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U. S. DEPARTMENT OF AGRICULTURE,

BUREAU OF CHEMISTRY—BULLETIN NO. 68.

H. W. WILEY, Chief of Bureau.

THE CHEMICAL COMPOSITION
OF
INSECTICIDES AND FUNGICIDES.

WITH AN ACCOUNT OF THE METHODS OF ANALYSIS EMPLOYED.

BY

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IN COOPERATION WITH THE DIVISION OF ENTOMOLOGY.

WASHINGTON:
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LETTER OF TRANSMITTAL

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., February 20, 1902.

SIR: I have the honor to transmit for your inspection and approval the manuscript of a bulletin prepared in this Bureau by Mr. J. K. Haywood, cooperating with the Division of Entomology. I recommend that this manuscript be published as Bulletin No. 68 of the Bureau of Chemistry.

Respectfully,

H. W. WILEY,
Chief of Bureau.

Hon. JAMES WILSON,
Secretary of Agriculture.

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THE CHEMICAL COMPOSITION OF INSECTICIDES AND FUNGICIDES.

INTRODUCTION.

Since the data on the chemical composition of insecticides and fungicides is extremely meager and no systematic work has been done, to the knowledge of the writer, in this direction by either State experiment stations, private individuals, or the National Government, it was considered that it would be interesting as well as instructive to make a chemical study of this class of compounds. It is true that several States have laws relating to the composition and sale of Paris Green, notably, Texas, California, Louisiana, and New York, and the last of these States publishes each year the composition of insecticides other than Paris Green, but such work as is done, excluding Paris Green, is, up to the present date, very meager. Even in those States having laws governing the composition of Paris Green, California is the only one, so far as the writer is informed, which requires that the free arsenious oxid shall be below a certain fixed percentage.

In pursuance of the study of the subject the chief of the Bureau of Chemistry in the spring of 1900 requested the chief of the Division of Entomology to collect samples of as many insecticides and fungicides as possible throughout the United States. This branch of the work was placed in the hands of Mr. R. S. Clifton, of the Division of Entomology. The following circular letter was sent to all State experiment stations and to several of the special agents of the Department in different localities:

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF ENTOMOLOGY,
Washington, D. C., March 28, 1900.

DEAR SIR: The Division of Chemistry of this Department is about to undertake, in collaboration with this Division, an extended investigation of the insecticides which are being sold on the open market in all parts of the country. With your experience you can readily understand that if the samples to be analyzed were purchased direct by this Department from the manufacturers we would have no guaranty that they would not be doctored, and it seems necessary therefore to avoid giving the information that they are purchased for chemical analysis. I have thought that the best way we can secure them is to ask for your assistance, together with that of other members of the American Association of Economic Entomologists. Can you,

without inconvenience, and will you, purchase through some farmer friend, who is not known to be in close relations with entomologists or chemists, a series of pound samples of the insecticides on sale in your vicinity and send them to this office or to the Chemist, with memorandum bill of expense? Samples may be sent under the inclosed franks, and if you wish more franks I will forward them to you. Accompanying each sample should be a statement of the date and place of purchase, price paid, and the name of the manufacturer.

All assistance will be gladly and publicly acknowledged, and I trust that you will be willing to assist in this interesting work. Of course, similar work has been done by some of the experiment stations, but it seems desirable that it should be done for the whole country. The idea originated in the Division of Chemistry, and this Division is simply collaborating so far as possible.

Most truly yours,

L. O. HOWARD, *Entomologist*.

Of the samples received, amounting to about 300, all those were rejected which did not give fairly complete data as to the price paid, name of manufacturer, and name of retailer. Again, many duplicate samples were received. In case such samples were in sealed packages and came from the same manufacturer only one was analyzed, but in case the samples came from the same manufacturer but were not sealed as many as were received were analyzed, since in the latter case alteration of the sample by the jobber or retailer was possible.

After sifting down the samples in this manner, 156 were finally chosen for analysis. It is thought that these samples fairly represent the present state of the market in the United States.

It is not the intention in this work to go into the various applications of these compounds to the treatment of insect pests and plant diseases, as this is a field which belongs more directly to the entomologist or plant pathologist. We simply desire to give the chemical composition of such preparations, and thus show the public what they are buying. Along with this are given the methods of analysis employed (many of which were devised especially for this work), and some discussion as to whether such compounds are injurious to plants or will serve the purposes for which they are intended.

The insecticides and fungicides are arranged in groups as far as possible, and they will be considered in the following order: (1) Paris greens; (2) London purples; (3) insecticides and fungicides, other than paris greens and London purples, that contain arsenic, copper or both; (4) soaps; (5) hellebores; (6) pyrethrums; (7) mixtures containing borax; (8) mixtures containing free phosphorus; (9) tobacco extracts; (10) miscellaneous solid insecticides and fungicides; (11) miscellaneous liquid insecticides and fungicides.

SOURCES AND DESCRIPTIONS OF SAMPLES.

Before proceeding with the discussion of the groups, Table I, on the next page, is introduced to show the source and description of all samples examined.

TABLE I.—Name of sample, with name of retailer, manufacturer, price, and form in which received.

Serial No.	Name.	Retailer.	Manufacturer.	Retail price.	Form in which received.
19524	Hammond's Slug Shot.....	W. I. Brooke & Co., Washington, D. C.	Benj. Hammond, Fishkill on the Hudson, N. Y.	5 cents per lb.	Sealed.
19525	Paris Green.....	F. A. Tschiffely, jr., Washington, D. C.	Leggett & Bro., New York City.	25 cents per lb.	Do.
19526	do.....	do.....	Name not given.	do.	Do.
19527	London Purple.....	do.....	do.....	do.	Bulk.
19528	Black Flag Insect Powder.....	Z. D. Gilman, Washington, D. C.	Nathan Howe, Boston, Mass.	10 cents per box.	Sealed.
19529	Buhach, C. N. Milco's California Universal Insect Exterminator.	do.....	Buhach Producing and Manufacturing Co., Stockton, Cal.	do.	Do.
19530	Persian Insect Powder.....	do.....	Gilman's Drug Store, Washington, D. C.	do.	Do.
19531	Dry Bordeaux Mixture.....	P. Mann & Co., Washington, D. C.	Leggett & Bro., New York City.	20 cents per lb.	Do.
19532	Paris Green.....	P. H. Heiskell, jr., & Co., Washington, D. C.	Hirshberg, Hollander & Co., Baltimore, Md.	25 cents per lb.	Do.
19533	do.....	do.....	do.....	do.	Do.
19534	do.....	L. G. Orndorff, Washington, D. C.	John Lucas & Co., New York City, Philadelphia, Chicago.	30 cents per lb.	Do.
19535	Scheele Green.....	Mackell Bros., Washington, D. C.	New York Enamel Paint Co., Brooklyn, N. Y.	\$2.50 per lb.	Do.
19536	Bug Death.....	Elmer & Amend, New York City.	Danforth Chemical Co., Leominster, Mass.	15 cents per lb.	Do.
19537	White Hellebore.....	N. N. Tompkins, Lawrence, Mass.	Hemingway & Co., London, England.	20 cents per lb.	Bulk.
19538	Pyrethrum Insect Powder.....	J. Overstreet, Lexington, Ky.	J. A. Blanchard, New York City.	25 cents per lb.	Bulk.
19539	Paris Green.....	N. N. Tompkins, Lawrence, Mass.	S. W. Gould & Bro., Philadelphia, Pa.	do.	Do.
19540	do.....	do.....	Lauman & Kemp, New York City.	20 cents per lb.	Do.
19541	Hellebore, "Lion Brand".....	A. C. Bauer & Co., Lansing, Mich.	Morrison, Plummer & Co., Chicago, Ill.	do.	Sealed.
19542	do.....	Alsdorf & Son, Lansing, Mich.	J. A. Blanchard, New York City.	12 cents per lb.	Do.
19543	do.....	A. C. Bauer & Co., Lansing, Mich.	Leggett & Bro., New York City.	do.	Do.
19544	do.....	do.....	Lyman Elliot Drug Co., Minneapolis, Minn.	25 cents per lb.	Bulk.
19545	do.....	D. W. Burdick, Ithaca, N. Y.	Beckstein Bros., New York City.	15 cents per lb.	Sealed.
19546	do.....	Treman, King & Co., Ithaca, N. Y.	Put up by Leggett & Bro.; manufactured by Hemingway & Co.	do.	Do.
19547	do.....	do.....	do.....	do.	Do.
19548	do.....	J. B. Todd, Ithaca, N. Y.	A. B. Ansbacher & Co., New York City.	25 cents per lb.	Do.
19549	do.....	A. B. Brooks, Ithaca, N. Y.	Nichols Chemical Co., Syracuse, N. Y.	15 cents per lb.	Do.
19550	do.....	J. B. Todd, Ithaca, N. Y.	Hemingway's London Purple Co., London, England.	25 cents per lb.	Do.
19551	do.....	R. R. Harting, Lexington, Ky.	Steekeberg, New York City.	do.	Bulk.
19552	do.....	do.....	Adler Color and Chemical Works, New York City.	do.	Bulk.
19553	do.....	do.....	Swift & Co., Boston, Mass.	35 cents per lb.	Sealed.
19554	do.....	W. R. McGee, Stillwater, Okla.	G. H. Lee Co., Omaha, Nebr.	25 cents per lb.	Sealed.
19555	do.....	C. A. Johns, Lexington, Ky.	Hale, Justice & Co., Cincinnati, Ohio.	25 cents per lb.	Bulk.
19556	do.....	Robertson & Witall, Lexington, Ky.	Stein-Vogeler Drug Co., Cincinnati, Ohio.	10 cents per lb.	Do.
19557	do.....	F. W. Neal & Co., Dover, N. H.	Carpenter-Morton Co., Boston, Mass.	25 cents per lb.	Sealed.
19558	do.....	W. W. Rawson, Boston, Mass.	J. A. Blanchard, New York City.	20 cents per lb.	Do.
19559	do.....	Joseph Breck, Boston, Mass.	A. B. Ansbacher & Co., New York City and Chicago.	30 cents per lb.	Do.
19560	do.....	Columbus Pharmaceutical Co., Columbus, Ohio.	Highlands Chemical Co., New York City.	25 cents per lb.	Do.
19561	do.....	do.....	do.....	do.	Do.
19562	do.....	N. B. Danforth, Wilmington, Del.	Adler Color and Chemical Works, New York City.	30 cents per lb.	Do.
19563	do.....	James Bradford Co., Wilmington, Del.	James Bradford Co., Wilmington, Del.	27 cents per lb.	Do.
19564	do.....	J. J. Smith, Wilmington, Del.	Leon Hirsch & Son, New York City.	20 cents per lb.	Bulk.
19565	do.....	F. M. Victor, Lawrence, Mass.	Leggett & Bro., New York City.	Free sample	Sealed

TABLE I.—Name of retailer, manufacturer, price, and form in which received.—Continued.

Serial No.	Name.	Retailer.	Manufacturer.	Retail price.	Form in which received.
19676	"P. D. Q." Insect Powder.	Sanborn & Robinson, Lawrence, Mass.	Worcester Compound Co., Worcester, Mass.	25 cents per box.	Sealed.
19677	Death Dust for Insects	J. M. Reed, Morgantown, W. Va.	Winkelman & Brown Drug Co., Baltimore, Md	10 cents per box	Do.
19678	Columbian Insecticide	D. S. Hall & Son, Lawrence, Mass.	Columbian Insecticide Co. Boston, Mass.	15 cents per can	Do.
19679	Leggett's Roach Destroyer	do	Leggett & Bro., New York City	15 cents per box.	Do.
19680	Lambert's Death to Lice	F. M. Victor, Lawrence, Mass.	D. J. Lambert, Apopkaug, R. I.	25 cents per box.	Do.
19681	Scrofularia Powder.	do	Benj. Hammond, Fishkill on the Hudson, N. Y.	do	Do.
19682	Peterman's Roach Food.	D. S. Hall & Son, Lawrence, Mass.	Wm. Peterman, New York City	5 cents per box.	Do.
19683	Electric Vermin Exterminator.	J. J. Smith, Wilmington, Del.	F. B. Smith, Canton, Ohio	25 cents per box.	Do.
19684	Instant Louse Killer.	J. M. Reed, Morgantown, W. Va.	Dr. Hess & Co., Ashland, Ohio	25 cents per lb.	Do.
19685	Hellebore	Jenner's hardware store, Dover, N. H.	Leggett & Bro., New York City	25 cents per can	Do.
19686	Leggett's Killer	D. S. Hall & Son, Lawrence, Mass.	do	30 cents per can	Do.
19687	Hammond's Tobacco Extract.	do	Benj. Hammond, Fishkill on the Hudson, N. Y.	50 cents per can	Do.
19688	Hammond's Copper Carbolate	do	do	7 cents per lb.	Bulk.
19689	Grape Dust.	do	Whitney Co., Natick, Mass.	5 cents per lb.	Do.
19690	Tobacco and Sulphur.	do	Benj. Hammond, Fishkill on the Hudson, N. Y.	do	Do.
19691	do	Jenner's hardware store, Dover, N. H.	F. S. Sturtevant, Hartford, Conn.	25 cents per box.	Sealed.
19692	Paris Green	F. A. Sheridan, Chase, Md	Morris Herrmann & Co., New York City	25 cents per lb.	Do.
19711	do	G. L. Peck, Jamaica, L. I.	C. T. Reynolds & Co., New York and Chicago.	20 cents per lb.	Do.
19712	do	Peter Henderson & Co., New York City.	Lewis Berger & Sons, London and New York City	25 cents per lb.	Do.
19713	do	do	do	do	Do.
19714	Paragrene	H. Braun & Sons, Columbus, Ohio	Fred. L. Lavanburg, New York City	do	Do.
19715	Paris Green	S. B. Losh, Coloma, Md	do	do	Do.
19716	do	George & Ridden, Denton, Md	Lewis Berger & Sons, London, England	do	Do.
19717	do	P. L. Hargett & Co., Frederick, Md	I. Pfeiffer, New York City	30 cents per lb.	Do.
19718	do	Jacob's Pharmacy Co., Atlanta, Ga.	No name given	25 cents per lb.	Do.
19719	do	J. E. Bowman, Brighton, Md.	S. G. McCotter, New York City	do	Bulk.
19720	do	Gilman & Dorsey, Columbia, Mo.	Fred. L. Lavanburg, New York City	20 cents per lb.	Do.
19721	do	Peck & Co., Columbia, Mo.	Meyer Bros., St. Louis, Mo	35 cents per lb.	Do.
19722	do	Tillery & Prather, Columbia, Mo.	Hirshberg, Hollander & Co., Baltimore, Md.	30 cents per lb.	Do.
19723	do	do	Morris Herrmann & Co., New York City	50 cents per lb.	Do.
19724	do	Opera House Pharmacy, Columbia, Mo.	Sondheim, Alsberg & Co., New York City	do	Do.
19725	do	Jacob's Pharmacy, Atlanta, Ga.	Morris Herrmann & Co., New York City	35 cents per lb.	Do.
19726	do	Alexander Seed Co., Augusta, Ga.	G. S. Adler, New York City	50 cents per lb.	Do.
19727	London Purple	Meyer Bro.'s Drug Co., St. Louis, Mo.	Leggett & Bro., New York City	35 cents per lb.	Do.
19835	White Hellebore	Reynolds & Balke, St. Joseph, Mich.	Peter Henderson & Co., New York City	30 cents per lb.	Sealed.
19836	do	Jacob's Pharmacy, Atlanta, Ga.	David Landreth & Sons, Philadelphia, Pa	35 cents per lb.	Do.
19837	do	F. S. Hopkins, Benton Harbor, Mich.	Name refused	25 cents per lb.	Bulk.
19838	do	do	Fuller & Fuller, Chicago, Ill	25 cents per lb.	Do.
19839	do	do	R. Hillier & Sons, New York City	do	Sealed.
19840	do	do	Morrison, Plummer & Co., Chicago, Ill	20 cents per lb.	Do.

TABLE I.—Name of sample, with name of retailer, manufacturer, price, and form in which received—Continued.

