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# BULLETIN

Article 1

# The Effect of Petroleum-oil Sprays on Insects and Plants

M. D. FARRAR



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#### FOREWORD

IN 1919 the ILLINOIS NATURAL HISTORY SURVEY began an investigation of oil sprays for the control of insects. In 1925 the Crop Protection Institute established a research fellowship on oil sprays with the NATURAL HISTORY SURVEY. This fellowship was supported by funds from the Standard Oil Co. of Indiana. This paper brings together data from the eight years of investigation 1925-33, and includes the data that appear to illustrate progress in the knowledge of oil sprays.

#### Types of materials and tests

The experiments have included tests on about 310 oil or oil-emulsion formulae, of which about 16 per cent were soluble oils and 84 per cent stock emulsions. In addition to these, various chemicals were included in many of the formulae. For purposes of discussion and comparison of the various emulsions the information is grouped under general headings referring to the specific insects and plants used in the experiments.

A very limited number of emulsion formulae were tested under field conditions for more than one season. The greater portion were tested under field conditions for only one year. Laboratory studies were made upon all of the formulae to determine their physical properties and toxicity to bean foliage. A limited number of tests were performed on insects to evaluate the insecticidal properties of each emulsion. Those formulae which looked promising as a result of information gained in the laboratory were tested under field conditions the following season.

#### Present knowledge of oil sprays

As tangible results of the investigation, a miscible oil and a white-oil stock emulsion have been developed. In the investigation of these products and the study of their related formulae, a fund of constructive information has been compiled that has been of great assistance in the interpretation of results. This information has added greatly to our understanding of some of the factors affecting the reactions of petroleum oils on insects and plants.

Indicative of present knowledge concerning oil sprays are the following:

The use of oil emulsions for codling moth control in late-brood sprays, particularly when mixed with nicotine sulfate, is very promising.

Sprays of oil emulsions for late-brood codling moth larvae have given excellent results in the reduction of lead arsenate residues.

The larvicidal limits of oil emulsions are fairly well established.

The use of oil emulsions with lead arsenate sprays shows promise in the control of codling moth larvae.

The use of petroleum oil as a carrier for plant poisons is recognized.

Oil emulsions are giving a control of scale insects superior to all other sprays for scale.

### Acknowledgments

The author wishes to thank the Crop Protection Institute, its committee, and chairman Professor W. P. Flint for their guidance in the development of this project; the Standard Oil Co. of Indiana for the funds used in the project and for experimental samples prepared and furnished through its entomological staff and chemists; the ILLINOIS NATURAL HISTORY SURVEY, Urbana, for permission to use the equipment employed in the investigation; members of the staffs of the University of Illinois and lowa State College for experimental material and assistance in the carrying on of the investigation, taking of data and evaluation of results; and Dr. L. L. English, former investigator of this project, who has furnished much time and effort in the collection and evaluation of data.

# Definition of Terms

Concentration of emulsions refers to measurements by volume.

*Lead arsenate* is acid lead arsenate, the powdered commercial product marketed for insecticidal purposes.

Miscible or soluble oils are clear oil-like emulsions containing very little water. In these a soap emulsifier is dissolved into the oil. Upon the addition of water the miscible oils disperse to form a milky white emulsion of very small oil-droplet size. The commercial oils *Dendrol* and *Sunoco* are examples.

Paraffin oil or unsaturated oil is not as highly refined as technical white oil (which see) and contains unsaturated hydrocarbons in varying amounts depending on the degree of refinement. The degree of saturation is expressed as unsulfonated residue and is determined by a standard procedure known as the Whiting method.

Quick-breaking emulsions are made in the spray tank with or without addition of some emulsifying agent. The emulsion depends upon the agitation to break up the oil into droplets. The oil droplets are very large and loosely emulsified, so that the oil phase may separate either on standing or soon after application. For certain insects such as leaf rollers and casebearers, they are superior to the more stable emulsions.

Stock emulsions are oil emulsions of a pastelike consistency. They generally contain water as a separate phase. Boiled fish-oil soap emulsion is an example of this type.

*Technical white oil* is an oil from which the preponderance of unsaturated hydrocarbons has been removed, but it is not of medicinal grade. A technical white oil, or saturated oil, is practically inert chemically.

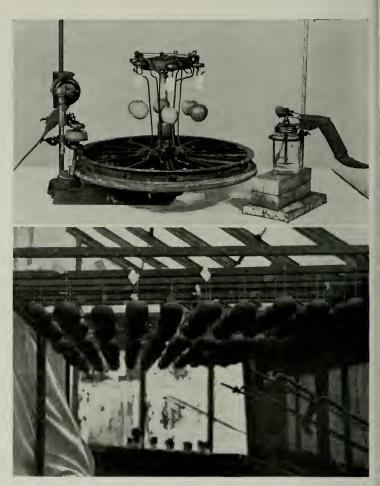
Viscosity, as measured by the Saybolt test, is the resistance to flow of a given volume of oil through a given orifice at  $100^{\circ}$  F, and is expressed in seconds.

Urbana January 1, 1936

M. D. F.

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TESTING CODLING MOTH SPRAYS.—Top, Apparatus devised by Farrar and McGovran to secure uniform deposit of spray on apples. At the left is shown a 110-volt, 60-cycle (1800 rpm) one-sixth horsepower stir motor with reduction gear. The stir rod buffers against a No. 13 rubbe stopper supported between two roller-skate wheels as bearings. On the lower end of the shaft carrying the stopper is a heavy-walled rubber tube extension that buffers against the rim of an auto wheel covered with friction tape to reduce slipping. The wheel is part of a whirligig that rotates a group of six experimental apples while revolving each apple separately. Moving at 8 rpm, the wheel turns six skate wheels, supporting an apple apiece, at 27 rpm against a fixed 6-inch buffer wheel. At right is a sprayer constructed for use with compressed air, spraying with 25 pounds pressure. The spray mixture in the jar is stirred by an agitator driven by a compressedair turbine. Bottom, Rack of test apples used in laboratory tests of codling moth sprays. VOL. 21, ART. 1

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# The Effect of Petroleum-oil Sprays on Insects and Plants

# M. D. FARRAR

#### PROPERTIES OF OIL EMULSIONS

The ratio between saturated and unsaturated hydrocarbons in an oil determines to a large extent the use of that oil as an insecticide on either dormant or nondormant plants. The viscosity, volatility and degree of dispersion are important properties which affect both plant tolerance and insecticidal efficiency. Spreading properties of unit volumes of spray are nearly identical as to coverage and time. Preparation of emilisions for these experiments was by the Whiting method.

ATURATION, or the ratio between saturated and unsaturated hydrocarbons, has sharply divided the usage of oil sprays into two distinct fields. The works of Grav & deOng (1926), deOng. Knight & Chamberlin (1927), English (1928) and others show that the oils containing the larger quantities of unsaturated hydrocarbons are the more likely to cause plant injury under comparable conditions. Extensive experiments indicate that the less highly refined oils are safe to apply on dormant trees if they are properly emulsified. Because of the lower cost of this grade of oil its use in dormant sprays has become general.

#### Plant Tolerance Limits Viscosity

The viscosity of oils used in tree sprays must be kept within the limits of plant tolerance. Oils of less than 40–50 seconds of viscosity are very difficult to combine into a stable emulsion. Although somewhat safer to use on plants, these light-viscosity oils disappear too rapidly to have a good insecticidal value. Knight, Chamberlin & Samuels (1929) feel that a 60-second viscosity oil represents about the upper limit of plant tolerance and that oils of higher viscosities must be used with caution if serious ultimate injury is to be avoided. Most authors agree that the lighter oils are safer on plants and generally recommend the lightest oil that will give proper control of the insect involved. Temperature under which an oil is used in the field should determine the proper viscosity, according to deOng (1931), although Green (1927) did not find viscosity a factor for toxicity in dormant spraying. Oils of 60–125 seconds viscosity are now being used in the manufacture of the greater part of the commercial brands of oil emulsions.

#### Less Stable Emulsions Most Toxic

Volatility is a property that some authors have considered important in petroleum oils for use in tree sprays. Although perhaps of some importance its significance has not been fully accepted by all workers. The property of volatility is by no means identical with that of viscosity, nevertheless it is closely associated with viscosity. According to the findings of Knight, Chamberlin & Samuels (1929), the effect of volatility "due to the enclosure of the oil in the intercellular spaces . . . is unquestionably negligible in comparison with translocation." In this paper the author has not considered volatility an independent property of the oils used in the emulsions tested.

The emulsifier plays a very important rôle in the performance of oil emulsions, as is shown by the work of deOng & Knight (1925), deOng (1926), deOng, Knight & Chamberlin (1927) and others. Oil emulsions that incorporate excessive amounts of emulsifier are very easily emulsified but are not as efficient for the control of scale as are less stable emulsions. English (1928), working with aphids, San Jose scale and oyster-shell scale, clearly demonstrated that high mortality was associated with emulsions exhibiting the quicker breaking properties.

#### Spray Methods Regulate Coverage

The total volume of any spray material required to cover a certain block of fruit applied. This fact is in accordance with the findings of Swingle & Snapp (1931), and it is brought out also in the records of commercial orchards which use large amounts of materials for each spray. In well-equipped orchards the variations can usually be accounted for by adverse weather conditions.

Additional proof that the volume of spray material necessary to cover a unit number of trees is relatively constant was given in a series of tests that made use of commercial oil 17<sup>1</sup> at 2 per cent concentra-

Type of Oil and Number	SPECIFIC GRAVITY AT 60° F grams per cc	Flash Point °F	Fire Point °F	Viscosity seconds al 100° F	Percentage of Evaporation 8 hours at 212° F	Percentage of Unsulfonated Residue
Technical white oil 32	. 801	174	206	32	35.1	100.
Technical white oil 83	. 857	345	395	83		99.
Technical white oil 95		366	410	95-100	8.6	99.5
Technical white oil 190	1.878	375	430	190	. 2	98.
Paraffin oil 32	.82	164	196	32	54.3	92.
Paraffin oil 83	. 883	335	385	83	1.7	91.
Paraffin oil 104	. 889	355	405	104	1.2	93.

Table 1.-Properties of experimental oils.\*

\* Technical white oils of viscosities other than those listed correspond to technical white oll 83 in unsuffonated residue. Parafiln oils correspond to parafiln oils 83 and 104 in unsuffonated residues. The data given above were suimitted from the laboratory of the Standard Oil Co. of

trees has not been a settled question in the minds of many horticulturists. They have been led to believe that there are wide differences in the spreading and covering properties of certain spray materials. Salesmen of insecticides often claim that this or that material will give a greater spread, and so reduce the actual cost of a spray to cover an orchard.

There are certain factors involved in the practice of spraying that influence the amount of material required to cover a certain number of trees. Some of these factors are the type of equipment, number and size of nozzles, the pressure, visibility, wind direction, type of workmen doing the work, and the care with which the workmen attempt to wet the trees thoroughly. If the workmen are given a standard set of equipment and have uniform weather conditions, the amount of material that they will use will vary but slightly regardless of the physical properties of the spray material Indiana, Whiting, Unsulfonated residue (degree of saturation) values given in this paper were determined by the Whiting method. These values are higher than those given by some other methods.

tion, commercial oil 2' at 6.5 per cent, "Illinois formulae" boiled fish-oil soap emulsion<sup>1</sup> at 3 per cent, and liquid lime sulfur solution at 10 per cent. In four tests 200 gallons of each material covered hetween 13.6 and 14.8 minutes for applicabetween 13.6 and 14.8 minutes for application. The test indicates that the volume of spray material necessary to cover a unit number of trees is relatively constant, irrespective of the nature of the spray material applied.

#### Stability Determines Mortality

By combining the proper grades of oils and emulsifiers in certain proportions it is possible to produce an emulsion that is stable under ordinary conditions of storage and handling and that will give a high kill of insects when it has been properly diluted. Addition of various chemicals to either the

<sup>4</sup>Laboratory designations of various experimental oils. Many others follow. oil, water or emulsifier phases of these emulsions often changed the degree of dispersion. The difficulty of classification made impractical the consideration of particle size in the experimental white-oil emulsions. It is generally recognized that the less dispersed oil emulsions separate faster and are more toxic to insects.

Most miscible oils and many of the stock emulsions were prepared by a standard technique worked out in the Standard Oil laboratories at Whiting, Ind. Stock emulsions incorporating inert emulsifiers were emulsified by colloid mills, capable of producing emulsions of uniform dispersion of oil droplets in the technical white-oil emulsions. (For analysis of oils used see Table  $1.)^2$ 

# EFFECT OF PETROLEUM OILS ON PLANTS

The physical properties of oil emulsions cannot be associated with some forms of plant injury that follow applications of oil sprays. Such plant disturbances are associated with general vigor, stage of growth, soil moisture and food relations.

