIMATION OF NAPHTHALENE

PHYSICAL PROPERTIES

Naphthalene, $C_{10}H_8$, is a white crystalline compound, occurring in coal tar and obtained by crystallization from the fraction boiling from 356° to 572° F. (180–300° C.). It crystallizes in shining plates that have a characteristic odor and a bitter aromatic taste. Insoluble in cold water and very slightly soluble in hot water, naphthalene is readily soluble in alcohol, benzene, and other organic solvents. It melts at a temperature of 176.2° F. (80.1° C.) and boils at 424.2° F. (217.9° C.). Roark and Nelson (*39*) reported that the specific gravity of a saturated atmosphere at 77° F. (25° C.) and normal pressure, as compared with air, was 1.0004. The vapor pressure is low, ranging from 0.02 mm at 32° F. (0° C.) to 0.21 mm at 95° F. (35° C.). Roark and Nelson (*38*) have calculated that 0.01 pound of naphthalene is required to saturate 1,000 cubic feet of air at 32° F. (0° C.); 0.03 pound at 59° F. (15° C.); 0.035 pound at 68° F. (20° C.); 0.04 pound at 77° F. (25° C.); 0.06 pound at 86° F. (30° C.); and 0.09 pound at 95° F. (35° C.). It is seen that under normal conditions only a small quantity of the compound is required to saturate the atmosphere.

QUANTITATIVE ESTIMATION

BY FORMATION OF NAPTHALENE PICRATE

When undertaking extensive experimentation with an insecticide, it is desirable to have some method for estimating quantitatively the concentration of the compound in order to determine the uniformity of dispersion and the persistence of the material under different conditions. A study was made of the method of Küster (*26, p. 1101*), as modified by Colman and Smith (*8*), to determine the reliability of the procedure for estimating small quantities of naphthalene. This method depends upon the precipitation of naphthalene as naphthalene picrate in the presence of excess picric acid.

Considerable variation was found in the recovery of naphthalene by the picric acid method, although in general the results were consistently low. This variation in the quantity of naphthalene recovered can be attributed to several factors: (1) Some naphthalene sublimed in the upper part of the flask, after the compound was heated with picric acid, although it was shaken occasionally; (2) some naphthalene was absorbed in the rubber stoppers used to seal the flasks; (3) the substitution of cork stoppers eliminated absorption but introduced the factor of leakage; (4) some naphthalene did not react with the picric acid because crystals of naphthalene were occluded by naphthalene picrate; (5) the temperature of the solution during the process of crystallization influenced the yield; (6) the quantity of water used to wash the excess picric acid from the precipitate was a factor because, although naphthalene picrate is not soluble in picric acid, it dissolves appreciably in water. It is obvious that the determination of naphthalene as naphthalene picrate is a relatively slow procedure, and careful standardization of each step in the procedure is necessary to obtain consistent, accurate results.

BY OXIDATION TO PHTHALIC ACID

Calcott, English, and Downing (*5*) have reported that sulphonated naphthalene is oxidized practically quantitatively to sulphophthalic acids by means of vanadic acid in 70 percent sulphuric acid. The quantity of vanadic acid reacting with naphthalene can be determined from the quantity of standard potassium permanganate required to reoxidize the reduced vanadic acid. In the presence of hot concentrated sulphuric acid naphthalene reduces vanadic acid to the blue divanadyl salt, according to the following equation:

$$18H_3VO_4 + 18H_2SO_4 + C_{10}H_8 = 9V_2O_2(SO_4)_2 + C_6H_4(COOH)_2 + 2CO_2 + 46H_2O$$

The treatment of divanadyl salts with potassium permanganate in hot dilute sulphuric acid changes them into vanadic acid according to the following equation:

$$5V_2O_2(SO_4)_2 + 2KMnO_4 + 22H_2O = 10H_3VO_4 + 2MnSO_4 + 2KHSO_4 + 6H_2SO_4$$

The above equations show that 5 molecules of naphthalene are equivalent to 18 molecules of potassium permanganate; therefore, 1 cc of N/10 potassium permanganate is equivalent to 0.7117 mg of naphthalene.

An experiment was carried on to determine the concentration of sulphuric acid necessary to sulphonate the naphthalene within 1 hour at a temperature of 212° F. (100° C.) Fifty milligrams of naphthalene were added to 10 cc portions of acid of different strengths and heated in closed tubes for 1 hour. At the end of this period, the degree of sulphonation was determined as outlined by Calcott, English, and Downing (*5*). The following results were obtained:

Strength of acid:	*Degree of sulphonation*
5 cc of concentrated sulphuric acid plus 5 cc of water	None
7.5 cc of concentrated sulphuric acid plus 2.5 cc of water	10 percent.
10 cc of concentrated sulphuric acid	100 percent.
10 cc of 15-percent oleum	100 percent.

It was found that sulphonation was incomplete when less than concentrated acid was employed; hot concentrated sulphuric acid, or oleum, appeared to be necessary for the sulphonation of naphthalene. It was concluded that some oleum should be used in the sulphonating mixture to insure that any water present in the sample of naphthalene would not reduce the concentration of the acid to a point where the compound was not completely sulphonated. The sulphonating mixture finally decided upon was composed of 40 parts by volume of concentrated sulphuric acid and 60 parts by volume of 15-percent oleum.

Another experiment was conducted to determine the minimum temperature that could be used in oxidizing the sulphonated naphthalene to the sulphophthalic acids. A standard oxidizing solution was prepared by adding 63 g of pure ammonium metavanadate to 220 cc of distilled water and adding gradually with agitation 780 cc of concentrated sulphuric acid. A standard napthalene solution was prepared by adding 1.13 g of resublimed naphthalene to 40 cc of concentrated sulphuric acid and 60 cc of 15-percent oleum and heating the mixture for 1 hour in a closed flask. The solution was then boiled to remove sulphur dioxide, cooled, and made up to a volume of 1 l with distilled water. Ten-cubic-centimeter aliquot portions of the standard naphthalene solution were then mixed with 10-cc portions of the standard vanadic acid solution and heated for 1 hour at different temperatures. At the end of the oxidation period each sample was diluted to 500 cc with distilled water and titrated with N/10 potassium permanganate solution. The following equation was used in calculating the results:

$$\text{Mg naphthalene} = \text{total cc N/10 potassium permanganate} - \text{cc N/10 potassium permanganate in the reagent blank} \times 0.7117$$

The degree of recovery of the 11.3 mg of naphthalene in the samples by oxidizing at the different temperatures is given in table 1. From a consideration of the results it appears that the oxidizing temperature should be at least 266° F. (130° C.). The oxidation of naphthalene at this temperature is practically quantitative, the average error being less than 1 percent.

TABLE 1.—*The effect of the oxidizing temperature on the recovery of naphthalene by the vanadic acid method*

Oxidizing temperature		Naphthalene taken for analysis	Naphthalene recovered			Average recovery
			Maximum	Minimum	Average	
° F.	*° C.*	*Mg*	*Mg*	*Mg*	*Mg*	*Percent*
212	100	11.3	9.0	8.1	8.82	78.05
230	110	11.3	10.6	9.6	10.02	88.67
248	120	11.3	10.8	10.0	10.50	92.92
266	130	11.3	11.4	11.2	11.26	99.64
275	135	11.3	11.4	11.0	11.26	99.64
284	140	11.3	11.5	11.4	11.44	101.24
302	150	11.3	11.5	11.4	11.44	101.24

The determination of naphthalene by suphonating and oxidizing to the phthalic acids is a relatively rapid procedure which gives results in close agreement; this procedure is free from the empirical factors and the careful standardization in the different steps of analysis required to obtain consistent results by the methods under which the naphthalene is finally estimated as naphthalene picrate.

INSECTICIDAL ACTION OF NAPTHALENE VAPOR ON IMMATURE STAGES OF THE JAPANESE BEETLE

PHYSIOLOGICAL EFFECT

Naphthalene is generally considered by entomologists to be a weak insecticide; actually, molecule for molecule, it is a more effective fumigant than carbon disulphide, carbon tetrachloride, paradichlorobenzene, and other widely used organic compounds. Tattersfield, Gimingham, and Morris (*47*) found the vapor of naphthalene more toxic than paradichlorobenzene to *Aphis rumicis* L. Lehman (*27, p. 965*) reported that naphthalene was 10 to 14 times more toxic than paradichlorobenzene at the same concentration to the confused flour beetle (*Tribolium confusum* Duv.). In 1924 Fleming (*13*) tested the comparative effectiveness of 43 organic compounds as fumigants against the larvae of the Japanese beetle and found that of these chemicals only benzyl chloride, phenol, and ortho-cresol were more effective insecticides than naphthalene. A molecule of naphthalene vapor was considerably more effective against the larvae than was a molecule of paradichlorobenzene, nitrobenzene, bromobenzene, chlorobenzene, hexachloroethane, carbon disulphide, or carbon tetrachloride.