Serial No.	Name.	Retailer.	Manufacturer.	Retail price.	Form in which received.
20499	Brown's Pink Powder Insecticide....	Brown's Insecticide Co., Syracuse, N. Y.	Brown's Insecticide Co., Syracuse, N. Y.	25 cents per can...	Sealed.
20500	Sure Destruction for Cockroaches and Ants.	Kuhn Drug Co., Omaha, Nebr.	Insect Exterminator Manufacturing Co., Council Bluffs, Iowa.	50 cents per box...	Do.
20501	Estes's Roach Powder....	do	N. T. Estes & Co., Omaha, Nebr.	\$1 per lb.	Do.
20502	Bliss's Cockroach Exterminator	Frank Bliss, St. Louis, Chicago, and Cincinnati	Frank Bliss, St. Louis, Chicago, and Cincinnati	40 cents per can	Do.
20503	Roach and Croton Bug Exterminator	Frank Bliss, St. Louis, Chicago, and Cincinnati	Perfection Manufacturing Co., Jersey City, N. J.	22 cents per can	Do.
20504	Brazilian Insecticide Powder	G. L. Peck, Jamaica, L. I.	I. Majors Co., La Mara, Brazil	25 cents per can	Do.
20506	An insecticide from Florida	C. O. Tyner, Atlanta, Ga.	do	25 cents per lb.	Bulk.
22282	Paris Green.	C. H. Bell, Hartford, Conn.	Cawley, Clark & Co., Newark, N. J.	do	Sealed
22283	do	Geo. J. Loeffler, Hartford, Conn.	Lehn & Fink, New York City	30 cents per lb.	Do.
22284	do	Rapelye & Palmer, Hartford Conn.	C. M. Childs & Co., Brooklyn, N. Y.	do	Do.
22285	do	N. N. S. Tompkins, Lawrence, Mass.	Harrison Bros., New York City	do	Do.
22286	White Arsenic.	F. Storer, Fort Collins, Colo.	Wholesale McPike & Fox, Atchison, Kans.	do	Do.
22287	White Arsenoid	Adler Color and Chemical Works, New York City.	Adler Color and Chemical Works, New York City.	do	Do.
22288	Extract of tobacco	W. W. Rawson & Co., Boston, Mass.	Louisville Spirit-Cured Tobacco Co., Louisville, Ky.	45 cents per can	Do.
22289	Nikoteen	do	Skabecura Dip Co., Chicago, Ill.	\$1 per bottle	Do.
22290	Royal Roach Powder.	do	H. C. Dubring & Co., Chicago, Ill.	50 cents per box	Do.
22291	North State Insecticide	T. J. King & Co., Richmond, Va.	North State Insecticide Co., Richmond, Va.	do	Do.
22292	Lee's Insect Powder.	Wm. Rust & Son, New Brunswick, N. J.	G. H. Lee Co., Omaha, Nebr.	25 cents per lb.	Do.
23491	Roachine.	do	Brown & Allen, Atlanta, Ga.	25 cents per box	Do.
23493	Red Cross Ant Destroyer	De La Mater & Todd, Atlanta, Ga.	Red Cross Chemical Co., Cincinnati, Ohio	15 cents per box	Do.
23501	Roach Sault.	G. L. Peck, Jamaica, L. I.	Barrett Chemical Co., New York City	10 cents per box	Do.
23721	Par Odium.	do	Imported by F. C. Boucher & Co., St. Paul, Minn.	30 cents per box	Do.
23722	Fleck's Lice Exterminator	M. A. Richards, Warsaw, N. Y.	J. J. Fleck, Tiffin, Ohio	45 cents per box	Do.
21 & W	Vaporizing and Fumigating Insecticide.	do	Eastern Chemical Co., Boston, Mass.	\$6 per lb.	Do.
41 & W	Sure Death to Insects	J. H. Green, Lawrence, Kans.	H. C. Dusenberry, New York City	25 cents per can	Do.
51 & W	Dr. Baker's Liquid Death Drops	G. L. Peck, Jamaica, L. I.	James Ainslie, Brooklyn, N. Y.	15 cents per can	Do.
61 & W	Heine's Liquid Insect Destroyer and Disinfectant.	do	Heine Chemical Co., Hollis, L. I.	25 cents per can	Do.
91 & W	Lemon Oil Insecticide	T. W. Wood & Son, Richmond, Va.	J. J. Pollexfen, Baltimore, Md.	do	Do.
111 & W	Thymo-cresol.	D. Landreth & Sons, Jamaica, L. I.	Ness & Co., Darlington, England	20 cents per bottle	Do.
121 & W	Dyke's Louse Paint	Raymond & Co., Lawrence, Kans.	Raymond & Co., Lawrence, Kans.	50 cents per bottle	Do.
131 & W	Chloro-naphtholeum	C. O. Tyner, Atlanta, Ga.	West Disinfecting Co., New York City	do	Do.
161 & W	Grub and Canker Worm Exterminator.	W. M. Rawson & Co., Boston, Mass.	G. H. Morrill & Co., Boston, Mass.	30 cents per can	Do.

METHODS OF ANALYSIS AND ANALYSES OF SAMPLES.

PARIS GREENS.

In analyzing the samples of Paris Green the following determinations were made: Moisture, sand, sodium sulphate, total arsenious oxid, copper oxid, soluble arsenious oxid, and soluble copper oxid. The methods follow.

METHODS OF ANALYSIS AND DISCUSSION.

Moisture.—Dry 1 to 2 grams for eight to ten hours at 105° to 110° C., and calculate loss as moisture.

Sand.—Dissolve the sample used for moisture determination in hydrochloric acid, filter, wash, dry, and finally burn filter, calculating the residue as sand.

Sodium sulphate.—Treat the boiling filtrate from the determination of sand with a boiling solution of barium chlorid, allow to stand until the precipitate settles, leaving a clear solution; filter, wash, dry, and burn, with the usual precautions used in determining barium sulphate; calculate the barium sulphate found to sodium sulphate, since it is in this form that sulphuric acid is supposed to be present.

Total arsenious oxid.—For this determination a standard iodine solution is needed, which is prepared in the following manner:^a Dissolve 12.7 grams of powdered iodine in about 250 cc of water to which has been added 18 to 25 grams of chemically pure potassium iodid, and make the whole up to a volume of 1 liter. To standardize this solution, weigh out 1 gram of dry, chemically pure arsenious oxid, transfer to a 250 cc flask by means of about 100 cc of a solution containing 2 grams of sodium hydrate in each 100 cc, and boil until all arsenious oxid goes in solution; cool, make to a volume of 250 cc, and use 50 cc for analysis.

This 50 cc portion is concentrated by boiling in a 250 cc flask to half its volume and allowed to cool to 80° C. An equal volume of concentrated hydrochloric acid is now added, accompanied by 3 grams of potassium iodid, mixed, and the whole allowed to stand for ten minutes (to reduce the arsenic oxid formed on boiling an alkaline arsenite to arsenious oxid). The brown solution is then diluted with cold water, and an approximately N/10 solution of sodium thiosulphate added, drop by drop, until the solution becomes exactly colorless. (This end-point is easy to read without the aid of starch.) This solution is then made slightly alkaline with dry sodium carbonate (using a drop of methyl orange to read the change), then made slightly

^a See article by Haywood, Jour. of the Amer. Chem. Soc., Vol. XXII, No. 9, September, 1900.

acid with hydrochloric acid, taking care that all lumps of sodium carbonate on the bottom are acted on by the hydrochloric acid. Sodium bicarbonate is now added in excess and the solution of iodine run in, drop by drop, using starch to read the end reaction. (Sometimes the solution gets dark toward the end of the titration. This must not be confused with the final dark-blue color given by the iodine and starch.)

From the number of cc of iodine solution used and the weight of arsenious oxide taken, the value of each cc of iodine in arsenious oxide can be determined.

Two grams of Paris Green are weighed out and transferred to a 250 cc flask and about 100 cc of water and 2 grams of sodium hydrate added. This solution is boiled for 5 to 10 minutes, or until all of the green particles have changed to red cuprous oxide. It is then cooled to room temperature and the volume made to 250 cc. The well-shaken liquid is filtered through a dry filter and 50 cc taken for analysis. The analysis is carried out from this point forward the same as when we standardize the iodine solution.

Copper oxide.—The cuprous oxide obtained in the above method by boiling the Paris Green with sodium hydroxide is poured on the filter (after taking an aliquot portion of the solution for the determination of arsenious oxide) and well washed with hot water. It is then dissolved with hot dilute nitric acid and made to a volume of 250 cc, one-fifth being taken for analysis. The copper in solution is determined either by means of the galvanic current or, when that is not handy, in the following manner: The nitric-acid solution is made alkaline with sodium carbonate, then made *slightly* acid with acetic acid, and about ten times the weight of the copper in potassium iodide added. When it is all dissolved, the free iodine is titrated with a standard solution of sodium thiosulphate, using starch as indicator.^a

Soluble arsenious oxide.—In making the determination of this constituent two methods of analysis were used, namely, the water extraction method and the sodium acetate extraction method.^b

The first of these methods is carried out as follows: Treat 1 gram of Paris Green with 1 liter of water (previously boiled to get rid of CO₂ and again cooled to room temperature) in a large flask. The flask is stoppered and shaken five times each day for 10 days. At the end of this time the solution is filtered off through a dry filter. Two hundred cc of this is treated with sodium bicarbonate and titrated with standard iodine. This gives the amount of arsenious oxide dissolved. Because of the fact that water breaks up Paris Green on standing in contact with it, some copper oxide always goes into solution at the same time as the arsenious oxide does. This was also determined

^aFor the details of this method, see Sutton's Volumetric Analysis, 3d ed., p. 133.

^bSee report of the associate referee in the Proceedings of the Association of Official Agricultural Chemists for 1901.

by the following method in the hope that at some future time the relation between the copper oxid dissolved and the Paris Green broken up might be determined: Another 200 cc of the above solution is treated with 5 cc of hydrochloric acid, heated to 70° C., and a current of hydrogen sulphid passed through until all arsenic and copper are precipitated. It is then filtered and washed quickly with hot water. The filter and its contents are burned in a porcelain crucible with powdered sulphur and weighed. In this way a mixture of CuO and Cu_2S is obtained. To calculate the amount of CuO dissolved, consider the residue as entirely made up of CuO . This is correct, since the percentage of copper in CuO and Cu_2S is the same.

The second or sodium-acetate-extraction method for determining the free arsenious oxid in Paris Green is made as follows: Digest over the flame 1 gram of Paris Green for about 5 minutes with 25 cc. of a solution of sodium acetate containing 12.5 grams of the crystallized salt. The solution is then cooled, made up to 100 cc. and 50 cc. filtered off and titrated with standard iodine in the usual way.^a

It will not be out of place at this point to discuss briefly these two methods of analysis. It appears from the work that has been done on the subject that the sodium-acetate method gives more closely the true percentage of free arsenious oxid in the sample of Paris Green, while the water-extraction method gives the percentage of free arsenious oxid in the green plus some arsenious oxid obtained by the decomposing action of the water on the Paris Green. In some very coarse samples of Paris Green, which are supposed not to be made as well as the finer samples, the author found recently that the sodium-acetate-extraction method gave very low figures for free arsenious oxid (showing that only a small amount of free arsenious oxid as such was present), while the water-extraction method gave very high figures (seeming to show that although only a small amount of arsenious oxid was present in the free state, there was present a certain portion of the green, which was in a very loose combination, and consequently was easily broken up). That portion of the green which was in such a loose combination would, in all likelihood, when applied to the plant soon break up and scorch the foliage. It therefore appears that while the sodium-acetate-extraction method, as before mentioned, gives more closely the actual percentage of free arsenious oxid present in the green, the water-extraction method gives some idea of its stability, and consequently will express more closely the value of the compound in actual orchard practice. It has recently been called to the author's attention by Mr. C. L. Marlatt, Assistant Chief of the Division of Entomology, that samples of Paris Green which do not scorch may be made to do so by grinding them up to a very fine powder and then applying them to

^aSee paper of Avery and Beans, Jour. of the Amer. Chem. Society, Vol. XXIII, No. 2.

the tree. This might be expected from the work done by Avery and Beans, which shows that for any particular green the finer the green the more arsenious oxid goes into solution in water in a given length of time. It is, therefore, apparent that the water-extraction method for determining free arsenious oxid does not distinguish between the arsenious oxid set free from a badly made sample of Paris Green and the arsenious oxid set free from a very fine sample. Too much arsenious oxid from either of these causes, however, is objectionable, although, perhaps, not equally so. The finely made greens stay in suspension in water much better than the coarser greens, and are, therefore, better adapted for spraying purposes, although, of course, there is a safety limit, above which the arsenious oxid extracted by water should not run for these as well as for the coarser greens. What constitutes the best Paris Green, then, for spraying purposes is a very fine sample which will stay in suspension in water but will not break up to a great extent upon standing in contact with it. Such greens as are mentioned can be made, since a number of those above analyzed were very fine, yet gave a very low figure for soluble arsenious oxid by the water-extraction method.

The amount of free arsenious oxid allowable in Paris greens varies somewhat in different climates, but in the East, generally, and in California 4 per cent is considered the maximum amount allowable. In Idaho^a 6 per cent has been adopted as the maximum amount. In all cases the 7-day or 10-day water-extraction method is the one to be employed in making determinations of this constituent. In this connection it may be stated that 4 per cent was adopted in the East as the maximum amount of free arsenious oxid allowable in Paris Green when the extraction with water was limited to 24 to 48 hours, but recent work has shown that all of the free arsenious oxid does not go into solution in this time, and that very likely where the old method gave from 3 to 4 per cent the new method would give at least 5 to 6 per cent. It is the opinion of the writer, then, that more work should be done upon this subject, using more modern methods of analysis, and that until such work is done the 6 per cent adopted by the Idaho station gives more closely the safety limit of the green (when a 10-day extraction is used for analysis and when lime is mixed with the green before using) than does the 4 per cent used in California and the East. The climatic conditions in different parts of the United States would most likely change this figure somewhat for different localities.

Again, while considering this point it seems, at any rate from a theoretical standpoint, that in preventing the scorching of foliage by adding lime to Paris Green it would be much better to mix the green, in suspension in water, with the lime about 10 days before use and stir the mixture occasionally, since the free arsenious oxid does not go

^aSee Bul. No. 25, Idaho Agricultural Experiment Station.

into solution in much less time than this, and consequently is not acted upon as well by the lime to form the less harmful compound calcium arsenite.

ANALYSES AND DISCUSSION.