Petroleum-oil emulsions formed with soap are generally toxic to foliage irrespective of the saturation or viscosity of the petroleum oil.

The amount of emulsifier present in an emulsion predetermines to some extent the physical nature of the emulsion and to a greater extent its insecticidal value. The emulsions which possess the larger oil droplets are the more toxic to insects and lass toxic to plants.

The viscosity of a petroleum oil determines to a limited extent its safety to growing plants. The lighter oils, 50 seconds of viscosity or less, are less toxic to plants but are not efficient insecticides. Oils of greater than 100 seconds of viscosity tend to create physiological disturhances within the growing plant.

A laboratory study of the larvicidal efficiency of emulsions indicates only a limited possibility of increasing the mortality of codling moth larvae by changes in the physical properties of an emulsion.

The technical white-oil emulsions are relatively safe on foliage if they possess the proper degree of saturation, viscosity, oil-droplet size, and an inert emulsifier.

Previous to 1900 many cases of plant injury were reported<sup>3</sup> from the spraying of plants with petroleum oils. It is not surprising to find injury occurring early in the

<sup>2</sup>Manufacturers of commercial oils used in these tests: Sun Oil Co., B, G. Pratt & Co., Sherwin-Williams Co., California Spray-Chemical Co., Shell Oil Co., Schaeffer Bros, & Powell, and Standard Oil Co. of Indiana.

<sup>4</sup>According to Lodeman (1896), Goeze was the inst to recommend the use of oils on plants. In 1762, J. A. E. Goeze worke, "performing many are must be taken in their use, since they are up the plant of the plant system as the first (1800) to call attention to the use (train (whale) oil against coccus or scale insects on plants.

The first record of petroleum oil as an insecticide in America is a recommendation in 1865 development of oil sprays. About 1900 the use of petroleum oils became more general and information became available as to the more suitable oils and emulsion types that could be safely used on growing plants. During this same period there was rapid development in the machinery necessary for the proper preparation and application of emulsions.

#### Oils the Modern Insecticide

Entomologists now recognize that oil sprays serve as very important weapons for the control of many dangerous pests, such as scales, mites, aphids, leaf rollers, casebearers, codling moth and oriental fruit moth. Petroleum-oil emulsions are now available that are adaptable for definite insecticidal purposes with a minimum of danger to the host plant.

Volck (1903) published the first comprehensive paper on the effect of petroleum oil when used on plants as an insecticide. In this paper he concluded, "by far the greatest cause of injury to vegetable tissue is brought about by the penetration of the oil applied, into the interior of the plant." He also found the lighter oils less injurious than heavy oils and that the degree of injury depended upon certain physical factors. Some of the factors listed by Volck are the condition of the plant, type of oil, amount of spray used, whether the spray is applied to the upper or lower leaf surface. and temperature and humidity at the time of spraying. He demonstrated a physical injury from oil that he attributed to "insulation" or sealing over of the parts of the plant; this insulation interfered with both respiration and transpiration of the plant. The chemical effects caused by the petroleum oils used by Volck can now be largely corrected by employing the highly refined white oils, which contain a minimum of unsaturated hydrocarbons.

to use kerosene undiluted. The oil was to be applied by means of a feather on citrus trees for the control of citrus scale. Kerosene was first employed as a spray in 1868 by Henry Bird, Newark, N. J. He used a mixture of kerosene, scap and water for the control of the currant worm. It was not generally used as an emulsion until recommended by A. J. Cook in 1878.

soap and water for the control of the curtant worm. It was not generally used as an emulsion until recommended by A. J. Cook in 1878. Hy 1882, H. O. Hubbard had developed a satisfactory formula for kerosene emulsion, using soap as an emulsifier. Kerosene and other light petroleum oils were used quite generally either as emulsions or as mechanical mixtures previous to 1960, followed by distillate-water meclanical institures in 1902 and by miscible oils in 1964, according to Mason (1928) and Essig (1953).

#### Tolerances Variable, Specific

Volck (1903) found that the important injuries to citrus leaves by oil are general and not local. This observation has been substantiated in more recent work by the determination of rather definite plant tolerances to oil sprays. No rules can be established relative to the tolerance of plant species to oils, but we do know within limits the amount of certain petroleum oils that can be applied with relative safety to the more important trees and plants. It is now well established that a dormant tree will withstand higher dosages of less refined oils than the growing tree. The type of oil that may be used with safety to the plant will depend to a great extent on the season of the year when it is applied.

The accumulative effect of oil sprays has been mentioned by Volck (1903), Yothers (1913) and others. It is pronounced during the active growth of the plant and becomes less evident as the plant approaches dormancy, at which time the effect apparently disappears.

Burroughs (1924) stated, "from theoretical considerations and from observations and experiments, it is quite certain that a covering of oil on the surface of the plant organ may affect the physiological processes of that organ. It is probable that under some conditions the effect of the oil may be infinitesimal."

#### Normal Metabolism Disturbed

Herbert (1924), working with prunes, found the greatest plant stimulation associated with the heavy types of miscible oils. He correlated the degree of stimulation with the moisture and food relations of the soil and the season of the year when the trees were sprayed with the oil. deOng (1926) also found a correlation between the season of spraving and the effect on French prunes. In his experiments, extending over two years, sprays applied in November before the trees were fully dormant slightly retarded development, December sprays had little effect, January and early February sprays stimulated, while sprays applied between February 15 and March 15 retarded development.

Knight, Chamberlin & Samuels (1929) determined histologically the distribution of the petroleum oil in the plant tissue following an oil spray. They found that the saturated white oils were absorbed by the tissues and not volatilized, as considered by some authors. The translocation of the absorbed oil was traced from the leaf surface to its final deposition in the large storage cells of the pith and the old wood fiber of the xylem. During the period of oil penetration and initial translocation, transpiration was sharply decreased and respiration enormously increased. Knight and his coworkers attributed the metabolic disturbances to physical rather than chemical handicaps imposed by the intrusion of the saturated petroleum oils into the plant tissue. Ginsburg (1929) found that apple foliage sprayed four times between July 12 and August 24 increased in chlorophyll content from 28 to 47 per cent in the two apple varieties tested. This secondary effect of oil sprays, that of intensifying the green appearance of the oil-sprayed foliage, has been reported by numerous workers with oil emulsions. Working with excised twigs, Kelley (1930a) found that "Saturation of the heavier oils, comparable to those used in commercial spraying, was not important in either the dormant or delayed-dormant periods. It was relatively unimportant in foliage applications."

#### Measure of Injury Unsettled

The works of Volck (1903), Yothers (1913), deOng (1926, 1928b, 1931), English (1928), Knight, Chamberlin & Samuels (1929), Kelley (1926, 1930a, b), Ginsburg (1929, 1931a, b) and others demonstrate that oils applied to foliage produce certain physiological effects on the trees. As yet no one has advanced a good criterion for classifying these physiological effects as injury resulting from an oil spray. Woodworth (1930) proposes certain terms which may be useful in classifying these effects. It is true that certain oils will injure or burn more than other oils under a comparable set of conditions, but it has never been possible to show that a certain property of an oil, if present in an emulsion, will result in foliage injury. In this paper, injury is considered as visible changes in the normal leaf tissue following the application of an oil spray. This type of injury usually is evident on the margins or tips of the terminal and adjoining leaves.

A part of the difficulty in dealing with emulsions lies in the fact that every emulsion prepared is an individual colloidal system possessing properties distinctive from every other emulsion. Although this condition is literally true, the emulsions tested in this work were approximately the same since they were prepared in the same way and from very similar materials. It was found possible to duplicate quite closely formulae that had been used in previous tests.

#### Annual Dormant Sprays Safe

Consistent annual spraying of deciduous fruit trees in the dormant stage with good oil emulsions has produced no apparent injury to them, according to Yothers (1918), Burroughs (1924), Newcomer & Yothers (1927) and Swingle & Snapp (1931). This fact is well established even though many individual cases of injury

Table 2.—Relation between the properties of emulsions and their relative safety when applied on apple foliage at a concentration of 2 per cent.

İTEM	Type of Emul- sion	Oil	Сомро Per- centage Unsulfo- nated Residue	SITION Emul- sifier	Addi- tional Material	NO. Form- ulae Tested	Total No. Tests	Cases of No Injury	Cases of Slight Injury	Cases Of MODER- ATE INJURY	Cases Of Severe Injury
1	Stock	Technical white oil	99.	Inert	None	7	75	69	3	2	1
2	Stock	Technical white oil	99.	Inert	Misc."	8	19	19			
.3	Stock	Technical white oil	99 _	Inert	Nicotine	5	17	17			
4	Stock	Technical white oil	99.	Inert	Derris	3	9	9			
5	Stock	Technical white oil	99.	lnert	Pyrethrum	8	41	37		2	2
6	Stock	Technical white oil	99	Inert	Copper	7	17	9	6	1	1
7	Stock	Technical white oil	99.	Inert	Sulfur	10	43	12	12	9	10
8	Stock	Technical white oil	99.	lnert	So <b>dium</b> fluosilicat	e 2	5	2	2		1
9	Stock	Technical white oil	99.	Soap	None	2	4	4			
10	Miscible	Technical white oil	99.	Soap	None	7	22	19	1		2
11	Commercial miscible	Paraffin	90.+	Soap	None	4	20	5	4	3	8
12	Experimen- tal mis- cible	Paraffin	91.	Soap	None	8	53	9	16	19	9
13	Experimen- tal stocks	Paraffin	91.	Soap	None	4	14	5	3	1	5
14				Soap	None	5	22	8	4	5	4
15	Water contr	rol				0	0	0	0	0	0

\* Petroleum compounds with nitrogen or oxygen, furfuramide, chinchona alkaloids.

In addition, wide variations in individual trees, localities, weather conditions, etc., exist in most orchard experiments. These variations have led to the publication of a vast amount of data covering experiments with the use of oil emulsions. In many of the experiments the results cannot be duplicated by the workers themselves. This has filled the literature with numerous discussions of individual experiments most of which prove little one way or the other relative to the toxicity of oils to plants. have been reported during certain experiments, particularly with nondormant trees. Vigorous trees are very tolerant to oil sprays and are seldom injured by them if the emulsions are properly prepared and applied. Where oils or other spray materials are applied under abnormal conditions, numerous foliage reactions may set in, according to Overley & Spuler (1928) and Dutton (1932). The accumulative effect of petroleum oils applied to foliage has been observed by Yothers (1913) and others. Overholser & Overley (1930) found that more than three applications of medium to heavy-viscosity oils applied to apple trees carrying a heavy crop resulted in a decrease in the size of fruit and a poor set of buds and fruit the following year. These effects usually follow the too-frequent application of emulsions to foliage early in the growth of new tissue. Delay of such sprays until growth has about stopped and the tissues hardened greatly reduces the abnormal physiological changes. such as destruction of tissue, yellowing, premature leaf-drop, and change in chlorophyll content.

#### Unsaturated Oils Highly Toxic

The lower-viscosity oils have from the first been considered less toxic to foliage than the heavier oils. The usual explanation is based on the fact that the heavier oils are less volatile and persist on the plant over a longer period than do the lighter oils. Detection by Gray and deOng in 1915, (Gray & deOng 1926), that the unsaturated hydrocarbons present in untreated oils are largely responsible for the injury to plants has contributed more toward the development of emulsions for use on growing plants than any other discovery.

Associated with plant injury are additional, less important factors: the use of inert emulsifiers, the limits of the size of the oil droplets, and the effect of oil-droplet size on quick breaking. All of these properties have a bearing on the emulsions and predetermine to a limited extent the physical properties of an emulsion suitable for tree-spray purposes.

The stage of growth in the plant, weather conditions at the time and following spray application, time and method of application, and a host of other factors so influence the results from application of an oil spray that an emulsion incorporating all the desirable properties of a summer oil will not insure uniform performance of an insecticide without injury to the host plant.

#### Saturated Oils Generally Safe

With these conditions in mind one is in position to attempt to judge foliage injury following the use of an oil spray. Table 2 presents data summarizing most of the results obtained from the use of oil sprays on foliage over a five-year period. Particu-

lar attention should be given to item 1, covering the work with a summer oil that possessed all the physical properties deemed necessary for safety to foliage. This oil emulsion in 75 field tests gave one case of severe injury, two of moderate, three of slight injury and 69 cases where no injury was observed. When given adverse conditions, this oil emulsion, although relatively safe, produced severe injury or burn to apple foliage. A very similar result was obtained with the emulsions under item 5. If given a sufficient number of trials under varying conditions, there is no doubt that the other emulsions listed in the table would have shown a similar number of cases where injury occurred.

