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STEPHEN A. FORBES, *Chief*

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Recent Insecticide Experiments in Illinois with Lubricating Oil Emulsions

BY

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ERRATA

Page 2, line 6, for *loan* read *loam.*

Page 4, line 18 from bottom, beech (after water beech) should be followed by a semicolon.

Page 138, line 10 and line 14 from bottom, for *Dane* read *Dann.*

Page 139, line 5, for *Dane* read *Dann.*

Page 180, line 5 from bottom, delete *D.*

Page 198, line 19 from bottom, for *March* read *March 16, 1918.*

Page 221, line 22, for *data* read *date.*

Page 278, lines 17 and 18 from bottom in right-hand column, for *150* read *158.*

Page 285, line 24 in left-hand column, for *Franeh* read *French.*

Page 321, table III, center column, for *1.02* read *1.00+*; for *1.04* read *1.02;* for *1.06* read *1.04.*

Page 411, line 4, for *pupation* read *breaking dormancy* .

STATE OF ILLINOIS

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Article II.—*Recent Insecticide Experiments in Illinois with Lubricating Oil Emulsions.* By S. C. Chandler, W. P. Flint, and L. L. Huber.

Introduction

From 1919 to 1922 inclusive, the San Jose scale caused more damage in southern Illinois than in any equal period since it was first established in this state. Following the work of Dr. Forbes and his assistants in 1900, 1901, and 1902, liquid lime-sulfur had been considered the standard remedy for San Jose scale control. Previous to 1919, it had not failed to give a satisfactory commercial control where thoroughly applied at dilutions of from 1 to 6, to 1 to 8. During 1920 and 1921, some of the best and most careful orchardists in southern Illinois lost trees from scale although these trees had been thoroughly sprayed with lime sulfur. In some instances the failure to control with it could be accounted for by the fact that the trees had been poorly sprayed, or an insufficient amount of material had been applied. In other cases, however, the applications had been made as thoroughly as seemed possible and enough material had been put on to cover the trees thoroughly. During the years mentioned above, a series of mild winters following warm late falls had allowed the scale to increase at an unusual rate, so that trees having a small amount of live scale remaining upon them in spring were heavily infested by fall. Because of the failure of lime sulfur to give a satisfactory control of scale, a series of experiments to test other scalecides was made by the Natural History Survey during the winter of 1922.

These experiments were planned to give a comparison of commercial liquid and dry lime-sulfur with commercial miscible oils and homemade lubricating-oil emulsions. The lubricating oil emulsion used, was of the type developed by W. W. Yothers, of the United States Bureau of Entomology for combating citrus scale insects in Florida, and was made by boiling together:

Potash-fish-oil soap	1 pound
Water	½ gallon
Light grade lubricating oil........	1 gallon

The mixture was boiled for about five minutes, removed from the fire, and pumped twice under a pressure of about seventy-five pounds, and it was then diluted at the rate of three gallons of the stock emulsion to ninety-seven gallons of water.

Experiment in Jolly Orchard, Olney, Illinois, Spring of 1922

A block of twenty-five-year old Ben Davis and Grimes Golden apple trees was chosen for this experiment in what was known as the Jolly orchard at Olney. These trees were heavily infested, most of them having a considerable portion of the tree incrusted. Those selected for the experiment were divided into blocks five rows long by four rows wide, the center row being Grimes Golden, and the two outside rows Ben Davis. The infested branches, which were later cut for scale examinations, were taken from the inside rows of the blocks.

The first sprays were applied on March 28, all blocks being treated within a week. Special attention was given to the application of the sprays. All trees were sprayed with rods, and the operator was followed by a third man to see that no part of the tree remained unsprayed. In the course of this work, we found that slightly more of the oil sprays was required to cover trees of a given size than was the case with the lime sulfur sprays. For the twenty-five-year old trees, twenty gallons per tree of the oil were required and fifteen to seventeen gallons per tree of lime sulfur. Forty-seven days after the treatment, samples of scale-infested twigs were taken from various parts of the trees on the inner rows of all of the blocks and examined for living and dead scale. The results of these examinations, expressed in percent of living scale, are shown in Table I.

Table I

Treatment	Percent of live scale
Scalecide (1 to 15)	.5
Spray Emulsion (1 to 15)	.4
Diamond Paraffin oil, fish-oil soap emulsion (2%)	1.5
Junior Red Engine-oil, fish-oil soap emulsion (2%)	7.
Commercial liquid lime-sulfur (32 Baumé, 1 to 8)	11.
Soluble sulfur, Niagara (15 lbs. to 50 gals. water)	18.5
Dry lime-sulfur (15 to 50)	41.
Check, no treatment	50.

The following conclusions, standing in the order of their importance, may be noted:

1. The oil sprays were superior to the sulfurs.
2. The oil emulsion made from Oil No. 1, the brand used in most cases in Florida, was almost as effective as the miscible oils.
3. Dry lime sulfur was not as good as liquid lime sulfur.

The fact that even after the most careful spraying, 11% of the scale was still alive on trees treated with lime sulfur, explained the failure of some growers to control scale where this material had been used. No further experiments were made in this orchard during 1922, but the remainder of it was sprayed thoroughly with commercial lime sulfur, used at 1 to 6, and in some cases at 1 to 4, dilution.

By fall of that year, the scale had so increased that but little fruit in the orchard was salable, and in the spring of 1923, the orchard was so badly infested by scale that no further attempt was made to save the trees by spraying. On May 4 of that year, however, the only trees of the orchard which were blooming, and practically the only trees alive, were those which had been given the oil sprays the previous spring. These blocks of trees were in a fairly vigorous condition, while practically all of the lime-sulfur sprayed trees around them were dead. The entire orchard was cut down during the next year because nearly all the trees had been killed by San Jose scale. (See Fig. 1 and 2.)

