

# THE COMPARATIVE INSECTICIDAL EFFICIENCY AGAINST THE CAMPHOR SCALE OF SPRAY OILS WITH DIFFERENT UNSULPHONATABLE RESIDUES<sup>1</sup>

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## INTRODUCTION

The percentage of the more chemically active constituents in a given mineral oil, as indexed by the unsulphonatable-residue test recommended by Graham (5, 6)<sup>3</sup> may indicate the approximate content of olefines or other unsaturated hydrocarbons, including aromatic, naphthenic, and resinous substances, as well as branched-chain paraffin compounds of high molecular weight. The large number of makes and grades of spray oils of the lubricating or engine oil type employed at present, together with the uncertainty about the chemical composition of each oil, prevents the making of a trustworthy comparison of the relative toxic effects of the above-mentioned active constituents on scale insects. However, although the different active constituents cannot be separated by the sulphonation test, it is possible to determine (by experiments) whether the more active compounds, as so measured collectively, have any effect upon the insecticidal action of the oils. Swingle and Snapp (8) have obtained data regarding the effect of several oils having different percentages of sulphonatable constituents, on the control of the San Jose scale (*Aspidiotus perniciosus* Comst.) on peach trees at Fort Valley, Ga., and report that wide variations in the content of sulphonatable materials produced little difference in toxicity to that insect. Their tests were conducted with oils containing sulphonatable portions ranging between 100 and 31.2 percent by volume. On the other hand, English (4) states that under some conditions the unsaturated oils may be more toxic.

Since any investigation of the toxicity of the sulphonatable constituents must be made with oils of both unknown and differing composition, it should be borne in mind that the results obtained in using a small number of oils are not necessarily applicable to all such oils. With recognition of this limitation, the laboratory and field experiments described below were planned. The objective was to determine whether or not three mineral oils containing markedly different percentages of sulphonatable material would produce measurably different mortalities of the camphor scale, *Pseudonidia duplex* (Ckll.), at New Orleans, La. Spuler, Overley, and Green (7) report that the insecticidal value increases with the viscosity of oils; and, if such is the case, the effects of the sulphonatable portions, provided they influence the insect, should be separated from effects attributable

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<sup>2</sup> Authors' names are given in alphabetical order; the sequence indicates no seniority.

<sup>3</sup> Reference is made by number (italic) to Literature Cited, p. 878.

to viscosity. Hence oils were selected which had substantially the same viscosity and volatility in order that the effect of variations in these two properties would not obscure differences in results due to variations in the sulphonatable content.

## EXPERIMENTAL METHODS

### SPECIFICATIONS OF OILS

All sprays were prepared from two oils purchased from a well-known manufacturer and designated for experimental purposes as WR and L-1778. The Saybolt viscosities, the percentages of non-volatile and of sulphonatable portions, and the specific gravities were separately determined and are presented in table 1.

TABLE 1.—*Characteristics of oils used in experiments with sprays against the camphor scale, New Orleans, La., 1932*

Oil designation	Saybolt viscosity at 100° F.	Nonvolatile material (24 hours at 100° F.) <sup>1</sup>	Sulphonatable material (by volume)	Specific gravity at 68.0° F.
	Seconds	Percent	Percent	
WR.....	94.9	98.6	6	0.8512
WR/L-1778.....	97.8	98.6	16	.8679
L-1778.....	101.9	98.4	33	.8846

<sup>1</sup> Evaporation tests were made according to methods proposed by Dawsey (2).

Distillation ranges were not determined, but according to the manufacturer's specifications the WR oil began distillation at 654° F. with 89 percent off at 750°, and the L-1778 began distillation at 630° with 87 percent off at 750°. The L-1778 was stated to be a blend of two or more straight-run oils. The oil designated as WR/L-1778 consisted of equal parts of the first and third oils, compounded by mixing them before preparing emulsions therefrom.

### PREPARATION OF EMULSIONS

The emulsions consisted of the mineral oils dispersed in an aqueous solution of sodium oleate. Concentrated emulsions containing 66% percent of oil by volume were made according to the process suggested by Dawsey,<sup>4</sup> wherein emulsification takes place spontaneously and the emulsifying soap is formed simultaneously. These emulsions were then usually diluted to an intermediate concentration of 40-percent oil before the highly diluted sprays containing from 0.9 to 2 percent of oil were prepared. The stock emulsions used throughout the work were numbered consecutively from 1 to 9, inclusive, and the sprays made from them were designated in a like manner.

Table 2 shows the composition of the sprays applied to potted camphor-tree (*Cinnamomum camphora*) plants in the laboratory. The stock mixtures were prepared 1 day prior to application of the sprays, the sprays being made up at the time of application by dilution with distilled water.

<sup>4</sup> DAWSEY, L. H. SPONTANEOUS EMULSIFICATION IN THE PREPARATION OF OIL SPRAYS. Unpublished manuscript.