Emulsions listed in items 3 and 4 are similar to those in item 1 with the exception that extracts of tobacco or derris have been added to the emulsions for the purpose of increasing their insecticidal efficiency. The incorporation of these materials into an inert white-oil emulsion did not materially increase foliage injury.

The injury shown in items 6, 7 and 8 was probably caused by the copper, sulfur or sodium fluosilicate mixed with the whiteoil emulsions. The formulae containing copper injured in 51 per cent of the cases, and those with sulfur in 70 per cent of the cases, where they were tested.

Technical white oils, item 9, made up as a stock emulsion with a soap emulsifier did not injure in the limited number of trials given. The same oils combined as a miscible oil injured in 14 per cent of the tests.

#### Smaller Droplets More Injurious

Petroleum oils containing as much as 9 per cent of unsaturated hydrocarbons will cause injury in most cases if they are combined with soap emulsifiers. The soap emulsifier, item 14, is no doubt responsible for some of the injury obtained with emulsions containing soap. The miscible or soluble oils, items 11 and 12, give relatively more injury than do the stock emulsions, item 13. The smaller oil-droplet size and greater stability of the miscible oils are responsible to some extent for the injury that follows the use of miscible-oil emulsions. It is evident that, other factors being equal, the emulsions with the smaller oil droplets are the more likely to cause injury to foliage.

In Table 2 a large number of cases are reported where no injury was observed with any of the sprays listed in items 1 to 14. These occurrences are significant because they explain to some extent the confusion resulting from reports of injury occurring in the individual experiments of all workers.

Some of the recent work of dcOng. (1926), dcOng, Knight & Chamberlin (1927), and Smith (1929, 1930 and 1931) has again raised the problem of applying oils to plants without the aid of an emulstability of such an emulsion was almost negative and the oil droplets so irregular and unstable that they were difficult to measure. The oil drops were very large and coalesced rapidly. Neither of these oils injured foliage at 1½ per cent concentration but the less saturated oil spotted the fruit of apple.

#### Emulsifier Controls Droplet Size

In items 3, 4 and 5 a technical white oil was used that contained a product known as "butylacetyl resinoleate," which has been

Table 3.—Relation between emulsifier, droplet size, saturation and foliage	e injury to apple.
Sprays applied to foliage at 1 <sup>1</sup> <sub>2</sub> per cent concentration of oil in the water phase.	

<b>I</b> TEM	Oil Formula			MPOSITI			Oil- droplet Size	INJURY TO FOLIAGE	Injury to Fruit
		Oil	Viscosity seconds at 100° F	Saturation percentage	Emulsifier	Stability	microns		
1	Technical white oil	Technical white oil	83	99.	None	None	4-24	None	None
2	Paraffin oil	Paraffin oil	104	91.	None	None	4 - 24	None	Trace
3	13550	Technical wbite oil	83	99.	2% Bar*	Very quick breaking	1-12	Trace	None
4	13720	Technical white oil	83	99.	4% Bar	Very quick breaking	1 12	Light	None
5	13740	Technical white oil	8.3	99	6% Bar	Very quick breaking	1 12	Moderate	None
6	Stock 200	Technical white oil	83	99.	Gum	Quick breaking	2-6	None	None
7	Soluble oil 8800	Technical white oil	60	99.	Soap 15	Quick breaking	18	Severe	Severe
8	Soluble oil 8790	Technical white oil	83	99.	Soap 15	Quick breaking	1-8	Severe	Severe
9	Soluble oil 17	Paraffin	83	91.	Spap 15	Quick breaking	1-6	Severe	Severe
10	Control							None	None

A material which, when added to an oil, permits dispersion of the oil into the water by agitation.

sifier or with only sufficient emulsifier to permit dispersion by agitation in the spray mixture. Such a method of application has several advantages over emulsions containing emulsifiers, as expressed by deOng & Knight (1925). It has been demonstrated that it is possible with the proper machinery to use oils without emulsifiers, but the construction of spray machinery now in operation will not warrant the general adoption of this method of applying oils to deciduous trees.

Some further evidence on the relation of saturation, the amount of emulsifier present in an oil, and oil-droplet size is shown in Table 3. In items 1 and 2 a technical white oil and a less saturated oil were applied without the aid of an emulsifier. The shown to impart to an oil the property of dispersion in water with limited agitation. This material was added to the oil at 2, 4 and 6 per cent respectively, to study the effect of this emulsifier on droplet size and foliage injury. The range in oil-droplet size does not present a true picture of the condition existing in the dilute sprays of these oils. Oil 13740, item 5, contains many more small oil droplets than oil 13550, item 3. With each increase of this emulsifier there is a corresponding reduction in the average oil-droplet size. This reduction in oil-droplet size produced increased injury to foliage, as is shown in items 3 to 5. None of these emulsions caused injury to the fruit.

Stock emulsion 200 was used in this

experiment as a comparison with the other emulsions. It contains a technical white oil with an inert emulsifier, has quick-breaking properties and is relatively safe on apple foliage.

Soluble oils 8800 and 8790 contain the same ratio of emulsifier as soluble oil 17 but are prepared with 60 and 83-second-viscosity white oils respectively. In items 7, 8 and 9 there was no marked difference between the technical white oils and the less saturated oil in their injury either to fruit or foliage when they were emulsified with certain soaps.

Considering the graduation in the oildroplet size and stability between items 1, 3, 5 and 8, all containing technical white oils, it is evident that the emulsions with the smaller oil droplets and greater stability are the more injurious to apple foliage. Similar differences are exhibited between items 2 and 9, both of which contain oils with a lower sulfonation test.

#### INSECTICIDE TESTS WITH THE EMULSIONS

#### **Codling Moth**

#### Carpocapsa pomonella (Linn.)

White-oil emulsions are practical in the late-brood coding moth sprays in order to avoid arsenical residues on the fruit. The white-oil emulsions alone are not equivalent to lead arsenate in larvicidal efficiency hut when comhined with nicotine sulfate they are comparable to lead arsenate in the control of the late-brood coding moth larvae.

Addition to emulsions of extracts of plant poisons such as pyrethrum, derris or tohacco materially increases toxicity of the emulsions as contact insecticides. Of the plant poisons, nicotine alone was stable to exposure under field conditions. Toxicity to foliage was not influenced by the addition of plant extracts to the emulsions.

White-oil emulsions can be used as ovicides in codling moth control either alone or included in the lead arsenate sprays. The combined spray has excellent ovicidal and larvicidal properties.

#### FIELD TESTS IN CONTROL OF CODLING MOTH

The codling moth is by far the most destructive insect encountered in the production of apples. The annual abundance of codling moth and the difficulty of securing an adequate control by the use of poison sprays have given this pest a rôle as limiting apple production, particularly in the sections where apples are grown commercially. In seasons of abundance the latebrood larvae will destroy 10–40 per cent of the marketable fruit. This condition persists in orchards receiving six to 12 sprays of lead arsenate.

Arsenical sprays were introduced against codling moth in recommendations by Cook (1881) and have continued to be the most satisfactory method of control, provided a sufficient coating of spray is maintained on the fruit. The number of arsenical sprays applied has increased in many apple-growing sections and as a result the harvested fruit carries residues of arsenic in excess of the legal tolerance (See Table 4).

Table 4.—Analyses\* of harvested fruit for arsenical residue from plots treated in the latebrood codling moth sprays with 2 per cent white-oil emulsions or the recommended strengths of lead arsenate (2-4 pounds per 100 gallons) and hydrated lime (4-8 pounds per 100 gallons). The legal tolerance for 1932 was 0.1 grain of arsenic trioxide per pound of fruit.

YEAR		IBER OF	GRAINS OF ARSENIC TRIOXIDE PER POUND OF FRUIT					
	Oil	Lead Arsenate	Oil	Lead Arsenate				
1928	3	1	. 0046	.006				
1929	1	1	.006	.013				
1930	19	4	.0053	.025				
1931	1	2	.008	.0185				
1932	3	6	. 005	,036				
Totals Average	27	14						
RESIDUES			.0058	.0199				

\* By the U. S. Department of Agriculture, Food and Drug Administration, St. Louis, Mo.

This condition has forced either the development of more efficient arsenical sprays or the use of nonarsenical sprays that can be substituted as sprays for lead arsenate. The most promising development has been the use of summer-oil emulsions either as direct substitutes for lead arsenate sprays or their inclusion with the lead arsenate sprays. Regan & Davenport (1928) used an oil emulsion and lead arsenate mixture in their experiments for the first time in 1925, and reported results favorable to the use of oil emulsions added to the regular lead arsenate sprays. Spuler & Dean (1930) found that fish oil and certain other drving oils were as satisfactory as petroleum oils with lead arsenate sprays.

#### Addition of Petroleum Oils to Lead Arsenate Sprays

With the development of adequate washing machinery, orchardists are in position to take advantage of the insecticidal efficiency of oil emulsions with lead arsenate. Spuler & Dean (1930) state, "when oils are combined with lead arsenate the resultant spray has an ovicidal value equal to that of oil alone and a larvicidal value greater than that of lead arsenate alone." In their tests a three-fourths per cent actual oil gave 80–95 per cent kill of codling moth eggs. Newcomer & Yothers (1932) suggest use of "a medium oil, having a Saybolt test of 65–75 seconds and a high sulfonation test applied in a propor-



Fig. 1.—Five years' tests of sprays to control codling moth in Illinois.

tion slightly less than 1 per cent." It should not be added to more than three or four of the lead arsenate sprays and in those sprays that occur when the highest percentage of eggs is on the trees and fruit. This statement is in accord with the report of the Western Cooperative Oil Spray Project (1932), and the Washington experiment station recommendations for 1932.

#### White-oil Emulsions as a Substitute for Lead Arsenate

The development of highly saturated white oils emulsified with inert emulsifiers offered the first oil sprays that could be used on foliage during the growing season. The safety of emulsions of this type has been demonstrated by deOng (1926), English (1928), and Headlee, Ginsburg & Filmer (1930). Results of Headlee, Ginsburg & Filmer (1930), Flint (1930), and Newcomer & Yothers (1932) have shown that emulsions for the full season, without the use of lead arsenate, cannot be used to control codling moth. There are several reasons for this situation:

1. Oil emulsions are primarily contact sprays and cannot be applied sufficiently often to kill all hatched larvae without disturbing the physiological development of the fruit and tree.

2. The ovicidal effect of oils can be utilized only occasionally.

3. Summer-oil sprays cannot be used with the necessary fungicides early in the season.

4. Summer oils are too expensive when compared with lead arsenate sprays.

The larvicidal effect of an oil deposit is inferior to that given by lead arsenate, as is shown in the results of Flint & Farrar (1931), Lathrop & Sazama (1932) and Newcomer & Yothers (1932). Nevertheless, all field tests conducted in many states where oil and lead arsenate sprays have been compared favor the limited use of oil sprays in the control of late-brood codling moth larvae. In most cases the leadsprayed fruit has had fewer codling moth entrances than the oil-sprayed fruit; but there has not been sufficient difference in control to justify the use of lead arsenate in late sprays in orchards unequipped to wash the fruit. In this regard oil emulsions have served as a valuable aid to codling moth control during the period in which growers have been reorganizing their methods of harvesting and packing of fruit.

The results of Spuler & Dean (1930), Headlee, Ginsburg & Filmer (1930), Flint & Farrar (1931) and Talhert & Swartwout (1931) indicate that oil emulsions can be substituted for lead arsenate in the late-brood sprays. Oil sprays applied at this season do not produce the injurious effects on tree growth that are brought about when the same sprays are used earlier in the season. Two to four applications of 2 per cent oil emulsions have given protection in the late-summer sprays.

Spray recommendations for most sections suggest the direct substitution of oil sprays in place of the usual lead arsenate sprays. This has been done without regard for the most efficient use of oil sprays as ovicides on the codling moth eggs. Newcomer & Yothers (1932) found that from 80 to 97.6 per cent of the eggs failed to hatch if sprayed with a 2 per cent heavy-oil emulsion and that 64.6 per cent of the eggs did not hatch if laid within seven days after application of an oil spray. The ovicidal effect obtained from oil sprays will explain in many cases why the same oil spray has given variable results in succeeding years and in different orchards.

Since 1927, orchard tests have been conducted to compare the efficiency of oil-emulsion sprays with that of lead arsenate sprays. The schedule followed for dates of furnished the fruit used in scoring spray performance. Apple pickers gathered the fruit from the inside quarters of these trees. A 1000-apple sample was taken from this fruit to be graded in scoring the plot. The population of codling moth varied in the test orchards from light to extremely heavy, including in the five years of records representative orchards in the commercial applegrowing sections of the state.