FIGURE 1

Outer rows of trees on separate plots, Olney, Ill., May 4, 1923. Row on the left had been sprayed with oil emulsion; row on the right, with lime sulfur. Lime-sulfur-sprayed trees all killed by San Jose scale

EXPERIMENTS IN UNIVERSITY ORCHARD, OLNEY, ILLINOIS, WINTER OF 1922-23

The objects of the investigation in the winter of 1922-23 were as follows:

1. To determine the best formula for making a stock oil-emulsion.
2. To determine the possibility of combining the oil emulsions with other spray materials.
3. To determine the efficiency of homemade oil-emulsions and commercial oil-emulsions.

Most formulae for oil emulsions have been the result of practical experience, for we know but little in regard to the various theories involved in a study of colloids, but some knowledge of the theory of emulsification is necessary if we would proceed intelligently.

THE THEORY OF EMULSION

The theory of emulsion is based on the assumption that we are dealing with two-phase systems, the oil and the water being the respective phases. The oil is the disperse or internal phase, the water is the continuous or external phase, and the two phases of the system are separated by surfaces of contact, or interfaces.

The external phase may be either a solid or a liquid. The first part of this discussion is concerned with a system in which both phases are liquids; but the latter part, with a system in which one phase is a liquid and the other a solid.

An emulsion is defined by Clayton as a system containing two liquid phases, one of which is dispersed as globules in the other. Making lubricating oil emulsion is essentially a mechanical process which has for its purpose the breaking up of the oil in the water, but as oil and water are immiscible an emulsifying agent is needed, and here again there may be two kinds of emulsions, "oil in water" and "water in oil", depending upon the nature of the emulsifying agent. The lubricating oil emulsions are of the former kind.

THE USE OF A SOLID AS AN EMULSIFIER

It has been long known that any substance that will go into the interface and thus increase viscosity, will cause emulsification. In other words, the insoluble solid, if finely enough divided, will yield results similar to those produced by a gelatinous colloid, such as soap. Pickering was one of the first to point out that the basic sulfate precipitated in Bordeaux mixture consists of just such particles. These particles have only a slight tendency to unite with one another, and are more readily wetted by water than by oil. When the oil is added to the Bordeaux mixture and broken up by agitation, the finely divided precipitate surrounds the oil globules, thus holding them in suspension. Theoretically there seems to be no reason to expect unfavorable results with the soapless emulsions. Indeed, in instances where a weak Bordeaux is advised when the soap emulsions are to be diluted with hard water, we get, in reality, what amounts to a soapless Bordeaux-oil emulsion.

THE USE OF SOAP AS AN EMULSIFIER

Bancroft holds that if the emulsifying agent is such that it will lower the interfacial tension of the water more than that of the oil so that the film bends convex to the oil, there will be a tendency to emulsify the "oil in water," but if the absorption of the emulsifying

FIGURE 2

Trees on reader's right had been sprayed with lime sulfur; those on left, with oil emulsion. The pictures were taken at Olney, Ill., May 4, 1923, without moving the tripod of the camera. The lime-sulfur-sprayed trees were all killed by the San Jose scale agent brings about an opposite condition, a "water in oil" emulsion will result. Hence, to make an "oil in water" emulsion it is necessary to use a water-soluble colloid; and to make a "water in oil" emulsion an oil-soluble colloid must be used. More briefly still, to get an "oil in water" emulsion the emulsifying agent must be such that it is wetted more by water than by oil. Potash and sodium soaps are such water-soluble colloids.

The excellence of an emulsion is judged primarily by its stability, which is very dependent on its viscosity. Upon what then does the viscosity depend?

Possibly the greatest single factor affecting viscosity is the volume ratio of the oil and water, or the concentration of the oil. While it is possible to make an emulsion with potash-fish-oil soap and oil alone, or by the addition of an unusual amount of water to the oil and soap, experiment has shown that there is an optimum proportion of ingredients.

Another factor affecting viscosity is the degree to which the ingredients are mechanically agitated. Orchardists have noted that the more often the mixture is run through their pumps and the greater the pressure, the more viscous is the product. This is due to the fact that the oil is reduced to more minute globules and the extent of the oil-water surface is thus greatly increased.

A deficiency in the proportion of soap results in lower viscosity owing to the prevalence of larger oil globules, regardless of agitation. These large globules have a tendency to coalesce by breaking the film which surrounds them.

The size of the oil globules is further dependent on the temperature of the mixture when it is agitated. A cold mixture will yield an emulsion physically inferior to a hot mixture. However, field and laboratory tests have demonstrated that there is nothing to be gained by continued application of heat. Heating facilitates emulsification only by lowering the viscosity of the mixture and reducing the interfacial tension between the two phases, thus aiding mechanical agitation.

In comparative spray-tests we have often failed to take proper cognizance of the fact that an emulsion undergoes some quantitative changes during the process of manufacture. Unless these changes are taken into consideration there is apt to be an element of doubt as to the accuracy of our results. In this regard, it is well to keep in mind that a mixture may, or may not, increase in volume with emulsification.

INCREASE IN VOLUME OF BOILED EMULSIONS

In making up the large series of emulsions which was necessary for these experiments, it was noticed that there was a greater increase in volume in the case of some boiled emulsions than in others, although exactly the same proportions and length of time for boiling was allowed in all cases. In one case the volume was increased by 13%, even though the outlet hose was put into the liquid so that large amounts of air could not be mixed with the emulsion. The increase was much greater where the hose was held out of the liquid, allowing the discharge to fall through the air. In most cases, this increase in volume is not very significant after the emulsion has cooled. The grower making his own emulsion should bear this point in mind when he comes to the dilution of his product.