The effect of naphthalene vapor on the immature stages of the Japanese beetle is not clearly understood. When the creamy-white eggs or larvae of this insect are exposed to the vapor of naphthalene they take on a reddish color which ranges in intensity from light pink to mahogany brown, depending on the period of exposure. In view of the results reported by Shull, Riley, and Richardson (*41*) with the oriental cockroach (*Periplaneta*) *Blatta orientalis* L., this discoloration of *Popillia* eggs and larvae is probably not caused by any change in the chemical composition of the blood. It is possible that the change in the color is due to the reaction of the vapor with the fat bodies in the insect, naphthalene being recognized as a fat solvent. After larvae are exposed to naphthalene vapor for several hours they are unable to walk, but move the head and body convulsively at periodic intervals. As the treatment is prolonged, the larvae gradually become completely paralyzed. The paralyzing effect of vapor of naphthalene has also been observed by Rico (*37*) with the parasitic roundworm *Ascaris lumbricoides* L. This paralysis is followed by death.

RELATIVE RESISTANCE OF EGG, LARVA, AND PUPA OF THE JAPANESE BEETLE

A study was made of the resistance of the different immature stages of the Japanese beetle to the insecticidal action of naphthalene vapor. The insects were placed in suitable cages, without soil, and put into 1-l Florence flasks. Balls of naphthalene were suspended from the stoppers in such a manner that the insects in each flask were subjected to the vapor without coming in contact with the solid chemical. The flasks were maintained at a temperature of 70° F., and at intervals some insects were removed to determine the effect of the vapor. The eggs were placed on the surface of moist peat, kept in the dark, and examined daily until all of them had hatched or decomposed; the larvae and pupae were placed on the surface of moist soil in cross-section trays (*15, pp. 6–9*) and observed until the effect of the treatment could be determined. With each test, groups of insects were placed in flasks without naphthalene in order to eliminate the errors introduced by mechanical manipulation. In this experiment 3,800 eggs, 1,300 larvae, and 1,000 pupae were used. The mortality caused by different periods of exposure was determined by the following formula:

$$\text{Percent mortality} = \frac{\text{Number of untreated alive} - \text{Number treated alive}}{\text{Number of untreated alive}} \times 100$$

The results of this experiment are summarized in table 2:

TABLE 2.—*Effect of naphthalene vapor on immature stages of the Japanese beetle at 70° F.*

Period of exposure (hours)	Mortality of—			Period of exposure (hours)	Mortality of—		
	Eggs	Larvae	Pupae		Eggs	Larvae	Pupae
	Percent	*Percent*	*Percent*		*Percent*	*Percent*	*Percent*
12	23.5	5.0	8.0	60	59.4	100.0	
24	20.0	82.5		72	100.0	100.0	8.2
36	55.0	98.7		96	100.0		8.2
48	66.5	100.0	10.9	120	100.0		54.0

It was found that the different stages of the Japanese beetle vary in resistance to the vapor of naphthalene. It is apparent that the larva is the stage most susceptible and the pupa the least susceptible to the vapor.

EFFECT OF TEMPERATURE ON INSECTICIDAL ACTION

The rapidity of insecticidal action of fumigants on the immature stages of the Japanese beetle is known to be appreciably affected by the temperature of the atmosphere. An experiment was carried on with 6,000 larvae, at temperatures ranging from 50° to 80° F., to determine the extent to which temperature affected the period required for an atmosphere saturated with naphthalene to kill them. The larvae were removed from soil and exposed at the different temperatures to the vapor in the manner described while determining the resistance of the different immature stages. The results of the different tests are summarized in table 3. It was found that the period of time required for naphthalene vapor to kill the larvae varies inversely to the temperature. The prolonged exposure at the lower temperatures is probably necessary because of the decreased activity of the insect and the smaller quantity of the compound in a unit volume of air. At a temperature of 80° an atmosphere saturated with naphthalene contains 0.77 mg of the compound per liter. This quantity is reduced to 0.55 mg at 70°, 0.42 mg at 60°, and 0.36 mg at 50°. It is apparent that even if the activity of the insect were not reduced at the lower temperatures, a much longer period of time would be required for it to absorb an insecticidal dose of the vapor.

TABLE 3.—*Effect of temperature on insecticidal action of naphthalene vapor on larvae of the Japanese beetle*

Period of exposure (hours)	Mortality of larvae at—				Period of exposure (hours)	Mortality of larvae at—			
	50° F.	60° F.	70° F.	80° F.		50° F.	60° F.	70° F.	80° F.
	Percent	*Percent*	*Percent*	*Percent*		*Percent*	*Percent*	*Percent*	*Percent*
2				2.0	24	0	74.0	82.5	100.0
4				37.5	36	3.5		98.7	
6				65.8	48	32.6		100.0	
8				94.8	72	42.0	100.0	100.0	
12		5.0	6.0	100.0	96	90.3	100.0	100.0	
15				100.0	120	100.0	100.0		
18	5.0			100.0					

EFFECT OF RELATIVE HUMIDITY ON INSECTICIDAL ACTION

The relative humidity of the atmosphere generally does not affect the insecticidal action of a fumigant as markedly as does the temperature, but it was expected that the relative humidity would modify to some extent the toxicity of naphthalene vapor. An experiment was carried on with 5,400 larvae at a temperature of 70° F. to determine the extent to which the minimum lethal exposure in an atmosphere saturated with naphthalene was modified by the relative humidity. The larvae were placed in Florence flasks which were then conditioned for humidity by passing a stream of air of known humidity through them for a period of 24 hours. A relative humidity of 100 percent was obtained by bubbling the air through distilled water, a relative humidity of 56 percent by bubbling the air through a saturated solution of sodium bromide, a relative humidity of 26 percent by bubbling the air through a saturated solution of calcium chloride, and dry air was obtained by passing the air through concentrated sulphuric acid. After the flasks had been aerated for 24 hours the flow of air was stopped and a ball of naphthalene was suspended in each flask. It is realized that when the movement of the conditioned air through a flask was stopped the relative humidity in the flask was probably modified by the respiration of the insects, but it is believed that this factor was not of great importance. Groups of larvae were removed from the flasks at intervals to determine the effect of the treatment. The results of this experiment are summarized in table 4.

TABLE 4.—*Effect of relative humidity on insecticidal action of naphthalene vapor on larvae at 70° F.*

Period of exposure (hours)	Mortality of larvae at—			
	Zero relative humidity	26-percent relative humidity	56-percent relative humidity	100-percent relative humidity
	Percent	*Percent*	*Percent*	*Percent*
18	----------	24.0	40.0	86.0
24	73.3	83.1	95.0	94.7
48	100.0	100.0	100.0	100.0

The mortality of the larvae exposed to naphthalene vapor was increased with the increase in the relative humidity of the atmosphere. As the period of treatment was prolonged there was less difference between the mortality in the different atmospheres until, with an exposure of 48 hours, all larvae were dead.

TREATMENT OF POTTING SOIL WITH NAPTHALENE CRYSTALS

Nurserymen have a definite need for a method of treating soil to destroy larvae of the Japanese beetle before the soil is used for potting greenhouse plants. The use of soil that has had an insecticidal treatment to destroy this insect is required of nurserymen who ship plants with soil about their roots to points outside the quarantined area. Fleming and Baker (*14*) state that steam sterilization and fumigation with carbon disulphide have been found satisfactory for this purpose.

DOSAGE EFFECTIVE AGAINST LARVAE PRESENT AT TIME OF TREATMENT

It was desired to know whether the application of naphthalene crystals to potting soil could be used as an alternative method. A series of experiments were conducted with sandy loam, clay loam, and a mixture of clay loam and peat moss to determine the possibility of using naphthalene crystals for this purpose. These soils were carefully screened to remove large lumps, stones, and debris, and were then mixed in a moist condition with resublimed naphthalene in quantities ranging from 0.2 pound to 2.1 pounds of the chemical to a cubic yard of soil. The treated soil was then placed in 3-inch pots and set in the greenhouse. On the same day the treatment was applied to the soils, 5 active third-instar larvae were placed in each pot. A total of 3,300 larvae were used in this test. These pots of soil were maintained for 21 days at approximately optimum moisture for growing plants in a greenhouse where the temperature ranged from 45° to 66° F. Ten days after the larvae were placed in the soils, the pots were examined, and a record was made of the number surviving in each treatment. When the examination was completed and all larvae were removed from the pots, the pots were reinfested and allowed to stand for another 10-day period. At the end of this time another examination was made to determine the number of larvae surviving in each treatment. The mortality resulting from each treatment was then calculated according to the previously mentioned formula. A summary of these data is given in table 5.