A summary of five years of data is given in Table 5 and fig. 1. The relative control given by all of the sprays as compared with no spraying is striking, especially in the

Table 5.—Codling moth control in the orchard over a five-year period. Orchard tests were conducted in commercial orchards of western, central and southern Illinois. A test was a count made on 1000 apples taken from the four center trees of a test block.

Îtem	SPRAY TREATMENT FOR SECOND AND THIRD-BROOD LARVAE	TOTAL NO. FIELD TESTS					А		e Per it Ent	Cent g erêd	ıF	FIVE-YEAR AVERAGE No. Per Tests Cent	
		'27	'28	'29	'30	'31	'27	'28	'20	'30	'31	Tests	Cent
1*	Lead arsenate (2)†, lime (4), water (100)†	3	10	6	6	3	1.8	3.8	2.7	18.4	5.1	28	6.6
2	Oil emulsion stock 200 at 2%	1	5	3	6	10	14.	3.2	6.	15.9	6.2	25	8.2
3	Oil emulsion stock 5 at 2%		3	2	6			3.7	5.3	21.9		11	13.8
4	Oil emulsion stock 200 plus pyrethrum, at 2%	2	5	2			15.9	3.3	17.2			9	9.2
5	Oil emulsion stock 200 plus nicotine (Ni-SO <sub>4</sub> ½ pt. per 100 gals.), at 2%		t	2	6	7		.3	3.3	10.5	5.1	16	6.6
6	Oil emulsion stock 200 plus derris, at 2%		2	2	7	1		3.3	7.	15.1	25.8	12	12.7
7	Control, no spray for late- brood larvae				1	4				25.8	33.7	5	27.
8	Control, no spray all season	2	4	3	4	2	56.7	46.1	62.8	42.5	36.4	15	49.3

\* The early-season spray schedule for items 1-7 was 3-4 pounds of lead arsenate and 6-8 pounds of hydrated lime per 100 gallons of spray, in the calyx and two or three cover sprays. In

spraying was recommended for 1928 to 1932 by the Illinois Agricultural Experiment Station. Close supervision of application of the sprays insured a uniform covering in all of the experimental orchards. Experimental blocks of apples were made up of mature trees 25-40 years of age. Each block contained 25-30 trees of the varieties Jonathan, Grimes Golden, Staymen Winesap or Ben Davis. The spray plots were conducted in duplicate or triplicate in each orchard.

When the fruit was ready for harvesting, the performance of the individual sprays was graded by members of the entomology and horticulture staffs. These men at the time they scored each plot were unfamiliar with the spray treatment the plot had received. The four center trees of the plot late-brood treatments of 1931, item 1, the lead arsenate was increased to 4 pounds per 100 gallons.

† Pounds per gallons.

second and third brood. In seasons when the fruit is plentiful and prices are low the cost of second-brood sprays must be low in order to justify their application.

All of the spray mixtures tested show a seasonal variation in their relative efficiency. When codling moth is abundant and the season favors its development, the number of larvae entering the fruit will increase in spite of consistent and careful spraying. This has led to the general conclusion, as put forth by Headlee (1932), that spray materials and spraying alone will never give adequate control of this insect unless the population of moths can be kept within reasonable limits by the use of supplementary measures of control, such as orchard sanitation and tree banding.

Item I can be used as a basis of comparison since this treatment conforms with the recommendations for the control of second and third-brood codling moth larvae. Lead arsenate was applied at 2 pounds per 100 gallons in 1927, 1928, 1929 and 1930, and at 4 pounds per 100 gallons in 1931. The change in 1931 gave lead arsenate an advantage, as no change was made in the oil-spray schedule. In items 1 and 2 a direct comparison is shown between lead arsenate plus hydrated lime (1 pound of lead arsenate to 2 pounds of lime) and oil emulsion at 2 per cent concentration. The results in 1927 were decidedly unfavorable to the oilsprayed plot. The years 1928 and 1930 showed 5 and 16 per cent fewer entrances in the oil-sprayed fruit. In 1929, 55 per cent, and in 1931 18 per cent, more larvae entered the oil-sprayed fruit. For the five-

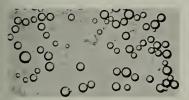


Fig. 2.—Oil stock 200. ×490.

year average, 19.5 per cent more larvac entered the oil-sprayed than the lead arsenate-sprayed fruit.

Items 2 and 3 show the performance of two commercial white-oil emulsions. The droplet size of these two oils is illustrated by microphotographs, figs. 2 and 3. The oil-droplet size in stock 200, fig. 2, is much larger. The emulsion contains 15.6 per cent less oil, has a relatively low wetting property, and gives a very spotted type of covering. Commercial oil stock 5, fig. 3, has small oil droplets, high wetting, and leaves a smooth, even oil covering. If the protection given fruit from codling moth larvae and the safety to fruit and foliage be considered, oil stock 200 is superior to the other oil emulsion for second-brood codling moth control.

#### White-oil Emulsions with Pyrethrum and Derris

Poison plant-extracts from pyrethrum flowers, tobacco or derris root, mixed with stock emulsion 200, have been given many

field trials. Data covering the work with codling moth are given in Table 5, items 4, 5 and 6. Emulsions containing extracts of derris and pyrethrum were found to be less toxic to codling moth larvae under field conditions than the nonimpregnated emulsions. Exposure of derris or pyrethrum products to the action of sunlight and oxygen destroyed their activity toward insects, as is clearly demonstrated by laboratory tests. These same oil emulsions, when tested against codling moth larvae in the laboratory after a relatively short exposure to the air, consistently gave performance superior to nonimpregnated emulsions.

#### White-oil Emulsions with Nicotine

The use of nicotine with oil emulsions is finding a place in the oil-spray program

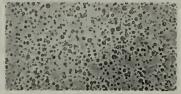


Fig. 3.—Commercial oil stock 5. ×490.

for second-brood codling moth, according to Herbert (1931). The field results with nicotine and oil sprays, Table 5, item 5, show it to be equal or superior to lead arsenate. This is in line with the results of Herbert & Leonard (1929), Regan (1930), Leonard (1930), Spuler & Dean (1930) and Webster (1931).

The most favorable mixture of oil and nicotine has been that of summer-oil emulsion at 1 per cent concentration with nicotine sulfate in dilutions of from 1:800 parts to 1:1600 parts. Free nicotine used in place of nicotine sulfate will give a somewhat quicker kill but is not so effective a mixture as is the oil with nicotine sulfate. The relative persistence of the two nicotine products has not been tested in the field under Illinois conditions. The 1932 report of the Western Cooperative Codling Moth Conference recommends oil and nicotine sprays as the most practical sprays for latebrood codling moth larvae where lead arsenate cannot be used.

#### LABORATORY TESTS IN CONTROL OF CODLING MOTH

Standardized laboratory tests were used in making close comparisons between oil emulsions. Many combinations of insecticides were tried in the search for improved killing power of the oil sprays. White-oil stock emulsion 200 served as a basis of comparison for oil emulsions. This formula contains 64.4 per cent of a technical white oil of 83 seconds viscosity, with an inert emulsifier. The formula was varied with respect water to a 2 per cent concentration by volume and applied at once to test apples by standardized methods to be described.

#### Laboratory Technique

The method used in rearing codling moth larvae for larvicidal tests was described by Farrar & Flint (1930). The apparatus and technique for handling the apples and the larvae were developed in the present work.<sup>4</sup> The method of applying the spray to the fruit was standardized as

Table 6.—Larvicidal efficiency of oil sprays tested in the laboratory against newly hatched codling moth larvae.

ltem	Type of Spray Tested	FORMULA VARIATIONS. MATERIALS ADDED TO OIL STOCK OR DILUTE SPRAY	No. Formulae Tested	Average Relative Efficiency
1	White oil 200	Pyrethrum products. Viscosities 40-150 seconds	10	87.8
2	White oil 200	Nicotine products	7	77.6
3	White oil 200	Miscellaneous group III*	4	68.7
4	White oil 200	A pyrethrum product plus six types of fungicides	10	66.6
5	White oil 200	Derris products	14	58.8
6	White oil 200	Four inert emulsifier combinations	4	58.5
7	White oil 200	10% fluosilicates	3	47.
8	White oil 200	Miscellaneous group II*	6	45.7
9	Commercial white- oil stocks		3	36.3
10	Lead arsenate (2) <sup>†</sup> lime (4) water (100)	t	3	36.3
11	White oil 200	Viscosities 40-200 seconds	15	34.2
12	White oil 200	Five types of fungicides	8	33.6
13	White oil 200	Miscellaneous group I*	6	25.
14	Control, water		1	0.

\* Miscellaneous Group I.—An oxidized oil base, 1 per cent cresylic acid, 1 per cent sald, 10 per cent waste sulfite liquor, 4 per cent Lethaneous Group II.—One per cent dark cresylic acid, 5 per cent straw cresylic acid, 1 per cent Penetrol, 5 per cent salol, 1 per cent acid oil, 8 per cent Alcotatc, 5 per cent soluble plac oil, Miscellaneous Group III.—One per cent low-

boiling petroleum nitrogenous bases, 5 per cent creexite acid, 1 per cent high-boiling petroleum nitrogenous bases, 1 per cent high and low (composite) petroleum nitrogenous bases. These groups are for convenience in evaluation and do not necessarily contain chemically related formulae.

† Pounds per gallons.

to those ingredients which were thought capable of improving toxicity. In the series of emulsions tested the oil-droplet size varied from 1 to 12 microns in diameter. Where the oil-droplet size was not disturbed by changes within the emulsion or by the addition of chemicals to the emulsion, the size remained relatively constant hetween 1 and 6 microns, averaging about 4 microns, fig. 2. The greater number of emulsions tested did not depart far in oildroplet size or in other physical properties from those exhibited by oil-emulsion stock 200. All emulsions were diluted with tap to distribution, time and pressure. Twentyfour hours after spraying, each apple was infested with 10 newly hatched larvae and then placed in a control chamber maintained at a temperature of  $80^{\circ}$  F and a relative humidity of 65–70 per cent. This set of conditions was found to be optimal for the entrance of codling moth larvae into untreated fruit. The infested fruit was kept under controlled conditions for 24 hours and then placed under laboratory conditions until it was checked for larval entrance. Three apples with a total of 30  $^{4}$  See frontispiece.

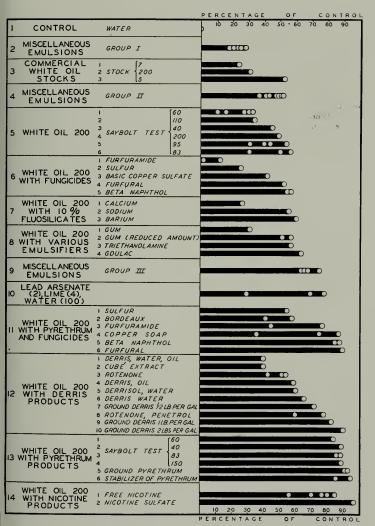


Fig. 4.—Laboratory tests of sprays to control codling moth in Illinois, showing relative efficiency of 13 groups of materials. Each white dot marks the average efficiency of a series of tests.

larvae were considered a series and a check apple with 10 larvae was included with

every two series. Seven series of tests were conducted with each emulsion. In the total test a material thus received 21 or more trials, in which 210 coding moth larvae were given opportunity to enter the sprayed fruit. Because of the natural variation of larval vitality it was found advisable to conduct the seven smaller tests of three apples each over a period of days rather than to make a large single test.

Scoring of treated and untreated fruit was done five days after infestation. All data obtained were calculated against the number of larvae entering untreated fruit. The relative efficiency for each emulsion has been calculated on the basis of the control afforded by the untreated fruit.

For purpose of discussion, emulsions that are related in certain ingredients are grouped together, as shown in Table 6. The emulsions within each group, and the several groups as a whole, have been arranged to show the relative efficiency of the sprays against codling moth larvae. Figure 4 is a graphical illustration of the exact performance of each material tested. Each white dot in the columns of this graph represents the average efficiency of a series of laboratory tests on a respective emulsion. the length of the column then showing the range of efficiency.

#### White-oil Emulsions

The property of an emulsion can be altered by changing the viscosity of the oil. The average efficiency given by 15 changes in viscosity was 34.2 per cent. The changes included cover the entire range in oil viscosities suitable for tree-spray oils. This control is only 2.2 per cent greater than that given by the 83-second oil, which is the same as commercial oil stock 200. The total range in the results by changes of viscosities alone is not much greater than that exhibited by the commercial white-oil emulsions. Laboratory data, fig. 4, item 5, would indicate that oil viscosities under 83 seconds are not so satisfactory as the higherviscosity oils for the killing of codling moth larvae.

