

Circular 153

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Report on Commercial Insecticides and Fungicides

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Connecticut
Agricultural Experiment Station
New Haven

Examination of Insecticides, Fungicides, Etc.

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INTRODUCTION

THE Legislature of 1923 passed an act concerning the manufacture, sale and transportation of adulterated insecticides and fungicides. The text of the law and regulations, made as provided therein for its enforcement, are given in Bulletin 258 issued by the Station in 1924. Both the law and such regulations as have been made are substantially the same as the federal law and regulations so that articles of this class which satisfy the requirements of interstate commerce will be accepted in this State.

The law requires this Station to make analyses of samples which may be collected by the Dairy Commissioner or by our station agent. Evidence of adulteration or misbranding is required to be reported to the Dairy Commissioner who is responsible for enforcement of the law. Analyses and such other information regarding the character, composition and use of these materials as may be of interest are required to be published in bulletins of this Station, either annually or at other intervals as may be advisable. The law carries no specific appropriation for the inspection work and a complete survey of the entire field of insecticides and fungicides each year is not thought to be advisable or necessary.

Seven bulletins [Bulletins 157 (1907); 242 (1922); 258 (1924); 272 (1925); 300 (1929); 346 (1933); 398 (1937)] and one circular [Circular 136 (1939)] have been published giving analyses of insecticides made by this Station. Bulletins 300, 346 and 398 also include analyses obtained from other sources.

The present circular includes analyses made at this Station in the period 1939 to 1941, inclusive.

CLASSIFICATION OF MATERIALS

The samples analyzed are classified as follows:

Materials	No. of samples
Arsenate of lead	2
Bordeaux—lead arsenate	1
Clay	4
Copper preparations	1
Lime	1
Lime—sulphur	2
Lime—sulphur--nicotine	1
Mineral oils and emulsions	26
Nicotine preparations	9
Pyrethrum preparations	1

Rotenone preparations	9
Soap	1
Sulphur	2
Miscellaneous	24
Total	84

RESULTS OF INSPECTION AND ANALYSIS

Arsenate of Lead

3162. *Chipman Hi-Test Lead Arsenate*. Chipman Chemical Co., Inc., Bound Brook, N. J. Analysis was as follows:

	Guaranteed Percent	Found Percent
Total arsenic oxide (As_2O_3)	32.50	32.36
Water-soluble arsenic oxide (As_2O_3)	0.38	0.05
Lead oxide (PbO)		63.68

970. *Ledogrub*. Golf and Lawn Supply Corporation, Scarsdale, N. Y. This was a mixture of lead arsenate and fertilizer material. Analysis was as follows:

	Guaranteed Percent	Found Percent
Lead arsenate ($PbHAsO_4$)	19.40	17.5
Total arsenic (As)	3.90	3.82
Water-soluble arsenic (As)	0.25	0.15
Total lead (Pb)		10.32
Total nitrogen (N)		4.88
Total phosphoric acid (P_2O_5)		2.90
Total potash (K_2O)		0.42

Bordeaux Mixture—Lead Arsenate

717. *Dry Bordeaux Mixture with Lead Arsenate*. Manufacturer unknown. Examined qualitatively for identification. Copper, calcium, lead and arsenic found present.

Clay

Four samples were analyzed for iron with results as follows:

	Ferric oxide (Fe_2O_3) percent
2624. <i>Daltonclay</i> . United Clay Mines Corp., Trenton, N. J.	8.46
2505. <i>Hall clay</i> . United Clay Mines Corp., Trenton, N. J.	11.06
2701. <i>Red clay</i> . Southeastern Clay Co., Aiken, S. C.	6.87
2623. <i>Red slate dust</i> . Manufacturer unknown.	7.95

Copper Preparation

7382. *Copper Sulphate Monohydrate*. Manufacturer unknown. Contained 37.04 percent water of crystallization. The theoretical