TABLE 2.—Composition of oil sprays used against the camphor scale on potted camphor-tree plants in the laboratory June 14 and Aug. 24, 1932

Stock emulsion no. <sup>1</sup>	Oil used	Date of spraying	Composition of sprays			
			Spray no. <sup>2</sup>	Volume of stock emulsion taken	Oil content of spray	Normality of sodium oleate in aqueous phase×10 <sup>4</sup>
			Cc		Percent	
1.....	WR.....	June 14	f1a.....	50	1.0	6.62
			f1b.....	85	1.7	11.34
2.....	WR/L-1778.....	do.....	f2a.....	50	1.0	6.62
			f2b.....	85	1.7	11.34
3.....	L-1778.....	do.....	f3a.....	50	1.0	6.62
			f3b.....	85	1.7	11.34
4.....	WR.....	Aug. 24	f4a.....	45	.9	5.95
			f4b.....	75	1.5	9.98
5.....	WR/L-1778.....	do.....	f5a.....	45	.9	5.95
			f5b.....	75	1.5	9.98
6.....	L-1778.....	do.....	f6a.....	45	.9	5.95
			f6b.....	75	1.5	9.98

<sup>1</sup> Each stock emulsion was made of 200 cc of oil, 4.3 cc of 3.05/N oleic acid, and 300 cc of distilled water, and had an oil content of 40 percent by volume.

<sup>2</sup> The total volume of the spray after dilution was 2,000 cc.

For the field sprays, both the oil concentration and the soap concentration were higher than in the laboratory sprays. The oil concentration was 2 percent and the emulsifier concentration (normality of sodium oleate in the aqueous phase) was  $24.7 \times 10^{-4}$ . The effect of increasing the oil content is to deposit a heavier film of oil on the tree. The effect of increasing the soap concentration is to decrease the oil deposit, but in this particular case, where 100 gallons of spray was made at a time, it was necessary to use New Orleans tap water, and an excess of soap was necessary to overcome its hardness and at the same time keep the oil globules in suspension.

Field-spray experiments (1) conducted in 1931 had shown that the kills most suitable for accurate mortality determinations (50 to 75 percent) might be obtained by using a spray containing 2 percent of oil (emulsified with potassium fish-oil soap) and having a soap normality of  $26.3 \times 10^{-4}$ . Effort was therefore made to approximate that composition in the field sprays. For each stock emulsion the oil was emulsified by the spontaneous method into a mixture containing 40 percent of oil. Two gallons of the oil was poured into a 15-gallon oak keg, to which a stirrer driven by a  $\frac{1}{4}$ -horsepower, 1,750-revolutions per minute motor was clamped; 0.0793 gallon (300 cc) of 3.05 normal oleic acid was stirred into the oil; 0.0817 pound of C. P. sodium hydroxide was dissolved in 1 gallon of distilled water, and this alkali solution was slowly added to the oleic acid-mineral oil mixture with fast mixing. After emulsification, 2 gallons more of distilled water was added and the whole 5 gallons of emulsion stored overnight in a large milk can for use in field spraying the following day.

These stock emulsions, nos. 7, 8, and 9, each containing 40 percent of oil (WR, WR/L-1778, and L-1778, respectively), were diluted with tap water to a final volume of 200 gallons to form sprays 7F, 8F, and 9F, each containing 2 percent of oil. The emulsifier concentration

(normality of sodium oleate in the aqueous phase) of  $24.7 \times 10^{-4}$  includes oleates of calcium, magnesium, etc., formed with undetermined quantities of mineral salts in the dilution water.

#### APPLICATION OF LABORATORY SPRAYS

The potted camphor-tree plants to which the laboratory sprays were applied had previously been artificially infested by the "box" method (1); so the populations were of uniform age distribution, and differences in the mortality of scales attributable to variation in age susceptibility were eliminated. The insects were in the adult stage at the time of spraying.

The laboratory spraying outfit consisted essentially of apparatus already described, and standardized conditions of spray application, like those described in the earlier paper (1), were maintained. The applications were made with a compressed-air sprayer consisting of a 2-liter metal tank, to which was attached a short piece of rubber hose fitted with a vermorel nozzle. The air-inlet pipe reached nearly to the bottom of the tank, so the incoming air provided sufficient agitation to insure an even distribution of oil within the tank. The infested plants were placed upon a revolving platform, at a distance of 1 meter from the nozzle, and each plant was sprayed for 24 seconds at a constant pressure of 40 pounds per square inch. The six sprays of the first set, numbered 1a to 3b (table 2), and containing 1 and 1.7 percent of oil, respectively, were applied on June 14. The other six sprays, designated 4a to 6b, were applied on August 24, but the oil concentrations were reduced to 0.9 and 1.5 percent, respectively, for the two series of the second set, because the scales of this brood were not so old as those of the earlier brood. Each set was treated as an independent experiment, since the applications were made at different times of the year. The number of plants composing the June 14 set was 46, of which 8 were sprayed with distilled water as checks; the number of plants composing the August 24 set was 42, of which 7 were sprayed with water as checks. The plants were from 15 to 20 inches in height and about 2 years old when sprayed.