Table II, following, gives the composition of forty-five samples of Paris Green:

TABLE II.—*Composition of Paris greens.*

Serial number.	Moisture.	Sand.	Sodium sulphate.	Total arsenious oxid.	Copper oxid.	Acetic acid by difference.	Soluble arsenious oxid by water-extraction.	Soluble copper oxid by water-extraction.	Soluble arsenious oxid by sodium-acetate extraction.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
19525.....	0.37	0.08	1.72	58.15	29.88	9.80	4.56	1.00	0.98
19526.....	.33	.08	.72	56.45	30.71	11.71	9.30	1.90	.88
19532.....	.37	.05	.87	57.05	30.29	11.37	4.56	1.00	.98
19533 ^a	.23	.05	.51	56.20	30.71	12.30	8.81	1.45	1.17
19534.....	.99			57.54	29.79		4.86		
19614.....	.70	.09	1.98	58.03	29.04	10.16	4.56	.90	.98
19616.....	1.24	.09	.83	58.03	29.15	10.66	4.80	.80	.68
19610.....	.80	.06	.69	58.03	30.09	10.33	3.84	.85	.98
19613.....	.92	.06	1.44	57.54	29.46	10.58	5.28	.70	1.37
19624.....	.72	.07	1.66	58.15	30.09	9.31	4.41	1.05	1.08
19668.....	.76	.06	2.26	57.42	28.83	10.67	4.80	.65	1.86
19669.....	.50	.16	3.59	58.27	28.83	8.65	4.32	.55	1.17
19670.....	.83	.12	.97	57.78	29.88	10.42	4.56	.75	1.17
19671.....	.66	.09	2.05	61.61	29.15	6.44	2.16	.90	1.08
19672.....	.74	.08	1.16	57.29	29.88	10.85	2.88	.45	.88
19673.....	1.01	.09	.93	57.91	30.09	9.97	5.52	.95	1.17
19711.....	.57	.05	.98	57.18	30.09	11.13	5.81	.80	1.27
19712.....	.60	.06	.45	61.19	27.58	10.12	10.53	.60	8.91
19713.....	.67	.04	.40	57.66	29.88	11.35	4.60	.70	1.08
19715.....	.84	.03	.78	57.54	30.50	10.31	4.60	.60	1.08
19716.....	.73	.06	.40	57.42	30.50	10.89	4.11	.65	1.17
19717.....	.84	.04	.80	57.42	30.29	10.61	3.63	.50	1.17
19718.....	.43	.06	1.10	56.93	31.13	10.35	6.37	.65	1.76
19719.....	.81	.04	1.85	57.05	29.32	10.93	5.88	.55	1.57
19720.....	.48	.08	1.42	57.42	29.71	10.89	3.92	.55	.88
19721.....	1.06	.08	.97	56.93	30.70	10.26	5.57	.90	1.27
19722.....	.63	.09	.80	57.42	30.11	10.95	5.39	.85	.98
19723.....	.61	.12	.94	57.42	29.71	11.20	5.88	1.05	1.37
19724.....	.62	.12	.78	57.66	30.11	10.71	3.14	.70	.98
19725.....	.56	.13	.98	57.18	29.52	11.63	6.12	.75	1.47
19726.....	.97	.07	.98	58.15	29.32	10.51	5.64	.65	3.13
20359.....	1.08	.23	.95	57.02	29.91	10.81	3.19	1.65	1.08
20360.....	.90	.09	1.86	58.70	30.50	7.95	4.45	1.05	1.47
20361.....	1.09	.08	1.09	57.38	30.30	10.06	4.00	.90	1.17
20362.....	.98	.06	1.12	58.82	30.11	8.91	4.39	.90	.88
20363.....	1.02	.02	.64	58.96	29.71	9.65	4.00	.75	.88
20364.....	1.00	.05	.77	57.52	30.11	10.55	4.41	.80	.88
20365.....	.84	.20	1.47	56.67	29.52	11.30	6.01	1.50	1.37
20366.....	.73	.06	1.91	56.79	29.32	11.19	4.16	.45	1.37
20367 (lost).....									
20368.....	.59	.05	.82	59.68	28.53	10.33	9.79	.85	6.37
20372.....	.70	.06	.42	58.96	30.50	9.36	4.09	.95	.78
22282.....	.90	.00	.35	56.80	30.76	11.19	2.69	.55	.80
22283.....	.71	.87	1.02	57.05	29.77	10.58	2.81		.98
22284.....	.72	.00	1.26	57.78	30.86	9.38	2.93		.88
22285.....	.94	.00	1.06	57.54	31.16	9.30	2.93		1.03

^aSee correspondence with manufacturers, page 61.

On examining the analyses in Table II, it will at once be seen that neither the amount of moisture nor sand is in any case sufficient to be objectionable.

As to sodium sulphate, which is always present in samples of Paris Green on account of the method of manufacture, there is no reason why a sample which had been at all well washed should have more than 1 per cent present. The table shows that 16 of the samples of Paris Green have between 1 and 2 per cent, 2 have between 2 and 3

per cent, and 1 has between 3 and 4 per cent, making a total of 19, or 43 per cent, which have more sodium sulphate than should be allowable. Of course, it is understood that this compound does not injure the green in any way for the purposes intended, but adds weight, and causes a high price for a cheaper article.

The figures for total arsenious oxid show that no green contains less than 56.20 per cent. There are 7 greens containing between 56 and 57 per cent, 24 containing between 57 and 58 per cent, 11 containing between 58 and 59 per cent, and the remaining 3 containing between 59 and 62 per cent. These figures would seem to indicate that there is no reason for a sample of Paris Green containing less than 56 per cent of total arsenious oxid, and that the States having laws regarding this subject should change them so as to require the presence of 56 instead of 50 per cent of arsenious oxid.

The figures for copper oxid vary from a minimum of 27.58 to a maximum of 31.16 per cent. More than half of the samples contain between 30 and 31 per cent.

If the Eastern and California standard of 4 per cent be adopted for free arsenious oxid, and the 10-day water-extraction method be used, there are only 13 samples, or 29 per cent, of the above Paris greens that would pass, but if the 6 per cent limit, which we consider the better, be used, 38 samples, or 84 per cent, of the greens would be accepted. Of the others, 3 samples contain between 6 and 7 per cent, 1 sample between 3 and 9 per cent, 2 samples between 9 and 10 per cent, and 1 sample 10.53 per cent.

A column of figures is given, representing the amount of copper oxid going into solution along with arsenious oxid when the green is treated with water for 10 days. At present we do not know the relation between the amount of copper oxid thus extracted and the amount of Paris Green broken up, so these figures can not be used to correct the soluble arsenious oxid for the amount corresponding to Paris Green broken up. These figures do, however, to some extent, seem to indicate which samples give high figures for arsenious oxid because of the breaking up of the Paris Green, and which give high figures because of the presence of free arsenious oxid in the original sample. Hence, the copper oxid determination seems to help us judge of the stability of the green in question. For example, we would say that samples 19526, 19533, 20359, and 20365 give high figures for free arsenious oxid because the green is unstable or is very fine, while samples 19712, 19718, 19726, and 20368 give high figures because the green contains more free arsenious oxid originally than the average sample. This course of reasoning is to some extent justified by the figures obtained by the sodium-acetate-extraction method, which is supposed to indicate more or less closely the amount of free arsenious oxid originally present in the green. For samples 19526, 19533, 20359, and 20365 the

sodium-acetate-extraction figures are only 0.88 per cent, 1.17 per cent, 1.08 per cent, and 1.37 per cent, while the sodium-acetate-extraction figures for samples 19712, 19718, 19726, and 20368 are 8.91 per cent, 1.76 per cent, 3.13 per cent, and 6.37 per cent, showing that the first four samples did not contain originally any more free arsenious oxid than an average sample, while the second four samples did contain more arsenious oxid than the average, with possibly one exception. Although this course of reasoning may not apply to all Paris greens, it is at least suggestive, and consequently is worthy of note.

The figures representing the soluble arsenious oxid by the sodium-acetate-extraction method seem to show that most samples of Paris Green really contain very little arsenious oxid as such, and that in the great majority of cases where an amount of free arsenious oxid manifestly above the average is shown by this method these samples are either above or approaching to the safety limit of 6 per cent, as shown by the water-extraction method. Examples of this are samples 19613, 19712, 19718, 19719, 19723, 19725, 19726, 20360, 20365, and 20368, while exceptions are samples 19668 and 20366.

LONDON PURPLES.

In analyzing samples of London Purple the following determinations were made: Moisture, sand, total arsenious oxid, total arsenic oxid, calcium oxid, soluble arsenious oxid, soluble arsenic oxid, and soluble calcium oxid. The methods follow.^a The dye is determined by difference.

METHODS OF ANALYSIS AND DISCUSSION.

Moisture.—Dry 1 to 2 grams at 100° C. for from 12 to 14 hours. Report loss as moisture.

Sand.—Use sample from moisture determination. Dissolve in hydrochloric acid; filter, wash, dry, and burn, calculating residue as sand.

Total arsenious oxid.—Two grams of London Purple are dissolved in about 80 cc of water and 20 cc of hydrochloric acid at a temperature of from 60 to 70° C. (not higher for fear of driving off arsenious chlorid), filtered and washed to a volume of 300 cc. One hundred cc of this is treated in a 500 cc flask, with sodium bicarbonate in excess. The contents of the flask is then brought to the mark with water, using a few drops of ether to destroy bubbles, and 250 cc filtered off. To this is added starch solution, and the standard iodine solution,^b until the blue color appears. The result is the arsenious oxid, as such, in 50 cc of the original solution, that is, in 0.3333 gram.

^aSee article by Haywood, Jour. of the Amer. Chem. Soc., Vol. XXII, No. 12.

^bThe standard iodine used is prepared in the same manner as that used in the analysis of Paris Green.

Total arsenic oxid.—Fifty cc of the hydrochloric acid solution (representing 0.3333 gram), spoken of above, is heated to 80° C. on the water bath and then taken off, and 50 cc of hydrochloric acid and 3 grams of potassium iodid added. The mixture is allowed to stand for at least 15 minutes, the “ic” arsenic thus being reduced to “ous” arsenic by the action of the potassium iodid in acid solution, iodine being set free. The solution is then rinsed out in a large beaker, diluted, and tenth-normal sodium thiosulphate added, drop by drop, to get rid of the iodine. The end-point here is rather difficult to read on account of the very dark color of the solution, but with a little practice one can determine it very easily by proceeding as follows:

The sodium thiosulphate is run in a little at a time and occasionally a drop of the solution is added to a drop of starch paste. This will, of course, give a blue color with the starch, which becomes fainter and fainter as the iodine is used up. Finally, when a drop of the solution only gives the slightest blue color with the starch, a little starch paste is added directly to the whole solution, and the blue color dissipated with a few drops of thiosulphate. With a little practice one can in this way get the exact end-point every time. The solution is immediately made alkaline with solid sodium carbonate. It is again made slightly acid with hydrochloric acid, taking care that all of the solid particles of the sodium carbonate on the bottom are neutralized by the acid, and finally made alkaline with sodium bicarbonate. Starch paste and tenth-normal iodine are now added until the blue color appears. This end-point is easily read if the beaker is placed on a white surface between the eye and the light and iodine run in until a distinct purple color appears. The figure thus obtained gives the total amount of arsenic in the solution as arsenious oxid. Subtracting the figure obtained in the determination of arsenious oxid above from this we have the amount of standard iodine corresponding to arsenic oxid in 50 cc of the original liquid.

Calcium oxid.—A portion of London Purple is dissolved in hydrochloric acid (an aliquot portion of the 300 cc used in the determination of arsenious and arsenic oxides will do) and hydrogen sulphid passed through. The precipitate is well washed, the filtrate is evaporated to small bulk and transferred to a 200 cc flask, when it is treated with ammonia (to precipitate the iron, etc.,) and made to the mark. A 100 cc portion of this is filtered off and treated with ammonium oxalate in the usual way.

Soluble arsenious oxid.—Extract 1 gram of the sample for 10 days with 500 cc of carbon-dioxid free water at room temperature, shaking occasionally each day. Pass through a dry filter and use 100 cc for analysis. Add sodium bicarbonate and starch paste and titrate to the appearance of a blue color. This will give a number of cc of standard iodine corresponding to the arsenious oxid in 0.2 gram of the sample.

Soluble arsenic oxid.—An aliquot portion of the liquid from the method above, for soluble arsenious oxid (say 200 cc) is transferred to a flask, made slightly alkaline with NaOH, and evaporated to about 25 cc on a hot plate. The flask is then removed and allowed to cool to about 80° C., and an equal volume of concentrated hydrochloric acid and 3 grams of potassium iodid added. It is allowed to stand 15 minutes, the iodine set free, exactly used up with tenth-normal thio-sulphate, using starch if necessary, and the solution neutralized with sodium carbonate. It is again made slightly acid with hydrochloric acid, taking care that all lumps of sodium carbonate are acted on, then made alkaline with an excess of sodium bicarbonate, and titrated with iodine, using starch as an indicator. From this figure is subtracted the number of cubic centimeters of standard iodine corresponding to the soluble arsenious oxid in 200 cc of the filtrate, and the remainder is calculated as arsenic oxid.

Soluble calcium oxid.—One hundred cc of the liquid from the method above for soluble arsenious oxid is treated with hydrochloric acid and hydrogen sulphid passed through. The solution is filtered, ammonia added, and the solution filtered again. Calcium is determined in the final filtrate by means of ammonium oxalate in the ordinary manner.

ANALYSES AND DISCUSSION.

Table III, following, gives the composition of 11 samples of London Purple:

TABLE III.—Composition of London purples.

Serial number.	Moisture.	Sand.	Total arsenious oxid.	Total arsenic oxid.	Calcium oxid.	Dye by difference.	Soluble arsenious oxid.	Soluble arsenic oxid.	Soluble calcium oxid.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
19527.....	1.87	3.54	8.16	33.60	25.09	27.74	2.43	15.81	7.80
19556.....	4.07	2.61	17.31	26.50	23.59	25.92	13.66	7.12	6.60
19615.....	2.76	2.46	10.47	32.85	24.55	26.91	3.88	12.56	7.00
19618.....	2.73	3.55	6.40	35.62	25.03	26.67	1.44	19.56	10.80
19674.....	3.77	3.26	6.98	35.79	26.41	23.79	2.67	14.96	
19727.....	2.50	2.92	4.94	38.80	23.41	27.43	.96	28.51	13.20
19840.....	3.02	3.12	3.50	40.40	23.82	26.14	.96	18.17	7.20
19841.....	3.58	2.89	17.50	26.60	24.38	25.05	12.03	5.03	4.50
19842.....	4.90	1.36	16.33	26.55	24.38	26.48	10.83	5.03	4.30
19843.....	2.93	9.74	8.17	27.09	22.45	29.62	6.50	6.99	5.00
19844.....	2.81	3.05	3.80	38.53	24.19	27.62	.96	16.49	8.00

The columns in Table III representing moisture and sand show that these two substances vary greatly in different samples of London purples. The former varies from 1.87 to 4.90 per cent and the latter from 1.36 to 9.74 per cent. It would be natural to expect some variation in an article like London Purple, which is obtained as a by-product in the dye industry. No fault can be found on this score with any of the samples analyzed, with the exception of sample 19843, which contains so much sand as to suggest that perhaps this substance has been added to gain weight, or that, at least, the sample has been very carelessly handled.

Up to a very recent date it was generally supposed that arsenic was present in London Purple in the form of calcium arsenite. During the course of this investigation, however, it was found that the arsenic is present both as calcium arsenite and calcium arsenate. The amount of these two substances varies very much, but where both the arsenious and arsenic oxids are calculated to arsenic it is found that the percentage of this constituent is fairly constant in different samples. For example, the percentage of arsenic in the samples above taken in serial order is as follows: 28.09, 30.39, 29.35, 28.07, 28.63, 29.04, 28.99, 30.59, 29.68, 24.55, and 28.00. Sample 19843 is the only one low in arsenic, and this is because it contains the large amount of sand previously spoken of.

As to the amounts of calcium oxid and dye, these two substances vary in the samples above about as much as might be expected, with the possible exception of 19843, which contains more dye and less calcium oxid than the average sample. As a whole, all of the samples are of about the same strength with the exception of 19843, which is somewhat weaker.

In the consideration of the soluble portion of London Purple a surprise awaits the investigator. According to the old method of considering that London Purple was composed mainly of calcium arsenite and only determining the arsenious oxid in a water extract, 7 of the above samples would have been considered as excellent for spraying purposes, since they contained only very small quantities of soluble arsenious oxid, but on looking at the figures for soluble arsenic oxid, it will be seen that wherever a sample only contains a small quantity of soluble arsenious oxid it contains a very large quantity of soluble arsenic oxid, so that the total amount of arsenic that is dissolved from any sample of London Purple is very great. This perhaps explains the unaccountable manner in which London Purple often scorches the foliage when an analysis has shown that only a small quantity of arsenious oxid is present. But even though London Purple has often unaccountably scorched foliage, there are many cases where it has been used with most excellent results. The only conclusion that can be drawn from this (if we consider that the above 11 samples fairly represent the London Purple as sold on the American market, and there is no reason to believe that they do not) is that plants can stand a vast amount more soluble arsenic than we have before thought possible. It is highly probable that if all of this soluble arsenic were present as free arsenious or arsenic acid *all* plants would be seriously burned, if not entirely defoliated, by applying such a substance, but where a large part of the soluble arsenic is present as the calcium salts of arsenious and arsenic acid, as shown by the fact that large amounts of lime also go into solution, it appears that this alters the case and that plants can endure much more of these salts than they can of the free acids. Work along this line is much needed.