TABLE 5.—*Effectiveness of naphthalene crystals in destroying larvae in different types of potting soil, the chemical being thoroughly mixed with the soil*

Crystals applied per cubic yard (pounds)	Mortality of larvae in—					
	Sandy loam		Clay loam		Clay loam and peat	
	When put in soil at time of treatment	When put in soil 10 days after treatment	When put in soil at time of treatment	When put in soil 10 days after treatment	When put in soil at time of treatment	When put in soil 10 days after treatment
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
0.2	97.4	0	94.3	0	47.5	13.5
0.4	93.4	0	97.2	0	74.4	39.0
0.6	100.0	0	100.0	0	85.9	7.7
0.8	100.0	4.3	100.0	0	100.0	22.3
1.0	100.0	0	100.0	0	98.7	29.0
1.2	100.0	0	100.0	0	100.0	22.3
1.5	100.0	18.0	100.0	0	87.2	30.6
1.7	100.0	0	100.0	0	100.0	27.7
1.9	100.0	0	100.0	0	100.0	17.7
2.1	100.0	0	100.0	0	100.0	5.0

EFFECT OF ORGANIC MATTER ON LARVICIDAL DOSAGE

It was found that when larvae were introduced into the soil immediately after the application of the chemical the insects were destroyed within 10 days. Naphthalene used at the rate of 0.6 pound per cubic yard was sufficient to kill the larvae in the clay loam and sandy loam; it had to be applied at the rate of 1.7 pounds per cubic yard in order certainly to destroy the larvae in the mixture of clay loam and peat. It is apparent that the organic matter in the soil modifies the effectiveness of naphthalene; the higher the concentration of organic matter in the soil, the less effective the compound is in destroying the larvae. The insecticidal value of the treatment was practically dissipated during the first 10 days. When larvae were introduced into the soils 10 days after the application of the chemical, very few of them were killed, even when the naphthalene was applied at the rate of 2.1 pounds per cubic yard. In view of the results obtained later in the chemical analyses of soils containing naphthalene, no satisfactory explanation can be given for the increased mortality of the larvae introduced into clay loam and peat 10 days after treatment. It will be noted, however, that all results obtained with clay loam and peat are somewhat variable.

DOSAGES REQUIRED TO KILL LARVAE INTRODUCED SUBSEQUENT TO TREATMENT

Another series of experiments was carried on to determine the quantities of naphthalene that would have to be applied to potting soil in order that there would be enough of the compound in the soil to destroy larvae introduced 7, 14, and 21 days after treatment, because it was anticipated that under some conditions the protection of the soil from reinfestation might not be possible. Naphthalene crystals were intimately mixed with moist sandy loam at rates ranging from 0.2 pound to 25 pounds per cubic yard. The soil was then placed in the greenhouse and infested with larvae, a total of 8,400 larvae being used in the experiment. At the end of 7 days the larvae were removed from the soil and the mortality from each dosage was determined. The soil was then reinfested and examined again after a lapse of 7 days. The process was repeated for periods of 14 and 21 days after the initial application of the chemical. The following concentrations of naphthalene per cubic yard of soil were necessary: 0.6 pound to kill larvae introduced at the time of treatment; 17.5 pounds to kill those introduced 7 days after treatment; and 20 pounds to kill those introduced 14 days after treatment. A dosage of 25 pounds per cubic yard destroyed only 32.5 percent of the larvae introduced 21 days after treatment. Apparently the insecticidal value of the treatment is rapidly lost in soil. Relatively large quantities of the chemical must be applied to insure the destruction of any larvae introduced subsequent to treatment. The use of these relatively large quantities appears impractical. Obviously, when only sufficient naphthalene is applied to destroy any larvae present at the time of treatment the soil must be protected from reinfestation.

RELATION OF SOIL LUMPS AND MOISTURE TO REQUIRED DOSAGE

The treatment of potting soil cannot always be carried out under relatively ideal conditions where the chemical is intimately mixed, where the soil is moist but not wet, and where there are no lumps large enough to harbor the insect. An experiment was conducted to determine the effectiveness of the treatment under less ideal conditions. Moist and wet sandy loam were obtained in the field and, without sifting, mixed with from 2 to 10 pounds of naphthalene per cubic yard. The moist sandy loam was of optimum moisture, containing 12 percent of water, and the wet sandy loam was practically saturated, containing 23 percent of water. It was not possible to determine that the natural lumps in the soil contained larvae without breaking the lumps and destroying their value for experiment; therefore, artificially infested lumps of moist and wet sandy loam were prepared in the following manner: A 3-inch pot was partially filled with soil, five larvae were placed on the soil, and sufficient soil was added to fill the pot. The pot was then inverted on washed muslin and carefully removed so that the form in which the soil was molded was not destroyed. The muslin was then wrapped tightly about the soil and tied, forming a lump of soil approximately 3 inches in diameter. Twenty-five hundred of these artificially infested masses of soil, containing 12,500 larvae, were prepared and buried in the different treated soils. During the following week, the temperature of the soils fluctuated between 45° and 60°, averaging 51° F. At the end of 7 days, the larvae were removed and the mortality was determined according to the previously mentioned formula. A summary of these results is given in table 6.

TABLE 6.—*Effectiveness of naphthalene crystals in destroying larvae in soil lumps embedded in moist and in wet*[1] *sandy loam treated with different quantities of the chemical*

Crystals applied per cubic yard (pounds)	Mortality of larvae after 7 days in—		Crystals applied per cubic yard (pounds)	Mortality of larvae after 7 days in—	
	Moist soil	Wet soil		Moist soil	Wet soil
	Percent	*Percent*		*Percent*	*Percent*
2	99.28	97.84	7	100.00	80.85
4	99.98	99.19	8	100.00	90.80
5	100.00	78.00	9	100.00	77.40
6	100.00	68.95	10	100.00	85.20

[1] The authors consider a soil as wet when it contains sufficient water to be puddled readily on manipulation; as moist, when about half saturated with water and in a friable condition.

It was found that in this treatment, when the crystals were not in contact with the larvae, naphthalene had to be applied at the rate of 5 pounds per cubic yard to destroy all of the larvae in moist soil. It should be noted, however, that a dosage of 2 pounds applied directly to moist soil killed 99.28 percent of them. In wet soil the treatment was not effective, even when the chemical was applied at the rate of 10 pounds per cubic yard.

From the data obtained, it was concluded that naphthalene crystals can be relied upon to destroy all larvae in soils to be used subsequently for potting plants, provided the following conditions are satisfied: (1) That the soil is moist, but not wet; (2) that it is free from large lumps; (3) that the temperature is maintained above 50° F.; (4) that the chemical is mixed with the soil at the rate of 5 pounds per cubic yard; (5) that the soil is left undisturbed for 1 week; and (6) that the soil is stored under conditions that will prevent subsequent reinfestation.

DECOMPOSITION OF NAPHTHALENE IN SOIL

That under favorable conditions naphthalene disappears rapidly when mixed with soil is well known. This fact has been recorded by Tattersfield (*46*) and Speyer and Owen (*45*). The disappearance of the compound from soil is usually too rapid to be attributed entirely to volatilization. In fact, Tattersfield (*46*), Thornton (*53*), and Gray and Thornton (*19*) have shown that the loss of naphthalene from soil is due largely to bacterial action. It was of considerable interest to know the rate of disappearance of the compound in soil, particularly when the soils were to be used subsequently for the growing of plants. A series of experiments were therefore carried out in the laboratory to determine by chemical analysis the rate of disappearance of naphthalene from the soil.

Before undertaking a study of the decomposition of naphthalene in soil, several experiments were conducted with known quantities of the compound mixed with soil to determine the best method for removing the chemical for analysis and also to learn how much naphthalene could be recovered. Vaporizing the naphthalene in the soil and carrying it to an absorbing material appeared to be the best procedure for removing the compound from the soil. Naphthalene was mixed intimately with moist sandy loam at the rate of 50 mg to 100 g of soil. The sample was placed in a 500-cc Erlenmeyer flask and heated on a water bath to 212° F. A stream of air, passed through the soil for 18 hours, carried the naphthalene vapor to the absorber, where it was absorbed in concentrated sulphuric acid at a temperature of 320°.