#### APPLICATION OF FIELD SPRAYS

In the field sprays an entirely different technique was employed, the applications being made with a power sprayer of a type used in commercial work. The camphor-trees to be sprayed were located in Metairie Cemetery on both sides of a driveway running southwest and northeast. They were heavily infested with adult scales and ranged from 10 to about 25 feet in height. All were in the open and exposed to the sun at all times of the day. The trees along the drive were numbered from 1 to 10, going in the northeast direction, the numbers alternating from one side of the road to the other. Nos. 1, 6, and 9 were designated as check trees and were left unsprayed. The remaining seven trees were sprayed between 9 a. m. and noon on November 22. Spray 7F was applied on trees 2, 3, and 5; spray 8F on trees 4 and 7; and spray 9F on trees 8 and 10. Applications were

made with a power spray machine at 350 pounds' pressure. The time during which each tree was sprayed depended upon the size of the tree, and an attempt was made to secure uniform coverage or wetting of all parts of each tree. At the time of spraying a number (from two to five) of small branches on each tree were tagged as suitable for making counts and mortality determinations of the scales located thereon.

In order to determine whether any separation of the oil occurred in the spray tanks, samples were taken from the nozzle at the beginning and end of the application of each spraying and analyzed for oil content. These samples showed from 1.9 to 2 percent of oil. Since a difference of 0.1 percent is within the limits of accuracy of the method of sampling and analysis, it was concluded that no breakdown of the emulsions occurred while spraying, and that all trees received substantially the correct oil concentration.

## RESULTS

### OIL DEPOSITED BY SPRAYS

Since the previous work done in a study of the insecticidal action of emulsions at this laboratory (1) has shown that the percentage of insects killed is closely related to the quantity of oil deposited on the plant surface by the spray, analyses were made to determine the quantity of oil deposited on camphor-tree foliage by all sprays described herein.

At the time the potted plants were treated on June 14, portions of the same sprays were applied to pairs of branches from green camphor-trees. The number of leaves on the branches usually amounted to about 1,000. After being sprayed, the branches were allowed to dry, and from each branch (there were duplicate branches for each spray) approximately 500 leaves were clipped. These leaves were thoroughly mixed, and from each lot 200 disks 2 cm in diameter were stamped by means of a leaf die. The disk samples were then extracted with ether as the first step in the determination<sup>5</sup> (3) of the quantity of oil deposited by the respective sprays.

In the laboratory tests of August 24 the method of application of sprays to foliage more closely approximated the method by which the potted plants were sprayed. Branches cut from trees in the field were brought to the laboratory, and the smaller twigs were cut off and formed into bunches, which were then stuck into pots to simulate the potted plants. These were placed on the revolving platform and sprayed in the same way as the plants, except that the time of application was lengthened to 45 seconds because of the greater quantity of foliage. Table 3 shows the average volume of oil found per square centimeter of foliage, listed together with the sprays producing the deposit. (See table 1 for composition of these sprays.)

<sup>5</sup> DAWSEY, L. H. THE DETERMINATION OF UNREFINED MINERAL OILS RETAINED BY LEAF SURFACES FOLLOWING SPRAYING. Unpublished manuscript.

TABLE 3.—Quantities of oil deposited on camphor-tree foliage by laboratory sprays, showing analyses of duplicate samples for each spray

Sprays applied June 14, 1932				Sprays applied Aug. 24, 1932			
Spray no.	Analysis sample no.	Volume of oil deposited per square centimeter of leaf surface at 20° C.		Spray no.	Analysis sample no.	Volume of oil deposited per square centimeter of leaf surface at 20° C.	
		Volume found	Average volume			Volume found	Average volume
		Cc×10 <sup>5</sup>	Cc×10 <sup>5</sup>			Cc×10 <sup>5</sup>	Cc×10 <sup>5</sup>
1a.....	{ 3	4.59	4.4	4a.....	{ 3	4.04	4.1
	{ 4	4.22		4a.....	{ 4	4.09	
2a.....	{ 15	4.22	4.3	5a.....	{ 15	4.17	4.1
	{ 16	4.36		5a.....	{ 16	4.04	
3a.....	{ 9	4.20	4.2	6a.....	{ 9	3.77	3.8
	{ 10	4.19		6a.....	{ 10	3.77	
1b.....	{ 5	6.04	6.7	4b.....	{ 5	5.74	5.7
	{ 6	7.26		4b.....	{ 6	5.59	
2b.....	{ 17	5.58	5.6	5b.....	{ 17	5.74	5.7
	{ 18	5.55		5b.....	{ 18	5.69	
3b.....	{ 11	6.18	6.2	6b.....	{ 11	4.85	4.8
	{ 12	6.12		6b.....	{ 12	4.79	

The deposits produced by sprays 1a to 3a should be slightly greater than those of sprays 4a to 6a, inasmuch as the oil concentration in the former was 1 percent while in the latter it was only 0.9 percent. This appears generally to be the case for corresponding preparations in the sprays applied June 14 and those applied August 24. On the other hand, the average oil deposits of the "b" sprays, in each series, were higher than those of the "a" sprays in the same series, because the oil concentrations were much higher.