TABLE I. ANALYSIS OF MINERAL OILS AND MINERAL OIL EMULSIONS

Station No.	Manufacturer or distributor and brand	Oil		Total ash	insol- uble ash	Alkalinity of soluble ash As K ₂ CO ₃	Constants of separated oil		Remarks	Probable emulsifier
		Guar- anteed	Found				Saybolt viscosity, 100° F.	Un- sulpho- nated		
		%	%	%	%	%	Seconds	%		
2560	Calif. Spray Chemical Co. Kleenup		62.1	0.73		0.35			No potassium or sulphate; odor of creosote	Sodium soap
7580	Calif. Spray Chemical Co. Kleenup		75.3	0.29		0.12	127	72.4	No potassium	
7582	Calif. Spray Chemical Co. Kleenup		73.1	0.40		0.12	121	61.0	No potassium	
6688	Calif. Spray Chemical Co. Kleenup Ready Mix						126	59.0		
715	Calif. Spray Chemical Co. Kleenup Ready Mix						131	57.2		
713	Calif. Spray Chemical Co. Kleenup Soluble	70	83.9	0.81	0.02	0.20	175	52.8	Considerable sulphate present	Sodium alkyl sulphate
6687	Calif. Spray Chemical Co. Kleenup Soluble Oil R. M.		87.8	0.73		0.32	162	56.0	No potassium; sulphate present	Soap plus sodium salt of sulphonated oil (or alkyl sulphate)
710	General Chemical Co. Dormasol						133	10.1		
709	General Chemical Co. 83 Flowable		72.0	0.51	0.46	0.01	133	12.6		Bentonite
7577	General Chemical Co. Oil Emulsion 83	81.3	74.6	0.26		0.17			No potassium; ammonia 2.52 percent	Ammonium soap
7581	Manufacturer unknown. Ortho-K		77.1	0.30		0.10	54	87.0	No potassium	
7579	Manufacturer unknown. Ortho-K Medium		75.2	0.29		0.05	66	89.0	No potassium	
7576	Manufacturer unknown. White Oil from Summer Emulsion						93	96.8		
706	Niagara Sprayer & Chemical Co. Emulso		72.5	0.09	0.04	0.04	123	61.6		Sulphonated oil (?)
705	Röhm and Haas Co. Inc. Lethane Dormant Oil						108	57.2		

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707	Shell Oil Co. Dormant Miscible		84.3	0.70	0.02	0.17	167	45.0	Considerable sulphate present	Sodium alkyl sulphate
704	Shell Oil Co. Neutrol Dormant Emulsible						125	45.6		
708	Shell Oil Co. Neutrol Emulsion		73.7	0.47	0.41	0.01	132	42.6		Bentonite
3422	Shell Oil Co. Repellocide No. 5+ Emulsifier							78.3		
7257	Shell Oil Co. Summer Spray Oil—Light						62			
711	Sherwin-Williams Co. Flo Mulsion	83 ¹	78.5	0.04	0.02	0.01	123	55.4	Ammonia present	Ammonium caseinate
714	Sherwin-Williams Co. Flo Mulsion Type D	83 ¹	80.7	0.50	0.46	0.01	128	55.6		Bentonite
712	Sherwin-Williams Co. Free Mulsion	83 ¹	76.8	0.05	0.02	0.02	125	55.0	Ammonia present	Ammonium caseinate
7578	Sherwin-Williams Co. Sherwin-Williams Free Mulsion	83 ¹	72.2	0.14		0.12	111	71.0	No potassium; ammonia 0.03 percent	
7575	Standard Oil Co. Industrial White Oil—Tecktol Socony Vacuum							92.0		
7574	Standard Oil Co. Standard Oil Paraffin Oil (Tree Spray Oil)						123	77.4		

¹ Percent by volume.

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water content of copper sulphate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) is 37.0 percent, so sample was the pentahydrate instead of the monohydrate.

Lime

3163. *Spray Lime*. Lime Corp., Lee, Mass. Found: calcium oxide (CaO) 45.51 percent; magnesium oxide (MgO) 32.11 percent; fineness, 100 mesh, 38 percent; 200 mesh, 9 percent; 300 mesh, 1 percent.

Lime—Sulphur

3439. *Formula 33*. Manufacturer unknown. Examination showed that sample was essentially a 4 percent solution of calcium polysulphide in 16 percent alcohol. Found: total solids 4.04 percent; alcohol by volume 16.19 percent.

6463. *Liquid Lime and Sulphur*. Manufacturer unknown. Found: specific gravity 33.0° Baumé.

Lime Sulphur—Nicotine Preparations

976. *Japanese Beetle Spray*. S. Demontis Insecticide Co., Bridgeport, Conn. Qualitative analysis showed nicotine, creosote, lime sulphur, some oil and possibly a little soap present. Only part of the oil was mineral oil; the boiling range of most of it ($155\text{--}180^\circ\text{C}$) indicated the possible presence of turpentine.

Mineral Oils and Mineral Oil Emulsions

Twenty-six samples were examined. Analyses are given in Table I.