The most efficient white-oil emulsions were obtained by varying the concentration or type of emulsifier. The most efficient emulsion contained the emulsifier *Goulac*, lignin pitch, which according to Hurt (1931) is a by-product of the paper industry. Physically this emulsion has undesirable properties. It is relatively unstable, has large irregular oil droplets, and the stock tends to jell on standing.

The amount of emulsifier included in an emulsion will determine within certain limits the type of emulsion that will be formed. When the percentage of emulsifier to the oil and water phases is reduced, the size of the oil droplets tends to increase. In the cases of the emulsions shown in fig. 4, item 8, where the gum emulsifier was greatly reduced, the emulsion formed was stiff, had larger oil droplets, and was more difficult to dilute than was oil emulsion 200, item 5. Although the physical change brought about by reduction of the emulsifier resulted in somewhat higher kill of codling moth larvae, the characteristics of this gum emulsion make it impractical.

In general, under laboratory conditions the oil emulsions containing the smaller oil droplets did not give so high a kill of codling moth larvae as did those with the larger oil droplets. The emulsion containing a triethanolamine emulsifier, item 8, did not follow this trend as did some of the other white-oil emulsions.

Need of a fungicide that can be safely combined with an oil emulsion as a summer spray has long been recognized. In this study a number of possible fungicides were tested. Fungicides typical of those tested are shown in fig. 4, items 6 and 11. Fungicides mixed with an emulsion tend to reduce the efficiency of the emulsion below that of the emulsion without the fungicide. This fact is particularly true of those fungicides that have the property of adsorption of a part of the emulsion to the surfaces of their particles. Examples of such fungicides are sulfur and Bordeaux. Furfural, beta naphthol and copper soaps do not seem to have this reaction of adsorption, neither do they reduce larvicidal efficiency as do the more bulky materials. In this study there was no fungicidal material tested that could be included in an emulsion in sufficient quantity to be toxic to the fungi of apple scab, l'enturia inaequalis (Cke.) Wint., or apple bitter-rot, Phyllosticta solitaria Ell. & Ev., under field conditions.

The commercial white oils include two of the most successful summer-oil emulsions. The laboratory efficiency of these oils was 24, 32 and 53 per cent, with an average of 36.3 per cent, Tahle 6, item 9. Stock oil 5, fig. 3, with the highest efficiency

-53 per cent-has 15.6 per cent more oil, smaller oil droplets and better wetting properties than the other two emulsions. Stock 200, fig. 2, is uniform physically but has low wetting power. Stock 7 is one with poor physical properties, including poor wetting, irregular oil droplets and low stability. The results obtained with these commercial white oils show that they are typical of results that may be obtained with any satisfactory white-oil emulsion. The variations used in experimental emulsions were not sufficient to show marked superiority over the standard emulsions. The physical changes made in emulsions, Table 6 and fig. 4, express about the physical limits possible with strictly white-oil emulsions for use in tree sprays applied to foliage.

#### White-oil Emulsions plus Pyrethrum

Pyrethrum extracts, 1-3 pounds per gallon of oil, combined with the oil phase of emulsions, produced the most efficient codling moth larvicides. Ten formulae containing pyrethrum gave an average relative efficiency of 87.8 per cent. The consistent superiority of pyrethrum emulsions is shown in fig. 4, item 13.

Addition of fungicides to pyrethrum emulsions reduced average efficiency of the latter 21 per cent. Furfural and beta naphthol did not reduce the kill as greatly as did copper soap, furfuramide, copper Bordeaux or sulfur, Table 6, item 4, fig. 4, item 11.

#### White-oil Emulsions plus Nicotine

Oil emulsions with nicotine produced the second most efficient codling moth spray, Table 6, item 2. The seven formulae tested averaged 10 per cent less efficient than the pyrethrum sprays, although the range in control shown by the oil-nicotine sprays is greater than that of pyrethrum sprays, fig. 4, item 14. All sprays of oil emulsion with nicotine were more efficient than white-oil emulsions alone. The use of oil emulsionnicotine sprays in the field has shown the residue to be as toxic and nearly as persistent for codling moth larvae under field conditions as that of lead arsenate. Oil emulsions containing nicotine are the only mixtures of oil and a plant poison that have withstood exposure to weather approximately as well as lead arsenate.

#### White-oil Emulsions plus Derris

Derris extracts act more slowly and are more stable to oxidation than are pyrethrum extracts. It was with the hope of finding a more stable insecticide than pyrethrum that derris was tried extensively in laboratory and field tests. The results of these tests were disappointing. In many cases emulsions containing derris were inferior to those containing pyrethrum. The efficiency of derris, Table 6, item 5, fig. 4, item 12, is shown to be widely distributed, illustrating the very erratic results obtained with this material. The derris sprays tested are not a dependable group when incorporated with white-oil emulsions for killing codling moth larvae.

#### White-oil Emulsions plus Other Insecticides

Fluosilicates at 10 per cent mixed with oil emulsions are more toxic than white oils alone, but such combinations have very undesirable properties which do not make them practical mixtures, Table 6, item 7, fig. 4, item 7.

In the miscellaneous group 111, Table 6, item 3, is a substance called petroleum nitrogenous base. The nitrogenous bases used in the tests have a boiling range between 180 and  $520^{\circ}$  F. This is the only material other than the extracts of the plant poisons which offered promise as an insecticide with oil. The other materials listed under the miscellaneous groups 1 and 11 do not show sufficient promise to justify further study. The materials included in the miscellaneous groups are not all chemically related, which makes the grouping artificial.

#### Laboratory Tests with Lead Arsenate

Two pounds ot lead arsenate plus 4 pounds of hydrated line killed codling moth larvae in the laboratory as efficiently as this spray did in the field. It is of interest to note that with the most uniform coating obtainable with lead arsenate on apples, the efficiency of this spray over unsprayed apples was only 30.3 per cent. This means that relatively 63.7 per cent of the larvae penetrated a coating of lead arsenate and gained entrance to the fruit. The highest control with lead arsenate was 88 per cent, or identical in control with the average efficiency for the pyrethrum-impregnated emulsions, Table 6 and fig. 4, item 10.

#### Red Spider

#### Tetranychus sp.

The most satisfactory control of red spider was obtained by the use of a technical white-oil emulsion at a concentration of 1 per cent.

#### ON CONIFERS

Conifers are often attacked by red spiders to such an extent that the younger trees are killed. Where the mites are not sufficiently abundant to kill the trees, they so destroy the chlorophyll in the tissues that the trees appear brown. Many of the needles become dry and drop off, leaving the tree very unsightly.

than in a film, as is commercial oil stock 5. This irregular type of coating reduces the danger of injury to foliage and leaves the oil spots scattered over the surface to entangle the red spiders as they crawl about the needles. Immediate kill of red spider by oil stock 200 is not apparent, but its residual effect gives the most satisfactory control of any emulsion tested. Addition of a material to give this oil emulsion higher wetting properties did not cause foliage injury but did reduce protection afforded against red spider.

In two cases oil stock 6990 gave very

Table 7 .--- Degree of safety and red spider control for oil emulsions on the foliage of conifers.

ITEM	Oil Formula No.		PROPERTIE	s	Oil- droplet Size	Degree of Red Spider Control	AT	. of Te Per C entra	ENT	DEGR TO	PERIMEN EE OF S. FOLIAGE PER CEN CENTRA	AFETY AT T
		Vis- cosity	Emul- sifier	Wetting Power	microns		.5	1.	2.	5.	1.	2.
1	6990	32	Gum	Low	1-5	Low		5	3		60	100
2	5220	40	Gum	Low	1-5	Low	1	4	1	100	100	100
3	5230	60	Gum	Low	1-5	Low	1	4	1	100	100	100
4	200	83	Gum	Low	1-5	High	1	17	5	100	94	80
5	$200 + Blood^{\dagger}$	83	Gum	Very high	1-5	Low		10			100	
6	Commercial oil 5		Inert	High	1-3	Medium	1	8	3	100	75	
7	Control		Water			None	17	17	17	100	100	100

\* Field-test blocks contained several species of conifers and 50 to 3000 trees. † Blood is a powdered residue sold as blood

In general it has not been considered safe to apply oil sprays to the foliage of conifers in the summer months or after growth has started. Conifer foliage is more sensitive to oil than deciduous foliage. This does not mean that oil sprays cannot be prepared for relatively safe use on conifers, particularly on the more resistant species, such as juniper, pine, arbor vitae and some forms of spruce. In the course of this project field tests were carried on each year to determine the effect of certain oils on conifer foliage. In Table 7 a summary of these tests is given wherein several facts are brought out regarding foliage tolerance and red spider control. Oil stock 200, fig. 2, gave the best control of red spider of any of the materials tested. This control can be largely attributed to the type of coating given by this oil, as stock 200 does not wet conifer foliage but adheres in the form of drops. When this type of emulsion dries, the oil is deposited in tiny spots over the surface rather

1

albumen, a by-product of the meat industry. It has about four times the wetting power of sodium oleate.

severe injury, possibly because of its low viscosity and rapid penetration. Oil-stock emulsions 5220 and 5230 were not as persistent as oil stock 200 and did not control red spider. This relation between persistence and control of red spider is in accord with the findings of deOng (1930). Neither oil 5220 nor 5230 cause injury to conifer foliage.

The tolerance of conifers to oils depends on species of the trees, season of the year, concentration, saturation and viscosity of the oil, and the wetting power of the dilute spray. Sprays with high wetting power are more toxic to foliage than sprays without this property. This excludes the use of oils with soap emulsifiers, such as miscible oils and inert emulsified oils with high wetting power. All oils should be of technical white grade and have a viscosity greater than 60 seconds and less than 100 seconds Saybolt. Concentration of the spray should be the lowest that will control the pest involved.

The technical white oils, properly emulsified, may be applied with relative safety at any time of the year except in extremely hot weather. Care should be taken in applying oil sprays to blue spruce, compact arbor vitae and all new plantings. Drenching of conifer foliage with oil sprays should be avoided. Pines and junipers will tolerate one or more applications of 1-2 per cent oil emulsion; spruce, arbor vitae and similar tender species from one-half to 1 per cent oil emulsion. One, or at most two, applications of the proper oil emulsion have given excellent protection for the season against the common red spiders attacking evergreens.

#### ON RASPBERRIES

Oil emulsions have been tested on raspberries for two seasons. In these tests one



**Fig. 5.**—Soluble oil 16. ×490.

to three applications of stock oil 200 at 1 per cent concentration gave excellent control of red spider without injury to the foliage. The sprays were applied with a power sprayer at a pressure of 200 pounds. For satisfactory results it was necessary to use care to wet the under surfaces of all leaves. For this purpose a short rod carrying three fine nozzles turned upward permitted excellent spraving from the underside. Stock oils 5220 and 5230 did not injure the foliage but failed to control the mites. The low viscosity of these oils, 40 and 60 seconds respectively, would suggest that they penetrated the tissues readily and did not leave a persistent oil film to entangle the crawling spiders.

Favorable results with oils on red spider are reported by Vinal (1917) on cucumbers, by Newcomer & Yothers (1927) on fruit trees, by Whitcomb & Guba (1928) on cucumbers, and by Compton (1931) on greenhouse crops. Yothers & Mason (1930) had unfavorable results in controlling the egg stage of the citrus rust mite, *Phyllocoptee oleivorus* Ashm., by the use of oil sprays.

# San Jose Scale

#### Aspidiotus perniciosus Com.

For control of San Jose scale, the most efficient emulsions contained relatively large oil droplets associated with high wetting and quick hreaking properties.

Oil emulsions have been used against scale since about 1878. Because of the wide distribution of San Jose scale it has received much attention in the development of control measures. Great impetus was given to the study of oil emulsions for this insect following the complete destruction of many orchards by this scale from 1919 to 1922, according to Ackerman (1923), Davis (1924) and Chandler, Flint & Huber (1926).



Fig. 6.—Soluble oil 90. ×490.

The scale affords such natural protection that any insecticide to be effective must gain access to the living insect. Ability of oil sprays to form films or coatings that are toxic to scale has made oil emulsions a reliable source of control. The work done in the control of this insect has demonstrated the physical and chemical properties necessary in an emulsion for high efficiency against a number of insects other than scale.

According to Ackerman (1923) and Davis, Yothers, Ackerman & Haseman (1926, 1927), most workers find that at least a 2 per cent actual oil is required to give satisfactory control of scale. The proper concentration will depend entirely on the properties of the emulsion, as is shown in Table 8. The data were calculated according to the formula of Abbott (1925). Abbott (1926) found that mortality counts on San Jose scale made 30 days after the treatment by sprays gave accurate indices of performances. In these tests the mortality counts were made from four to six weeks after the oil-spray treatments were applied.