The disagreement between deposits from the sprays of equal strength, as shown in table 3, is not thought to be due to large errors in chemical analyses, since check samples containing known quantities of each oil were run simultaneously with the test samples. The lack of agreement is more likely to have been caused by nonuniform spraying of the duplicate tree branches, in spite of the precautions used in applying the sprays. A second source of error probably lay in the random difference in leaf surface as it affected the quantities of oil deposited. The question of lack of uniformity in coverage as being due to changing oil concentrations in the sprays during application was separately investigated for the laboratory sprays applied August 24. Samples of stock mixtures 4a to 6b (table 2) were placed in the reservoir of the spray apparatus, and the same volumes of spray liquids as were applied to each infested group of camphor plants were sprayed from the nozzle and caught in beakers for determination of the percentage of oil in each portion. There were six 300-cc portions in each case, including the liquid ejected at the beginning when the spray tank was full and the liquid at the end when the tank was nearly empty. If the oil globules actually had collected toward the top of the tank, the first portions of the spray would have been lower in oil content than the last portions, since the outlet pipe projected to the bottom of the spray reservoir. All tests made in this manner showed no change in the percentage of oil in the liquids, hence the trouble could not be located at this point. On the contrary, the oil-percentage determinations indicated that the quantity of oil

in the sprays as they left the nozzle of the apparatus could be controlled with high precision.

In preparation for the determination of the oil deposits produced by the field sprays, certain portions of each sprayed tree that offered the proper degree of infestation had been tagged prior to the spray application. Since mortality counts were to be made on these twigs, leaves from these twigs and from the immediate vicinity were detached after the sprays had dried, and disks 2 cm in diameter were cut from them. Duplicate samples of only 150 disks were taken from each tree, since the oil deposits on the foliage appeared to be heavy. The tagged, infested twigs, upon which the tips and younger shoots were left untouched, were left growing on the tree. The quantities of oil found in these samples are shown in table 4. Again, in these field sprays, differences in samples were found, but they are likewise attributed to small errors in analyses or to larger and unavoidable discrepancies due to nonuniform spray coverage.

TABLE 4.—Quantities of oil deposited on camphor-tree foliage per square centimeter of leaf surface in field spraying,<sup>1</sup> as shown by analyses of duplicate samples from each tree sprayed

Tree no.	Spray 7F	Spray 8F	Spray 9F	Tree no.	Spray 7F	Spray 8F	Spray 9F
	Cc×10 <sup>5</sup>	Cc×10 <sup>5</sup>	Cc×10 <sup>5</sup>		Cc×10 <sup>5</sup>	Cc×10 <sup>5</sup>	Cc×10 <sup>5</sup>
2.....	{ 9.6	-----	-----	7.....	{ 10.2	-----	-----
	{ 9.8	-----	-----		{ 9.4	-----	-----
3.....	{ 10.6	-----	-----	8.....	{ -----	-----	10.2
	{ 9.0	-----	-----		{ -----	-----	9.6
4.....	{ -----	9.4	-----	10.....	{ -----	-----	10.1
	{ -----	8.9	-----		{ -----	-----	10.6
5.....	{ 8.7	-----	-----		{ -----	-----	-----
	{ 10.4	-----	-----		{ -----	-----	-----

<sup>1</sup> Each spray contained 2 percent of oil. The soap concentration (normality of soap in the aqueous phase) of  $24.7 \times 10^{-4}$  includes oleates formed with sodium, calcium, magnesium, etc., in the dilution water.

#### DETERMINATION OF MORTALITY

Because of unavoidable delays in securing the proper oils, the scales at the time (June 14) of the first laboratory tests were reproducing by the time they were sprayed. If they had been kept under summer temperatures, many of the living scales would have deposited all their eggs and died before the mortality counts could have been completed, so it was necessary to retard the rate of development by subjecting the insects to low temperatures. The sprayed plants were placed in a cold room, where they were held at temperatures of 11° to 13° C. until the mortality had been determined. Counts were started June 29 and completed July 2, 18 days after spraying.

Earlier tests had shown that even lower temperatures did not cause any mortality of camphor scales when kept in the cold room for 3 weeks. In these preliminary cold-room tests, made earlier in the year, infested twigs were taken from camphor-trees in the field, the leaves removed, and the twigs (with the cut ends in water) were placed in the cold room, which was held at a temperature of 8° to 11° C. The natural mortality for one sample of these twigs was determined at the time of cutting, and for two other samples 15 and 21 days later. The results are given in table 5.