The data of the tables tend to prove that the amount of water and soap as well as this increase in volume are factors to be considered if final results are to be used in a comparative way.

TABLE II

1. Formula: Oil 1 gallon, water ½ gallon, soap 2 pounds

Emulsion	Water	Percent of oil in spray solution	Percent of dead scale
1½ gal.	100 gal.	.007	73.
3 "	100 "	.015	97.6
4½ "	100 "	.022	98.3
6 "	100 "	.030	100.

2. Formula: Oil 1 gallon, water ¼ gallon, soap 1 pound

Emulsion	Water	Percent of oil in spray solution	Percent of dead scale
1½ gal.	100 gal.	.010	89.
3 "	100 "	.020	98.5
4½ "	100 "	.030	99.4
6 "	100 "	.040	100.

DE-EMULSIFICATION

Sometimes an apparently good emulsion de-emulsifies, or "breaks", and the commercial orchardist generally finds that this is due to one or more of the following causes:

It is to be expected that an emulsion which contains a water phase would be injured by freezing temperatures which by breaking the external or water phase frees the oil. However, it is entirely possible to make a stock emulsion that will withstand continued zero temperature with only negligible damage. Injury is proportional to an excess amount of water in the emulsion.

A most carefully made emulsion may, after a long period, begin to de-emulsify. The air coming into contact with its surface causes evaporation of the water and results in the cracking of the film around the oil globules, and the droplets then coalesce.

The presence of acids or large amounts of lime, as in lime sulfur, leads to the breaking down of an emulsion in such way that re-agitation will not restore it.

Perhaps the most common source of de-emulsification is the presence of calcium or magnesium in the water that is used as a diluent. The presence of these salts with the oil leads to the formation of an insoluble calcium or magnesium soap, which are products of reversion and tend to form "water in oil" emulsions. The addition of Bordeaux mixture to the diluted spray solution prevents this reaction. This recommendation is based upon a statement made in the fore part of the discussion, that a colloid solution has two phases—a solid and a liquid. A fuller explanation is given in the following paragraphs.

Although emulsions can be made with even extreme amounts of water or soap, there is a practical optimum amount of each of these constituents. Judging from our experiments, the optimum amount of soap is from one to two pounds per gallon of oil, and that of water from ¼ to ½ gallon for each gallon of oil. It has been found necessary, in order to get a good emulsion, to use from 1½ to 2 pounds of soap with many of the waters used by orchardists for spraying in this state. Emulsions made with varying amounts of soap and water were tested as to their ability to kill scale. The following tables show the results.

TABLE III

Strength of oils	Amount of soap	Number of experiments	Number of scale examined	Number alive	Percent alive
2%	1 pound	2	1554	19	1.2
2%	2 pounds	2	2083	31	1.4

TABLE IV

Strength of oils	Amount of soap	Number of experiments	Number of scale examined	Number alive	Percent alive
2%	½ gallon	4	4092	77	1.8
3%	½ gallon	4	4076	28	.6
2%	¼ gallon	3	2082	50	2.4
3%	¼ gallon	4	3500	16	.4

These tables would seem to indicate that as far as kill of scale is concerned, there is little to choose between an emulsion made with either one or two pounds of soap and ¼ gallon or ½ gallon of water per gallon of oil, but laboratory experiments showed conclusively that a stock emulsion made with ¼ gallon of water to each gallon of oil was less likely to be broken down from cold, because of a lower freezing point. In addition to this, an emulsion made with ¼ gallon of water requires less space for storage. Using this amount of water in the stock emulsion, a dilution of three gallons in a hundred of water would give slightly more than a 2% solution, and for a 3% strength, four gallons to the hundred would be sufficient.

COMBINATION OF OIL EMULSION WITH OTHER MATERIALS

For practical reasons, it is desirable that a spray may be mixed with as many other spray materials as is possible. Obviously, it is highly desirable that Bordeaux mixture, a fungicide, should be one of the compatible sprays. As already indicated, any substance that will go into the interface and increase the viscosity will cause emulsification, and the basic sulfate which is precipitated when lime and copper sulfate are poured together, does exactly this thing. The small particles of the precipitate have only a slight tendency to unite with one another and are more readily wetted by the water than by the oil; hence they surround the oil globules in the spray solution, thus aiding the soap in holding them in suspension.

Pickering, in 1907, discovered that oil could be emulsified with Bordeaux mixture. Some of his work was therefore duplicated in 1922 in our laboratory, and the product tried out in the field the following year.

Since such a combination is theoretically and practically sound, an oil emulsion made by the formula, one gallon paraffin oil, 90 viscosity, one-fourth gallon of water, and two pounds of potash-fish-oil soap, was used at varying strengths with Bordeaux mixture at strengths of 3-9-50, 9-3-50, and 4-4-50. The results are shown in Table V.

TABLE V

Bordeaux	Percent of oil	Number of scales examined	Number alive	Number dead	Percent alive
3–9–50	2%	1088	20	1068	1.8
3–9–50	4%	1000	1	999	.1
3–9–50	8%	1000	0	1000	0
9–3–50	2%	1000	0	1000	0
9–3–50	4%	1000	0	1000	0
9–3–50	8%	1000	0	1000	0
4–4–50	2%	1000	5	955	.5
4–4–50	3%	1000	0	1000	0
4–4–50	4%	1000	0	1000	0
4–4–50	8%	1000	0	1000	0
Check	...	1015	669	346	65.9

This shows no decrease in the effectiveness of the lubricating oil emulsion as a scalecide when combined with Bordeaux mixture.