Nicotine Preparations

Nine samples were examined. Analyses are given in Table II.

TABLE II. ANALYSES OF NICOTINE PREPARATIONS.

Station No.	Manufacturer or distributor and brand	Nicotine	
		Guaranteed %	Found %
816	Manufacturer unknown. Nicotine Spray Solution		0.14
7366	Manufacturer unknown. Tobacco		0.63
1020	Manufacturer unknown. Tobacco Dust ¹		1.26
3310	Manufacturer unknown. Tobacco Dust		0.59
7358	Manufacturer unknown. Tobacco Stems		0.69
3389	Mechling Bros. Chemical Co., Camden, N. J. Mechling's Powdered Tobacco	1.0	1.30
1254	Tobacco By-Products and Chemical Corp., Inc., Louisville, Ky. "Black Leaf" 155 Micronized Nicotine Dust	2	2.25
1255	Tobacco By-Products and Chemical Corp., Inc., Louisville, Ky. "Black Leaf" 155 Micronized Nicotine Dust	3	2.97
1256	Tobacco By-Products and Chemical Corp., Inc., Louisville, Ky. "Black Leaf" 155 Micronized Nicotine Dust	4	3.93

¹ Fineness: 100 mesh 92 percent; 200 mesh, 54 percent; 300 mesh, 26 percent.

Pyrethrum Preparations

4148. *Pyrethrum Insecticide*. Connecticut Chemical Co., New Haven, Conn. Claimed: clear kerosine, odorless, 50 parts by volume; cosmetic grade white oil, 10 parts by volume; pyrethrum, 20 extract, $3\frac{1}{2}$ parts by volume. Found: pyrethrin I 0.0326 gram per 100 cc.

The declared formula is equivalent to 13.2 grams of pyrethrum per 100 cc. On the basis of an average of 0.41 percent pyrethrin I in pyrethrum (Gnadinger, "Pyrethrum Flowers," p. 95), sample was about 60 percent of its declared strength.

Rotenone Preparations

Nine samples were examined. Analyses are given in Table III.

TABLE III. ANALYSES OF ROTENONE PREPARATIONS.

Station No.	Manufacturer or distributor and brand	Rotenone	
		Guaranteed %	Found ¹ %
4303	Apothecaries Hall Co., Waterbury, Conn. Rotenone Dust	0.5	1.11
8505	Apothecaries Hall Co., Waterbury, Conn. Rotenone Dust	0.5	1.16
1262	Bonide Chemical Co., Inc., Utica, N. Y. Bonide Greentox Rotenone Garden Spray		0.22
1676	Manufacturer unknown. Ground Derris Root		9.57
7706	Manufacturer unknown. Rotenone Dust	0.5	0.96
7708	Manufacturer unknown. Rotenone Dust	0.5	1.33
7707	Manufacturer unknown. Rotenone Dust	1.0	1.65
7709	Manufacturer unknown. Rotenone Dust	1.0	1.74
7439	Seacoast Laboratories. Rotenone Dust	1	1.74

¹ These values were obtained by the colorimetric method of Goodhue, J. Assn. Off. Agr. Chem., 19, 118 (1936), and include deguelin as well as rotenone. The true rotenone percentages are probably about half these figures.

Soap

7417. *Ivory Flakes*. Procter and Gamble, Cincinnati, Ohio. Qualitative analysis showed no silicate, phosphate or borate present.

Sulphur

6683. *Flotation Sulphur*. Public Service Electric & Gas Co., Camden, N. J. Found: sulphur 41.49 percent.

7303. *Koppers Dry Flotation Sulphur*. The Koppers Co., Pittsburg, Pa. Found: sulphur 91.21 percent.

Miscellaneous

6685. *3-5 Dinitro Cresol in Oil*. Standard Chemical Co. Analysis showed 3, 5 dinitro-o-cresol 3.92 percent; oil 92.18 percent; sulphonation test on oil, 67.4 percent.

6684. *Dispersing Oil—Rotenone Product*. Standard Chemical Co. Sample was a brown oil with a sassafras odor. Analysis showed

rotenone 1.14 percent, ash 0.37 percent, alkalinity of ash as Na_2CO_3 0.25 percent, extractable by alkali from ether solution 1.60 percent, neutral oil 93.56 percent. The oil had a saponification number of 72 and gave a sulphonation test of 23.6 percent.

From the above analysis it was considered probable that sample contained rotenone-bearing material and soap in an oil which was a mixture of mineral oil, a fatty oil and camphor or sassafras oil.