TABLE 5.—Proportion of adult scales found alive on sample twigs on different dates, 1932

Date counted	Period in cold storage	Overwintering adults counted	Adults alive
	Days	Number	Percent
Jan. 14.....	0	800	86.8
Jan. 29.....	15	779	84.2
Feb. 4.....	21	811	86.7

No significant changes in mortality were found; in fact, the counts made January 14 and February 4 showed unusually close agreement for duplicate samples from the field.

The counts were made in the manner described in a previous paper (1), but the methods of analyzing the data differed from those used

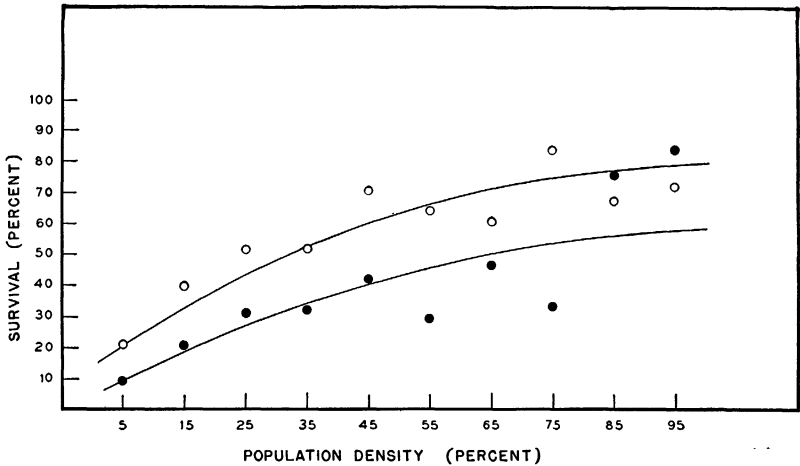


FIGURE 1.—Survival of camphor scales on potted camphor-tree plants sprayed June 14, 1932. The upper curve represents the average survival for the 1a, 2a, and 3a sprays, which contained 1 percent of oil. The lower curve represents the average survival for the 1b, 2b, and 3b sprays, containing 1.7 percent of oil. Open circles represent the average survival at different population densities for the 1-percent sprays; solid circles, the average survival for the 1.7-percent sprays.

in the earlier work. After determining the natural mortality at each population-density interval, the numbers of dead and living scales at each density were totaled for the plants sprayed with the same oil concentration. Corrections were made for natural mortality, and the percentages killed by the sprays estimated. In this way curves were obtained (fig. 1) which represented the average survival for the three oils at each oil concentration. Then the number surviving each oil was compared with the average survival for the three oils at the corresponding concentration, and the deviation from the average curve found at each density interval. To illustrate this procedure, a sample summary is shown in table 6. By taking the algebraic sum of the deviations, the differences in survival for the three oils were found. These differences were finally expressed as percentages of the total number of scales alive before spraying, as shown in table 7.



TABLE 6.—Sample summary used in the estimation of relative effectiveness of sprays made from oils differing in sulphonatable content

(The number of scales alive before spraying was obtained by multiplying the total number of scales at each population density by the percent alive in the check. The number expected to survive was estimated by multiplying the number alive before spraying by the average rate of survival at the corresponding density as shown in fig. 1. The data given are for spray 1a, containing 1 percent of oil)

Population density	Scales alive before spraying	Scales surviving	Scales expected to survive	Deviation	Population density	Scales alive before spraying	Scales surviving	Scales expected to survive	Deviation
	<i>Number</i>	<i>Number</i>	<i>Number</i>	<i>Number</i>		<i>Number</i>	<i>Number</i>	<i>Number</i>	<i>Number</i>
5 percent	82	14	17	-3	55 percent	96	57	64	-7
15 percent	113	45	37	8	85 percent	50	31	39	-8
25 percent	104	53	46	7	95 percent	92	73	74	-1
35 percent	82	46	43	3					
45 percent	146	102	88	14					

TABLE 7.—Comparison of rates of survival of camphor scales on potted camphor-tree plants treated with sprays made from oils differing in sulphonatable content; sprays applied June 14, 1932

Spray no.	Proportion of oil sulphonatable	Oil concentration in spray	Average oil deposit per square centimeter of leaf surface	Plants	Scales alive before spraying	Deviation from average survival curve	
						In scales surviving <sup>1</sup>	In rate of survival
	<i>Percent</i>	<i>Percent</i>	<i>CcX10<sup>5</sup></i>	<i>Number</i>	<i>Number</i>	<i>Number</i>	<i>Percent</i>
1a	6	1.0	4.4	6	765	+13	+1.7
2a	16	1.0	4.3	6	504	+19	+3.8
3a	33	1.0	4.2	7	662	-18	-2.7
1b	6	1.7	6.7	6	671	+2	+3
2b	16	1.7	5.6	6	557	-26	-4.7
3b	33	1.7	6.2	7	582	+26	+4.5

<sup>1</sup> The curves of fig. 1 were fitted so as to make the sum of the residuals approximately zero. Since in calculating the residuals the deviations from the curves were weighted by the square root of the number of scales at each density, the sum of the actual deviations as shown in this column will not necessarily be zero.