The emulsion was also combined with lead arsenate (basic lead) at the rate of one pound to fifty gallons of diluted emulsion. The results are shown in Table VI.

TABLE VI

Treatment	Total number of scales examined	Number alive	Number dead	Percent alive
2%	1000	9	991	.9
4%	1000	0	1000	0

COMPARISON OF BOILED EMULSIONS WITH LIME SULFUR AND VARIOUS MISCIBLE OILS

During the spring of 1923, a number of tests were made with the various spray materials listed below. As the question of thoroughness of application has often been raised in connection with control of San Jose scale, it was obvious that if our final results were to be depended on, all treated branches must have been very carefully covered. All sprays were applied with a small hand-sprayer, previously marked-off areas on the various branches being sprayed individually and from all angles, and spraying was continued until the operator was sure that the solution had covered every scale. This method eliminated the chance —always present in orchard experiments—of taking samples from a part of the tree which had been missed or only partly wet in spraying. Four to eight weeks after the sprays were applied, a number of scales, usually a thousand from each treatment, were examined under a binocular microscope to determine the percent of scale surviving. These examinations were made by at least two persons within a few days after the sample branches were cut from the tree. The scales on these trees were carefully examined in the beginning of the experiment as were also the untreated checks on the same trees at the end of the work.

TABLE VII

Treatment	Number scale examined (total)	Number alive	Number dead	Percent alive
Lime Sulfur (1–8)				
320 Baumé	1000	110	890	11
Lime Sulfur (1–4)	1000	93	907	7
Sun Oil Co.'s Emulsion 1–10	1000	0	1000	0
1–15	1000	1	999	.1
1–30	1042	182	862	17.2
Good's Mistoil				
1–10	1000	0	1000	0
1–15	1000	0	1000	0
1–30	1000	137	862	13.6
Pratt's Scalecide				
1–10	300	0	300	0
1–15	500	0	500	0
1–30	500	11	489	2.2

Table VII gives the results of applications of lime sulfur and of various miscible oils. Since these were applied in the same manner as the oil emulsions mentioned in Tables II to VI inclusive, an inspection of those tables will also give a comparison with the homemade emulsions.

Table VIII

Percent of oil in emulsion	Number of experiments	Number of scales examined	Number alive	Number dead	Percent alive
1	4	4558	894	3664	19.6
2	4	3637	50	3587	1.3
3	8	7576	44	7532	.5
4	8	7600	2	6598	.02
8	4	3600	0	3600	0.0
Check	...	1015	669	346	65.9

From the experiments made in 1922-23, it is apparent that in most cases, a 2% lubricating oil emulsion will give a satisfactory kill of scale if made from the proper oils and thoroughly applied. The effectiveness of the same oil at different strengths, is shown in Table IX. Judging from this record of treatment and of the examination of a fairly large number of scale coming from several experiments, it is evident that a 3% emulsion would be advisable in orchards where scale is very abundant and increasing, but that a 2% emulsion will take care of ordinary infestations when thoroughly applied.

Experiments in Hartline Orchards, Anna, Illinois, Winter of 1923-24

After the spring of 1923, oil emulsions enjoyed a greatly increased popularity due to their cheapness as compared with other oil sprays and to their efficiency as scalecides as proved in trial tests by growers and experiments in this and other states. A large number of oils were advertised as suitable for making the stock emulsions, and insecticide companies took advantage of the general turn from lime sulfur to oil sprays and pushed the sale of their prepared oils. Cold-mixed emulsions also were attracting more attention from the growers, and inquiries began coming in for more information in regard to them. To find an answer to these questions, to gain further information, and to corroborate points brought out during the work of the two previous seasons, a series of experiments was planned for the winter of 1923-24. These were located in the apple orchards of Willis Hartline at Anna, and the sprays were applied to trees that had become badly infested. The same method of application was employed as in 1922-23, all treatments being made with a hand sprayer, and great care taken that every scale should be covered. Table IX gives the results of this work. It will be noticed that some of the fall sprays were repeated in spring. In most cases this was done to check up on fall applications which showed an appreciably higher percent of live scale than did oil emulsion made with a certain 90 viscosity paraffin oil, which we use as a standard because of the large number of tests which have been made with it. In Table IX, viscosity was ascertained by the Saybolt test. Good emulsions were obtained with all the oils used.

TABLE IX

Oils and dosage (Oils used in all cases at 2% strength unless otherwise specified)	Emulsifier and amount per gallon of oil				Date of treatment, number of scale examined, and percent living		
	Potash-fish-oil soap	Calcium caseinate	Bordeaux mixture	Iron sulfate-lime	Date of treatment	Number of scales examined	Percent of living San Jose scale 6 to 8 weeks after treatment
No. 1. Oil, 90-100 viscosity, .42% volatility	1½ #				12/24–26/23	1569	1.6
No. 9. Same oil as No. 1			4–6–50 1 gal.		"	1500	1.8
No. 23. " " " " "			6–8–50 1 gal.		1/8/24	1000	3.5
No. 30. " " " " "				4–6–50 1 gal.	"	1000	1.6
No. 31. " " " " "		4 oz.			1/29/24	1501	2.5
No. 32. " " " " "		4 oz.			"	1537	5.9
No. 35. " " " " "			4–6–50 1 gal.		"	1500	1.5
No. 49. " " " " "			(Copper sulfate 2 oz. lime 2 oz.)		1/30/24	1300	3.6
No. 54. " " " " "	¼ #				1/31/24	1500	1.6
No. 55. " " " " "	½ #				"	1500	1.
No. 56. " " " " "	1½ #				"	1500	1.
No. 62. " " " " " 3%			1–1¼–50 1 gal.		3/17–19/24	1500	0
No. 64. " " " " "				4–6–50 1 gal.	"	1500	1.5
No. 65. " " " " "		4 oz.			"	1500	.2
No. 66. " " " " " 3%		4 oz.			"	1500	.06
No. 70.* " " " " "		4 oz.			"	1500	0
No. 80. " " " " "	¼ #				"	1500	0
No. 81. " " " " "	¾ #				"	1500	0
No. 82. " " " " "	1½ #				"	1500	.06
No. 2. Oil, 98-100 viscosity, .44% volatility	1½ #				2/17–23/24	1543	3.2

* Lime sulfur (1-8) added after emulsification.