Several different types of absorbers were tried with varying degrees of success. The most satisfactory was a glass tower 5 feet long, the vapors being introduced at the bottom, where they met a stream of hot sulphuric acid that was trickling slowly downward over glass beads with which the tower was filled. A ground-glass connection fused to the bottom of the tower was fitted into a 60-cc round-bottomed Pyrex flask, which caught the acid coming from the tower. The inlet for the air carrying the naphthalene vapor was just above this fused connection. Resistance wire was wrapped closely around the outside of the glass tower to heat the acid, so that when it finally passed into the glass reservoir it was at a temperature of approximately 320° F. A 60-cc dropping funnel was placed above the absorbing tower and so adjusted that a drop of acid was discharged every 15 seconds. As the air carrying the naphthalene vapor passed up the absorbing tower, the naphthalene reacted with the sulphuric acid and was carried into the reservoir, where it was maintained at a temperature between 266° and 320°. After the soil had been aerated for 18 hours, the flow of air and acid was stopped. The acid in the absorbing tower was allowed to drain for 30 minutes into the reservoir. The reservoir was then removed and the absorbing tower washed with distilled water until the washings discharged from the bottom of the tower were neutral to litmus. These washings were then combined with the acid in the reservoir and made up to a definite volume. Aliquot samples were then taken and oxidized with the standard vanadic acid and the quantity of naphthalene was determined by the procedure previously described.

The results of these preliminary tests showed that practically all of the naphthalene could be recovered from soil by this procedure. When 50 mg of the compound were mixed with soil, the results of analysis showed from 48 to 52.2 mg, indicating at this concentration an error of ±2 percent. The average quantity recovered was 49.6 mg, or 99.2 percent. An attempt was made to reduce the period of aeration below 18 hours by raising the temperature of the soil from 212° to 266° F. It was found that at these temperatures a black, tarry substance was distilled from the soil and deposited in flakes on the inside of the absorbing tower. This material was insoluble in hot concentrated sulphuric acid, but any of the material carried down mechanically in the washing operation dissolved in the vanadic acid, thus introducing a possible error in the analysis. In order to avoid this difficulty the soil was not heated above 212°.

EFFECT OF ORGANIC MATTER

In order to study the rate of decomposition of naphthalene in the soil two soil mixtures were obtained from a commercial nursery. One was a clay loam mixed with well-rotted cow manure; the other, a mixture of this soil with an equal volume of peat moss. The clay loam had a moisture content of 19.8 percent, an apparent specific gravity of 1.132, and weighed about 1,900 pounds per cubic yard; the mixture of clay loam and peat had a moisture content of 40.3 percent, an apparent specific gravity of 0.878, and weighed about 1,480 pounds per cubic yard. These soils were passed through a 10-mesh sieve.

One portion of each soil remained untreated; the other portions were intimately mixed with naphthalene crystals at the rate of 5 pounds per cubic yard. The naphthalene was added at the rate of 2.6 g to each 1,000 g of the clay loam and at the rate of 3.37 g to each 1,000 g of the clay loam-peat mixture. The treated and untreated soils were placed in 1-gallon stone jars and covered with lids. The loss of moisture in each of the jars was determined daily and sufficient distilled water was added to replace the loss from evaporation. At intervals the soil was removed from one of the jars, thoroughly mixed, and three 100-g samples were taken for analysis. At the same time three samples were taken from one of the jars with soil containing no naphthalene, to serve as a blank in the titration. The samples were analyzed according to the previously described procedure and the results expressed in pounds of naphthalene per cubic yard. The data are summarized in table 7.

TABLE 7.—*Rate of loss of naphthalene from moist clay loam and from a mixture of clay loam and peat at room temperature*

Period after treatment (days)	Concentration of naphthalene per cubic yard in—					
	Clay loam			Clay loam and peat		
	Maximum	Minimum	Average	Maximum	Minimum	Average
	Pounds	*Pounds*	*Pounds*	*Pounds*	*Pounds*	*Pounds*
0	----------	----------	5.00	5.04	4.20	4.68
1	4.40	4.38	4.43	4.21	3.28	3.60
2	4.45	3.06	4.22	3.84	3.40	3.07
3	3.84	3.30	3.81	1.48	.95	1.19
4	3.65	2.50	3.50	.34	.19	.26
5	2.07	1.05	1.40	.17	.00	.07
6	.83	.78	.82	.03	.00	.01
7	.23	.00	.00	.02	.02	.02
8	.02	.00	.01	.00	.00	.00
9	.00	.00	.03	.00	.00	.00

The quantity of organic matter does influence appreciably the decomposition of naphthalene in the soil. In the clay-loam soil the concentration was less than 0.1 pound on the average on the seventh day; with the mixture of clay loam and peat this condition was obtained on the fifth day. Apparently the more organic material there is in a soil the more rapidly the naphthalene disappears.

EFFECT OF REPEATED APPLICATIONS

After soil has been treated with naphthalene at the rate of 5 pounds per cubic yard and stored in such a manner as to prevent reinfestation, there generally would be no necessity for re-treating it; but occasionally the fumigated soil becomes reinfested and then re-treatment is necessary. In such instances the nurseryman should know the rate of decomposition of naphthalene in the soil in order to determine when the soil can be used for potting plants. A mixture of clay loam and peat was treated with naphthalene at the rate of 5 pounds per cubic yard, placed in stone jars, and analyzed at daily intervals until the compound had disappeared. The soil was left undisturbed for 5 days, re-treated with naphthalene at the same rate, and analyzed again at daily intervals. Five days after the second application had disappeared, the procedure was repeated. The results of these tests are given in table 8.

TABLE 8.—*The effect of re-treatment on the persistence of naphthalene in soil*

Period after treatment (days)	Concentration of naphthalene per cubic yard after—								
	First treatment			Second treatment			Third treatment		
	Maximum	Minimum	Average	Maximum	Minimum	Average	Maximum	Minimum	Average
	Pounds	*Pounds*	*Pounds*	*Pounds*	*Pounds*	*Pounds*	*Pounds*	*Pounds*	*Pounds*
0	5.04	4.20	4.68			5.00			5.00
1	4.21	3.28	3.66	4.23	3.90	4.11	4.24	3.92	4.10
2	3.84	3.49	3.67	2.33	1.70	1.97	3.36	3.03	3.19
3	1.48	.05	1.19	.85	.40	.69	2.84	2.47	2.65
4	.34	.19	.26	.08	.00	.03	2.42	1.91	2.16
5	.17	.00	.07						
6	.03	.00	.01						
7	.02	.02	.02				1.21	.94	1.09
8							.92	.60	.79
9							.69	.53	.61
10							.80	.35	.59
11							.48	.19	.38
15							.05	.00	.03
16							.03	.00	.01

Practically all traces of the initial treatment disappeared in 6 days; the second application disappeared in 4 days; but the third application remained in the soil for 16 days. Filamentous fungi developed rapidly during the second treatment but were not noticeably present following the first or third applications. It is probable that the first application stimulated the development of this organism so that it was sufficiently numerous to decompose the second application more rapidly; but the extensive development of fungi following the second treatment probably exhausted the essential food elements in the soil and reduced the fungi population so that the naphthalene was decomposed more slowly. The rapid development of fungi in treated soil gives ground for the suspicion that fungi, like bacteria, have a role in the decomposition of naphthalene in the soil.

The decomposition of naphthalene in the soil is influenced by many factors, the most important of which are the organic matter, the moisture content, the temperature, and the degree of aeration. Under conditions in commercial nurseries the soil should be free from all traces of the compound within 2 weeks after the application of crystals.

EFFECT OF TREATMENT ON SUBSEQUENT GROWTH OF PLANTS

A preliminary experiment was carried on with radishes as indicators to determine the effect of treating soil with naphthalene crystals on the subsequent growth of plants. Sandy loam was treated with naphthalene at rates ranging from 5 to 15 pounds of the crystals per cubic yard of soil. Soil was also treated with mixtures of naphthalene and lime and with mixtures of naphthalene and sulphur to determine the possible effect on the growth of the plants of incorporating these chemicals with naphthalene. The mixtures of naphthalene with lime or sulphur were made up in the ratios of 3 to 1, 1 to 1, and 1 to 3, but because of the similarity of the results obtained when the same quantity of naphthalene was mixed with different quantities of the other chemicals, all of the results with the naphthalene-lime mixtures and with the naphthalene-sulphur mixtures are grouped together. Immediately after the soil had been treated it was placed in pots in the greenhouse and 100 radish seeds were sown in each pot. The same number of seeds was also sown in untreated soil. Two weeks after sowing, the seedlings in each pot were counted and the effect of the treatment was determined by comparing the number of plants growing in the treated soil with those in the untreated soil. The number in each treatment was expressed as the percentage of the number in the untreated soil. After the observation was completed the plants were removed from the pots and the soil was left fallow for 2 weeks. Each pot was again sown with 100 radish seeds and the process repeated. The results of this experiment are summarized in table 9.