The methods of counting and analysis of the results of the sprays applied August 24 were the same as described for the June 14 sprays. Figure 2 shows the average survival curves for each of the two oil

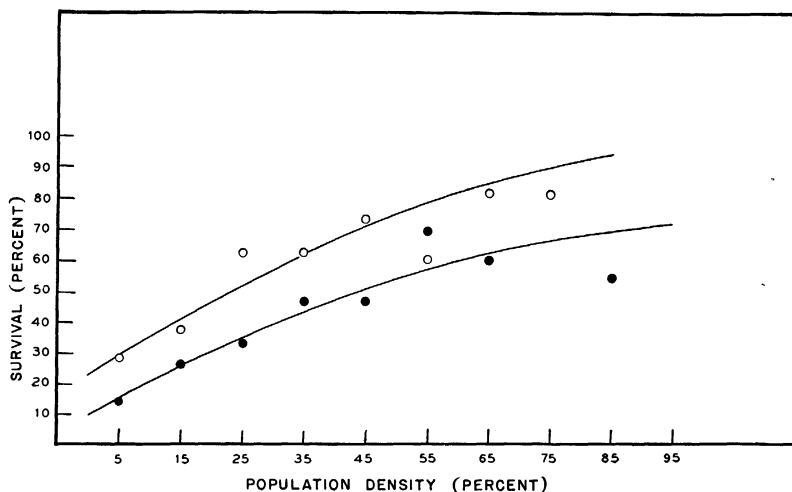


FIGURE 2.—Survival of camphor scales on potted camphor-tree plants sprayed August 24, 1932. The upper curve represents the average survival for the 4a, 5a, and 6a sprays containing 0.9 percent of oil. The lower curve represents the average survival for the 4b, 5b, and 6b sprays containing 1.5 percent of oil. Open circles represent the average survival for the 0.9-percent sprays; solid circles, the average survival for the 1.5-percent sprays.

concentrations. The deviations from these curves of the survival rates for each spray are given in table 8.

TABLE 8.—Comparison of rates of survival of camphor scales on potted camphor-tree plants treated with sprays, made from oils differing in sulphonatable content; sprays applied Aug. 24, 1932

Spray no.	Proportion of oil sulphonatable	Oil concentration in spray	Average oil deposit per square centimeter of leaf surface	Plants	Scales alive before spraying	Deviation from average survival curve	
						In scales surviving	In rate of survival
	Percent	Percent	Cc×10 <sup>5</sup>	Number	Number	Number	Percent
4a.....	6	0.9	4.1	5	1 281	-9	-3.2
5a.....	16	.9	4.1	5	697	+6	+ .9
6a.....	33	.9	3.8	6	839	-8	-1.0
4b.....	6	1.5	5.7	6	574	-42	-7.3
5b.....	16	1.5	5.7	5	1,093	+58	+5.3
6b.....	33	1.5	4.8	6	829	-22	-2.7

<sup>1</sup> Figures for this spray do not include 1 plant which showed only 21 percent dead (natural mortality in checks was 11.5 percent) and was inconsistent with the other 5 plants in this group. The reason for the low kill is unknown. Possibly the plant had a roughened bark instead of the usual smooth green bark. On such plants the kill is always lower. If this plant is included, the 4a spray shows a deviation from the average curve of 14.7 percent; the effect, therefore, would be to increase the variability of the results but not to indicate a difference due to the sulphonatable portion of the oil.

At the time the field sprays were applied the living overwintering scales were all adults, about 95 percent being in the pink and gravid stages. In making the counts from the field material the dead adults of the older broods and those attacked by predators or fungous diseases were classified separately. They were used in estimating the population density, but were then eliminated from further calculations. The proportion of dead immature scales was negligible, and such scales were not included in the counts.

Immediately before applying the sprays, infested branches were cut from the trees to be sprayed and the natural mortality was determined. No systematic variations in the proportion of dead scales were found throughout the rows. Counts were also made on branches of the check trees at intervals until the spray counts were completed, and no changes in the natural mortality were found during this time; accordingly, all the check counts were averaged to find the natural mortality at each population density. The proportion of dead scales in the unsprayed branches increased from 3 percent, when 5 percent of the twig area was covered by scales, to 8 percent when the population density was 95 percent.

From the natural mortality curve the number of scales estimated to be alive before spraying was found for each of the sprayed twigs, and from the number actually found alive the survival was calculated as a percentage of the number of scales alive before the application. The survival percentages are given in table 9 and are shown graphically in figure 3.