TABLE IX—Continued

Oils and dosage (Oils used in all cases at 2% strength unless otherwise specified)	Emulsifier and amount per gallon of oil				Date of treatment, number of scale examined, and percent living		
	Potash-fish-oil soap	Calcium caseinate	Bordeaux mixture	Iron sulfate-lime	Date of treatment	Number of scales examined	Percent of living San Jose scale 6 to 8 weeks after treatment
No. 10. Same oil as No. 2			4–6–50 1 gal.		2/17–23/24	1500	2.8
No. 58. " " " " "	1½ #				4/14–22/24	1500	.2
No. 3. Oil, 91 viscosity, .13% volatility	1½ #				12/24–26/23	1512	.4
No. 11. Same oil as No. 3			4–6–50 1 gal.		"	1300	2.9
No. 4. Oil, 177 viscosity, .04% volatility	1½ #				"	1504	1.1
No. 12. Same oil as No. 4			4–6–50 1 gal.		"	1500	4.
No. 37. " " " " "	1½ #				1/30/24	1500	1.7
No. 41. " " " " "			4–6–50 1 gal.		"	1500	3.3
No. 6. Oil, 108 viscosity, .28% volatility	1½ #				12/24–26/23	1510	2.8
No. 14. Same oil as No. 6			4–6–50 1 gal.		"	1500	4.
No. 60. " " " " "	1½ #				3/17–19/24	1500	.3
No. 7. Oil, 93 viscosity, .34% volatility	1½ #				12/24–26/23	1502	2.0
No. 15. Same oil as No. 7			4–6–50 1 gal.		"	1500	4.
No. 8. Oil, 55 viscosity, 9.84% volatility	1½ #				"	1506	.9
No. 16. Same oil as No. 8			4–6–50 1 gal.		"	1500	3.1
No. 17. Oil, 152 viscosity, 1.36% volatility	1½ #				1/8/24	1000	.6
No. 27. Same oil as No. 17			4–6–50 1 gal.		"	1000	1.7

No. 18. Oil, 130 viscosity, 1.56% volatility	1½ #				"	1000	1.7
No. 26. Same oil as No. 18			4–6–50 1 gal.		"	1000	2.3
No. 20. Oil, 147 viscosity, .77% volatility	1½ #				"	1000	1.7
No. 25. Same oil as No. 20			4–6–50 1 gal.		"	1000	1.7
No. 21. Oil, 100 viscosity, .59% volatility	1½ #				"	1000	.6
No. 24. Same oil as No. 21			4–6–50 1 gal.		"	1000	3.2
No. 22. Oil, 212 viscosity, 1.35% volatility	1½ #				"	1000	2.5
No. 28. Same oil as No. 22			6–8–50 1 gal.		"	1000	4.4
No. 33. " " " " "		2 oz.			1/29/24	1501	.5
No. 34.* " " " " "		2 oz.			"	1500	.1
No. 36. " " " " "			4–6–50 1 gal.		1/30/24	1500	.6
No. 50. " " " " "					1/31/24	1400	2.6
No. 57. " " " " "	1½ #				3/17–19/23	1100	.4
No. 63. " " " " " 3%			4–6–50 1 gal.		"	1500	.1
No. 67. " " " " "		4 oz.			"	1500	.8
No. 68. " " " " " 3%		4 oz.			"	1500	.2
No. 69.* " " " " "		4 oz.			"	1500	.5
No. 39. Oil, 100 viscosity, .33% volatility	1½ #				1/30/24	1500	.9
No. 43. Same oil as No. 39			4–6–50 1 gal.		"	1500	2.1
No. 40. Oil, 189 viscosity, .50% volatility	1½ #				"	1500	1.2
No. 44. Same oil as No. 40			4–6–50 1 gal.		"	1500	1.8
No. 51. Oil, 179 viscosity, .90% volatility					1/31/24	1500	1.6
No. 52. Same oil as No. 51 4%					"	1500	.4
No. 53. " " " " " 8%					"	1500	0
No. 74. " " " " " 3%					3/17–19/23	1500	.06

* Lime sulfur (1-8) added after emulsification.