TABLE 9.—*Effect of naphthalene and of mixtures of naphthalene with lime and sulphur on the subsequent growth of radishes*

Concentration of naphthalene per cubic yard (pounds)	Number of plants developing					
	When planted immediately after treatment with—			When planted 30 days after treatment with—		
	Naphthalene alone	Naphthalene and lime	Naphthalene and sulphur	Naphthalene alone	Naphthalene and lime	Naphthalene and sulphur
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
5	63.2	83.1	60.4	78.2	95.0	97.1
6	73.4	71.0	50.0	92.7	97.5	90.6
7	59.1	69.4	47.9	95.0	98.5	95.6
8	62.2	63.2	33.6	98.5	86.0	100.0
9	40.8	57.1	34.7	91.5	95.0	80.8
10	39.8	53.0	44.9	92.7	85.2	70.8
11	34.7	41.8	35.7	75.3	75.3	81.0
12	35.7			71.0		
13	31.6			68.1		
14	25.5			79.7		
15	23.4			79.7		

It was found that when radish seeds were planted in soil immediately after naphthalene crystals had been applied the germination and growth of the plants were seriously retarded. The number of plants was reduced progressively from 63.2 percent in the soil treated at the rate of 5 pounds per cubic yard to 23.4 percent in the soil treated with 15 pounds per cubic yard. The addition of lime or sulphur did not materially change the germination or growth of the plants in the naphthalene-treated soil. When the soil was seeded again 30 days after the naphthalene had been applied there was practically no difference in the development of the plants in the soil treated with different quantities of the chemical up to 10 pounds per cubic yard. Some residual retarding action was apparent because in some pots 20 to 25 percent fewer plants developed in the treated soil. In general, however, the detrimental effect of the naphthalene had largely disappeared from the soil within 30 days after treatment.

From the results obtained with radishes it was expected that treatment of potting soil with naphthalene at the rate of 5 pounds per cubic yard would have little effect on the growth of greenhouse plants and nursery stock, provided the soil was aerated after treatment. A mixture of clay loam and manure was treated with naphthalene crystals, left undisturbed for 7 days, and then spread out in a thin layer for 2 days. At the end of this time no odor of naphthalene could be detected. The soil was then placed in pots and seeded with pot marigold (*Calendula officinalis*) and common snapdragon (*Antirrhinum majus*). No retardation in the germination of the seeds in the treated soil was apparent; the plants grew normally; and those in the treated soil could not be distinguished from those in the untreated soil. The treatment did not affect the quality or the quantity of the blossoms. Commercial nurserymen applied naphthalene crystals to their potting soil at the recommended rate and used it successfully to grow all varieties of potted plants, among which Norfolk-Island-pine (*Araucaria excelsa*), a species of *Cyclamen*, *Dracaena fragrans*, a species of *Aspidistra*, India rubbertree (*Ficus elastica*), English ivy (*Hedera helix*), Forster palm (*Howea forsteriana*), screwpine (*Pandanus* sp.), *Poinsettia pulcherrima*, bowstring hemp (*Sansevieria zeylanica*), and roses (*Rosa* spp.). At the laboratory, *Azalea amoena* and *A. indica*, *Hydrangea opuloides*, a species of *Deutzia*, *Poinsettia pulcherrima*, and a rose (Dorothy Perkins) have been grown successfully in soil previously treated with naphthalene crystals. In addition it was found that a species of *Dahlia*, a species of *Iris*, and a species of *Phlox* grew normally in soil that had been fumigated with naphthalene.

These tests indicate that plants can be grown successfully in commercial nurseries in soil previously treated with naphthalene crystals. It is expected that if the soil is properly aerated after treatment the commercial nurseryman and greenhouseman will have no difficulty in growing the different varieties of plants.

TREATMENT OF POTTED PLANTS WITH NAPHTHALENE CRYSTALS

One of the problems in the control of the Japanese beetle is the development of a method for destroying the larvae in the soil of potted plants. It has been demonstrated many times that the soil of potted plants left in the field becomes infested with this insect during the summer months. It was thought that a practical method for destroying this infestation might be developed with naphthalene.

HYDRANGEAS IN 6-INCH POTS

A series of experiments were carried on with *Hydrangea opuloides*, growing in 6-inch pots. A group of 600 potted plants of this species were infested by placing 25 larvae on the surface and permitting them to burrow into the soil. One week was allowed for the larvae to become established throughout the soil in the pot. Napthhalene crystals were then applied, using dosages of 14.5, 42.5, and 123 g per pot. In half of the pots the chemical was applied at the surface and in the remainder it was placed under the soil mass. Two weeks after treatment all of the soil was removed from the pots, sifted, and examined to determine the number of living larvae. The mortality caused by each treatment, calculated according to the previously stated formula, is given in table 10. It was found that applying even 123 g of naphthalene at the surface or putting the crystals under the soil in the pots did not kill all of the larvae within this period. In the examination some of the larvae were found within one-half inch of the naphthalene crystals, apparently unaffected by the chemical. The application of the crystals at the surface or under the soil mass in the pots did not appear to have any immediate detrimental effect on the hydrangeas, but it did seriously interfere with the natural drainage of the pots, and probably over an extended period would have proved injurious to the growth of the plants.

TABLE 10.—*Effectiveness of naphthalene crystals in destroying larvae in 3-inch pots of hydrangeas when applied at the surface or under the soil in the pots*

Crystals applied per pot (grams)	Mortality of larvae—	
	When chemical was applied at surface of soil	When chemical was applied under soil mass
	Percent	*Percent*
14.5	93.29	77.20
42.5	75.50	94.15
123.0	65.60	95.15

HYDRANGEAS IN 4-INCH POTS

It was still considered possible that crystals of naphthalene might be applied effectively to hydrangeas growing in 4-inch pots. One thousand of these plants were infested, each with 5 larvae, in the manner described in infesting the larger pots. Naphthalene crystals were then applied, using 5, 10, 15, and 20 g of the chemical per pot. Some of the plants were treated by applying the chemical at the surface of the soil, some were treated by putting the chemical under the soil in the pots, and others were treated by applying the chemical both at the surface and under the soil. After treatment the soil in half the pots was kept relatively dry and in the others it was kept wet for a period of 2 weeks. At the end of this period, the mortality resulting from each treatment was determined in the usual manner. These data are given in table 11. It was found that the infestation was destroyed in the pots kept relatively dry by applying 15 g of the crystals at the surface or 10 g under the soil, or by putting 2.5 g on the surface and an equal quantity under the soil in the pots. When the soil was kept wet, the infestation was destroyed only when the chemical was applied both at the surface and under the soil. The application of naphthalene in this manner prevented the natural drainage of the pots. The soil, which was watered, remained puddled throughout the period of 2 weeks, a condition undesirable for the proper growth of these plants. In view of the results obtained, it did not seem possible to use naphthalene crystals to destroy an infestation of this insect in pots where plants were growing without causing serious damage to the plants.

On the possibility of destroying the infestation in the soil about the roots of hydrangeas in the process of potting the plants, naphthalene crystals were mixed with soil at the rates of 5, 7.5, 10, 12.5, and 15 pounds per cubic yard. Six hundred hydrangeas were dug in the field, most of the soil was shaken from the roots, and 500 were potted in the treated soil and 100 in untreated soil. The naphthalene was applied to the soil just before it was used in the field in order that there might be enough of the chemical to destroy any larvae present in the small masses of soil adhering to the roots. Two days after being potted the plants with naphthalene about their roots began to show signs of injury and continued to grow worse until at the end of 2 months all were dead or dying. The untreated plants during this period continued to grow vigorously. Two months after treatment a record was made of the condition of the plants, considering a plant "normal" when the top was growing vigorously and the roots practically filling the pot, "stunted" when it had made only a feeble growth, and "dead" when the roots had made no growth and the top was wilted. A summary of these results is given in table 12. The application of naphthalene crystals in this manner was found to be very detrimental to hydrangeas; none of the treated plants grew normally after the crystals had been placed about their roots. Apparently it is not safe to apply treated soil about the roots of hydrangeas until practically all traces of naphthalene have disappeared.