Just at this point it might be well to mention one function that lime perhaps performs when used with Paris Green to prevent scorching of the foliage. It is known, of course, that lime is added to change the free arsenious oxid in the green to the less harmful salt calcium arsenite, but it also seems probable that lime has a second function. As the Paris Green is gradually broken up by the action of dew and rain, both charged with carbon dioxid, lime would form with the arsenious oxid, set free the more insoluble and less harmful compound calcium arsenite, and thus prevent scorching of the foliage. Even when the lime had finally all changed to calcium carbonate by the action of carbon dioxid in the air, when water containing carbon dioxid came in contact with it, it would render enough of the carbonate soluble as the bicarbonate to act upon the arsenious oxid set free from the Paris Green and form calcium arsenite.

INSECTICIDES AND FUNGICIDES, OTHER THAN PARIS GREEN AND LONDON PURPLE, THAT CONTAIN ARSENIC OR COPPER, OR BOTH, AS THEIR ACTIVE CONSTITUENT.

The methods of analysis for this group of compounds are so many and diverse that but a few general principles applying to most of the mixtures coming under this heading can be given.

METHODS OF ANALYSIS AND DISCUSSION.

The methods of analysis of Green Arsenoid and Scheele Green are the same as those described for Paris Green. For White Arsenoid, the same methods are used as described in connection with London Purple, except that the barium is determined by precipitating from hydrochloric-acid solution with sulphuric acid. For Rough on Rats some of the London-Purple methods are also used. For those compounds which contain arsenic, copper, calcium, magnesium, iron, and sulphur trioxid the following methods are used:

Copper oxid.—Dissolve the compound in hydrochloric acid and filter; heat the solution to 70°C . and pass hydrogen sulphid through until all copper sulphid and arsenious sulphid are precipitated; wash quickly with hot water, dry, and finally burn in a weighed porcelain crucible; add some powdered sulphur, cover crucible, and burn again; finally, weigh the copper as the mixed subsulphid and oxid. The percentage of copper being the same in both, the whole may be considered as copper oxid.

Arsenious oxid.—Proceed just as in the determination of copper down to the point where the arsenious and copper sulphids have been washed with hot water (taking the extra precaution, however, of not using too high a degree of heat in getting the original substance in solution, for fear of driving off arsenious chlorid); spread the filter out and transfer

the whole to a 200 cc. flask by means of a jet of hot water. This transference, with care, can be made very complete. Add sodium sulphid and digest on the water bath until all arsenious sulphid has gone into solution, leaving the copper sulphid behind; cool, make to the mark, mix thoroughly, and filter off 100 cc. through a dry filter, rejecting the first few cc. The solution thus obtained is warmed, and made acid with hydrochloric acid, which reprecipitates the arsenious sulphid and a large amount of free sulphur; filter and wash; extract the still moist precipitate on the filter with ammonia; wash the residual sulphur; reprecipitate the filtrate with hydrochloric acid, without heat; filter in a Gooch crucible; wash; dry at $100^{\circ}\text{C}.$; extract the remaining free sulphur with carbon disulphid; dry at $100^{\circ}\text{C}.$, and weigh; repeat extraction and drying until constant weight is obtained. From the arsenious sulphid compute arsenious oxid. While this method leaves much to be desired, it is the best we could find to work on mixtures of this kind. The old method, as described in many text-books, of treating the mixed sulphids of copper and arsenic with sodium sulphid and filtering the entire arsenic solution thus obtained from the copper sulphid left behind did not work in the hands of the author, since in most cases the copper sulphid would commence to run through the filter directly after washing was begun.

Iron.—The iron in these compounds is determined by precipitating with ammonia directly in the presence of arsenic and copper.

Calcium and magnesium.—These two substances are determined by first precipitating out the copper and arsenic with hydrogen sulphid, then precipitating the iron with ammonia, and finally determining calcium by means of ammonium oxalate in the usual way, and magnesium in the filtrate by means of sodium phosphate in the ordinary manner.

Sulphur trioxid.—This constituent is determined as barium sulphate by precipitating sulphur trioxid from the hydrochloric acid solution of the substance in the ordinary manner.

In determining the lead and arsenic oxids in "Swift's Lead Arsenate," the compound is first dissolved in fuming nitric acid to get rid of all organic matter. It is then evaporated to drive off most of the acid and transferred to a measuring flask, where it is made to a volume, aliquot portions of which are taken for analysis. Sulphuric acid is added to one portion and the sample evaporated on the steam bath till the odor of nitric acid ceases to come off. It is now diluted with water and filtered, washing with water slightly acidified with sulphuric acid, till the precipitate is free of arsenic. This filtrate is saved. The filter and contents are washed with 60 per cent alcohol till free of sulphuric acid, dried, and finally burned and weighed as lead sulphate, using the usual precautions mentioned for this compound. To the filtrate add ammonia till slightly alkaline and magnesia mixture in the usual man-

ner, and finally weigh the arsenic acid as magnesium pyro arsenate, after having ignited with the precautions mentioned in Fresenius. The results will be slightly low in both cases.

GREEN ARSENOID,^a

[Serial No. 19625.]

Composition of Green Arsenoid.

	Per cent.
Moisture.....	2.56
Sand	1.30
Sodium sulphate	2.02
Arsenious oxid.....	61.43
Copper oxid	29.67
Blue coloring matter (by difference).....	3.02
	100.00
Arsenious oxid, soluble by water extraction.....	5.88
Copper oxid, soluble by water extraction	.50

This sample has as much copper oxid as the ordinary sample of Paris Green and even more arsenious oxid, so that it is as strong or stronger in those constituents which kill insects.

It is high in both sand and sodium sulphate, faults which could easily be remedied by more careful handling and more washing.

As to the free arsenious oxid, this is above the standard used in California and the East, but below the 6 per cent standard. Since this compound has been used in practical experiments with good results by a number of the experiment stations, this is only one more point in favor of adopting the 6 per cent rather than the 4 per cent limit for free arsenious oxid in Paris greens and other compounds.

SCHEELÉ GREEN.

[Serial No. 19535.]

Composition of Scheele Green.

	Per cent.
Hygroscopic moisture	5.27
Water of constitution that should be present to form	
Cu HAs O ₃	4.32
Arsenious oxid.....	51.33
Copper oxid	38.14
	99.06
Arsenious oxid extracted by water in 10 days	15.00
Arsenious oxid extracted by water in 36 days	17.70

^aSee correspondence with manufacturers, page 59.

Since Green Arsenoid is often spoken of as Scheele Green in publications by entomologists, it might be as well to call attention to the fact that these two compounds are not identical. Scheele Green is copper hydrogen arsenite and contains less arsenious oxid and more copper oxid than Paris Green, while Green Arsenoid is one of the copper arsenites not containing any hydrogen, but being richer in arsenious oxid than Paris Green and containing about the same amount of copper oxid. It is this neutral arsenite that has been recommended by the Department of Agriculture.

Although this sample contains large quantities of arsenious and copper oxids, it is worthless as an insecticide, since it contains such large quantities of soluble arsenious oxid.

PARAGRENE.

[Serial No. 19714.]

Composition of Paragrene.

	Per cent.
Sand	0.25
Water of crystallization and hygroscopic moisture.....	10.05
Ferric oxid	.20
Calcium oxid	12.13
Copper oxid	20.50
Sulphur trioxid	16.15
Arsenious oxid.....	33.14
Acetic acid.....	6.66
	<hr/>
	99.08
Arsenious oxid soluble by water extraction	6.12
Copper oxid soluble by water extraction.....	.90

This compound consists essentially of a mixture of Paris Green with about 35 to 36 per cent gypsum. The principal use of the gypsum appears to be to weaken the compound and add weight. It may also be of value in mitigating the injurious effects of the free arsenious acid. Since this compound contains even more than the 6 per cent limit of arsenious oxid, it can not be recommended as a high-grade insecticide. If the amount of soluble arsenious oxid could only be reduced to a slight extent this would doubtless be a good insecticide, since it contains enough poison to kill insects; it is at the same time in a very fine condition and will consequently stay in suspension in water better than the ordinary sample of Paris Green.

LAUREL GREEN.^a

[Serial No. 19617.]

Composition of Laurel Green.

	Per cent.
Sand	0.49
Ferric oxid	3.25
Copper oxid	12.53
Calcium oxid	27.50
Magnesium oxid	2.29
Arsenious oxid.....	6.92
Sulphur trioxid	24.65
Carbon dioxid	5.73
Water of crystallization, composition, and hygroscopic (by difference).....	16.64
	<hr/>
	100.00

This substance consists of large amounts of gypsum, calcium carbonate, and magnesium carbonate, amounting in all to about 65 per

^aSee correspondence with manufacturers, page 62.

cent, along with about 13 per cent calcium arsenite, 12.53 per cent copper oxid, and small amounts of other substances. It is of value as an insecticide as far as its 13 per cent of calcium arsenite is concerned. As to the copper, this seems to be present entirely as copper oxid. A full discussion of why copper in this condition can be of little or no value as a fungicide is given in Farmers' Bulletin 146 of this Department.

BORDEAUX MIXTURE AND PARIS GREEN.

[Serial No. 19675.]

Composition of Bordeaux Mixture and Paris Green.

	Per cent.
Carbon	0.96
Sand	0.41
Arsenious oxid	25.86
Sulphur trioxid	6.78
Carbon dioxid	9.48
Acetic acid	3.34
Copper oxid	16.93
Calcium oxid	24.00
Magnesium oxid	1.90
Water of crystallization, composition, and hygroscopic (by difference)	10.34
	<hr/> 100.00
Arsenious oxid by water extraction	6.86
Copper oxid by water extraction	0.40

This compound consists of a mixture of about 40 per cent Paris Green and 60 per cent of the ingredients used in making Bordeaux Mixture.^a

The 6.86 per cent of soluble arsenious oxid at once condemns this compound for all spraying purposes.

NORTH STATE INSECTICIDE.

[Serial No. 22291.]

Composition of North State Insecticide.

	Per cent.
Insoluble in hydrochloric acid	24.91
Copper oxid	12.43
Ferric oxid and alumina	13.98
Calcium oxid	9.07
Arsenious oxid	20.32
Sulphur trioxid	12.95
Acetic acid and water (by difference)	6.34
	<hr/> 100.00

This compound is of value as an insecticide as far as its content of Paris Green, amounting to about 36 per cent, is concerned. The remainder, consisting of calcium sulphate, sand, ferric oxid. and alumina, is inert in its action on insects.

^a For a discussion of the causes why Dry Bordeaux Mixture thus prepared can in all likelihood never be a success, see Farmers' Bulletin No. 146, of this Department.

SLUG SHOT.^a

[Serial No. 19524.]

Composition of Slug Shot.

	Per cent.
Water of crystallization and hygroscopic.....	18.37
Sand	4.09
Sulphur trioxid	39.65
Calcium oxid	27.75
Copper oxid	.58
Arsenious oxid	1.58
Ferric oxid	1.98
Sulphur and carbolic acid.....	4.00
Tobacco (by difference)	2.00
	<hr/> 100.00

The sulphur, carbolic acid, tobacco, arsenious oxid, and copper oxid, in so far as they go (about 8 per cent) are of value as either insecticides or fungicides, but the remainder is inert in its action on insects. It is possible that the gypsum present in this compound is used to act as a mitigating agent upon the active substances mentioned above.

BLACK DEATH.

[Serial No. 19846.]

Composition of Black Death.

	Per cent.
Carbon	17.11
Sand	5.86
Moisture.....	10.18
Carbon dioxid	12.39
Sulphur trioxid	23.15
Arsenious oxid.....	.97
Copper oxid	.59
Ferric oxid	.46
Calcium oxid	24.97
Magnesium oxid	5.03
	<hr/> 100.71

The only substances present in this compound which have any value as insecticides are copper oxid and arsenious oxid, and these are present in very small quantities. If mixtures containing as small amounts of arsenious oxid and copper oxid as this will kill insects, it would be much better for the consumer to buy a pound of Paris Green and mix it with about 75 pounds of gypsum, thus obtaining 76 pounds of the preparation at a cost of about 50 cents, while if it was bought already prepared, it would cost retail about \$3.80.

^aSee correspondence with manufacturers, page 61.

SWIFT'S LEAD ARSENATE.

[Serial No. 19626.]

Composition of Swift's Lead Arsenate.

	Per cent.
Moisture.....	1.39
Lead oxid ^a	58.90
Arsenic oxid ^a	25.62
Organic.....	13.00
	<hr/> 98.91

The organic portion of this substance consists of dextros and dextrin, showing that glucose sirup has been added to cause the lead arsenate to adhere to the foliage.

This compound is the least soluble of all the arsenical insecticides tested, and consequently is not liable to scorch the foliage. It has been used as an insecticide with good results.

WHITE ARSENOID.^b

[Serial No. 22287.]

Composition of White Arsenoid.

	Per cent.
Moisture	1.90
Sand	.38
Chlorine	4.46
Carbon dioxid	8.88
Arsenic oxid.....	1.13
Arsenious oxid.....	33.67
Sodium (joined with part of chlorin)	1.25
Barium (to join with part of the chlorin).....	4.89
Barium oxid.....	42.19
	<hr/> 98.75
Arsenious oxid dissolved by cold water.....	33.67
Large amount of barium salts also dissolved by cold water.	

The two compounds which give this substance any value as an insecticide are the arsenious and arsenic acids. Since, however, all of the arsenic is present in a soluble condition, an application of this mixture to the foliage would burn it badly, consequently the use of this insecticide can not be recommended. It is understood that the results from this mixture have been so unsatisfactory that it has been withdrawn from the market. One point in connection with White Arsenoid which has not been mentioned in any of the State experiment station reports on the subject is the probable toxic effect of barium in a soluble condition on plants sprayed with it.

^aThe methods for determining PbO and As₂O₅ give rather low results.

^bSee correspondence with manufacturers, page 59.

WHITE ARSENIC.

[Serial No. 22286.]

Composition of White Arsenic.

	Per cent.
Sand	0.04
Moisture.....	.12
Arsenious oxid.....	99.16
	<hr/> 99.32

This is an excellent full strength sample of White Arsenic, and could be used with good effect along with lime in preparing homemade calcium arsenite.

ROUGH ON RATS.^a

[Serial No. 20482.]

Composition of Rough on Rats.

	Per cent.
Fine sand.....	1.59
Moisture.....	.20
Arsenious oxid.....	71.83
Barium oxid.....	20.22
Carbon dioxid.....	5.81
	<hr/> 99.65

This mixture contains as its active poisonous constituents arsenious oxid and barium carbonate, and will be apt to serve the purposes for which it is intended. Great care should be taken not to leave any of this compound carelessly around a house, since it might be stirred up with the dust, breathed by persons, and finally cause arsenic poisoning; also, in the presence of organic matter, the same action might take place, as has been noted in the case of arsenical wall papers, resulting in the formation of a very poisonous gas, arsine.

ROYAL ROACH POWDER.

[Serial No. 22290.]

Composition of Royal Roach Powder.

	Per cent.
Borax	30.94
Arsenious oxid ^b	.73
Arsenic oxid ^b	4.69
Some calcium. ^b	
Some dyestuff. ^b	
The remainder appears to be Persian Insect Powder or pyrethrum.	

This mixture may be effective in killing and driving away roaches, ants, etc., but the advisability of scattering it around indiscriminately, as is usually done with roach powders, is very doubtful, since it contains arsenic, which might rise with the dust of the room, and

^aSee correspondence with manufacturers, page 62.^bCorresponding to about 12 to 13 per cent London Purple.

being breathed, cause arsenic poisoning; also, as in the case of Rough on Rats, the arsenic might under certain conditions be converted to arsine.

LEE'S INSECT POWDER.^a

[Serial No. 22292.]

Composition of Lee's Insect Powder.

	Per cent.
Sulphur	47.93
Arsenious oxid.....	5.00
Remainder appears to be tobacco and pyrethrum.	