Soluble oils 16, fig. 5, and 90, fig. 6, contain the same amount of emulsifier, but oil 16 contains a highly saturated white oil. This emulsion is less stable than the emulsion containing the unsaturated oil. The effect of saturation on the stability of soluble oil 16 is reflected in the difference between these two emulsions in the mortality in this comparison between soluble oils 47, 48 and 49. All are less stable than the 83second-viscosity emulsions even though the lighter emulsions contain the emulsifier in greater quantity.

deOng & Knight (1925) found as they decreased the amount of soap emulsifier used in relation to the volume of oil that "the results showed a progressive increase of kill as the amount of soap was decreased.

Table 8.—Relation between viscosity, saturation, ratio of emulsifier, and droplet size, for miscible-oil sprays tested on San Jose scale.

Item	Oil Formula No.	,	Сомр	0517102	Ratio of	Oil- droplet Size	С	N at P once	ER (				AT 1	VE EFF Per Ce centr.		
		Vis- cosity	Satu- ration	Emul- sifier	Emul- sifier to Oil	microns	1.	1.5	2.	2.5	3.	1.	1.5	2.	2.5	3.
1	Soluble oil 49	32	100	Sodium oleate	1-1.5	1-5	1		1			37.6		45.1		
2	Soluble oil 47	32	100	Sodium oleate	1-4	1-11	i		1			46.5		63.6		
3	Soluble oil 48	32	100	Sodium oleate	1-5.6	1-20	1		1			44.7		83.2		
4	Soluble oil 16	83	100	Sodium petro- leum soap	1-6.7	1-6	1	1	1		1	93.1	100.	99.7		99.
5	Soluble oil 90	83	91	Sodium petro- leum soap	1-6 7	1-3	1	1	4		1	57.1	87.5	91.8		99.1
6	Soluble oil 22	83	91	Sodium petro- leum soap	18	1-5	I		7			82.6		96.6		
7	Soluble oil 17	83	91	Sodium petro- leum soap	1-9	1-6	1	3	7	4	2	91.2	94.	97.4	98.8	97.
8	Control, w 33.3 per	ater cent o	of scale	alive			5	5	5	5	5	0.	0.	0.	0.	0.

\* Items 1 to 4 contain technical white oil, 1 to 5 and item 7 are paraffin oils.

of scale. The less stable soluble oil 16 is the more toxic.

Emulsions containing oils of 32 seconds of viscosity are not sufficiently persistent to give a satisfactory control of San Jose scale. The effect of the ratio of emulsifier to the oil phase is demonstrated in this series of low-viscosity oils. Soluble oil 49, fig. 7, contains a high ratio of emulsifier to the oil and is less toxic than soluble oil 48, fig. 8, in which the amount of emulsifier is reduced. Soluble oil 47 is intermediate between the other two emulsions in ratio of emulsifier and toxicity to scale.

A comparison of items 1–3 with items 4–7, Table 8, shows decided superiority for the 83-second-viscosity emulsions. Viscosity and not stability is the dominant factor  $\dot{\uparrow}$  A test is a field series in which 1000 to 4000 overwintering scales were counted. Data include six years' results.

but with the least amount of soap used. namely 1 per cent, there was not a complete kill of the scales." deOng (1926) gave additional data substantiating this principle. Soluble oils 90, 22 and 17, Table 8, items 5, 6 and 7, demonstrate the effect of the ratio of the emulsifier and oil on droplet size, breaking and toxicity to San Iose scale. Soluble oil 90 contains the most emulsifier, has smaller oil droplets and lower efficiency than either oil 22 or 17, figs. 9, 10. Soluble oil 17 possesses the correct balance between oil and emulsifier. It is an emulsion with relatively large oil droplets, is quick breaking, and has superior kill of San Jose scale. Soluble oils 16 and 17 have similar stability and compare favorably in toxicity to San Jose scale.

It is evident from these data that efficiency is influenced by viscosity, saturation and oil-droplet size. These variations give to an emulsion certain properties that in with their respective degrees of control, are shown in Table 9. Certain relationships are lacking but those most influential in control of scale are included. Soluble oils



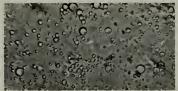


Fig. 7.—Soluble oil 49. ×490.

Fig. 9.-Soluble oil 22. ×490.

Table 9.—Relation between viscosity, saturation, ratio of emulsifier and oil-droplet size for commercial oil sprays tested against San Jose scale.

Item	Com- mercial Oil No.	MERCIAL OIL NO. COMPOSITI Vis- Emul-			Average Range of Oil- droplet Size	No, Tests at Per Cent Concentration†				Relative Efficiency at Per Cent Concentration					
		cosity		sion to Oil	microns	1.	1.5	2.	2.5	3.	1.	1.5	2.	2.5	3.
1	Soluble oil 2	47	Soap	High	1-3		1	1		1		52.6	94.5		88.5
2	Soluble oil I	350	Soap	High	1-3		2	1	2	1		99.	89.2	99.	93.7
3	Soluble oil 17	83	Soap	Low	2.5-3.5	1	3	7	4	2	91.2	94.	97.4	98.8	97.
4	Government formula BFOS	104	Potash fish-oil soap	Low	3-4	1	1	2	ı	1	91.1	98.4	95-9	100.	98.5
5	Illinois formula BFOS	104	Potash fish-oil soap	High	2-3					4					95.4
6	Stock oil 5	80	Inert	Low	2.5-4.5	1	1	1	1	1	88.9	97.	95 5	99-9	99.
7	Stock oil 8		Gum	Low	1-6			1		2			98.9		98.2
8	Control, water 33.3 per cent scale alive					5	5	5	5	5	0.	0.	0.	0.	0.

\* Item 6 contains a technical white oil. Items i to 5 and item 7 are paraffin oils.  $^\dagger$  A test is a field series in which 1000 overwintering scales are counted. Data include six years' results.



Fig. 8.—Soluble oil 48. ×490.

turn influence the degree of control which the emulsion will give against San Jose scale.

The physical properties of proprietory emulsions tested against San Jose scale,



Fig. 10.-Soluble oil 17. ×490.

1. 2. 17, items 1, 2 and 3, are typical of soluble oils marketed in Illinois for dormant spraying of fruit trees.

Soluble oil 2, fig. 11, with an oil of 47seconds viscosity is inferior to soluble oil 1 containing a 350-second oil. The oil-droplet size is not greatly different in these two emulsions. Soluble oil 17 has a lower ratio of emulsifier to the oil than the other solu-



Fig. 11.-Commercial soluble oil 2.  $\times 490.$ 

ble oils and is a quicker-breaking emulsion. This oil is consistently superior to the others in its control of scale.

Boiled fish-oil soap emulsions, fig. 12, were as efficient against San Jose scale as was soluble oil 17. Both of the boiled emulsions, Table 9, items 4 and 5, have larger oil droplets and somewhat quicker breaking qualities than soluble oil 17. The government formula<sup>5</sup> contains half as much soap emulsifier and considerably more water than the Illinois formula5. This condition imparts to the government formula a slightly larger oil-droplet size and quicker breaking qualities.

There are certain sections in Illinois orchard areas where the extremely hard water will prevent this formula from emulsifying properly. For this reason the Illinois formula is recommended and used. This emulsion contains sufficient soap to permit emulsification in most Illinois waters. Both of these emulsions contain about one third

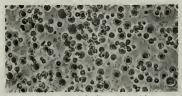


Fig. 12 .- Boiled fish-oil soap emulsion stock, Illinois formula. ×490.

<sup>5</sup> Government formula: Potash fish-oil soap......1 pound .....2 Water Light-grade lubricating oil.....1 gallon lilinois formula: Potash fish-oil soap......1-2 pounds  of their volume in water and soap and are therefore recommended for use at a concentration of 3 per cent, which places them about on a basis of comparison with a soluble oil at 2 per cent concentration. When boiled-soap emulsions are tested and compared with miscible oils on their oil basis they are as effective in their kill of scale as the soluble oils. This accords with findings of Ackerman (1923), Davis (1924), Chandler, Flint & Huber (1926), English (1928) and Swingle & Snapp (1931).

Stock emulsions 5 and 8, Table 9, items 6 and 7, are emulsions containing inert emulsifiers. In both, the ratio of emulsifier to the oil is sufficiently low to impart quick-

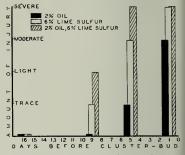


Fig. 13 .- Relation in days between bud development of apple and safety to the tree when sprayed with a 2 per cent soluble-oil emulsion, 6 per cent liquid lime sulfur, or a combination of both. The first spray was applied in the following periods of tree development: dormant, tip-green, delayed-dormant, and cluster-bud.

breaking properties, as shown by their respective oil-droplet size. The control of San Jose scale exhibited by these emulsions is comparable with the quick-breaking soluble oils and boiled fish-oil soap emulsions.

Factors other than insecticidal efficiency have entered into public acceptance of the several commercial emulsions. Many of those who have made and used the boiled fish-oil soap emulsions will continue to do so, but there is an increasing trend toward the commercially prepared quick-breaking soluble oils. This has come about from the relatively low cost of soluble oils, the ease with which they can be stored and diluted, and the uniform performance given by such oils as soluble oil 17 in the control of San Iose scale.

#### Delayed-dormant Applications of Oil Sprays

For a number of years there has been a tendency to delay the application of oil sprays for the control of San Jose scale as late as possible in the season in order to kill such aphids as were hatched. Very few cases of injury are reported from stable oil sprays applied when the trees are strictly dormant. Felt (1913) reported less injury when oils were applied just before growth started in the spring than in fall applications. deOng (1926) observed that the blooming date was influenced by the season of application, recording that the have been obtained. In the test are included an unsaturated miscible oil at 2 per cent, applied in the fall, winter and spring dormant, and the delayed-dormant stages; boiled fish-oil soap emulsion at 3 per cent ; and the miscible oil at 8 per cent concentration. The last two are applied in the spring dormant. In one year the fish-oil soap emulsion, and for three years the 8 per cent miscible oil, have shown a retarding effect on leaf development. In all cases the retardation was no longer noticeable six weeks after the buds started to open. There have been no injurious effects on any of

Table 10.-Relation between oil-droplet size of a miscible oil and its insecticidal efficiency as tested against aphids.\*

Item	Formula	Oil	COMPOSITION		Ratio of Soap	Oil- droplet Size	(10 AT	OF TH 00 Aph Per C Centr	IDS) ENT	RELATIVE Efficiency at Per Cent Concentration		
		Oil	Viscosity	Emulsifier	of Soap to Oil	microns	.5	1.	2.	.5	1.	2.
1	Soluble oil 18	Paraffin	83	Soap 15	1-2.6	1-3	3	7	7	96.5	89.4	80.6
2	Soluble oil 90	Paraffin	83	Seap 15	1-6.7	1-3	3	43	33	96.	80.3	95 5
3	Soluble oil 17	Paraffin	8.3	Soap 15	1-9	1-6	3	17	12	98.	90.	98.
4	Soap 15			Sodium petrole- um soap			3	17	7	98	82.3	81~
5	Control, water						50	50	50	9.3	9.3	9.3

\* H. setariae, A. pomi, A. spiraecola.

early winter applications produced a stimulating effect, whereas applications after the start of bud development had a retarding effect. Kelley (1930a) suggests that the injury observed from early fall applications was due to winter injury that followed the breaking of the rest period by an application of oil.

In Illinois commercial practice, the dormant spraying of apple and peach with oils for scale has been followed for 10 consecutive years without measurable effects on crop production. Experimentally, 10 annual dormant applications of a 2 per cent miscible oil have not produced neticeable changes in tree development. A parallel test, where an 8 per cent oil was applied, gave a distinct retardation of bud development for about seven days. This retardation, although very apparent at the time, was completely covered by normal tree growth in about six weeks after bud develooment started.

In a series of tests that has been conducted for seven years, quite similar results the trees receiving the 2 per cent oil spray in the dormant stage, regardless of the season at which they were sprayed.

As shown in fig. 13, if the oil spray is delayed until the leaves begin to unfold or later, injury in the form of burning of the new leaves may result.

#### Aphids

#### Aphis spiraecola Patch

#### .1phis pomi DeG.

#### Hysteroneura setariae (Thos.)

The efficiency of emulsions against aphids depends on high wetting and low stability or the presence of limited quantities of nicotine. An emulsion containing nicotine was more efficient when it contained a substance to promote high wetting.

English (1928) found that "an emulsion in order to be effective against aphids must have 'high wetting' ability coupled with unstability. Either of these factors may vary so as to be dominant. A relatively 'poor wetting' unstable emulsion may be more effective on aphids than a 'good wetting' stable emulsion. If the stability of two emulsions is about the same, then the one with the greater wetting ability is the more effective on aphids."