TABLE IX—Concluded

Oils and dosage (Oils used in all cases at 2% strength unless otherwise specified)	Emulsifier and amount per gallon of oil				Date of treatment, number of scale examined, and percent living		
	Potash-fish-oil soap	Calcium caseinate	Bordeaux mixture	Iron sulfate-lime	Date of treatment	Number of scales examined	Percent of living San Jose scale 6 to 8 weeks after treatment
No. 76. Same oil as No. 51					3/17–19/23	1500	.06
No. 77. " " " " " 4%					"	1500	0
No. 45. Oil, 48 viscosity, 7.72% volatility			4–6–50 1 gal.		1/30/24	1500	5.2
No. 46. Same oil as No. 45	1½#				1/31/24	1500	3.6
No. 61. " " " " "	1½#				3/17–19/23	1400	1.7
No. 71. Sherwin-Williams Free-mulsion 1-50					"	1500	.6
No. 72. Same oil as No. 71 1-25					"	1500	0
No. 73. Sunoco, 1-20					3/17–19/23	1500	0
No. 75. Same oil as No. 73					"	1500	.06
No. 38. Oil, 115 viscosity, .28% volatility					1/30/24	1500	2.2

A Comparison of Oils for Oil Emulsion

Good emulsions were made with all grades of oil used, but five of them show a somewhat too high percent of live scale in the fall tests. Comparing the scale kill with the analyses of oils, it will be found that the most effective oils fall within certain limits. After a conference with government entomologists who had been working on the control of San Jose scale with oil emulsions at Bentonville, Arkansas, and at Vincennes, Indiana, and with the Entomologist of the Purdue Agricultural Experiment Station, a joint statement was issued in the fall of 1924 to the effect that the best results had been obtained with oils within the following limits:

Specific gravity ....87 to .93 at 20° C.
Volatility ....Not above 2% at 110° C. for 4 hours. (Saybolt test.)
Viscosity ....90 to 250 seconds at 100° F. (Saybolt test.)

A Comparison of Boiled Fish-oil-Soap Emulsions and Cold-mixed Emulsions

1. Scale Kill

Table X, summarizes all the tests made in the Hartline orchard with boiled fish-oil-soap emulsions of 2% strengths and all those with cold-mixed emulsions except where lime sulfur was combined with them. This table shows the cold-mixed emulsions to be not quite so effective as the boiled emulsions. This table, however, gives only a rough comparison of all types of cold-mixed emulsions used with certain types of boiled emulsion.

Table X

Type of emulsion	Number of tests made	Number of scales examined	Number of live scales	Percent alive
Boiled F. O. soap, 2%....	25	33,747	513	1.5%
Cold-mixed, 2%...........	28	37,202	996	2.7%

2. Cold-mixed Oil Emulsions

The argument in favor of cold-mixed emulsions is the ease with which they can be made. They do not require boiling, nor handling while hot, and they can usually be more cheaply made than a boiled soap emulsion. In most cases they do not have as high a wetting power as the soap emulsions, and this makes them much less effective against certain kinds of insects, such as aphids.

3. Ease of Dilution

The ease with which any spray material mixes in the tank is an important consideration. Some stock emulsions look good, but upon dilution with water, free oil, which may be injurious to plants, appears on the surface.

In the case of the cold-mixed emulsions with Bordeaux mixture as the emulsifying agent, the stock emulsion rises to the top of the spray solution, though no free oil may appear. This difficulty can be overcome by diluting the stock emulsion with a weak Bordeaux instead of water. A 1-1¼-50 Bordeaux holds it at an equilibrium. It is possible that with the agitator in a spray tank running, this difficulty would not be so serious, but the stock emulsion rises quickly, and it is not at all certain that with the agitator in the bottom of a full tank, a good mixture could be made.

4. Stability

On the whole, cold-mixed emulsions are not as stable as boiled emulsions, as shown by our experience of the past four years. The cold-mixed stock emulsions, upon standing, break down faster than the boiled emulsions, especially in cold weather. For this and other reasons, there is a greater likelihood of injury with the cold-mixed emulsions than with the boiled emulsions.

5. Compatibility with Fungicides

The boiled soap-oil emulsions will mix with Bordeaux, but not with lime sulfur. Most cold-mixed emulsions will mix with both Bordeaux and lime sulfur. While there is some precipitation in the cold-mixed emulsions with lime sulfur, yet effectiveness does not seem to be impaired, as will be seen by applications 69 and 70 in Table IX (pp. 115, 117).

6. Kinds of Cold-mixed Emulsions and Methods of Making

Bordeaux.—Cold-mixed Bordeaux-oil emulsion is made by pumping together, without heating, oil and Bordeaux mixture. Most of that used in our experiments was made with equal parts of oil and Bordeaux. Three pumpings gave a product appreciably better than that made with two. In most of our work, a 4-6-50 Bordeaux (using hydrated lime) was used.

Calcium Caseinate.—Kayso, or any form of calcium caseinate, usually makes a good emulsion. It is probably the easiest to make of any of the commonly used cold-mixed emulsions, and one of the cheapest. The formula generally used is two gallons of oil and one gallon of water in which is mixed four ounces of calcium caseinate. Calcium caseinate should be used fresh to get the best results.

Iron Sulfate-Lime.—Iron sulfate and lime can be used in place of the copper sulfate and lime of the Bordeaux mixture. In our experiments, this emulsion was made up in exactly the same way. The same difficulty of the emulsion rising to the top appeared, but was overcome by diluting with a 1-1¼-50 iron sulfate-lime mixture instead of water.

Colloidal Clays.—Certain colloidal clays—Kaolin, Fuller's earth, Bentonite, and several others—have been used successfully for making cold-mixed oil emulsions. Those made with these clays were only tested in a very limited way in the work here recorded, but very good results were obtained. Work of the entomologists of the Bureau of Entomology and in other states indicates that excellent emulsions can be made with these colloidal clays. In some respects these are superior to most other types of cold-mixed oil emulsions, and they are much cheaper than the boiled soap oil emulsion. They are made up in the form of a thin paste rather than a fluid, and this is objectionable for some uses.

VEGETABLE-OIL-SOAP EMULSIONS

In treatments Nos. 54, 55, 56, 80, 81, and 82 of Table IX (see p 115), the results of spraying with emulsions made with vegetable-oil soap as a substitute for fish-oil soap are given. They are apparently just as effective scalecides as the emulsions made with potash-fish-oil soap, and are slightly cheaper.