TABLE 11.—*Effectiveness of naphthalene crystals in destroying larvae in 4-inch pots of hydrangeas when applied at the surface and under the soil in the pots*

Crystals applied per pot (grams)	Mortality of larvae—					
	In soil kept dry			In soil kept wet		
	Chemical applied at surface of soil	Chemical applied under soil mass	Chemical applied both at surface and under soil mass	Chemical applied at surface of soil	Chemical applied under soil mass	Chemical applied both at surface and under soil mass
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
5	100.00	90.34	100.00	70.90	80.95	94.75
10	97.44	100.00	100.00	61.60	70.70	100.00
15	100.00	100.00	100.00	83.60	94.65	100.00
20	100.00	100.00	100.00	19.10	92.80	100.00

TABLE 12.—*Effect on subsequent growth of hydrangeas of mixing naphthalene crystals with soil in process of potting*

Crystals applied per cubic yard (pounds)	Condition of plants 2 months after treatment			Crystals applied per cubic yard (pounds)	Condition of plants 2 months after treatment		
	Dead	Stunted	Normal		Dead	Stunted	Normal
	Percent	*Percent*	*Percent*		*Percent*	*Percent*	*Percent*
5.0	43	57	0	12.5	92	8	0
7.5	67	33	0	15.0	93	7	0
10.0	92	8	0	0	0	0	100

The foregoing experiments show that naphthalene crystals cannot be applied to the soil of potted hydrangeas to destroy the larvae of the Japanese beetle without causing serious injury or death to the plants.

FUMIGATION OF THE SOIL OF POTTED AND BALLED PLANTS WITH NAPTHALENE VAPOR

Naphthalene vapor has been used successfully in greenhouses to control the common red spider (*Tetranychus telarius* L.), the sugar-beet thrips (*Heliothrips femoralis* Reut.), the onion thrips (*Thrips tabaci* Lind.), the greenhouse whitefly (*Trialeurodes vaporariorum* Westw.), and different species of aphids on cucumbers, tomatoes, strawberries, carnations, lilies, azaleas, deutzias, hydrangeas, sedums, arborvitae, and several other kinds of plants, without causing any serious damage to the plants (*9; 22; 23; 24; 33; 34; 36, p. 69–75; 43; 44, p. 49–50*). In view of these results, it was believed that the immature stages of the Japanese beetle might be destroyed in the soil about the roots of potted plants and balled nursery stock by placing the infested stock in a chamber filled with naphthalene vapor.

VAPORIZATION OF CRYSTALS AND DISPERSION OF VAPORS

Before undertaking the insecticidal investigation, a study was made of the vaporization and the dispersion of the vapor in a 1,000-cubic-foot fumigation chamber. The naphthalene was vaporized by two different methods: (1) By placing the crystals in a metal pan directly upon a hot plate, and (2) by placing the crystals in a metal pan which was floating in hot water. In both methods the relative humidity of the air was maintained at practically 100 percent at a temperature of 80° F. by boiling water continuously in the chamber. When the naphthalene crystals were placed directly on the hot plate the compound vaporized so rapidly that the metal pan was empty in 30 minutes. Under these conditions the compound sublimed very quickly, crystals settled on the walls and ceiling of the chamber, and the air was filled with floating crystals. Fifteen hours after the naphthalene had been vaporized and when no crystals were floating in the air, samples of the air were withdrawn from points 2.5, 5, and 7.5 feet above the floor to determine the concentration of vapor in the atmosphere. Ten liters of air were withdrawn from each point at the rate of 1 l in 4 minutes. The air was then analyzed to determine the concentration of naphthalene. It was found that 15 hours after the naphthalene was vaporized on the hot plate, the average concentration of the vapor in the chamber was 0.44 mg per liter. The quantity of naphthalene theoretically required to saturate a liter of air at a temperature of 80° was calculated by the formula:

$$W = \frac{M}{RT} \times \frac{p}{760} \times 1,000$$

where W = weight of naphthalene in milligrams per liter, M = 128.06 the molecular weight (128.06) of the compound, R = the gas constant (0.08204 liter atmosphere per molecule per degree), T = temperature in absolute degrees, and p = 0.113, the vapor pressure of naphthalene in millimeters at 80° F. It was calculated that at a temperature of 80° 0.77 mg of naphthalene was required to saturate 1 l of air. When the average concentration was 0.44 mg per liter the air was only 57 percent saturated with the vapor.

The rapid sublimation of the compound, when the naphthalene was vaporized directly on a hot plate, was considered undesirable. The experiment was repeated, vaporizing the naphthalene in a pan which was floating in water maintained at a temperature of 130° to 140° F. At the end of 15 hours a large part of the crystals still remained in the vaporizing pan. The continuous boiling of water in the chamber to maintain a relative humidity of 100 percent resulted in the accumulation of moisture on the walls and ceiling of the chamber. This moisture appeared to have a cooling effect on the naphthalene vapor, causing crystals of the compound to form on the walls and ceiling. An analysis of the air in the chamber 15 hours after beginning the vaporization showed that the average concentration was 0.46 mg per liter. With an average concentration of 0.46 mg per liter the air was 59 percent saturated with naphthalene. Vaporizing the compound over hot water reduced the rapidity of sublimation but did not prevent the formation of crystals on the sides of the chamber.

Another experiment was carried on, vaporizing the naphthalene in a chamber with the relative humidity of 90 to 95 percent, to determine whether the absence of moisture on the sides and ceiling of the chamber would reduce the sublimation. At the end of 15 hours, when samples of air were taken for analysis, no crystals of naphthalene were on the walls or ceiling of the chamber. The average concentration was 0.81 mg per liter at a distance of 2.5 feet above the floor, 0.83 mg at a distance of 5 feet, and 0.79 mg at a distance of 7.5 feet above the floor. It was apparent that by maintaining the relative humidity just below the saturation point and vaporizing the compound over water at a temperature of 130° to 140° F. practically a saturated atmosphere of naphthalene vapor could be obtained.

MORTALITY OF LARVAE IN SOIL BALLS AT DIFFERENT TEMPERATURES

An experiment was conducted to determine the period of time required for an atmosphere, with a relative humidity of 90 to 95 percent and saturated with naphthalene vapor, to kill larvae in masses of moist sandy loam, approximately 3 inches in diameter, at temperatures ranging from 75° to 90° F. A total of 1,020 masses of soil were prepared and each was infested with five larvae in the manner previously described. The infested soil was then placed in the 1,000-cubic-foot fumigation chamber and exposed to the naphthalene vapor for periods ranging from 18 to 96 hours. At the end of the predetermined period larvae were removed from the soil balls and placed on trays for observation to determine the effect of the treatment. The mortality resulting from each treatment was calculated according to the previously described formula. These data are summarized in table 13.

TABLE 13.—*Effectiveness of an atmosphere with 90 to 95 percent relative humidity, and saturated with naphthalene vapor, in killing larvae in 3-inch balls of soil*

Period of exposure (hours)	Mortality of larvae at—			Period of exposure (hours)	Mortality of larvae at—		
	75° F.	80° F.	90° F.		75° F.	80° F.	90° F.
	Percent	*Percent*	*Percent*		*Percent*	*Percent*	*Percent*
18			36.4	72	97.5	100.0	100.0
24	18.0	40.0	90.0	96	100.0	100.0	100.0
48	80.2	98.7	100.0				

Apparently this treatment might be effective in destroying the larvae in small masses of soil clinging to the roots of deciduous plants and in the soil balls about the roots of small evergreens, provided the vapor was not injurious to the plants.

MORTALITY OF LARVAE IN POTS

The treatment was then applied at a temperature of 80° F., for periods ranging from 48 to 144 hours, to 3-, 4-, and 6-inch pots of moist soil infested with larvae. At the end of the predetermined exposure larvae were removed to determine the effect of the treatment. These data are given in table 14. It was found that an exposure of even 144 hours was not sufficient to destroy all of the larvae. Apparently very little of the vapor penetrated the walls of the clay pots. The rate of penetration of the vapor from the surface of the soil appeared to be too slow to make the procedure of practical commercial value.