Griffin, Richardson & Burdette (1927) found that "under conditions of comparable concentrations and type of oil, miscible



Fig. 14.—Soluble oil 18. ×490.

breaking emulsion and a higher mortality of aphids. In soluble oil 17, fig. 10, the emulsifier has been reduced over that of soluble oil 90 with a still greater efficiency against aphids. This oil exemplifies about the maximum killing power obtainable with a miscible oil that is sufficiently stable to be marketed commercially. In all of the tests the toxicity to aphids is greater at the lower concentrations of the emulsions or the emulsifier. This condition illustrates the principle that excess wetting causes a run-off of the spray material and a reduction in the kill of some contact insecticides.

Table 11 includes the results obtained

Table 11.—Relation between droplet size and insecticidal efficiency of commercial oils tested against aphids.\*

ltem	Com- mercial Oil No.	Composition Oil Viscosity Emulsifier		WETTING PROPERTY	Oil- droplet Size	No. of Tests (1000 Aphids) at Per Cent Concentration			Eff P	RELATIVE EFFICIENCY AT PER CENT CONCENTRATION		
		Oil	VISCOSILY	Enuismet		microns	.5	1.	2.	.5	1.	2.
1	Stock 5	Technical white	80	Inert	Low	1-6	3	35	17	93.4	67.2	91.1
2	Stock 4	Paraffin		Soap	Low	1-21	3	17	17	93.4	82.4	93.8
3	Soluble oil 17	Paraffin	83	Soap	Medium	1-6	3	17	12	98.8	90.	98.
4	Illinois formula BFOS	Paraffin	104	Soap	High	3-8	3	35	35	86.1	61.8	84.
5	Soluble oil 1	Paraffin	350	Soap	High	1-4	3	7	7	85.6	74.4	84.7
6	Control, water				Low		50	50	50	9.3	9.3	9.3

\* II. setariac, A. pomi, A. spiraecola.

oils are probably the less toxic to insects than the ordinary soap emulsions, because they contain smaller oil droplets and the oil therefore adheres to the plant and (no doubt to the insect) less effectively."

Data given in Table 10 include the factors of wetting, quick breaking and droplet size. The miscible oils in items 1, 2 and 3 contain the same oil and emulsifier as in item 4, but are combined with their emulsifier in concentrations that give them entirely different physical properties, Soluble oil 18, fig. 14, is heavily emulsified and the oil droplets are so small that they are hardly visible under high magnification. This emulsion has as high wetting properties as does the emulsifier in item 4. The emulsion is extremely stable and gives a mortality of aphids comparable with the emulsifier. In soluble oil 90, fig. 6, the emulsifier has been reduced to give a quick-

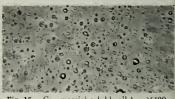


Fig. 15.—Commercial soluble oil 1. ×490.

with five commercial enulsions that have a wide range of physical properties. The killing power of these emulsions at concentrations of one-half, 1 and 2 per cent is in line with Table 10, where the mortality was highest at a one-half per cent concentration, lowest at 1 per cent, increasing again at 2 per cent. This effect is produced by the volume and concentration of spray material adhering to the bodies of the insects. The physical properties of wetting and stability of each emulsion determine to a large extent these factors. The effect of the wetting properties of emulsions on their efficiency can be illustrated by comparing stock oils 5 and 4. Stock oil 5, Table 11, item 1, and fig. 3, contains an inert emulsifier and has quick breaking properties. It is less effective than stock oil 4, which contains a soap emulsifier and has higher wetting and quicker breaking properties.

Soluble oil 17 combines the property of higher wetting than that possessed by the former emulsions with almost as good breaking properties. This oil, however, dilute emulsion for increasing its toxicity against aphids but soap is not compatible with the stock emulsion when incorporated with it as a concentrate.

Free nicotine is compatible with this emulsion in the concentrate when used in proportions of not more than 1 per cent. Such a combination makes an emulsion that is very efficient against aphids. When onefourth of 1 per cent of soap is added to the dilute emulsion to give it increased wetting, as in item 6, the mortality of aphids is higher than that given by the emulsion without soap but containing twice the

Table 12.—Laboratory	tests on aph	nids with gum	emulsions,	demonstrating th	ie necessity
for increasing wetting powe	r or for incor	porating nicoti	ne into gum	emulsions to con	itrol aphids.

Item	Spray Base	MATERIAL Added to Stock Emulsion	Per Cent Soap in Dilute Spray	No. Combina- tions	Tests (1000 Aphids Each)	RANGE IN PERCENT- AGE OF KILL
1	Water	None	0.			0-5.
2	Water	None	. 25	3	4	17.
3	Stock 200 at 2%	None	0.	5	24	35-86.
4	Stock 200 at 2%	None	. 25	3	7	97.
5	Stock 200 at 2%	.5-1% nicotine	0.	4	13	96-100.
6	Stock 200 at $2\%$	. 24% nicotine	. 25	2	6	99.
7	Stock 200 at $2\%$	1 lb. pyrethrum per gal.	0.	1	4	63.
8	Stock 200 at $2\%$	1 lb. pyrethrum per gal.	. 25	3	4	96.
9	Stock 200 at 2%	1 lb. cubé per gal.	0.	1	2	94.
10	Stock 200 at $2\frac{c_r}{c_0}$	1–5% sulfur; petrol- eum nitrogenous bases; chinchona alkaloid salts; pine oil	0.	7	17	25-82.

gives maximum kill. Of the commercial emulsions, the Illinois formula of boiled fish-oil soap emulsion contains an excess of soap necessary for emulsification in hard waters. This excess soap gives to this emulsion very high wetting which offsets its advantages of larger droplet size and quicker breaking properties. Soluble oil 1, fig. 15, is both high wetting and slow breaking, a combination of properties which would seldom give a satisfactory control of aphids at comparable concentrations.

The addition of one-fourth of 1 per cent of soap, Table 12, item 2, to diluted stock emulsion 200, item 3. increased the mortality of aphids 52 per cent over the use of the emulsion alone, and 80 per cent over soap alone. A soap may be added to the actual nicotine, item 5. Free nicotine and nicotine sulfate both tend to reduce the stability of a stock emulsion, especially in the higher concentrations. For this reason the more practical combination would be one that contains a very low nicotine content to be used with soap at the time of application for increased wetting.

Extracts of pyrethrum, derris or cubé are not as efficient as nicotine when mixed with oil emulsion against aphids. The addition of soap will increase the killing power of an oil containing such extracts, item 8, but not enough to warrant the added cost of the extracts.

Sulfur (2–5 per cent), petroleum nitrogenous bases, chinchona alkaloid salts, or pine oil did not increase the toxicity of the oil emulsions to aphids. Nicotine, added to either the stock emulsion or the diluted spray, was the only really efficient spray for aphids. These tests demonstrate the toxic nature of nicotine to aphids. Experiments were conducted with 25 different oil emulsions, most of which were tested at 5, 8 and 10 per cent concentrations. The results are shown in Table 13. These experiments demonstrate clearly

Table 13.--Results of laboratory tests with various concentrations of oil emulsions on eggs of oyster-shell scale.

Item	TYPE OF EMULSION TESTED	NO. Emulsions Tested		NO. TES T PER CE NCENTRAT	INT	Relative Efficiency at Per Cent Concentration			
			5.	8.	10.	5.	8.	10.	
1	Commercial oils	6	4	2	24	25.4	80.	88.	
2	Experimental miscible oils	9	9	4	16	50.	52.	94.	
3	Boiled fish-oil soap emulsions	6	4		13	32.2		96.4	
4	Experimental oils (White-oil stocks)	4			14			92.5	
5	Control, water	1	б	б	б	0.	0.	0.	

 $\ast$  A test is considered 10 twigs with 10 over-wintering oyster-shell scales each. Each of the

# Oyster-shell Scale

### Lepidosaphes ulmi (Linn.)

Oyster-shell scale was adequately controlled with a 2 per cent white-oil emulsion applied about the time the young scales hathed. A miscihle or soluble oil applied in the dormant stage of development at high concentration gave a satisfactory control of this insect.

The tests of dormant-oil emulsions against the eggs of the oyster-shell scale

100 scales per test contained from 40 to 80 eggs.

that only a high concentration (10 per cent) of oil emulsion is effective against the eggs of this scale. Even at high concentration good wetting must be secured if control is to be obtained.

Under field conditions somewhat lower dilutions have given good results due to the effect of the residual oil left on the bark following an application of oil spray. The

Table 14.—Results of field tests for efficiency of oil sprays on oyster-shell scale at time of hatch.

Item	Formula	Сомро: Emulsion	Emulsifier	lnjury to Foliace	No. Formulae Tested	No. Tests at 2 Per Cent Con- centra- tion	RELATIVE CONTROL
1	Soluble oil 17	Paraffin oil, miscible	Petroleum soap	Yes	1	3	99.3
2	Stocks 3050, 3060	White oil stock, pyrethrum, sulfur	Gum	Yes	2	14	99.7
3	Stock 430	White oil stock, pyrethrum	Gum	No	1	6	96.
4	Commercial	White oil stocks	lnert	No	2	11	95.5
5	Control	Water		No	0	8	0.

were conducted in the laboratory by a standard procedure. Twigs having overwintering scale were collected, graded, sprayed and placed in moist sand to grow until the young scale hatched. The young scales were removed and counted as fast as they hatched. presence of oil on the twigs destroys many of the young migrating scale.

The best results in control of this insect were secured by the application of an oil spray at the time the young scales were hatching. At this season the young scales are sensitive to oil and are easily killed by oil sprays. The results of field trials are shown in Table 14. Young oyster-shell scale hatch at a season of the year (May-July) when their host plant is in full foliage. Although practically any oil applied at 2 per cent concentration will give control of this scale, the oil selected for application should be safe to apply on foliage. The white-oil emulsions marketed as summer oils have proved the most satisfactory. similar to soluble oil 17, at 5 per cent concentration, have been tested against this pest. This oil has given a very high degree of control where good wetting is obtained and when the oil is applied in the spring before the clm foliage appears.

The control of young European elm scale by summer-oil sprays has not been as satisfactory because of the wide distribution of the young over the leaves. To obtain

Table 15.—Results of leaf roller ovicide tests in the laboratory at three concentrations of emulsions. Each egg mass averaged 57 viable eggs.

ITEM	Formula No.	GENERAL COMPOSITION	Vis- cosity			PHYSICAL PROPERTIES OF DILUTE SPRAY	I	Egg M Sested Per Ce Centr	AT	Eff	Relative Efficiency at Per Cent Concentration	
					microns		4.	б.	8.	4.	б.	8.
I	Stock	Diamond paraffin oil in Bor- deaux*	104	1-20	1-11	Very quick breaking	50	30		100.	100.	
2	Stock	Diamond paraffin oil in Kayso†	104	1-20	1-19	Very quick breaking	10			100.		
3	Commercial oil 7	Soluble	140		0-100	Very quick breaking	20	20		100.	100.	
4	lllinois formula BFOS	Diamond paraffin oil in BFOS	104	1-5.7	3-8	Ve <b>ry</b> quick breaking	40	30	10	96.	100.	100.
5	8350	Soluble	225	1-9	1-2	Very quick breaking	50	20		95.	88.	
6	5110	Soluble +5% cresylic acid	83	1-6.7	1-5	Quick breaking	40	40		79.	83.	
7	22	Soluble	83	1-8	1-5	Quick breaking	30	40	20	72.	94.	98.
8	8340	Soluble	104	1-9	1-3	Quick breaking	40	20		62.	57.	
9	8360	Soluble +1% free nicotine	83	1-9	1-4	Quick breaking	30	30		61.	89.	
10	8370	Soluble + petroleum bases	83	1-9	1-6	Quick breaking	40	20		52.	58.	
11	17	Soluble + nicotine sulfate 1:800	83	1-9	1-6	Quick breaking	30	10	10	50.	69.	94.
12	17	Soluble	83	1-9	1-6	Quick breaking	50	60	30	47.	75.	96,
13	Commercial oil 1	Soluble	350		1-4	Not quick breaking	30	20	10	26.	61.	67.
14	Commercial oil 2	Soluble	47		1-4	Not quick breaking			20			6.
15	Control	Water					110	110	110	0.	0.	0.

\*4:4:50 Bordeaux.

†2 ounces Kayso per gallon of oil.

#### European Elm Scale

#### Gossyparia spuria (Mod.)

A miscible or soluble oil used at a concentration of 4 per cent adequately controlled European elm scale when applied as a delayed-dormant spray. Some difficulties were encountered in controlling this insect with summer sprays.

The rapid spread of European elm scale in nurseries and cities has attracted considerable attention. Dormant-oil emulsions the highest degree of control by summer applications (at the time of hatch), the oil must possess a high wetting power and should contain nicotine at the same concentration as that recommended for aphids. Complete covering of the foliage with the spray is essential if a satisfactory control is to be obtained.