8506, 9071, 9072. *DN Dust*— $\frac{1}{2}\%$. Dow Chemical Co., Midland, Mich. These three samples were supposed to contain dinitro-o-cresol. Analysis showed, however, that dinitrocyclohexylphenol was present instead of dinitro-o-cresol. The following amounts were found:

No.	Dinitrocyclohexylphenol percent
8506	1.45
9071	0.58
9072	1.31

6881. *Dolco Nomole*. C. B. Dolge Co., Westport, Conn. Analysis showed that sample was whole canary grass seed impregnated with 0.15 percent strychnine.

6686. *Dowspray Dormant*. Dow Chemical Co., Midland, Mich. Analysis showed dinitrocyclohexylphenol 4.24 percent; oil 93.14 percent; sulphonation test on oil 54.4 percent.

6681. *Du Pont Contact Insecticide IN*—2018. E. I. du Pont de Nemours & Co., Inc., Grasselli Chemicals Dept., Wilmington, Del. Claimed: active ingredients 3.6 percent (rotenone 0.95 percent, other derris extractives 2.65 percent); inert ingredients 96.4 percent (includes solvents, emulsifying and stabilizing agents). "Contact insecticide IN-2018 contains an efficient stabilizer for rotenone, preventing deterioration on long standing. This stabilizer also has a definite synergistic effect on rotenone, which substantially increases its ability to kill red spider, as compared to a rotenone spray of the same concentration but without the stabilizer."

Sample consisted of a brown liquid containing some crystalline sediment. It had an odor of sassafras. Quantitative analysis showed rotenone 0.14 percent, ash 0.001 percent, neutral oil 83.70 percent, organic bases 0.47 percent, and resins and fatty acids 3.37 percent. The neutral oil had the following constants: index of refraction, 25° C., 1.4771; saponification number 81; saponifiable matter 26.33 percent; sulphonation test, 19 percent. Qualitative investigation did not identify the organic base present, although an acetyl derivative was prepared from it which melted at 111—118° C. Examination of the neutral oil fraction indicated the probable presence of safrole. A trace of ammonia was present.

So far as our analyses revealed, sample appeared to contain derris extract, an unidentified organic base, a trace of ammonia, mineral oil, a fatty oil and either camphor or sassafras oil.

The crystalline sediment which was present in the sample was obtained after recrystallization from alcohol as fine yellow needles melting at 203—204° C. It may be surmised that these crystals were not originally present but were a mixture of dehydrorotenone and rotenone formed on standing by oxidative decomposition of the rotenone in solution. (See Jones and Haller, *Jour. Am. Chem. Soc.* 53: 2320. 1931.)

3440. *Go-West*. Manufacturer unknown. In an earlier analysis (*Oregon Agr. Exp. Sta. Cir.* 84: 15. 1927.) this product had been found to contain calcium arsenate. Qualitative analysis of the present sample indicated that its composition has been changed so that now it is composed of apple peelings impregnated with a fluosilicate. No arsenic was found.

7416. *H. P. C. Rosin Residue Emulsion Sticker*. Manufacturer unknown. This product was supposed to be made by the following formula: casein 4 pounds, water 45 pounds, ammonium hydroxide, sp. g. 0.90, 0.75 pound, rosin residue 50 pounds. Analysis showed non-volatile chloroform extract 89.03 percent, total nitrogen 0.36 percent, ammonia nitrogen 0.20 percent. Comparison on a percentage basis between the formula and the composition shown by analysis is as follows:

	Formula calls for, percent	Found percent
Casein	4.01	1.02
Ammonia	0.21	0.24
Rosin residue	50.14	89.03
Water	45.64	9.71

Sample therefore contained more rosin residue and less casein and water than formula called for.

7498. *J. B.-7 Japanese Beetle Repellent and Insecticide*. Colton Insecticide Co., Orange, N. J. From the analysis, sample appeared to be an emulsion containing nicotine 0.56 percent, water 5.98 percent, petroleum naphtha, heavier mineral oil and the sodium salt of a sulphonated oil.

3341. *Moth Liquid*. The Birchard System, Inc., Hartford, Conn. Claimed to contain kerosene, "Sunco spirits (sub. turp.)", oil of cedar and phenol. Analysis showed that kerosene and phenol and probably painters' naphtha (though not over 11 percent) were present. Sample contained methyl salicylate instead of oil of cedar.