TABLE 14.—*Effectiveness of an atmosphere with 90 to 95 percent relative humidity, and saturated with naphthalene vapor, in killing larvae in pots of soil at a temperature of 80° F.*

Period of exposure (hours)	Mortality of larvae in—			Period of exposure (hours)	Mortality of larvae in—		
	3-inch pots	4-inch pots	6-inch pots		3-inch pots	4-inch pots	6-inch pots
	Percent	*Percent*	*Percent*		*Percent*	*Percent*	*Percent*
48	88.0	92.0	66.6	120	96.7	98.0	81.4
72	91.4	92.0	82.0	144	98.7	98.7	81.4
96	94.0	94.0	92.6				

EFFECT OF TREATMENT ON PLANTS

Although the insecticidal results were not very encouraging, some tests were made with azaleas, hydrangeas, and barberry to determine the resistance of these plants to the action of the vapor. It was planned to expose these plants to a saturated atmosphere of naphthalene for 1 week at a temperature of 80° F. Plants were removed from the fumigation chamber at intervals of 24 hours and placed in the greenhouse. At the end of 24 hours the leaves on the plants began to show injury from the vapor and all plants exposed to the vapor for 48 hours, or longer, were damaged so seriously that they were of no commercial value.

These experiments demonstrated that the vapor of naphthalene in insecticidal concentration was relatively slow in penetrating small balls or pots of soil. A comparatively long exposure was required to assure the destruction of the larvae. The vapor proved so detrimental to the plants, even when they were exposed for shorter periods than were required to kill the insect, as to preclude its use in commercial nurseries.

NAPHTHALENE AS A REPELLENT TO PREVENT FEEDING BY THE ADULT JAPANESE BEETLE

An experiment was conducted in 1925 to determine the value of naphthalene for protecting ripening peaches from injury by the adult Japanese beetle. Thirty-six balls, each 2.5 inches in diameter, were prepared by pouring liquefied naphthalene into spherical molds and permitting it to crystallize. These balls were hung on the branches of small peach trees on which fruit was ripening. A few beetles were in the tree when the chemical was placed there at 10 a.m. and, although the odor was very pronounced, the beetle population had not diminished at the end of 7 hours. The tree was kept under close observation for 6 days, the temperature ranging from 69° to 95° F. At the end of this period the population of beetles in the tree had greatly increased and all of the fruit was damaged. It was apparent that naphthalene used in this manner is of no practical value.

Metzger and Grant (*30*) made a smudge of naphthalene by mixing the crystals with a slowly burning base of wood flour and potassium nitrate. This smudge was found to be only slightly repellent to the Japanese beetle in orchards of ripening peaches; moreover, the wind in the orchard dispersed the smudge so rapidly as to give no satisfactory control.

The blossoms of hybrid tea roses are among the favored foods of the adult beetle and there is at present no satisfactory method for protecting these plants from injury. An attempt was made to protect the blossoms by periodic applications of naphthalene to the ground under the plants. A bed 5 feet wide and 10 feet long, containing 18 roses of the varieties commonly grown throughout the infested area—Mme. Edouard Herriot, Golden Ophelia, Souv. de Georges Pernet, Norman Lambert, John Russell, Red Radiance, Mrs. William C. Egan, Imperial Potentate, Mme. Jules Bouché, Hadley, Angele Pernet, Los Angeles, Sir David Davis, Caroline Testout, Mme. Léon Paine, Mme. Butterfly, Souv. de Claudius Pernet, and General MacArthur—was selected for experimentation. When, on June 15, 25 rose chafers (*Macrodactylus subspinosus* Fab.) were found on the plants, 1 pound of naphthalene crystals was spread on the surface of the ground under the plants. One hour later the odor was very pronounced. All of these beetles left, but on the following day more were present. The treatment was repeated on June 21, June 28, and July 3, therefore definite odor of the compound was always about the roses during this period. The first Japanese beetle was found on the roses on June 28. The number of beetles increased on the plants, notwithstanding the odor, 25 being removed on July 5, 65 on July 6, and 50 on July 7. The application of naphthalene crystals to the surface of the ground under the plants apparently afforded no appreciable protection.

NAPHTHALENE AS A REPELLENT TO PREVENT OVIPOSITION BY THE JAPANESE BEETLE AND AS A CONTROL MEASURE IN THE FIELD

Many references to naphthalene as a repellent and control for soil-infesting insects are found in the literature, but the reports are often so conflicting that it is difficult to determine definitely the value of the chemical for this purpose. Favorable results with naphthalene against wireworms, larvae of Elateridae, have been reported by Theobald (*50*) and Zappe (*54, pp. 163-165*). Gray and Wheldon (*20*) reported that it was of little value in reducing the number of wireworms. Bencomo (*3*) discounted its value for this purpose, and Theobald (*51, p. 213*) in a later paper stated that it had only temporary value as a repellent against these insects. Tattersfield (*46*) and Miles (*31, pp. 17-21*) concluded that naphthalene was of only limited value because the action of the chemical was so dependent upon thorough distribution in the soil, and the effect was temporary because of its relatively rapid decomposition in the soil.

Blakeslee (*4*) and Peterson (*35*) placed naphthalene crystals about the base of peach trees in the usual manner for killing larvae and repelling adults of the peach borer (*Aegeria exitiosa* Say). They found that it had little larvicidal action and did not prevent the adults from ovipositing. Chamberlin (*6*) fround crude naphthalene of little value against the black gooseberry borer (*Xylocrius agassizii* Lec.), when applied about the base of the plants.

Feytaud (*12*) and Hutson (*25, p. 122*) found that spreading naphthalene on the surface of the ground was effective in repelling mole crickets. Some protection of newly planted tobacco against attack by tenebrionid beetles has been obtained by spreading naphthalene about the plants (*10, p. 146*). This treatment has also been found effective in protecting grafted slips of grapevines from attack by these insects (*1*).

Smith and Wadsworth (*42*), Gray (*18*), and Thompson (*52*) found that applying naphthalene between rows of onions killed a high percentage of the onion fly (*Hylemyia antiqua* Meig.). Smith and Wadsworth (*42*), Glasgow (*16*), and Thompson (*52*) reported that the application of naphthalene between the rows of carrots greatly lessened the injury by the carrot rust fly (*Psila rosae* Fab.), although the degree of control obtained by this method was not sufficiently uniform to warrant its use on a commercial scale. Naphthalene has been reported by Thompson (*52*) to be effective against the cabbage maggot ((*Phorbia*) *Hylemyia brassicae* Bouché). The celery fly (*Acidia heraclei* L.) has been repelled by the application of the compound to the soil (*2*). Theobald (*48, Rpt. 1925–26, p. 7*) reported that vegetables were protected from damage by ortalid larvae, (*Myodina*) *Seoptera vibrans* L., by hoeing naphthalene into the soil about the plants. Collinge (*7, pp. 17–18*) found that the larvae of the March flies *Bibio marci* L. and *B. johannis* L., which are introduced into soil with manure or leaf mold and attack vegetables and young coniferous plants, can be controlled by treating the ground with naphthalene.

Glendenning (*17*) found that the migrants of the elm-currant aphid (*Eriosoma ulmi* L.) were repelled from black currant bushes by hoeing naphthalene about the plants. Collinge (*7, p. 7*) obtained some control of the woolly apple aphid ((*Schizoneura*) *Eriosoma lanigerum* Hausm.) by applying naphthalene to the soil about infested trees.

Naphthalene has been used with varying results in the control of white grubs in the soil. McKinney and Milam (*28*) found that applications of naphthalene to tobacco beds gave little control of the larvae of the green June beetle (*Cotinis nitida* L.). Escherich (*11*), Novak (*32*) and Romanovsky-Romanko (*40*) reported that the application of naphthalene to the surface of soil prevented the adult cockchafer (*Melolontha melolontha* L.) from ovipositing. Marie (*29*) stated that applying the crystals at the rate of 1 ounce to 10.75 square feet repelled the females of this insect but did not kill the larvae; Guyot (*21*) reported that this treatment was effective against cockchafers, both as a larvicide and as a repellent. Theobald (*48, Rept. 1922–23, p. 12*) stated that leatherjackets, cutworms, and chafer larvae were killed by naphthalene at the rate of 3 hundredweight to the acre, but concluded (*49, p. 43*) that the use of naphthalene could not be depended on to control the cockchafer.

The value of naphthalene as a repellent for soil-infesting insects was reported to vary considerably. The fact that under some conditions the chemical apparently repelled or controlled some insects in the soil led to the conclusion that it might be of value against Japanese-beetle larvae.