This is another compound which may be effective in killing and driving away fleas, lice, croton bugs, etc., but as to the advisability of using it around human habitations the same may be said of it that has been said of Royal Roach Powder.

DRY BORDEAUX MIXTURE.

[Serial No. 19531.]

Composition of Dry Bordeaux Mixture.

	Per cent.
Sand	0.41
Carbon	1.05
Carbon dioxid	3.55
Copper oxid	15.46
Sulphur trioxid	15.00
Calcium oxid	30.12
Magnesium oxid	17.52
Water of constitution, crystallization, and hygroscopic (by difference).....	16.89
	<hr/> 100.00

This sample was originally prepared by using approximately equal portions of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and unslaked lime. The reasons why such a compound can hardly be successful have already been stated (p. 27).

HAMMOND'S COPPER SOLUTION.

[Serial No. 19688.]

Composition of Hammond's Copper Solution.

	Grams.
Ammonium hydroxid, per 100 cc.....	32.9060
Copper oxid, per 100 cc.....	5.5657
Sulphur trioxid, per 100 cc.....	1.1397

A considerable amount of carbon dioxid is also present. This solution was evidently made from strong ammonia, copper sulphate, and copper carbonate. The value of a mixture of this kind as a fungicide is well known and requires no further discussion.

^aSee correspondence with manufacturers, page 61,

COPPER SULPHATE.

[Serial No. 20369.]

Composition of Copper Sulphate.

	Per cent.
Sand	0.05
Copper oxid	31.48
Sulphur trioxid	32.13
Water of crystallization and hygroscopic.....	36.00
	<hr/>
	99.66

This compound is an excellent sample of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

GRAPE DUST.^a

[Serial No. 19690.]

Composition of Grape Dust.

	Per cent.
Sand	1.64
Sulphur	58.82
Copper oxid	.90
Calcium oxid	11.93
Sulphur trioxid	17.04
Water to form $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	7.67
Tobacco (by difference)	2.00
	<hr/>
	100.00

This substance contains sulphur and copper oxid probably in the form of copper sulphate, which is of value in treating surface mildew on the vine. The amount of tobacco is too small to be of any value. The gypsum adds weight, and serves as an agent to mitigate the action of the sulphur and copper sulphate.

SOAPS.

In making an analysis of this class of compounds, the following determinations were made: Moisture, total fatty matter, total alkali, free alkali, and in certain cases calcium oxid, neutral fat + unsaponifiable matter, and resin. The methods^b follow:

METHODS OF ANALYSIS AND DISCUSSION.

Moisture.—Weigh a 100 cc beaker containing about one-half inch of recently ignited sand and a small glass rod. Add about 5 grams of the sample, and weigh again. Dissolve the soap by means of 25 or

^aSee correspondence with manufacturers, page 60.

^bMost of the methods are from "Oils, Fats, and Waxes," by Benedikt and Lewkowitsch, with slight modifications.

more cc of strong alcohol on the water bath. If the soap is very hard it may be as well to leave it in contact with the alcohol overnight, so as to soften it. Evaporate off the alcohol and dry in the oven at $110^{\circ}\text{C}.$, either to constant weight or to the point where it only gains or loses a little during one-half hour extra drying.

Total fatty matter.—Five to 10 grams of the soap is dissolved in hot water in a beaker by means of gentle heat and constant stirring. When the liquid finally begins to boil, a few drops of methyl orange are added and dilute hydrochloric acid until the indicator shows an acid reaction. Continue the boiling and stirring until all fatty matter has collected in drops. Add about 5 grams of dry beeswax, accurately weighed on a weighed watch glass. Boil again till the fat collects on the top as a clear layer free of specks. Rinse off the rod and boil till the fat has again collected. Allow the contents of the beaker to cool until the fatty layer has entirely solidified. Remove this layer by means of a platinum spatula, wash with cold water, dry between filter papers, and transfer to the weighed watch glass previously spoken of. Gather together all particles of fat left behind by means of the platinum spatula, and when this is not possible by means of small weighed pieces of filter paper, add to the watch glass and contents, dry in a desiccator overnight, and weigh. The total weight thus obtained, minus the combined weights of the watch glass, beeswax, and pieces of filter paper, gives the weight of the total fatty matter. In the absence of resin, neutral fat, and unsaponifiable matter, all of this fatty matter is reported as fatty acids, but if any of the other three are present they are subtracted and the remainder reported as fatty acids, which multiplied by 0.965 gives fatty acid anhydrid.

Total alkali.—A weighed quantity of the soap is decomposed by hydrochloric acid and the water is filtered off from the fat, which is washed. Both potassium and sodium in the filtrate are first determined as the mixed chlorids in the ordinary manner, and the potassium then determined by means of platinum chlorid solution.

Free caustic alkali.—Dissolve 30 grams of the soap in strong or absolute alcohol. Filter rapidly by means of a hot-water funnel into a narrow-necked flask. Wash the filter with alcohol till clean. The filtrate is now titrated with N/10 hydrochloric acids, using phenol phthalein as indicator. In the case of all whale-oil soaps examined, an acid reaction was obtained at this point denoting free fatty acids.

Calcium oxid.—This was found to be present in one sample of soap and was determined by first acidifying a weighed quantity of soap in water solution with hydrochloric acid, just as in the determination of

potassium and sodium, washing the filter and contents, and precipitating the calcium out of the filtrate in the ordinary way with ammonia and ammonium oxalate.

Total neutral fat and unsaponifiable matter.—To determine these two substances, about 10 grams of the soap is weighed out and dissolved in alcohol. Fifty to 70 grams of acid washed, ignited sand is now added and the alcohol evaporated off. The mass is dried and transferred to a Soxhlet extractor, where it is extracted with ether. This extract contains neutral fat, unsaponifiable matter, and free fatty acids, which are weighed together. The free fatty acids are determined by means of standard alkali and phenol phthalein and subtracted from the other two.

Resin acids.—A rather large quantity of the soap is first dissolved in water and, after heating to boiling, enough dilute hydrochloric acid is added to set the fatty and resin acids free. The boiling is continued till the fat collects in oily drops. The vessel is cooled and the acid water poured off. The residue is again boiled with water, which is again poured off. The fatty matter now remaining is washed with cold water and transferred on a watch glass to a desiccator, where it is thoroughly dried. The resin acids are determined in a weighed quantity of this mixture by the Twitchell method. For this dissolve 2 or 3 grams of the mixture of acids prepared as above and place in a flask in about 10 times their volume of absolute alcohol. Pass a current of dry hydrochloric acid gas through the flask, which is immersed in ice water. Pass the hydrochloric acid through for about three-quarters of an hour, or until it is noticed that the gas is passing through the liquid unabsorbed. Set the loosely covered flask aside for one hour, dilute with 5 volumes of water, and boil until the water layer becomes clear. Transfer to a separatory funnel, washing out all the ethylic ethers and resin acids with ether. Shake well and run off the acid layer. Wash the etherial solution a number of times with pure water until all hydrochloric acid is removed. Now add 50 cc of alcohol and titrate the solution with standard potassium hydroxid, using phenol phthalein as indicator. The resin acids combine with the alkali and the ethylic ethers are practically not touched. To obtain the amount of resin, multiply the number of cc of normal alkali used by 0.346. One hundred parts of resin acids are equal to 97.32 parts of resin anhydrid.

ANALYSES AND DISCUSSION.

The table following (IV) shows the composition of various soaps:

TABLE IV.—*Composition of soaps.*

Serial No.	Name.	Moisture.	Fatty acid anhydride.	Neutral fat and unsaponifiable.	Resin acid anhydride.	Sodium oxid.	Potassium oxid.	Calcium oxid.	Total.	Free caustic alkali.
		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per cent.</i>
19557...	Whale-oil Soap, "Lion Brand"...	5.82	86.38			7.63			99.83	Acid reaction.
20455...	Whale-oil Soap ...	16.14	74.61			7.02			97.77	Do.
20456 ^a ...	do.	42.96	48.39				8.44		99.79	Do.
20457...	do.	13.68	73.27			8.52		1.06	96.53	Do.
20458 ^a ...	Permol Kerosene Soap	10.33	28.61	52.55		6.00			97.49	Do.
20459...	Whale-oil Soap	12.87	77.62			7.04			97.53	Do.
20460...	J. & M. Reliable Insecticide.....	34.30	25.30		34.67	2.92	1.82		99.01	Do.
20461...	Stott's Fir Tree Soap	26.00	12.06	49.21		5.05	3.03		95.35	0.22
20696...	Insecticide from Florida	78.82	7.50		11.36		2.67		100.35	.42
9 I. & W	Lemon-oil Insecticide.....	81.29	12.47				3.76		97.52	

^aSee correspondence with manufacturers, pp. 60 and 62.

REMARKS.

20458. The greater part of the unsaponifiable matter in this sample on being subjected to distillation distills over between 300° to 400° C., and is evidently composed of the higher boiling products of petroleum.

20460. It appears that one alkali is present in the fatty acid soap, and another has been used to saponify the resin.

20461. The unsaponifiable portion of this sample appears to consist of products from either the fir or pine, and closely resembles turpentine. Low results were obtained doubtless because some of the unsaponifiable matter was volatile with water.

9 I. & W. The fatty acids are mixed to quite a large extent with resin or resin oil. If there is any lemon oil, it is extremely doubtful, as its presence is not shown.

The soaps sold under the name of "Whale-oil soaps" are usually not really whale oil, but are made of some cheap fish oil. Marlatt^a says in regard to such soaps that they are especially valuable in destroying soft-bodied insects. He further remarks that a soap should be demanded which is made with potash rather than soda, since the former is more effective and more readily sprayed, and that not more than 30 per cent of water should be present. The figures for the whale-oil soaps in Table IV show that none come in this classification. While soaps 19557, 20455, 20457, and 20459 contain only a moderate amount of water, they are in every case hard or soda soaps. The only whale-oil soap that is soft and contains potash is 20456, and this contains entirely too much water.

Permol Kerosene Soap appears to consist of about 10 per cent moisture, 35 per cent soap, and 53 per cent crude petroleum products, especially paraffin. It will doubtless have the same good effect as the usual soap-petroleum emulsion.

J. & M. Reliable Insecticide is the well-known resin soap, containing in this case about 36 per cent resin soap, 27 per cent fatty acid

^aFarmers' Bul. 127, U. S. Department of Agriculture.

soap, and 34.30 per cent moisture. The merits of a resin, fatty acid soap mixture are well-known and so do not need repetition.

Stott's Fir Tree Soap evidently consists of a mixture of fatty acid soap and some fir or pine tree products. It may serve the purposes for which it is intended but has not been tested to the author's knowledge.

Sample No. 20696 is an insecticide forwarded from Florida by a private party through the Entomologist of the Department. It evidently consists of a mixture of resin soap and fatty acid soap dissolved in water. It has the advantage of having potash instead of soda as the alkaline principle, and will doubtless serve the purpose for which it is intended. A small amount of alkali is in the free condition, but hardly enough to injure the foliage.

Lemon-oil Insecticide is evidently a water solution of resin and fatty acid soap, with potash as the alkaline principle. Its name is misleading, in that we would expect to have some lemon oil present. Twenty-five cents per pint is a rather high price for such a mixture.

HELLEBORES.

The roots of *Veratrum viride*, or American hellebore, and the roots of *Veratrum album*, or European hellebore, when ground in a powder possess considerable insecticidal qualities.

In making an analysis of this class of compounds, a determination of the following constituents was made: Moisture, ash, to see if the roots had been properly freed of dirt, and alkaloids present, to see if they were the alkaloids that should be present in true hellebore roots.

METHODS OF ANALYSIS AND DISCUSSION.

Moisture.—Dry 1 to 2 grams of the sample at the temperature of boiling water for 12 to 14 hours. The loss in weight is reported as moisture.

Ash.—Burn the dried sample from the above determination at a low red heat to a white ash. The residue is reported as ash.

Alkaloids.—From *Veratrum viride*^a 6 well-defined alkaloids have been extracted, namely, jervine, pseudojervine, rubijervine, veratralbine, veratrine, and cevadine. All of these except cevadine are also present in *Veratrum album*. In extracting and testing the above samples, the following method is followed:^b The finely powdered substance is extracted for 3 to 4 hours with water weakly acidified with sulphuric acid at a temperature of 40° to 50° C. It is then filtered through a folded filter and the filtrate transferred to a separatory funnel. It is first extracted in the separatory funnel with petroleum ether to get rid of coloring matter, etc. It is then extracted with chloroform.

^a See Wright and Luff, Jour. Chem. Soc., 35, p. 401-426.

^b See "Poisons: Their Effect and Detection," 3d ed., Blyth.

This extraction with chloroform is repeated a number of times, thus extracting most of the alkaloids of hellebore, either partially or wholly. This solution, which we will call *A*, is put aside, and the contents of the separatory funnel is made alkaline with ammonia and extracted with petroleum ether to obtain any alkaloids that are left, particularly veratrine, making solution *B*.

Portions of solution *A* are placed in watch glasses, allowed to evaporate spontaneously, and finally tested with concentrated sulphuric acid. On employing two samples known to be pure, the following results are obtained: The residue left is amorphous. On treating it with a drop of sulphuric acid a yellow solution is first formed, changing to brown, then purplish red on the edges, and finally, after standing about half an hour, purplish red throughout the whole mass. Portions of *B* are now treated in the same way, and on two pure samples the following results are obtained: The residue is amorphous. On being treated with concentrated sulphuric acid a yellow solution first forms, which turns dark yellow, chestnut, and finally brown red, with sometimes a greenish color around the edges of the drop.^a

Of course, there are some substances that might give the same play of colors with sulphuric acid as those described, but probably not any that would likely be used to cheapen the product.

ANALYSES AND DISCUSSION.

The results obtained by the above methods of analysis are given in the following table:

TABLE V.—*Composition of hellebores.*

Serial No.	Name.	Moisture.	Ash.	Acid-chloroform extraction treated with concentrated sulphuric acid.	Ammonia - petroleum ether extraction treated with concentrated sulphuric acid.
19558	White Hellebore	<i>Per cent.</i> 6.44	<i>Per cent.</i> 15.71	Amorphous; yellow, brown, purplish red on edges, finally all purple red.	Amorphous; yellow, dark yellow, chestnut, brown red, greenish on edges.
19611	Hellebore, "Lion Brand,"	7.45	14.16	do.....	Do.
19635	White Hellebore	6.23	12.67	do.....	Do.
19685	Hellebore	6.43	9.16	do.....	Do.
19835	White Hellebore	8.87	9.72	do.....	Do.
19836	do	7.17	25.29	do.....	Do.
19837	do	7.00	24.57	do.....	Do.
19838	do	8.21	11.37	do.....	Do.
19839	do	7.17	14.92	do.....	Do.

^a According to Allen's Commercial Organic Analysis, the pure alkaloids should give the following play of colors with concentrated sulphuric acid:

Jervine: Yellow, brown yellow, bright green.

Pseudojervine: Yellow, bright green.

Rubijervine: Yellow, orange, dark red.

Veratralbine: Yellow, orange red, blood red with green fluorescence.

Veratrine: Exactly like cevadine except that the red solution is not fluorescent.

Cevadine: Yellow, brown red, blood red with greenish fluorescence; if allowed to stand in air long enough, becomes purple.

A glance at the moisture figures in Table V shows that the variation in the 9 samples is not very great, with a minimum of 6.23 per cent and a maximum of 8.87 per cent.

As to the percentage of ash, it must be remembered that these preparations are the powdered root of a plant. We would therefore expect to find a rather high ash content, which would be much increased if the roots had not been properly cleansed. The figures show that there is at any rate no reason that the ash content should be above 15 per cent, and evidently, with proper handling, this can be very much reduced. Adopting, then, 15 per cent as the maximum amount allowable, it will be seen that samples 19836 and 19837 are very much above this limit, and 19558 slightly above.

Since the alkaloidal principles in the above 9 samples, on being treated with concentrated sulphuric acid, act exactly similar to the alkaloidal principles of 2 samples known to be pure, and also follow fairly closely the color changes as indicated for the alkaloids of the hellebores in chemical literature, these samples appear to belong to the class of hellebores. The color changes seem to follow most closely those indicated for cevadine, which might be expected if the above samples are roots of *Veratrum viride*, since this alkaloid appears in quantities greater than the sum of all the other alkaloids.