TABLE 9.—Comparison of the rates of survival of camphor scales (at different population densities) on camphor-trees in Metairie Cemetery, after treatment, Nov. 22, 1932, with sprays 7F, 8F, and 9F, made from oils containing different proportions of sulphonatable compounds; together with data concerning quantities of oil deposited on leaves and some of the properties of the oils used

SPRAYS USED

Spray no. <sup>1</sup>	Oil used	Saybolt viscosity of oil at 100° F.	Proportion of oil volatilized (24 hours at 100° F.)	Specific gravity of oil at 68° F.	Proportion of oil sulphonatable	Average volume of oil deposited per square centimeter of leaf surface
		Seconds	Percent		Percent	Cc×10 <sup>3</sup>
7F.....	WR.....	94.9	1.44	0.8512	6	9.7
8F.....	WR/L-1778.....	97.8	1.42	.8679	16	9.5
9F.....	L-1778.....	101.9	1.59	.8846	33	10.1

RESULTS WITH VARIOUS SPRAYS

Population density (percentage of twig area covered by scales)	Checks		Spray 7F		Spray 8F		Spray 9F	
	Scales alive before spraying	Survival	Scales alive before spraying	Survival	Scales alive before spraying	Corrected survival	Scales alive before spraying	Corrected survival
	Number	Percent	Number	Percent	Number	Percent	Number	Percent
0-10.....	501	96.9	299	20.7	282	43.6	253	21.3
10-20.....	1,008	96.1	673	32.2	785	46.4	843	33.3
20-30.....	1,336	95.8	736	49.5	900	54.9	1,129	47.1
30-40.....	1,068	95.6	831	62.2	938	65.6	811	49.9
40-50.....	800	94.9	632	57.8	680	75.9	914	62.7
50-60.....	918	94.6	410	78.0	408	76.2	827	57.6
60-70.....	642	94.0	272	71.7	339	92.0	585	72.0
70-80.....	390	90.9	216	70.4	193	81.3	236	68.6
80-90.....	298	93.4	149	71.1	92	100.0	329	74.2
90-100.....	361	91.6	133	58.6	42	81.0	357	73.1

<sup>1</sup> Each spray contained 2 percent of oil to 98 percent of soap solution which was 24.6×10<sup>-4</sup> normal in soaps.

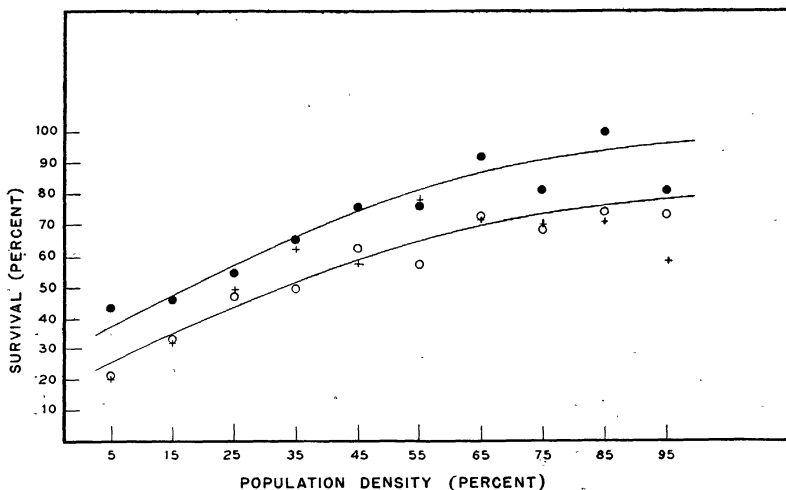


FIGURE 3.—Survival of camphor scales from oil sprays applied to camphor-trees. The lower curve represents the average survival for sprays 7F (WR oil) and 9F (L-1778 oil); the upper curve, the survival for spray 8F (containing a mixture of the two oils). Crosses represent survival from the WR oil spray; open circles, survival from the L-1778 oil spray; and solid circles, survival from the spray of the two oils mixed. Each spray contained 2 percent of oil.

From the curves of figure 3 it may be seen that the values at each density for the 7F and 9F sprays fell so close together that they could be fitted by one curve. The oils used in these sprays represented the two extremes in sulphonatable content. The survival from the 8F spray was uniformly higher over the entire range of population density. This oil was a mixture of the two other oils, and therefore intermediate in its properties. The average oil deposit from this spray was slightly lower than from the others, but the difference, although it may have been a contributing factor, was hardly sufficient to account for all of the difference in scale mortality. Other possible reasons might be differences in the texture of the bark, or more foliage on trees 4 and 7 to hinder the oil from reaching the twig surfaces where the scales were located.

### DISCUSSION OF RESULTS

From the curves shown in figures 1 and 2 it was found that the average survival for the laboratory sprays containing 0.9 percent of oil ranged from 29 percent at a population density of 5 percent to 90 percent at a population density of 75 percent, and for the spray with 1.5 percent of oil, from 15 to 67 percent at the same population densities; for the 1-percent spray the survival ranged from 21 to 80 percent within the population density limits of 5 to 95 percent, and for the 1.7-percent spray, from 9 to 59 percent within the same limits. In the field sprays, when 2 percent of oil was used, the two oils of 6-percent and 33-percent sulphonatable content killed practically the same proportion of scales, the survival ranging from 25 to 78 percent over the entire population-density range, while for the oil containing 16 percent sulphonatable material the survival was 36 to 96 percent (fig. 3). The oil deposit on the leaves was considerably greater in the field applications, and it appears probable that a larger proportion of the oil was deposited on the foliage relative to that deposited on the twigs, by the field sprays than by the laboratory sprays.