Cleveland (1931) found that stock emulsion 200 used at 2 per cent concentration gave excellent control of hatching scale when 4 pounds of 30 per cent potassium fish-oil soap were added to each 100 gallons of dilute emulsion. He found the addition of soap necessary in order to secure adequate wetting of the foliage and insects. No injury resulted from his sprays on American and Cornish elms, applied July 15 at Monroe, Mich.

#### Fruit-tree Leaf Roller

#### Archips argyrospila Walk.

Stability was the dominant physical property of an emulsion that was efficient in killing eggs of the fruittree leaf roller. The less stable emulsions were the most efficient.

The control of fruit-tree leaf roller by the application of lead arsenate sprays has not given uniformly satisfactory results,



Fig. 16.—Soluble oil 8350. ×490.

according to Regan (1923), Wakeland (1925), Flint & Bigger (1926) and Harman (1928). Feeding of the larvae can be checked with lead arsenate sprays, but special sprays using high dosages of lead arsenate are necessary. Authors agree that the logical method of control is one directed against the egg stage of the insect.

Oil emulsions have given the most satisfactory control of leaf roller eggs in the laboratory and in the field. Wide variations have been found to exist between the various oil emulsions tested. Under field conditions it is seldom that sprays can be applied thoroughly enough to strike every egg mass on the tree. For this reason field trials of the same oils and concentrations have not given so high a mortality as have laboratory tests. This is in accord with the findings of Penny (1921). Comparable results were obtained in field tests (not included in this paper) where the oil sprays listed in Table 15, items 1, 5, 6, 7 and 12 were given field trials.

Regan (1923) asserts that unless 75 per

cent of the eggs can be killed the expense of the spray is not warranted.

Experiments of Flint & Bigger (1926), Harman (1928), Tolles (1931), and Parrott, Hartzell, Glasgow & Harman (1931) show that less than 6–8 per cent actual oil in an emulsion will not give control of leaf roller eggs. These tests further indicate that the quicker-breaking emulsions will kill the eggs at a lower concentration than the slow-breaking emulsions. The coldmixed Bordeaux or Kayso emulsions have been the most efficient because of their quick-breaking properties.

Laboratory data on leaf roller eggs given in Table 15, items 1 and 2, illustrate the superior killing power of cold-mixed emulsions. With an actual oil content of 4 per cent, the 4:4:50 Bordeaux and calcium caseinate emulsions gave 100 per cent kill of eggs. Commercial soluble oil 7 is the only soluble oil giving complete kill at a low concentration. The non-miscible properties of this oil in hard water would not make it a practical commercial emulsion for other than leaf roller or similar insect control. Only a small portion of this emulsion would remain in suspension without constant agitation. Boiled fish-oil soap emulsion, item 4, with a large oil-droplet size and quick-breaking property, gave almost as high mortality of eggs as did the oils under items 1, 2 and 3.

Soluble oil 8350, item 5 and fig. 16, has the same emulsifier ratio as soluble oil 17, item 12, but contains a 225-second-viscosity oil. The higher-viscosity oil produces an emulsion that does not disperse as readily as does oil 17. As a result the diluted soluble oil 8350 tends to separate out of the water phase, giving to the emulsion a quickbreaking property. The same characteristic is exhibited to an even greater extent by commercial soluble oil 7. Neither oil has sufficient emulsifier to disperse the oil particles completely when diluted with hard water.

Emulsions included in items 6 to 11 vary in certain ingredients from soluble oil 17. The cresylic acid in soluble oil 5110 and extra emulsifier in soluble oil 22 did not increase the toxicity materially. The addition of 1 per cent free nicotine as in oil 8360, fig. 17, nicotine sulfate at 1:800, or petroleum base, as in oil 8370, fig. 18, did not increase the kill over that exhibited by soluble oil 17. In all of the experiments the quick-breaking soluble oils tested at 8 per cent concentration gave a satisfactory control of leaf roller eggs. This is significant because of the wide use of soluble oils commercially for the control of this insect.

As ovicides for leaf roller eggs, emulsions with large oil droplets, such as occur in cold-mixed emulsions, are more efficient than commercially prepared emulsions when compared at equal concentrations. Certain quick-breaking soluble oils will give a commercial control of leaf roller if they are carefully applied at a concentration of 8 per cent or greater. The viscosity

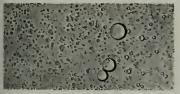


Fig. 17. Soluble oil 8360. ×490.

of oils between 83 and 350 seconds is not of major importance except as it affects the breaking property of the emulsion. A soluble oil containing an oil of 47 seconds of viscosity was not efficient against the eggs of the fruit-tree leaf roller.

#### Fleas

#### Pulex irritans Linn.

Oil emulsions at a concentration of 5 per cent gave excellent control of fleas on farmsteads.

The economic importance of fleas as a pest of rural communities is not generally recognized nor appreciated. The author made a survey of several counties in central Illinois and found a large number of farmsteads infested with fleas. Reports from farm advisers located in other counties indicated that similar conditions were present over a large section of the state.

Tests carried on over a period of three years indicate that soluble oil 17 at 5 per cent concentration will give a satisfactory control of fleas. Premises (interiors of barns, outbuildings, etc.) must be thoroughly cleaned of litter, dust and other refuse. The area is then sprayed with a 5 per cent oil emulsion and the floors thoroughly soaked. From 300 to 800 gallons of dilute spray will cover the average farmstead. One application is sufficient for light infestations but two or three treatments at intervals of two weeks are recommended in heavily infested areas.

#### OILS WITH FUNGICIDES

Dormant-oil emulsions can be used with sulfur fungicides in the dormant stage of tree development. If used after the buds start to swell, serious injury may result. There is danger of injury if oil emulsions are applied in the delayed-dormant stage of tree development, particularly when sulfur is to be used in the later sprays for the control of fungous diseases.

It is doubtful whether fungicides of the more common types can be used with summer emulsions without disturbing the efficiency of the emulsion or causing injury to the plant tissue when they are applied to foliage.

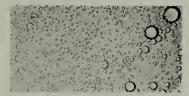


Fig. 18.--Soluble oil 8370. ×490.

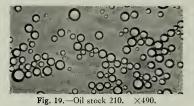
# Dormant Oils with Fungicides

Under some conditions it is practical to use sprays that contain an emulsion and a fungicide. The most common fungicides contain either copper or sulfur. Dormant peach and apple trees will withstand relatively high concentrations of either oil emulsion or lime sulfur without injury. It is therefore possible to spray dormant trees with oil emulsions mixed with some fungicides. Such sprays are very useful in the control of scale and peach-leaf curl on peach, and scale and apple scab on apple. The properties of such sprays will depend on the fungicide added.

The use of copper in the form of Bordeaux has been widely tested and has proved successful in most respects. Emulsions containing soap can be added to Bordeaux if the concentration of the uncombined copper or calcium is not so high as to reverse the type of emulsion.

Most soap emulsions flocculate in the presence of inert forms of sulfur. "Flotation," a type of very finely divided precipitated sulfur obtained in the manufactured-gas industry from a process known as liquid purification, combines with an inert emulsified oil, such as oil 210, to make an oil-sulfur combination possessing unusual properties, figs. 19, 20. These properties are described by Farrar & Smith (1930). Emulsions made with extremely stable emulsifiers, as oil 210, will mix with lime sulfur without separation. Such sprays can be used on dormant trees without noticeable injury.

The addition of sulfur to emulsions, particularly in the form of lime sulfur, has many objectionable features, the most serious of which is separation of the emulsion in the presence of lime sulfur. All miscible oils and stock emulsions that contain soap as the emulsifier will not mix with lime sulfur without separation of the emulsion and the liberation of free oil in the spray



mixture. This finding is in accord with the results of Ackerman (1923) and Yothers & Winston (1924). It is dangerous practice to apply partly separated emulsions to trees even in the dormant stages of their development. Yothers & Winston (1924) suggest the addition of stabilizers to oillime sulfur mixtures. Cutright (1929) describes the 10 oil-fungicide combinations which he thought possible to use under Ohio conditions. deOng (1930) suggests the use of organic sulfur in the cyclic series with oil emulsions to increase their fungicidal value.

The results of two years of tests with sprays containing lime sulfur and oil emulsion are shown in fig. 13. In these experiments three distinct types of emulsions were tested—a miscible oil at 2 per cent concentration, a proprietary emulsion recommended for use with lime sulfur, and stock 210. All were applied in the dormant, tipgreen, and delayed-dormant stages of tree development. No injury from any of the materials was apparent among those trees sprayed while dormant. With the trees in the tip-green stage,6 the emulsion alone did not injure, but lime sulfur either alone or mixed with oil emulsion caused very noticeable injury. In the delayed-dormant period, when the leaves were beginning to unfold. all of the sprays produced some injury. Least injury occurred on the oil emulsionsprayed trees; those receiving lime sulfur were badly burned, and those receiving the mixture of oil and lime sulfur were so severely burned that the leaves did not develop further. The oil emulsion sprayed in the cluster-bud stage resulted in very noticeable injury to the unfolding leaves and flower clusters. Dutton (1932) finds that "there is evidence that the presence of oil with lime sulfur renders lime sulfur injury more severe or causes its develop-



Fig. 20.—Oil stock 210 plus sulfur. ×490.

ment when line sulfur without oil would not produce injury." Although sometimes recommended, the spraying of fruit trees with oil emulsion and lime sulfur is not a safe practice.

In many sections spray schedules on apples call for a spray of lime sulfur very early in the season, for the control of apple scab. This spray is often applied in the cluster-bud stage of development. Figure 13 shows the effect of oil emulsion and lime sulfur on foliage. Serous burning will usually result. The same condition can be approached where the oil spray is delayed and followed soon after by an application of summer-strength lime sulfur. Figure 21 summarizes the experiments to determine the interval of time that should elapse between the oil spray and a spray of lime sulfur. It appears to be relatively safe to apply lime sulfur two weeks after an application of oil emulsion.

<sup>&</sup>lt;sup>6</sup> The tip-green stage of bud development is the time at which the bud scales have parted sufficiently to show the new green tissue that has been developing within the expanding bud. This stage precedes by a few days the rapid expansion of new leaves.

Yothers & Mason (1930) found that two or three weeks should elapse before an oil spray on citrus was followed by a sulfur spray or dust for the control of citrus rust mite. Overholser & Overley (1930) found under Washington conditions that oil sprays previous to July 1 caused injury to fruit and foliage following a delayed-dormant application of lime sulfur.

# Summer Oils with Fungicides

The need of an oil emulsion with fungicidal properties has been recognized. Many attempts have been made to combine sulfur,



Fig. 21.—The incompatibility of dormant-oil spray and sulfur makes necessary an interval of 10 days to two weeks between applications of the two mixtures. The graph shows the injury to the tree caused by applying a dormant oil too late in the season; that is, just before the cluster-bud stage.

copper and their derivatives with both summer and dormant-oil emulsions. Other materials that have exhibited fungicidal properties have been tested but most of them have been discarded as unsatisfactory. Although sulfur is widely used as a fungicide, it will produce injury even when used in small quantities with oil, according to deOng (1928c). Hoerner (1929) suggests that Penetrol is compatible with flowers of sulfur and safe on apple foliage if the two are mixed with water before they are combined. Talbert & Swartwout (1931) state, "Lubricating-oil emulsions have been used throughout the summer in applying the regular summer combination applications, using the oil at 1 per cent and 2 per cent with the standard insecticidal and fungicidal spray, lime sulfur and Bordeaux with lead arsenate."

This finding is not in accord with the general knowledge of sulfur and oil combinations. Oil emulsions containing certain forms of copper are relatively safe on foliage but the spray has low insecticidal efficiency and it is difficult to store because of its corrosive action on metal. Oil emulsions may be added to well-made Bordeaux without danger of injury but the Bordeaux reduces the efficiency of the oil emulsion, according to Porter & Sazama (1930).

Oil emulsions containing derivatives of furfural show promise but are not completely satisfactory as insecticides or fungicides.

To our present knowledge there is no material sufficiently toxic to fungi that can be added to an oil emulsion without interfering with either the insecticidal efficiency of the oil emulsion or with the toxicity of the fungicide to fungi. The reduced efficiency of emulsions with fungicides was demonstrated in experiments with codling moth larvae, Table 6 and fig. 4. In these experiments, fungicides added to the pyretbrum sprays reduced their average efficiency from 88 to 66 per cent, and that of the white oils without pyrethrum from 34 to 33 per cent. Fungicides that were partially soluble in the oil phase of the emulsion decreased efficiency of the sprays less than did the flocculent materials, such as flowers of sulfur or Bordeaux.

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