PYRETHRUMS.

The next group of substances to be taken up are the pyrethrums, which include *Pyrethrum roseum*, or Persian Insect Powder, and *Pyrethrum cinerariæfolium*, or Dalmatian Insect Powder, and Buhach.

The ground flower heads of these plants are used to kill insects. Such preparations contain as their active principle a volatile oil, which quickly oxidizes on exposure to the air to an inactive resin.^a This oil may be determined by extracting with ether and drying at a low heat. Four samples examined by Hilgard gave the following results:

Name of preparation.	Per cent ether extract.	Color and odor.
Imported pyrethrum	9.5	Greenish black, tarry; strong odor.
Buhach, or domestic pyrethrum	6.1	} Yellow, thin oil; strong odor.
Do	5.8	
Lyon's Magnetic Powder	4.9	Green-black, crumbly; slight odor.

Such powders sometimes contain either lead chromate, barium chromate, or tumeric, to give them a bright yellow color or to palm off some entirely different powder as one of the pyrethrums.

METHODS OF ANALYSIS AND DISCUSSION.

In making an analysis of these products the following determinations were made: Moisture, ash (a large amount of which indicates the

^a Hilgard. Report of the College of Agri. Univ. of California, 1879, p. 68.

presence of barium or lead chromate), ether extract, and qualitative tests for lead, barium, chromium, and tumeric.

Moisture.—This is determined by drying a weighed quantity of the powder for from 12 to 14 hours in hydrogen at the temperature of boiling water, and reporting the loss as moisture.

Ash.—Burn the dried, weighed portion from the moisture determination at a low red heat until a white ash is obtained. The residue is reported as ash.

Ether extract.—Extract a weighed portion of about 2 grams of the powder for 16 hours with ether in any of the ordinary extraction apparatus. Drive off the ether on the steam bath and dry at a temperature of about 70° C., not leaving the extraction flask in the oven longer than necessary. The residue is reported as ether extract.

Lead, barium, and chromium are tested for in a nitric acid solution of the ash by the ordinary methods employed, and chromium by the borax bead test, using a small quantity of the ash.

Turmeric.—Treat a portion of the powder with alcohol and filter off the extract. Saturate a piece of filter paper with the extract and dry on a watch glass over the steam bath. Repeat three or four times. Now dip the paper in a solution of borax slightly acidified with hydrochloric acid. Dry on a watch glass over the steam bath, and if tumeric is present a cherry-red color appears.

ANALYSES AND DISCUSSION.

In Table VI, following, are the analyses of 10 samples of pyrethrums or imitations thereof.

TABLE VI.—Composition of pyrethrum.

Serial No.	Name.	Moisture.	Ash.	Ether extract. ^a	Lead in ash.	Chromium in ash.	Barium in ash.	Turmeric.
		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>				
19528	Black Flag Insect Powder.....	7.21	8.01	8.91	None ...	None ...	None ...	None.
19529	Buhach	6.96	9.69	9.09	...do ...	...do ...	...do ...	Do.
19530 ^b	Perslan Insect Powder.....	5.82	12.02	5.15	Present.	Present.	...do ...	Do.
19559	Pyrethrum Insect Powder.....	7.39	7.80	8.38	None ...	None ...	...do ...	Do.
19677	Death Dust for Insects.....	6.75	7.74	10.68	...do ...	...do ...	...do ...	Do.
20371	Pyrethrum Powder	7.49	11.91	6.68	...do ...	...do ...	...do ...	Do.
20483	Dalmation Insect Powder.....	6.24	8.35	6.43	...do ...	...do ...	...do ...	Do.
20485	Tiger Insect Powder.....	7.68	6.47	6.22	...do ...	...do ...	...do ...	Do.
20486	Magic Insect Powder	6.47	5.90	6.35	...do ...	...do ...	...do ...	Do.
20487	Insect Powder	6.87	8.03	6.78	...do ...	...do ...	...do ...	Do.

^aCHARACTER OF ETHER EXTRACT.

19528. Black, tarry oil; strong smell of pyrethrum.
 19529. Brown oil; strong smell of pyrethrum.
 19530. Greenish-brown oil; not very strong smell of pyrethrum.
 19559. Greenish-brown oil; not very strong smell of pyrethrum.
 19677. Black, tarry oil; strong smell of pyrethrum.
 20371. Green oil; some smell of pyrethrum.
 20483. Dark green, tarry oil; strong smell of pyrethrum.
 20485. Light green oil; strong smell of pyrethrum.
 20486. Dark green, tarry oil; strong smell of pyrethrum.
 20487. Dark green, tarry oil; some smell of pyrethrum.

^bSee correspondence with manufacturers, page 60.

Table VI shows that the percentages of both moisture and ash are about normal and fairly close together in samples 19528, 19529, 19559, 19677, 20371, 20483, 20485, 20486, and 20487, with the exception of 20371, in which the ash is rather high. This high ash content might easily occur, however, in a sample that had not been tampered with in any way. The ether extract figures for the above numbered samples are much higher than those found by Hilgard for Buhach, and the residue has a strong characteristic smell, indicating that the samples are high in that active principle which kills insects. On the whole, all of the above-mentioned samples appear to be the true powdered pyrethrum flower.

Sample 19530 is a bright yellow powder, which has a higher percentage of ash and a lower percentage of ether extract than the average. The oil obtained by extracting with ether has not a very strong pyrethrum odor, and the presence of lead and chromium in the ash of this sample shows that lead chromate has been added to give the powder a brighter yellow color. This sample appears to be either only partially composed of the flower of pyrethrum, or composed of flowers of an inferior quality or of flowers mixed with stems. The use of lead chromate in powders of this class, which are to be blown out in the air of a room, does not appear to us as commendable, since they are, to a certain extent, breathed by human beings, and the lead chromate contained might give rise to troubles more or less serious.

MIXTURES CONTAINING BORAX.

Practically all of these mixtures are used for the destruction of cockroaches and water bugs, and some are further advertised to kill fleas, moths, ants, lice, etc. The substances contained in these compounds are so many in number and the methods of analysis so diverse that only the methods that are used for the determination of borax will be given. Where there is no interfering substance the following method^a is used:

METHODS OF ANALYSIS AND DISCUSSION.

Weigh 2 grams of the sample out into a 200 cc flask and shake well with water till all borax is dissolved. Make up to the mark, filter through a dry filter, throwing away the first 5 cc, and take aliquot portions of 50 cc for analysis. Add methyl orange and then dilute sulphuric acid drop by drop to the appearance of a pink color. Boil to get rid of carbon dioxide, cool, add a little more methyl orange and titrate back with fifth normal sodium hydroxid until the pink color just changes to yellow. All boric acid is now in the free state. Add

^aThomson. Sutton's Volumetric Analysis.

enough glycerin to amount to at least 30 per cent after the titration is finished; then add phenol phthalein and titrate with N/5 sodium hydroxid to the appearance of a permanent pink color. From the number of cc of soda used in the last titration the borax may be calculated.

The writer has found during the course of this work that the best way to standardize the sodium-hydroxid solution is against a weighed quantity of chemically pure $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, the titrations to be carried

FIG. 1.—Apparatus for determining boric acid.

through in exactly the same manner as described above. Sometimes the borax can not be dissolved directly from the sample and titrated because of interfering substances. In such a case as this the method following, as used in the food laboratory, Bureau of Chemistry, is applied.

Saturate a weighed quantity of the sample with a weak solution of sodium hydroxid dry, and burn at a low temperature. Transfer the ash by means of hydrochloric acid and water to a flask, add some methyl alcohol (previously boiled with strong potassium hydroxid and distilled to break up organic acids), and distill with an apparatus such as is shown in fig. 1.

The flask is heated by means of a paraffin bath to about 140° C. After the flask becomes dry remove the paraffin bath, let the flask cool, add 10 cc of methyl alcohol, restore the paraffin bath to its place, and distill again. Repeat this treatment until all of the boric acid has distilled over, as is shown by testing a drop or two of the distillate and afterwards the residue in the flask with tumeric paper. Now make the distillate slightly alkaline with sodium hydroxid and evaporate on the steam bath to dryness. Add some water, and from this point on proceed just as described above where a water solution of the substance is made at once; that is, first make acid with dilute sulphuric acid, boil, cool, titrate to a pure yellow color with standard sodium hydroxid, add glycerin and phenol phthalein, and titrate to a pink color with the standard sodium hydroxid.

ANALYSES AND DISCUSSION.

In the table on the next page are given the results obtained upon fourteen samples.

TABLE VII.—Composition of mixtures containing borax.

Serial No.	Name.	Borax	Sand.	Cloves.	Ether ex-tract.	Cane sugar.	Sodium car-bonate.	Sul-phur.	Cal-cium oxid.	Sul-phur trioxid.	Remarks.
19678	Columbian Insecticide.....	Per ct. 94.74	Per ct. 2.52	Per ct. 3.28	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	A small amount of blue coloring matter is also present. Remainder appears to consist of pyrethrum and a little blue coloring matter. Remainder consists of ether potato or pea meal and a little red coloring matter. Remainder consists of corn meal and a little red coloring matter. Consists of 20 per cent borax and the remainder pyrethrum. Remainder appears to consist of a small amount of cloves and pink coloring matter. Remainder appears to consist of a small amount of pink coloring matter. Remainder appears to consist of pink coloring matter in large quantities. Remainder appears to consist of pyrethrum and a small amount of pink coloring matter. Remainder appears to consist of pyrethrum, corn meal, and a small amount of pink coloring matter. Remainder appears to consist of pyrethrum and a large quantity of ultramarine. Remainder appears to consist of pyrethrum and blue coloring matter. Remainder appears to consist of sassaparilla root and water, corresponding to $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$.
19679	Leggett's Roach Destroyer.....	22.22									
19682	Peterman's Roach Food.....	20.60									
20453	Hooper's Fatal Food.....	92.44									
20484	Bliss's Bedbug Exterminator.....	20.00			6.79						
20498	Ant Destroyer.....	54.12									
20499	Brown's Pink Powder Insecticide....	88.55				44.80					
20500	Sure Destruction for Cockroaches and Ants.	99.50									
20501	Estes's Roach Powder.....	98.31									
20502	Bliss's Cockroach Exterminator.....	81.89									
20503	Roach and Croton Bug Exterminator	47.61									
20504	Brazilian Insecticide Powder.....	8.84					37.52	13.57			
23491	Roachine.....	90.00									
23493	Red Cross Ant Destroyer.....	11.07							13.12	18.56	

It will at once be seen from Table VII that nearly all of these mixtures are made up principally of one or more of the following substances: Borax, pyrethrum, cloves, and sassafras. The first two of these have been used for years in driving away vermin from the places they frequent. The second two, although not so often used, are doubtless efficacious, since it is commonly supposed that roaches, etc., are driven away by high-smelling oils and spices.

The analyses, as given in the table, in most cases explain themselves, so it is unnecessary to go into further detail. Two samples, however, are worthy of further note, namely, 20504 and 23493. The first of these appears to be a by-product or an unpurified product in the manufacture of ultramarine (hence the sulphur, sodium, carbonate, and ultramarine), along with small amounts of borax and pyrethrum. Three of these, sulphur, borax, and pyrethrum, amounting on the whole to not more than one-third of the mixture, give to it its only value as an insecticide. Sample 23493, besides containing borax and sassafras root, also contains about 39 per cent gypsum, which adds weight, and is valueless as a vermin exterminator. Very fine powders, such as powdered gypsum, are said by some to drive away ants, since they seem to dislike to crawl through finely divided substances.

ROACH PASTES.

According to formulas published in chemical receipt books and pharmaceutical journals, these pastes all have free phosphorus as their active poisonous constituent. This is sometimes mixed with molasses and flour or corn meal, sometimes with glucose sirup and flour or corn meal, and sometimes with sugar, lard, and flour or corn meal. These preparations are very efficacious in ridding the house of cockroaches and rats, since they eat the paste and are killed outright instead of being driven away to some other place. Great care should be taken in the use of such preparations, since when taken internally they are poisonous to human beings and household animals.

METHODS OF ANALYSIS AND DISCUSSION.

The methods of analysis used for this class of compounds are as follows:

Phosphorus.^a—Extract the phosphorus from a weighed quantity of the paste by repeatedly rubbing the sample up with small quantities of carbon bisulphid. Filter each time, keeping the filter covered to prevent oxidation. After all phosphorus is extracted treat the filtrate with a 5 per cent solution of silver nitrate, which will precipitate the phosphorus as silver phosphid. Oxidize the precipitated phosphid by treating with 30 to 40 cc of dilute nitric acid and shaking well. Finally, heat gently to oxidize the remaining phosphid and drive off

^a Chem. Zeit., 1893, XVII, 1244, 1245.

the carbon bisulphid. Boil for ten minutes. Precipitate out the phosphate formed with molybdate solution and wash. Dissolve on the filter with ammonia and determine the phosphorus with magnesia mixture in the ordinary manner.

While the accuracy of the above method is not great, it appears to be of sufficient value for the use to which it is put.

Microscopic examination.—This is made to determine whether wheat, corn, or any other form of starch is present.

Reducing sugars.—A qualitative examination of a water extract of the paste is made with Fehling solution to see if reducing sugars are present.

Polariscopic readings.—Treat a small unweighed quantity of the paste with water and pass the solution through a filter. Clarify with a little lead acetate and pass through another filter. Transfer 45 cc of the solution to a 50 cc flask, make up to the mark with water, mix, transfer to a 200 mm tube, and take a reading. Transfer another 45 cc of the solution to a 50 cc flask, add 5 cc of concentrated hydrochloric acid, mix, invert by heating to 68° C., taking fifteen minutes in so doing, filter, and again take a reading in a 200 mm tube at practically the same temperature as that of the former reading. If reducing sugars are present and the polariscopic reading is + before inversion and — after inversion, the presence of molasses is indicated. If reducing sugars are present and the polariscopic reading is + before inversion and practically the same + reading after inversion, glucose sirup is indicated. If reducing sugars are present and the reading is + before inversion and a less + reading after inversion, molasses which has been mixed with glucose may be present.

ANALYSES AND DISCUSSION.

Table VIII shows the results obtained by the above methods of analysis on five samples:

TABLE VIII.—*Composition of roach pastes.*

Serial No.	Name.	Phosphorous.	Microscopical examination.	Test for reducing sugars.	Reading before inversion.	Reading after inversion.
20489	Roach and Water Bug Ext-terminator.	<i>Pr. ct.</i> 1.37	Shows wheat starch..	Present.	+ 3.7	Less than 0
20490	Tiger Paste.....	1.93	do.....	do.....	+ 1.5	Do.
20491	Fidelity Cockroach Paste..	.77	do.....	do.....	+ 1.8	+ 1.3
20492	Allan's Lightning Roach Paste.	1.94	Shows corn starch.....	do.....	+24.3	+24.3
20493	Stearn's Electric Rat and Roach Paste.	1.46	do.....	do.....	+ 2.9	+ 2.8

Samples 20489 and 20490 appear to consist of phosphorus, flour, and molasses. Sample 20491 consists of phosphorus and flour with some

sweetening substance that appears to be molasses mixed with glucose sirup. Sample 20492 is evidently composed of phosphorus, corn meal, and glucose sirup, and sample 20493 appears to be composed of the same substances, with perhaps a little molasses.

TOBACCO EXTRACTS.

Only four samples of this class of goods were examined, using the following methods of analysis:

METHODS OF ANALYSIS AND DISCUSSION.