Summer Sprays with Oil Emulsion, 1923 and 1924

FOLIAGE TESTS, SUMMERS OF 1923 AND 1924

Oil emulsions had, of necessity, been used for a number of years on citrus trees while in foliage. During the summer of 1922, they were used on apple foliage with little or no burning in experimental work by the Bureau of Entomology in the Bentonville, Arkansas, section, and in work done by this office near Olney, Illinois. During the summer of 1923, foliage injury tests were made at Carbondale with a number of different trees, shrubs, and other plants. Apple, cherry, grape, lilac, mulberry, maple, peony, peach, pear, potato, rose, tomato, and walnut were sprayed during June on clear hot dry days, the temperatures ranging from 89° to 91° F., with 2% strengths of (1) boiled fish-oil-soap emulsion, (2) the same with Bordeaux mixture, 4-4-50, and (3) cold-mixed Bordeaux oil emulsion, and the only seen injury to plants in these tests was severe burning of the foliage on potato and tomato, and a slight blackening of a few leaves on rose and maple.

On cooler cloudy and humid days, with temperatures ranging from 80° to 83°, the following were injured.

With boiled fish-oil-soap emulsion alone

Peach	Slight to defoliation
Pear	50% of leaves specked black
Tomato	15% of leaves partly blackened
Rose	40% of leaves slightly burned
Walnut	50% of leaves peppered with black dots
Maple	2% of leaves slightly blackened

With boiled fish-oil-soap emulsion in 4-4-50 Bordeaux

Peach ....Same as on p. 121
Pear ....1% of leaves slightly burned
Walnut ....Same as on p. 121
Maple ....25% of leaves injured

With cold-mixed Bordeaux-oil emulsion

Peach ....Same as on p. 121
Pear ....90% of leaves burned, 15% severely, 8% killed
Tomato ....10% of leaves partly blackened
Rose ....50% of leaves burned
Walnut ....Same as on p. 121
Maple ....10% of leaves burned severely

During the summer of 1924 the following sprays were confined to apple, cherry, grape, peach, plum, potato, and tomato.

Boiled fish-oil-soap emulsion alone, 1% and 2% with paraffin oil of 90-100 viscosity.

Boiled fish-oil-soap emulsion, plus Bordeaux 4-6-50.

Boiled fish-oil-soap emulsion, plus Bordeaux 4-6-50, and arsenate of lead 2-50.

All the above repeated, using a paraffin oil of 212-220 viscosity.

Calcium caseinate cold-mixed emulsion, 1% and 2% with oil of 90-100 viscosity.

Skim milk cold-mixed emulsion, 1% and 2% with oil of 90-100 viscosity.

The injury, listed according to plants sprayed, was as follows:

Apple, Cherry, Grape...No injury by any spray under any condition of weather.

Peach ....From 30% to 90% defoliation with boiled fish-oil-soap emulsion, 90 viscosity, paraffin oil at 2% strength with and without the addition of Bordeaux and arsenate of lead. This occurred both in hot dry weather, and in cooler, cloudy, humid weather. Only slight injury with 1%. Using 1% with oil of 212-220 viscosity, no defoliation was observed. No injury with the cold-mixed emulsions.

Plum ....No injury.

Potato ....2% strengths of everything except the milk emulsions, gave moderate to severe burning. 1% strengths produced very little burning, and usually none.

Tomato ....In most cases injured moderately to severely, both the leaves and fruit, by both 1% and 2% applications.

The apples used in these tests were Winesaps, in the nursery row.

Larger trees in the University orchard at Olney were sprayed with the regular orchard equipment in the summer of 1923 by the Horticultural Department of the University of Illinois. On apples receiving from one to three summer applications, very slight burning of the foliage was seen in all blocks, but nothing serious. Dr. B. A. Porter, using summer sprays on various varieties of apples at Vincennes, found injury serious only on Grimes Golden.

Scale Tests with Oil Emulsion, Summer of 1923

Three series of tests were made during the summer of 1923 with 2% strengths of (1) boiled-fish-oil-soap emulsion; (2) boiled fish-oil-soap emulsion, with Bordeaux; and (3) cold-mixed Bordeaux-oil emulsion. In these experiments, the leaves were all removed from the sprayed branches so that every scale could be hit; and reinfestation was prevented, as far as possible, by bands of tanglefoot around the bases of the branches. In the first two of these series, the percent of scale found alive upon examination ranged from .2% to 2.5%. In an adjoining orchard which was being sprayed with a 3% strength of oil emulsion during the time of one of the tests, 16.8% of the scale was found alive, showing the effect of the foliage in preventing thorough application, and indicating that under orchard conditions, summer applications would not be very effective. The third series of tests gave 15% of the San Jose scale alive, even where the foliage was removed so that every scale was hit.

Experiments in Ed Kelley Orchard, Anna, Illinois, Winter of 1924-25

During the winter of 1924-25, a series of tests was run with the object of comparing the efficiency of light and heavy oils when used in boiled and cold-mixed emulsions. All applications were made with the hand sprayer, as previously described. The fall sprays were applied December 1-9, and examined from six to eight weeks later for live scale. The spring application was made February 7, and examined six weeks later. Table XI gives the results of these sprays.