4302. *Niagron Rotenone—Fumigant Spray*. Niagara Sprayer & Chemical Co., Middleport, N. Y. Claimed: inert ingredient water 19 percent. "Niagron contains 5 percent of active derris extractives including rotenone, combined with an organic fumigant material and a spreading and wetting agent."

Analysis indicated the presence of derris extract, an organic thiocyanate and sodium alkyl sulphate. Rotenone found 0.29 percent.

4844. *Niaproof*. Niacet Chemicals Corp., Niagara Falls, N. Y. Calculated composition from the analysis was as follows:

Aluminum acetoborate $[Al_2O(C_2H_3O_2)BO_2]$	63.52 percent.
Basic aluminum acetate $[Al_2O(C_2H_3O_2)_2 \cdot 4H_2O]$	0.57 "
Basic ferric acetate $[Fe(OH)(C_2H_3O_2)_2]$	0.22 "
Water by difference	34.19 "

4845. *Niaproof B*. Niacet Chemicals Corp., Niagara Falls, N. Y. Calculated composition from the analysis was as follows:

Basic aluminum acetate $[Al_2O(C_2H_3O_2)_2 \cdot 4H_2O]$	77.59 percent.
Aluminum hydroxide $[Al(OH)_3]$	5.48 "
Ferric hydroxide $[Fe(OH)_3]$	0.12 "
Water by difference	16.81 "

6682. *Nitro Kleenup*. California Spray Chemical Co., Watsonville, Calif. Found: 2, 4 dinitrophenol 2.68 percent; oil 72.95 percent; ash 0.53 percent; alkalinity of ash as Na_2CO_3 0.13 percent. Constants of separated oil: viscosity, 100° F., 105 seconds; unsulphonated, 69.4 percent.

208. *Nitro-Kleenup Powder*. California Spray Chemical Co., Watsonville, Calif. Claimed: active ingredient dinitro-o-cresol 50 percent; inert 50 percent. Found: 3, 5 dinitro-o-cresol 50.31 percent; acid-insoluble ash 20.54 percent. Some form of clay, possibly bentonite, present.

7573. *No-Weed*. Horticultural Research Lab., New York, N. Y. Calculated composition from the analysis was as follows:

Sodium chlorate ($NaClO_3$)	3.23 percent
Sodium sulphate (Na_2SO_4)	0.33 "
Trisodium phosphate (Na_3PO_4)	0.54 "
Sodium hydroxide ($NaOH$)	0.04 "
Potassium permanganate ($KMnO_4$)	0.05 "
Iron, calcium, chloride	traces

1400, 8980. *Orchard Brand Genicide*. General Chemical Co., New York, N. Y. Composition of No. 8980 was not declared. No. 1400 was claimed to contain dibenzo gamma pyrone 95 percent, dibenzo gamma pyrone derivatives 4 percent, inert ingredients 1 percent. Analysis showed No. 1400 to contain 95.30 percent, and No. 8980 68.83 percent, of xanthone. "Dibenzo gamma pyrone" has sometimes been used as a synonym for xanthone.

661. *Safe-N-Lead*. Sherwin-Williams Co., Cleveland, Ohio. Claimed: a prepared dry powdered patented zinc compound used to reduce the injurious effects of water-soluble arsenic found in arsenate of lead. Calculated composition from the analysis was as follows:

Zinc sulphate monohydrate ($ZnSO_4 \cdot H_2O$)	69.77 percent
Calcium hydroxide ($Ca(OH)_2$)	22.33 "

Excess water of crystallization	7.90 percent
Heavy metals, iron, magnesium	traces

3413. *Score #1-A*. Specialty Products Co., Jersey City, N. J. From the analysis, sample appeared to be an emulsion containing kerosene, mineral oil, creosote oil, soap, a little copper and a trace of lead. Found: ash 2.79 percent, oil 83.53 percent, phenols and fatty acids 0.56 percent, copper 0.23 percent, lead 0.03 percent.

1392. *Self-Emulsifying Q T H Oil*. Naugatuck Chemical Division of U. S. Rubber Co., Naugatuck, Conn. Analysis showed that sample was composed mostly of a high boiling mixture of organic chlorine compounds (58 percent boiled 300-336°, and 39 percent 330-336°). An unidentified organic base and possibly a little mineral oil were also present.

1391. *Spergone*. U. S. Rubber Co. Sample was a yellow powder which after recrystallization from amyl acetate was obtained as yellow crystals melting at 286° C. It was identified as chloranil (tetrachlorobenzoquinone).