Nicotine.^a—About 5 to 6 grams of the tobacco extract is weighed out in a small beaker. Ten cc of an alcoholic soda solution (containing 6 grams of sodium hydroxid, 40 cc of water, and 60 cc of 90 per cent alcohol) is added, followed by enough chemically pure powdered calcium carbonate to form a moist but nonlumpy mass. This is transferred to a Soxhlet extractor and extracted for about 5 hours with ether. The ether is evaporated off at a low temperature by being held over the steam bath, and the residue is taken up with 50 cc of a soda solution (containing 4 grams of sodium hydroxid in 1,000 cc of water). This is transferred by means of water to a Kjeldahl flask, capable of holding about 500 cc, and distilled in a current of steam, using a condenser through which water is flowing freely. A three-bend outflow tube is used and a few pieces of pumice and a small piece of paraffin are added to the Kjeldahl flask to prevent bumping and frothing. The distillation is continued till all the nicotine has passed over. The amount of distillate varies from 300 to 500 cc, according to the amount of nicotine present. When the distillation is complete only about 15 cc of liquid should remain in the distilling flask. The distillate is titrated with standard sulphuric acid, using phenacetolin as indicator. One molecule of sulphuric acid is equal to two molecules of nicotine.

Camphor.—The camphor in sample 2 I & W is determined in the following manner: Weigh out about 30 grams of the sample in a small beaker, add a large quantity of water to precipitate the camphor, filter and wash with cold water, spread the filter paper out on a flat surface and transfer the precipitate to thick dry filter paper by means of a thin spatula, and press between its folds. It is again transferred and pressed between filter paper. This is continued till it is dry. Transfer to a weighed watch glass and weigh again to obtain the weight of camphor. A correction is made based on the amount of camphor that is dissolved by a volume of water equal to that of the filtrate above. By this method low and only approximate results are obtained, but it is the best method we can find to work on mixtures of this description.

^a Wiley's Principles and Practice of Agricultural Analyses, Vol. III, p. 605.

ANALYSES AND DISCUSSION.

Table IX gives the results obtained by the above methods of analysis on four samples:

TABLE IX.—*Composition of tobacco extracts.*

Serial No.	Name.	Nicotine.	Camphor.	Alcohol, etc. (by difference).
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
19687	Hammond's Tobacco Extract	1.88		
22288	Extract of Tobacco	3.50		
^a 22289	Nikoteen.....	26.01		
^b 2 I & W	Vaporizing and Fumigating Insecticide.....	30.87	13.64	53.49

^aSee correspondence with manufacturers, page 62.

^bThe following formula for this substance is placed on the bottle: 37 per cent nicotine, 23 per cent camphor, 32 per cent pure alcohol, remainder, other essential ingredients. See correspondence with manufacturers, page 60.

Both samples 19687 and 22288 contain very small quantities of nicotine, and are consequently not good tobacco extracts. Sample 22289, containing 26.01 per cent nicotine, is a good tobacco extract. Although sample 2 I & W has not as much nicotine and camphor as are given in the formula on the bottle, as an insecticide it will be apt to give good results in actual practice.

MISCELLANEOUS SOLID INSECTICIDES AND FUNGICIDES.

The methods of analysis of the compounds under this heading are given in only a few cases, because they are so numerous and because most of them would immediately occur to anyone with a chemical training.

METHODS OF ANALYSIS AND COMPOSITION OF COMPOUNDS.

BUG DEATH.

[Serial No. 19555.]

Method of analysis.—The moisture determination is made by drying the substance for 12 hours at 100° to 105° C. The residue is heated to dull redness for volatile matter. For all of the other constituents 2 grams is dissolved in aqua regia by prolonged boiling. What remains is weighed as sand. The filtrate from this is made to 250 cc and one-half taken for analysis, about 3 or 4 cc of concentrated sulphuric acid are added, and the solution evaporated until white fumes come off and no odor of nitric or hydrochloric acid remains. It is diluted with water, filtered, washed with water containing a little sulphuric acid, and finally with 60 per cent alcohol. The lead sulphate thus obtained is burned and weighed with the usual precautions. Sodium carbonate is

added to the filtrate, and the precipitated zinc and iron washed carefully, dried, finally transferred to a weighed crucible, and carefully separated from the filter. The filter is burned in another crucible after treatment with concentrated nitric acid. The residue is again treated with concentrated nitric acid evaporated to dryness and burned. The other crucible is heated with the Bunsen flame, and the combined residues reported as zinc oxid + ferric oxid. The residue is dissolved by digestion with hydrochloric acid, and potassium hydroxid added till the iron is precipitated and the zinc has gone into solution. The precipitate is washed, dissolved in hydrochloric acid, precipitated again with ammonia, and finally washed, dried, burned, and weighed as ferric oxid.

Composition of Bug Death.

	Per cent.
Sand	4.35
Moisture.....	.73
Volatile, besides moisture (principally NH_4Cl and some KCl)	3.27
Lead oxid	1.93
Zinc oxid	85.17
Ferric oxid	4.38
	<hr/>
	99.83

This compound contains only 3.27 per cent of those constituents which would give it any value as a plant food, so that it would not give very good results in this direction. As to its value as an insecticide, it is reported as not having any appreciable effect on bugs when applied at the rate of 40 pounds to the acre. When applied at the rate of 100 pounds to the acre it kills the bugs but also burns some of the plant leaves. It is too expensive for general use.^a

VELTHA.

[Serial No. 19845.]

Composition of Veltha.

	Per cent.
Sand	32.73
Moisture.....	27.00
Carbon	2.31
Sulphur trioxid	19.90
Ferrous oxid.....	17.90
	<hr/>
	99.84

This substance is evidently a sample of partially dehydrated ferrous sulphate mixed with about 35 per cent sand and carbon. The green vitriol is very commonly used as a fungicide, but the other 35 per cent is inert in its action on insects.

^aSixteenth Annual Report of the Maine Agricultural Experiment Station (1900).

IRON SULPHATE.

[Serial No. 20370.]

Composition of Iron Sulphate.

	Per cent.
Sand	0.06
Ferrous oxid	31.65
Sulphur trioxid	34.61
Water of crystallization and hygroscopic	33.50
	<hr/> 99.82

This is evidently a good sample of partially dehydrated $\text{FeSO}_4\cdot 7\text{H}_2\text{O}$.

SCROFULARIA.^a

[Serial No. 19681.]

Composition of Scrofularia.

	Per cent.
Moisture	4.91
Ash	19.52
Ether extract	7.35

The oil extracted is black and tarry, and has a slight smell of both pyrethrum and tobacco. Small amounts of lead and chromium are also present.

This sample evidently consists of tobacco and pyrethrum, colored with lead chromate.

SULPHUR.

[Serial No. 19636.]

Composition of Sulphur.

	Per cent.
Moisture	0.58
Ash	.07
Volatile matter, other than moisture	99.35
	<hr/> 100.00

This is an excellent sample with a very small content of moisture and ash.

P. D. Q. INSECT POWDER.

[Serial No. 19676.]

Composition of P. D. Q. Insect Powder.

	Per cent.
Moisture	2.62
Sulphur	15.59
Ash insoluble in hydrochloric acid	64.51
Ash soluble in hydrochloric acid	15.23
What appears to be coal-tar products (by difference)	2.05
	<hr/> 100.00

^aSee correspondence with manufacturers, page 60.

The soluble portion of the ash consists of Ca, Mg, Fe, K, Na, SO_3 , and P_2O_5 . The sample appears to consist of sulphur, coal-tar products, and common earth. While the first two of these substances are of value in driving away and killing lice, the last is valueless, and 25 cents per pound is being paid for a substance that appears to consist of 80 per cent earth and 20 per cent cheap ingredients.

LAMBERT'S DEATH TO LICE. ^a

[Serial No. 19680.]

Composition of Lambert's Death to Lice.

	Per cent.
Moisture	3. 97
Ash insoluble in hydrochloric acid	35. 91
Ash soluble in hydrochloric acid	31. 40
Volatile matter, other than moisture	28. 72
	<hr/> 100. 00

The portion of ash soluble in hydrochloric acid contains a large amount of Ca, some Fe, Mg, K, Na, and CO_2 , and very small amounts of SO_3 and P_2O_5 . The volatile portion appears to consist of the organic portion of tobacco, some carbon dioxid, and what appears to be small amounts of coal-tar products. This sample on the whole appears to be a mixture of tobacco with some lime treated with coal-tar products. Either through negligence or on purpose a large amount of dirt is present. It does not appear that this mixture possesses much value as a louse killer outside of its content of tobacco, a like amount of which could be bought for much less than 25 cents, the price per box of the above mixture.

ELECTRIC VERMIN EXTERMINATOR.

[Serial No. 19683.]

Method of determining phenol.^b—About 50 grams of the powder is treated with 5 cc of water in a mortar and sulphuric acid (1-2) added, a few drops at a time, mixing well after each addition. The addition of acid is not fast enough to cause a sensible rise in temperature, but extends over several hours. The operation is complete when a small piece of the well-mixed contents of the mortar gives an acid reaction when placed on litmus paper. Enough sand is added to granulate the contents of the mortar, and the whole is allowed to stand for a few hours. The mass is transferred to a Soxhlet extractor and extracted with ether. The ether is distilled off until the contents of the flask acquires a temperature of 110°C ., and the residue is weighed as crude carbolie acid.

^aSee correspondence with manufacturers, page 61.

^bAllen's Commercial Organic Analysis, 2d ed., Vol. II, p. 548.

Composition of Electric Vermin Exterminator.

	Per cent.
Sand	0.69
Carbon dioxid	5.22
Phenol anhydrid	2.25
Calcium oxid	58.28
Magnesium oxid	12.77
Water of constitution, hygroscopic, and a small amount of pink dyestuff (by difference)	20.79
	<hr/> 100

This substance evidently consists of partially air-slaked lime, treated with some crude carbohic acid, and colored with a pink dyestuff. It does not possess much power as an insecticide, since even pure carbolate of lime is not particularly effective in this direction. Lime also has only weak insecticidal properties.

INSTANT LOUSE KILLER.

[Serial No. 19684.]

Composition of Instant Louse Killer.

	Per cent.
Moisture.....	1.21
Ash insoluble in hydrochloric acid	27.10
Ash soluble in hydrochloric acid	64.78
Some carbon dioxid, what appears to be coal-tar pro- ducts, and a little tobacco (by difference).....	6.91
	<hr/> 100

The portion of ash soluble in hydrochloric acid consists of large amounts of Fe, Al, Ca, and CO₂, some Mg, and small amounts of SO₃ and P₂O₅. This sample appears to consist of small amounts of carbolate of lime and tobacco and large amounts of lime, calcium carbonate, and probably clay. While the lime and carbolate of lime possess weak insecticidal properties, and the tobacco is of some value, the compound as a whole appears to have very little value. The price (25 cents per box) is also excessive.

WHITNEY'S SULPHON CARBOLATE OF LIME.

[Serial No. 19689.]

Composition of Whitney's Sulphon Carbolate of Lime.

	Per cent.
Sand	1.14
Carbon dioxid	8.60
Sulphur trioxid	.16
Calcium oxid	57.42
Magnesium oxid	7.37
Phenol anhydrid	1.68
Water of constitution, hygroscopic, and small amounts of pink dyestuff (by difference).....	23.63
	<hr/> 100

The same remarks made in regard to Electric Vermin Exterminator apply equally well to this compound.

TOBACCO AND SULPHUR.

[Serial No. 19691.]

Composition of Tobacco and Sulphur.

	Per cent.
Moisture.....	4.70
Sulphur	36.21
Ash insoluble in hydrochloric acid	19.40
Ash soluble in hydrochloric acid	14.17
Volatile matter, other than moisture and sulphur	25.52
	<hr/> 100

The soluble ash consists of Ca, Mg, Fe, K, Na, P_2O_5 , SO_3 , and CO_2 . The sample evidently consists of sulphur and tobacco, but a larger amount of sand is present than should be in a good sample of tobacco.

TOBACCO AND SULPHUR.

[Serial No. 19692.]

Composition of Tobacco and Sulphur.

	Per cent.
Moisture.....	6.57
Sulphur	3.51
Ash insoluble in hydrochloric acid	5.17
Ash soluble in hydrochloric acid	24.08
Volatile matter, other than moisture and sulphur.....	60.67
	<hr/> 100

The soluble ash consists of Ca, Mg, K, Na, SO_3 , P_2O_5 , and CO_2 . Besides the above constituents, this sample is quite rich in potassium nitrate. The sample, as claimed, evidently consists of sulphur and tobacco, the latter of which is exceedingly rich in potassium nitrate, which compound is excellent as a plant food.

FIBRO FERRO FEEDER.

[Serial No. 19847.]

Composition of Fibro Ferro Feeder.

	Per cent.
Water of crystallization and hygroscopic.....	7.80
Sand	5.56
Sulphur trioxid	22.37
Chlorin.....	7.70
Ferric oxid	16.06
Ferrous oxid.....	8.06
	<hr/> 67.55

The remainder of this substance was not estimated, but appears to consist of organic material resembling wood fibers. This mixture evi-

dently consists of organic matter, chlorid of iron, and ferrous sulphate, which has been partially dehydrated and partially oxidized to ferric sulphate. Both the chlorid of iron and ferrous sulphate present in this mixture are used as fungicides in certain cases of plant disease, but they can be purchased very cheaply unmixed with worthless and inert matter.

POTASSIUM CYANIDE.

[Serial No. 20480.]

A determination of the cyanogen in this sample, calculated to potassium cyanide, shows that only 27.60 per cent is present. The remainder appears to be composed principally of potassium carbonate with a little potassium cyanate. It is needless to say that this is a very impure sample of potassium cyanide.

TAR CAMPHOR.

[Serial No. 20481.]

This sample is prepared from the heavy fractions of coal tar, and is almost entirely composed of naphthalene. This substance is of value in driving away moths, lice, etc., but will not kill the insects.

FANCIER'S FRIEND.

[Serial No. 20488.]

Composition of Fancier's Friend.

	Per cent.
Moisture.....	5.03
Ash.....	7.21
Sulphur.....	30.77
	<hr/> 38.01

The remainder appears to be an imitation of pyrethrum, since it contains lead and chromium as lead chromate to give it a yellow color, yet the ether extract does not in the slightest degree have the characteristic odor of pyrethrum.

ROACHSAULT.

[Serial No. 23501.]

Composition of Roachsault.

	Per cent.
Sodium fluorid.....	85
Moisture.....	2.53
Alumina and ferric oxid.....	.27
Silica as fine sand.....	9.60
Sodium sulphate (by difference).....	2.60
	<hr/> 100

While sodium fluorid, which is the principal constituent of the above mixture, has been used for quite a while as an antiseptic, we are not aware that its value as an insecticide has been tested, but think that it might have considerable power in this direction.

PAR' OIDIUM.

[Serial No. 23721.]

Composition of Par' Oidium.

	Per cent.
Ash insoluble in hydrochloric acid	7.10
Carbon dioxid	2.88
Sulphur trioxid	17.12
Ferric oxid	10.63
Calcium oxid	15.58
Magnesium oxid	.73
Sulphur	19.12
Tobacco, water of hydration, and hygroscopic (by difference)	26.84
	<hr/> 100

This substance appears to be principally composed of gypsum, sulphur, and ferric oxid, with smaller quantities of sand, tobacco, etc. Nothing appears to be present besides the sulphur and small amount of tobacco that gives it any value as an insecticide. As far as its sulphur content goes, this compound would be applicable to surface mildews. The same amount of sulphur and tobacco, however, could be purchased for much less than 30 cents, the cost of a can of this preparation.

FLECK'S LICE EXTERMINATOR.

[Serial No. 23722.]

Composition of Fleck's Lice Exterminator.

	Per cent.
Insoluble in hydrochloric acid	4.20
Carbon dioxid	9.56
Sulphur trioxid	1.99
Ferric oxid	1.43
Calcium oxid	31.90
Magnesium oxid	21.61
Volatile matter, other than carbon dioxid	29.88
	<hr/> 100.57

The volatile matter consists of naphthalene, tobacco, pink coloring matter, and water of constitution and hygroscopic moisture. This sample evidently consists of partially air-slaked lime mixed with naphthalene and tobacco, and colored with a pink dyestuff. In so far as the naphthalene and tobacco are concerned, this compound will doubtless drive away lice. The lime also has weak insecticidal properties.

GRUB AND CANKER WORM EXTERMINATOR.

[Serial No. 16 I. & W.]