Table XI

Treatment (Oil emulsions, all at 2% strength)	Scale examined	Live scale	Percent alive
Paraffin oil, 90-100 viscosity .42% volatility			
Boiled fish-oil-soap emulsion	1500	7	.4
Boiled corn-oil-soap emulsion	1500	3	.2
Cold-mixed (with Bordeaux) emulsion..	1500	2	.1
" (" Kayso) " ..	1500	14	.9
" (" egg) " ..	1000	33	3.3
Paraffin oil, 100 viscosity .33% volatility			
Boiled fish-oil-soap emulsion	1500	8	.5
Cold-mixed emulsion (calcium caseinate)	1500	1	.06
Paraffin oil, 212 viscosity 1.35% volatility			
Boiled fish-oil-soap emulsion	1500	1	.06
Cold-mixed (with Bordeaux) emulsion..	1000	0	0
Boiled corn-oil-soap emulsion	1500	1	.06
Check, January 16......................	1000	293	29.3
Paraffin oil, 192 viscosity .12% volatility			
Boiled fish-oil-soap emulsion	1500	2	.1
Boiled corn-oil-soap emulsion............	1500	0	0
Cold-mixed (with Bordeaux) emulsion..	1500	2	.1
" (" calcium caseinate) emulsion	1500	2	.1
Cold-mixed (with egg) emulsion........	1500	60	4.0
Check, February 7......................	1500	195	13.0
Check, March 18........................	1628	170	10.0
Free-mulsion 1 to 10 (Sherwin-Williams Co.)	1000	1	.1

This table would seem to indicate that there is no difference in effectivness between oils within the range of those used in these experiments. Vegetable-oil soap-emulsions in these tests show as well as those made from fish-oil soap. Cold-mixed emulsions excepting the egg emulsion appear to be as effective as the boiled emulsions. Efforts to make an egg emulsion that would mix well and would stand up over twenty-four hours were unsuccessful with the waters available.

Sherwin-Williams Free-mulsion, while it gave a satisfactory "kill", showed a considerable amount of free oil.

The Effect of Cold Winters on San Jose Scale and Scale Sprays

An examination of the foregoing tables will show considerable variation from year to year in winter mortality. The counts of live scale for the four years on untreated branches were as follows:

Year	Percent alive
1921-22	50.4 (March)
1922-23	65.9 (April)
1923-24	41.4 (March)
1924-25	29.3 (January) 13.0 (February) 10.0 (March)

It would seem entirely plausible that with the weakening effect of a cold winter on scale, the sprays would be more effective. The tables presented here seem to indicate that this is true. In the fall tests in 1923, given in Table VIII, the percent of live scale runs higher than in the spring tests (1924) given in the same table.

During this season we had a rather unusual experience in making scale counts. Previously, after applying sprays, a month had been found long enough to wait for the drying up of the scales that had been killed. Following the fall applications of this year, however, there was a period of abnormally cold weather, and on starting our counts after the usual interval, the oil-sprayed branches showed from 22 to 36 percent of the scale apparently alive. After another four weeks, branches with the same treatment showed only 1.3% to 1.8% live scale, indicating that the scales had been kept in cold storage, as it were, the continuous cold preventing their drying sufficiently to show any discoloration. The winter of 1924-25 was the most severe on the San Jose scale of any winter since 1917-18, and the record of only 10% live scale on the check branches in southern Illinois in March is remarkably low. The effect of this winter-killing is indicated by the very small percent of live scale shown in Table XI for that year, in which none of the treatments, with the exception of two very poor emulsions, gave less than 99% dead scale.

Summary and Recommendations

This report gives the results of four years experiments on the control of San Jose scale at various points in southern Illinois.

The superiority of oil sprays over lime sulfur was demonstrated, 11% of the scale remaining alive after being hit with lime sulfur, as compared with less than 2% with most of the oil sprays.

Boiled emulsion was as effective as the various miscible oils used.

Cold-mixed oil emulsions were about as effective as the boiled emulsions, but somewhat more unstable.

The most reliable type of homemade emulsions are the boiled soap-emulsions.

Vegetable-oil soap was as effective in making the boiled emulsions as fish-oil soap.

Emulsions made from oils with viscosities below 80, have not shown uniformly good kill of scale. There were apparently no differences in effectivness on San Jose scale in emulsions made from oils of 90 to 220 viscosity.

Tests with boiled potash-fish-oil-soap emulsions in summer showed very little injury to apple foliage, considerable injury to peach, and to a few other plants under some conditions. Due to the difficulty in reaching the scale when the trees are in foliage, summer sprays are not recommended except in case of very severe scale infestation.

Where oil emulsions were properly mixed and applied, no injury to trees has resulted.

On the basis of these experiments and observations, the following recommendations are made:

1. Oil emulsion is recommended as a cheap and effective spray for the control of San Jose scale. The formula for the stock emulsion found best in our experimental work is as follows:

Oil	1 gallon
Potash-fish-oil-soap	1 to 2 pounds
Water	¼ gallon

Heat to boiling, and pump twice at a pressure of 75 pounds, or more. The strength recommended is 2.4% (3 gallons in 100), or, in case of severe and increasing infestation, 4 gallons in 100 gallons of water. The best oil to use, judging by our experiments and those of investigators in Indiana and Arkansas, is a lubricating oil coming within the following limits:

Specific gravity..	.87 to .93 at 20° C.
Volatility.......	Not above 2% at 110° C. for 4 hours.
Viscosity.......	90 to 250 seconds (Saybolt test) at 100° F.

2. If cold-mixed emulsions are used, they may be made according to the following formulae:—

Bordeaux, Cold-mixed

Pump together equal parts of oil and 4-4-50 Bordeaux mixture, sending the material at least three times through the pump. For a 2% strength, dilute four gallons in one hundred.

Calcium Caseinate, Cold-mixed

Pump together two gallons of oil and one gallon of water in which is dissolved four ounces of calcium caseinate. For a 2% strength, use three gallons in one hundred.