The data given in tables 7 and 8 show no systematic differences in the laboratory tests which could be attributed to the sulphonatable portions of the oils used. In table 8, sprays 5a and 5b, in which the oil used was intermediate in its sulphonatable content, show slightly higher rates of survival than the other sprays in their respective groups. Otherwise the differences in the oil deposit and insect mortality appeared to be entirely random and well within the limits of variation ordinarily encountered in the effects of sprays where a comparatively low mortality is obtained. The results of the field sprays (table 9 and fig. 3) agree with those of the laboratory sprays in failing to show any differences in insecticidal efficiency resulting from differences in the sulphonatable portion of the oils. In all of these tests, therefore, no evidence was found that the sulphonatable portions of the oils had any effect upon the insecticidal action when the other physical and chemical properties of the sprays were uniform.

### EFFECT OF OILS UPON PLANTS

Little concerning the relation between plant injury and refinement of the oil can be deduced from the laboratory spray tests, since small potted camphor-tree plants with few leaves and a heavy scale infestation may be much more easily injured than trees in the field. Of the

trees to which the field sprays were applied, only the two (nos. 8 and 10) sprayed with the oil containing 33-percent sulphonatable material exhibited any appreciable injury. On February 1 these had lost 75 to 80 percent of their leaves, whereas none of the others had had more than a 10-percent leaf drop. Freezing weather (20° F.) occurred on February 9, and on March 10 tree 10 was completely defoliated and tree 8 had only a few new leaves at the top. However, one of the trees sprayed with the mixed oil and one of the check trees had very few leaves also. The other trees were apparently normal. Other unsprayed trees throughout the city showed great variation in their resistance to the low temperature, and it is not known how much of the injury in the sprayed plot can be ascribed to increased susceptibility resulting from the oil sprays. However, the fact that trees 8 and 10 had by far the greater leaf drop before the freeze may indicate that under normal conditions the less refined oils are more toxic to camphor-trees.

#### SUMMARY

The comparative insecticidal value of three petroleum oils with 6, 16, and 33 percent by volume, respectively, of sulphonatable material was investigated in laboratory and field spraying tests. The oils were applied, in the form of emulsions containing from 0.9 to 2 percent of oil, sodium oleate soap being used as the emulsifier, to camphor-tree plants and trees infested with the camphor scale (*Pseudaonidia duplex* (Ckll.)). The other characteristics of the different oils were substantially the same, so that any detectable differences in scale mortality could be due only to differences in the sulphonatable portions.

When sprays which gave equal oil deposits were compared, the variations in mortality appeared to be entirely random, and no differences were found which could be attributed to the sulphonatable content of the oils.

For making comparisons between the toxicities of different oils, the first condition it was sought to satisfy was that of obtaining equal oil deposits on the plants. Analyses of the oil deposits left by sprays showed that substantially equivalent deposits were obtained when equal concentrations of the different oils were applied. In the laboratory tests the oil deposits produced were independent of the sulphonatable content of the oil but varied directly with the oil content of the spray, ranging from  $3.8 \times 10^{-5}$  cc per square centimeter of leaf surface for a 0.9-percent emulsion to  $6.7 \times 10^{-5}$  cc per square centimeter for a 1.7-percent emulsion. In the field spraying with emulsions containing 2 percent of oil the deposits ranged from  $9.5 \times 10^{-5}$  cc to  $10.1 \times 10^{-5}$  cc per square centimeter of leaf surface.

The average survival for the laboratory sprays containing 0.9 percent of oil ranged from 29 percent at a population density of 5 to 90 percent at a population density of 75 percent, and for the 1.5-percent sprays, from 15 to 67 percent at the same densities; for the 1-percent sprays, from 21 to 80 percent at densities of 5 to 95 percent; and for the 1.7-percent sprays, from 9 to 59 percent within the same density limits. In the field sprays, where 2-percent emulsions were used, the two oils containing 6 and 33 percent of sulphonatable material, respectively, killed the same proportion of scales, the sur-

vival ranging from 25 to 78 percent over the entire population-density range, while for an oil containing 16 percent of sulphonatable material the survival was from 36 to 96 percent.

No conclusion as to the effects of the oils on potted plants was practicable, inasmuch as such results would not be typical of reactions under field conditions; however, in the field tests the trees sprayed with 2-percent emulsions made with oil containing 33 percent of sulphonatable material showed a 75- to 80-percent leaf drop when observed 10 weeks after the spraying date. Trees sprayed with oils of lower sulphonatable content had a maximum leaf drop of less than 10 percent.

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