Method of analysis.—Weigh out about 10 grams of the sample in a beaker. Treat with petroleum ether, pouring the wash ether through a weighed filter, and continue washing till all oil is removed. Dry filter and contents and weigh. The contents of the filter represents

aniline dye, Prussian blue, and carbon. The difference between the contents of the filter and the original weight of substance taken is oil.

Treat the residue on the filter with 90 per cent alcohol, and wash until aniline dye is removed. Dry and weigh. The difference between the weight of the present and former contents of the filter represents aniline dye. None was present in this sample.

Treat the residue on the filter with about 200 cc of 2 per cent sodium hydroxid and wash with water until free of alkali. Treat the residue with 300 cc of 10 per cent hot hydrochloric acid, continue washing with more dilute hydrochloric acid until the filtrate is free of iron. Wash with hot water, dry, and weigh. The difference between this weight and the former is Prussian blue, and what remains on the filter is carbon.

Composition of Grub and Canker Worm Exterminator.

	Per cent.
Oil (appears to be linseed).....	87.28
Carbon	11.40
Prussian blue	1.32
	<hr/> 100

This substance is a sample of printers' ink, and when smeared around a tree or on a piece of paper tied around a tree, will doubtless prevent the adult females from climbing up the trees and depositing their eggs.

MISCELLANEOUS LIQUID INSECTICIDES.

METHODS OF ANALYSIS AND RESULTS OF DISTILLATION TESTS.

A few general methods of analysis are applicable to all of these substances. In the first place, a sample is subjected to distillation, and the temperature of the vapor as the distillation progresses and size of the fractions are noted. The odor of the various fractions is noted, much light often being thrown upon the subject thereby. The usual tests for various substances suspected of being present are made in the different fractions. Tests of the miscibility of the samples with different solvents and of the optical activity often allow us to distinguish certain oils from one another. This is especially true in distinguishing kerosene from turpentine and allied products.

LEE'S LICE KILLER.^a

[Serial No. 19627.]

Results of distillation test.

77°-100° C. About 3 per cent of light oils clouded with water, and having a pronounced odor of benzene, toluene, etc.

100°-180° C. Practically nothing comes over.

180°-220° C. Fifty-two per cent comes over, having the odor of and giving tests for phenol and cresols.

220°-250° C. Thick oil comes over, mixed with naphthalene, and finally at 250° C. the distillation tube become entirely clogged with naphthalene.

^aSee correspondence with manufacturers, p. 61.

The sample does not mix or form an emulsion with water. It is undoubtedly a sample of a coal-tar product, probably cresote oil. This oil is undoubtedly of value as a louse killer and a disinfectant.

LEGGETT'S KILLER.

[Serial No. 19686.]

Results of distillation test.

	Per cent.		Per cent.
164°-166° C	32	172°-200° C	12
166°-168° C	28	200°-220° C	3
168°-172° C	19	Remaining in flask and loss	6

The sample, including every fraction, has a strong odor of turpentine. In a 200 mm. tube it gives a +67° reading on the Schmidt and Haensch polariscope sugar scale. It also mixes entirely with glacial acetic acid.

Thorp's Dictionary of Applied Chemistry says oil of turpentine usually sold in the American trade begins to boil about 160° C. and distills over, leaving only a slight residue above 180°. It also mixes with glacial acetic acid.

This sample appears to be composed of American oil of turpentine. It is of value for the purposes intended, but 25 cents per can is a very high price.

SURE DEATH TO INSECTS.

[Serial No. 4, I. & W.]

Results of distillation test.

	Per cent.	
40°-60° C	24	} Light oils with odor of lower boiling fractions of petroleum.
60°-80° C	41	
80°-110° C	26	
110°-140° C	6	} A pleasant ethereal odor, as though some substance resembling oil of citronella were present.
Remaining in flask and loss.	3	

This sample has a strong odor of gasoline. It appears to consist of the lower boiling products of petroleum, commonly grouped together as gasoline, and perhaps a small quantity of some high-smelling oil with an odor resembling that of oil of citronella. Gasoline is known to be a good remedy in getting rid of bedbugs and other vermin, but 25 cents per can is an exorbitant price. The indiscriminate use of gasoline is very dangerous by reason of its extreme volatility and easy ignition.

DR. BAKER'S LIQUID DEATH DROPS.

[Serial No. 5, I. & W.]

Results of distillation test.

	Per cent.		Per cent.
40°-70° C	20	130°-150° C	5
70°-100° C	42	150°-168° C	3
100°-130° C	24	Remaining in flask and loss ..	6

This sample has a strong odor of gasoline, and every fraction has a strong odor of the lower boiling products of petroleum; appears to be composed of the lower boiling products of petroleum, commonly classed as gasoline. There is no indication of any other substance being present.

HEINE'S LIQUID INSECT DESTROYER AND DISINFECTANT.^a

[Serial No. 6, I. & W.]

Results of distillation test.

	Per cent.	
160°-170° C	11	} Strong odor of turpentine.
170°-180° C	14	
180°-190° C	15	
190°-205° C	18	
205°-225° C	14	} Thick oil; pungent burnt odor.
225°-268° C	19	
Remaining in flask and loss ..	9	

The sample, which has a strong odor of turpentine, is partially soluble in glacial acetic acid, and the reading in a 200 mm. tube on the Schmidt and Haensch polariscope scale is +25°. It appears to consist of a mixture of turpentine with some other oil which, from its boiling points and general properties, appears to be kerosene. Such a mixture is good both as an insect destroyer and to some extent as a disinfectant, but the price (25 cents) per can is too high.

THYMO-CRESOL.

[Serial No. 11, I & W.]

Results of distillation test.

85°-105° C. A fair-sized fraction comes over, which separates into a layer of water and a layer of oil having the odor of benzene, toluene, etc.

105°-180° C. Practically no liquid comes over, and at the latter temperature a semisolid mass is formed in the retort.

180°-220° C. A small amount of a thick oil having the odor of and giving test for phenol and the cresols.

220°-270° C. Thick oil, with odor and other properties of cresols.

270°-285° C. Thick, heavy oil, with odor and other properties of cresols.

Left in the retort—a solid mass.

Sodium oxid, 2.20 per cent.

Sample has a strong odor of coal tar, which is evidently the basis of the preparation. It appears, from the boiling points and size of the

^aSee correspondence with manufacturer, page 61.

fractions, that that fraction of coal tar containing phenol and the cresols, usually termed creosote oil, was principally used. The creosote oil has evidently been boiled with resin or fatty matter and sodium hydroxid, which causes it to form an emulsion with water instead of a separate layer. It is well known that creosote emulsions give good results, both as disinfectants and insecticides.

DYKE'S LOUSE PAINT.

[Serial No. 12, I and W.]

This sample has a coal-tar odor, and has a black substance in suspension, which appears to be carbon. What remains after filtering off the carbon is a green fluorescent oil.

Results of distillation test.

Per cent.	
80°-150° C.....	3 Light oil; odor of benzene, toluene, etc.
150°-180° C.....	10
180°-210° C.....	13
210°-240° C.....	14
240°-270° C.....	14
270°-300° C.....	18
300°-327° C.....	18
Remaining in flask and loss	10

In case this were a sample of pure coal tar the higher boiling fractions would be heavier than water. The sample appears to be mainly composed of petroleum along with some coal tar, or one of its fractions as creosote oil. Carbon is present in suspension. The results of using this as a louse exterminator would very likely be good. The preparation should not be applied in too concentrated a form, as it would burn the skin badly.

CHLORO NAPHTHOLEUM.^a

[Serial No. 13, I. and W.]

Results of distillation test.

Sample has a strong odor of coal tar.

80°-105° C. A fair-sized fraction, separating into a layer of water and a layer of light oils having the odor of benzene, toluene, etc., comes over.

105°-180° C. Only a few drops of liquid come over.

180°-220° C. A small amount of a thick oil comes over having the odor and giving tests for phenol and the cresols. At about 200° the mass in the retort becomes semisolid.

220°-225° C. A few drops of oil come over, and at the latter temperature the condenser becomes so stopped up with naphthalene that the distillation must be stopped.

Sodium oxid, 1.99 per cent, and traces only of sodium chlorid.

This sample appears to consist of creosote oil, perhaps enriched with naphthalene, which has been boiled with resin, fatty matter, and sodium hydroxid to cause it to form an emulsion with water.

^a See correspondence with manufacturers, page 62.

This investigation shows that there are many insecticides on the market which are nearly worthless, and many for which a very exorbitant price is paid that could just as easily be prepared at home. In fact there are very few of the above insecticides which could not either be prepared at home or for which a substitute could not be prepared at a less cost. The only way to obtain information regarding insecticides is by consulting bulletins dealing with the subject, or in case the composition of the insecticide in question is not published, to have the same analyzed.

CORRESPONDENCE WITH MANUFACTURERS.

In order that manufacturers might have a chance to comment upon analyses before they were published, the following circular letter was sent to each manufacturer whose goods had been examined:

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C.

DEAR SIR: The Bureau of Chemistry is preparing a bulletin giving the results of its examinations of insecticides and fungicides. While we feel sure that the data are practically correct, at the same time we know that one sample does not always represent the products of its manufacturer. We should, therefore, be pleased to have you make any comments that you think necessary on the data as given below.

It might be as well for you to know that in our forthcoming bulletin this Bureau has expressed an opinion in favor of a 6 per cent rather than a 4 per cent limit for soluble arsenious acid in insecticides containing this substance.

Respectfully,

H. W. WILEY, *Chief.*

The answers to the above letter, where they throw any light upon the subject under consideration, are as follows:

[Serial No. 19625.]

We note that the analysis of our Green Arsenoid shows 2.02 per cent sodium sulphate and 1.30 per cent sand, and we wish to explain that the sand gets into this material from the arsenic which is used in its manufacture. We have been using English arsenic, which at times contains quite a percentage of sand. This does not seem to be the case with the Continental arsenic, which we are now using exclusively. The sodium sulphate is, of course, a by-product, and the quantity in the arsenoid is dependent, in a large measure, upon the amount of washing that the arsenoid receives. We believe that the 2.02 per cent is quite exceptional, particularly in view of the fact that we are now washing the arsenoid more thoroughly than heretofore.

ADLER COLOR AND CHEMICAL WORKS.

[Serial No. 22287.]

We are in receipt of your analysis of White Arsenoid and would say that this substance is a product which we had used only in a tentative way. There is now none on the market and will be none in the future.

ADLER COLOR AND CHEMICAL WORKS.

[Serial No. 2, I & W.]

Following is the synthesis of the insecticide, which we have been as careful as possible not to deviate from in compounding:

	Per cent.
Nicotine	39
Camphor	23
Glycerin	10
Alcohol	28

EASTERN CHEMICAL CO.

Comment by chemist: The best method available for determining camphor gives very low results, but we are sure that our nicotine figure is correct for the sample examined.

[Serial No. 19530.]

We have the report on sample 19530, and would say that while we have always been aware that the powder in question was colored, there being a demand for this grade, we had no idea the quality is not of the best.

Z. D. GILMAN.

[Serial No. 20456.]

I notice the large amount of moisture. The original soap, which was an experiment, had about this amount, and the soap that I have been sending out for a long time should have less.

JAMES GOOD.

[Serial No. 19690.]

One-thirteenth part of the total weight of Grape Dust consists of tobacco.

BENJ. HAMMOND.

Comment by chemist: Another sample of Grape Dust was obtained directly from the manufacturer and analyzed. It was found to contain about the same amounts of sand, copper oxid, and gypsum as the previous sample, but the sulphur figure was 52.82 per cent and the tobacco figure 8 per cent.

[Serial No. 19681.]

Scrofularia consists of pyrethrum, naphthalene, tobacco, camphor, and about 0.1 per cent oil of cedar oil. We never put in any lead chromate at all.

BENJ. HAMMOND.

Comment by chemist: In the sample of Scrofularia examined in this laboratory pyrethrum and tobacco only were found, consequently upon hearing from Mr. Hammond a fresh sample was at once obtained from him, in which were found all the ingredients he mentions above, excepting oil of cedar, which was present in such small quantities that we were unable to find a good test for it, mixed as it was with the other constituents. In the sample originally examined the camphor and naphthalene had entirely volatilized. Mr. Hammond was informed that whoever was supplying him with pyrethrum was coloring it with lead chromate.

[Serial No. 19524.]

The arsenic percentage is above any we have used in many years. Our working formula is now under 1 per cent.

BENJ. HAMMOND.

[Serial No. 6 I & W.]

I use a high-boiling paraffin oil, but have never used turpentine. I have sent you by mail a sample of my insecticide and disinfectant.

HEINE CHEMICAL CO.

Comment by chemist: The sample sent by the Heine Chemical Company was received and analyzed and found to consist principally of one of the higher-boiling petroleum oils and sassafras, but the same was very different from the original sample analyzed, which evidently consisted of kerosene and turpentine. A sample of the oil was sent to Mr. Heine, stating the facts, and suggesting that perhaps someone was putting up goods under his name, since every test showed that turpentine was present. To this no reply was received.

[Serial No. 22292.]

Lee's insect powder contains one more ingredient not mentioned here.

GEO. H. LEE CO.

[Serial No. 19627.]

Lee's Lice Killer is a coal-tar product heavily charged with gases destructive to insect life. It can not be prepared of uniform strength and quality from bulk material obtained in open market, but requires fresh and uniform ingredients, special manipulation, and air-tight packages.

GEO. H. LEE CO.

[Serial No. 19680.]

We use the best and finest ground tobacco dust and lime chemically treated with the higher products of coal tar. If there is any sand in the mixture it goes in before the goods reach our hands.

D. J. LAMBERT.

[Serial No. 19533.]

You are correct in your surmise that one sample does not always represent the product of its manufacturer. We do not think that this can possibly be an examination of our standard article on account of the large percentages of acetic acid and soluble arsenious oxid.

JOHN LUCAS & Co.

Comment by chemist: An examination of a sample forwarded by Mr. Lucas was made with the following results:

	Per cent.
Moisture.....	0.90
Sand	.11
Sodium sulphate	1.01
Arsenious oxid.....	57.21
Copper oxid	31.00
Acetic acid	9.77
	<hr/>
	100.00
Soluble arsenious oxid.....	5.33

This showed that Mr. Lucas had improved his process since making the sample, the analysis of which has already been given.

[Serial No. 19617.]

We have ceased to manufacture this article.

THE NICHOLS CHEMICAL CO.

[Serial No. 20458.]

I should judge the sample you had must be an old one from the small percentage of water present. How there should be such a large amount of unsaponifiable matter I can not understand, unless the presence of petroleum has acted on the fatty acids used. When the soap is made a mixture of fat oils and resin is saponified nearly neutral and 25 per cent of the entire weight of the finished mass in kerosene oil is added.

You may have had some of the crude oil soap which had 25 per cent crude Pennsylvania petroleum in it.

POOLE & BAILEY.

Comment by chemist: The sample examined so evidently consisted of soap and unsaponifiable matter, resembling paraffin, in the proportions given in the table, that a sample was forwarded to Poole & Bailey for analysis if they so desired. No reply was received.

[Serial No. 22289.]

This material should average 30 per cent nicotine.

SKABCURA DIP CO.

[Serial No. 20482.]

We use the best commercial white arsenic. Some few years ago we used one-third barium carbonate; now we use one-seventh barium carbonate, and we shall soon discontinue it altogether.

E. S. WELLS.

[Serial No. 13 I & W.]

Chloronaptholeum is prepared from coal-tar creosote, which is obtained by fractional distillation of coal tar. After all the light oils have been distilled, they are collected separately and cooled by means of refrigerators, and then redistilled until all the impurities and free carbon have been eliminated. The oil has then the required specific gravity, and is free from carbolic acid. It is then treated in the presence of resin and fatty matter by means of alkali, containing a certain percentage of sodium chlorid, when a light-brown finished fluid is obtained.

WEST DISINFECTING CO.

Farmers and others desiring to have work done similar to the above should apply to the Bureau of Chemistry, laboratory of insecticides and agricultural waters, U. S. Department of Agriculture, Washington, D. C., for directions for taking and forwarding samples.

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