NUMBER OF EGGS DEPOSITED IN NAPHTHALENE-TREATED SOIL

A preliminary experiment was set up in the insectary to determine whether the presence of naphthalene in the soil would reduce oviposition by the beetle. Naphthalene crystals were thoroughly mixed with the soil at rates ranging from 0.54 pound to 10.80 pounds per cubic yard. The treated soil was placed in 12-inch pots and covered with cages; then 1,000 beetles were introduced into each cage and left with an abundant supply of foliage as food. At the end of 1 week, the soil was removed from each pot and the number of eggs deposited in each pot of treated soil was determined. The results of this experiment are presented in table 15. It was found that the reduction in the number of eggs in the treated soils, as compared with the number in the untreated soil, ranged from 30.8 percent, with a dosage of 0.54 pound, to more than 90 percent with the higher dosages. It was impossible from this experiment, however, to determine whether this apparent reduction in the number of eggs was due to the repellent action of the compound or to the insecticidal action on the insects after they had entered the soil.

Table 15.—*The effect on the number of eggs deposited by the female Japanese beetle of mixing naphthalene crystals with soil*

Crystals applied per cubic yard (pounds)	Eggs found in treated soil	Eggs found in untreated soil	Reduction in number of eggs in treated soil as compared with number in untreated soil	Crystals applied per cubic yard (pounds)	Eggs found in treated soil	Eggs found in untreated soil	Reduction in number of eggs in treated soil as compared with number in untreated soil
	Number	*Number*	*Percent*		*Number*	*Number*	*Percent*
0.54	1,777	2,567	30.77	5.40	6	112	94.64
1.08	1,437	2,567	44.02	6.48	5	112	95.53
2.16	389	2,567	84.85	7.56	39	112	65.18
3.24	356	2,567	86.13	8.64	10	112	91.07
4.32	268	2,567	89.55	9.72	9	112	91.97
4.32	0	112	91.07	10.80	22	112	80.30

RESULTS FROM TWO METHODS OF APPLICATION

Another preliminary test was made with naphthalene crystals applied to the surface of fallow ground and to turf. Trays 18 inches square and 3 inches deep were filled with fallow soil and with turf. Some were treated by applying naphthalene to the surface at the rate of 1,000 pounds per acre; others remained untreated. A treated and an untreated tray of each were placed in diagonally opposite corners of a large wire cage, 10 feet square and 8 feet high. Approximately 10,000 adult beetles were liberated in the cage and food was added as required. At the end of 1 week the treated and untreated turf and the fallow soil were examined to determine the number of eggs in each. It was found that the application of naphthalene crystals to the surface of the fallow soil or to turf had not caused a decrease in the number of eggs. When applied to the surface in this manner the crystals disappeared, owing to vaporization, before the experiment was completed.

This experiment was repeated, naphthalene being applied at the rates of 1,000 and 2,000 pounds per acre and mixed with the soil. An examination of this soil at the end of 1 week showed that there were no eggs in the treated soils, although a large number of dead beetles were found. A total of 233 eggs were found in the untreated soil in the cage. The naphthalene apparently did not prevent the females from entering the soil to oviposit but killed them before they laid their eggs.

An experiment was next conducted in a flower garden, heavily infested with adult beetles, to determine whether repeated applications of naphthalene to the soil about the plants would prevent the establishment of a larval population in the soil. The flower garden, 50 feet wide and 90 feet long, was planted in rows 2 feet apart with the following varieties: Pot marigold (*Calendula officinalis*), calliopsis (*Coreopsis tinctoria*), Chinese pinks (*Dianthus chinensis*), blue laceflower (*Trachymene caerulea*), African marigold (*Tagetes erecta*), common snapdragon (*Antirrhinum majus*), sweet scabiosa (*Scabiosa atropurpurea*), strawflower (*Helichrysum bracteatum*), common zinnia (*Zinnia elegans*), and the common nasturtium (*Tropaeolum majus*). In addition to these annuals, there were two rows of sweet-william (*Dianthus barbatus*), a row each of the phloxes Mrs. Jenkins and Champs Elysee, some iris, and several clumps of dahlia. When the Japanese beetle appeared in the garden on July 2, naphthalene was applied between the rows of plants at the rate of 1 pound to 100 square feet, and cultivated into the soil to a depth of approximately 2 inches. While the adult beetle was present in the garden the operation was repeated at weekly intervals up to August 20, a total of 8 pounds of naphthalene being applied to each 100 square feet. The odor of naphthalene was very pronounced in this garden during the summer but it did not repel the beetles from the flowers. Beetles could be found on all of the varieties, the feeding being largely confined, however, to the dahlias, marigolds, and zinnias. The application of naphthalene between rows of plants as in this experiment apparently did not affect the growth of these plants.

In November, a larval survey was made by digging two strips, 3 feet wide, 10 inches deep, and 90 feet long, through the garden and across the rows of plants. In this survey 161 *Popillia* larvae and 9 larvae of *Macrodactylus* were found in the rows of plants, and 30 *Popillia* and 1 *Macrodactylus* larvae between the rows of plants where the crystals were applied. In other words, there were 300 larvae of the Japanese beetle in each 1,000 square feet of this garden. It is not uncommon in the vicinity of this garden to have 750 to 1,500 larvae of the Japanese beetle in 1,000 square feet of soil. It should be noted that when this larval survey was made, 3 months after the last application of the crystals, the odor of the chemical was still pronounced and crystals were found in the soil. No doubt the application of naphthalene crystals did reduce the number of larvae in the garden; the results obtained, however, did not warrant recommending the treatment as a control measure.

SUMMARY AND CONCLUSIONS

Naphthalene has been tested extensively by entomologists as a control for different insect pests inhabiting soil, and in the past 10 years has been investigated as an insecticide and as a repellent against the Japanese beetle.

The methods of determining naphthalene quantitatively were investigated. The determination of naphthalene as naphthalene picrate is relatively slow and requires careful standardization of each step in the procedure to obtain consistent results. The determination of the compound by oxidation to phthalic acid is more rapid and gives results in close agreement.

The physiological action of the vapor on the immature stages of the Japanese beetle is not clearly understood. Eggs and larvae exposed to the vapor become reddish in color, the intensity depending on the period of exposure. The vapor paralyzes the immature stages and finally causes their death. The resistance of the different stages increases in this order: (1) Larva, (2) egg, and (3) pupa. The period of exposure necessary for a saturated atmosphere of naphthalene to kill depends upon the temperature, ranging from 12 hours at 80° F. to 120 hours at 50°. The relative humidity of the atmosphere influences to some extent the insecticidal action on larvae, the mortality increasing with the increase in relative humidity.

The larvae can be destroyed in potting soil by applying naphthalene crystals at the rate of 5 pounds per cubic yard, provided the soil is moist (but not wet) and free from large lumps; that the temperature is above 50° F.; and that the soil is left undisturbed for 1 week. Insecticidal tests and chemical analyses show that naphthalene is decomposed rapidly in the soil and under normal conditions would disappear within 14 days. Organic matter in the soil in the form of peat hastens the decomposition. Different potted plants, including *Araucaria excelsa*, *Cyclamen* sp., *Aspidistra* sp., *Poinsettia* sp., *Rosa* sp., *Hydrangea* sp., and *Deutzia* sp., have been grown successfully in soil previously treated with naphthalene crystals. It was found impossible to apply naphthalene crystals to the soil of potted hydrangeas in sufficient quantity to destroy the larvae without causing serious injury to the plants.

When infested pots of soil and infested soil balls were placed in a saturated atmosphere of naphthalene, the vapor penetrated only slowly into the soil. The best results were obtained by vaporizing the crystals over hot water in an atmosphere having a relative humidity of 90 to 95 percent. The plants, however, could not withstand the long exposure necessary to destroy the insect.

Naphthalene vapor did not protect either roses or ripening fruit from attack by the adult beetle.

Mixing naphthalene crystals with soil at the rate of 1,000 pounds per acre was found, under insectary conditions, to prevent oviposition but did not keep the females from burrowing into the soil. The periodic application and mixing of naphthalene into soil about the roots of annual flowers during the flight of the adult beetle reduced the larval population in the soil; however, the results were not sufficient to warrant recommending the procedure as a control measure.

From this investigation it was concluded that naphthalene when applied to soil is an effective insecticide for destroying the immature stages of the Japanese beetle. It cannot be used in insecticidal concentrations to destroy the insect in the soil about the roots of growing plants. Thus, its use is limited to conditions where the soil can be treated before the plants are set. The compound has little value as a repellent against the adult beetle.

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U.S. GOVERNMENT PRINTING OFFICE: 1934

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