After treatment, the oil showed a negative Kreis test and was returned to the original customer, who commented favorably upon the quality of this oil, and requested that future shipments be so selected to represent equal quality.

REFERENCES

1. Oil & Soap, Vol. 10, No. 6, June, 1933, pp. 105. 2. Industrial and Engineering Chemistry, 26, 3, pp. 245. 3. Oil & Soap, Vol. 13, No. 8, pp. 203 (Committee Report). 4. Journal Agri. Research, 26, 323 (1923).

METHOD REVISIONS

The 1936 revisions of the American Oil Chemists' Society official analytical methods are now available for distribution. The price per set of these revisions is 50c. Complete sets of methods revised to 1936 are available at \$3.00 per copy with binder and \$2.00 per copy without binder.

The Society maintains a list of members who desire to have revisions sent them each year. The Secretary advises that less than half of the membership is carried on this list. This is an extremely low figure in view of the fact that revisions are made annually, and in view of the rapid addition of new methods. All members should keep up to date on their methods. We therefore urge that all members not now on the Secretary's list for receipt of revisions each year, have themselves placed on the list immediately. Address correspondence to Mr. J. C. P. Helm, Secretary, 509 Tchoupitoulas Street, New Orleans.

SOAPS AND DETERGENTS

Committee D-12, Soaps and Detergents, of the American Society for Testing Materials, held an all day meeting and luncheon in the Hotel New Yorker, New York City, December 3, at which 46 were present.

The organization of the various subcommittees and sections was completed, and a program of work outlined. Plans were made for a spring meeting, at which all committees will report and all specifications on which work will have been completed by that time will be considered by the whole committee for adoption by letter ballot. Such specifications will then be submitted to the American Society for Testing Materials at their annual meeting for their consideration and action.

A number of the American Oil Chemists' Society members are active on this new committee and Mr. M. L. Sheely has been designated as our official representative. The methods of analysis of the American Oil Chemists' Society Soap Committee were considered by Committee D-12 and approved for submission for adoption by letter ballot.

USE OF MERCURATED FATTY COMPOUNDS AS WEED KILLERS*

By A. W. RALSTON, C. W. CHRISTENSEN and GEORGE JOSH

Armour and Company, Chicago, Ill.

I N A PREVIOUS paper presented before this Society the importance of finding new inedible uses for fatty acid derivatives was stressed. The object of this paper is to discuss the preparation of a series of mercury derivatives of fatty acids or their esters and the use of these compounds for the control of various weed pests.

The problem of weed control is one of considerable magnitude and large sums of money are expended annually for the killing of weeds along railroads and public rights of way. The usual methods employed by railroads are either to spray the weeds with a killing solution, such as a tar distillate, or else to actually burn the weeds by means of specially designed flame throwers. The principal objection to either of these methods is that the weeds are not permanently killed, due to the fact that the root system is left essentially unaffected and the weeds are quite apt to regrow.

Another interesting problem in this connection is the control of

weeds such as dandelions, plantain weeds and other common weeds in lawns, public parks, golf courses, etc. These are a constant source of trouble and the maintenance of the average lawn requires continual work to keep them under control. Here the problem is complicated by the fact that any proposed treatment must not have a destructive effect upon the grass and it is obvious that measures such as those employed by railroads cannot be used for the treatment of weeds occurring in lawns because such methods would kill the grass. The means of controlling weeds in lawns is essentially limited to various ingenious mechanical devices which have been developed, or to treating each individual root system with acidic substances, such as solutions of sulfuric acid.

From the above it appears that there is a very definite need for substances which will kill weeds by drying up the root system so that the weed cannot regenerate and is, therefore, permanently removed. It is also evident that if substances can be found which will preferentially kill weeds and not grass they can be employed for the control of weeds in lawns.

Realizing this need, a series of mercury derivatives of fatty acids was investigated for this purpose and it was found that very effective weed killers could be synthesized by the action of mercuric salts such as the acetate, chloride, nitrate, or iodide upon esters of unsaturated fatty acids. One of these compounds, methyl-9-acetoxy mercuri 10-methoxy stearate, has been extensively tested for this purpose. The method of preparation of this compound is as follows:

We start with 500 parts by weight of ethyl oleate dissolved in 500 parts by weight of methyl alcohol and to this we add 543 parts by weight of mercuric acetate. The reactants are placed in a flask equipped with a motor stirrer and reflux condenser and the flask is heated by means of a water bath to the refluxing temperature of the

*A paper presented at the Fall Meeting of the A. O. C. S., Chicago, October 8-9, 1936.

oil & soap

methyl alcohol. This refluxing is continued for from three to five hours until the mercuric acecompletely disappears, as tate shown by the fact that the solution becomes transparent. After the conclusion of this reaction the mixture is diluted with water and the heavy oil laver which separates to the bottom is washed with water until no further test for mercury is obtained in the wash water. The final product, which is methyl-9acetoxy mercuri 10-methoxy stearate, is a clear yellowish oil heavier than water. The reaction is as follows:

while the other half was treated with the emulsion or solution of the mercuric compound. It was found that the kerosene solution was much easier handled and more effective and the emulsion method of applying the compound was consequently abandoned. This was due to the fact that emulsions would break after several days' standing and also they were rather difficult to apply by spraying. The kerosene solutions, on the other hand, were extremely stable and no separation was noted after several months' standing. These plots contained both grass and weeds and

$$CH_{2} - 0 - CH_{2} + CH_{2}CH_{2}CH_{2}CH_{2} + Hg < 0 - CH_{3} + CH_{3}OH \longrightarrow 0$$

$$CH_{2} - 0 - CH_{3} + CH_{3}OH \longrightarrow 0$$

$$CH_{2} - 0 - CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{3} + HO - CCH_{3}CH_{3}$$

The theoretical percentage of mercury in this compound is 34.2 per cent and the product obtained by us contained 33.8 per cent.

This compound is very soluble in organic solvents such as alcohol. ether, naphtha, kerosene, benzene, etc.

Several compounds quite similar in structure have been prepared, among which may be mentioned ethyl-9-acetoxy mercuri 10-ethoxy stearate, methyl - 9-chloromercuri 10-methoxy stearate and ethyl 9chloromercuri 10-ethoxy stearate, all of which were heavy oils. A series of similar products has been prepared employing esters such as ethyl linoleate. When triglycerides were employed the products were pasty solids soluble in the usual organic solvents. Preliminary tests indicated that all of these compounds possessed marked weed killing power, and it was decided to run a series of exhaustive tests upon one of them to determine its specificity for this purpose.

For this series of tests we selected the methyl 9-acetoxy mercuri 10-methoxy stearate. Two general methods of procedure were at first employed: First, a water emulsion of the compound was prepared by using soaps or other emulsifying agents and this emulsion used as a spray; and, second, we dissolved the compound in kerosene and sprayed the solution. Test plots were selected and one-half of the plot sprayed with the blank

they were sprayed with solutions of various strengths until concentrations were found which would destroy the common weeds, such as dandelions or plantain, without any noticeable effect upon the grass. As a result of this study it was found that a 0.1 per cent solution of the methyl 9-acetoxy mercuri 10-methoxy stearate in kerosene completely destroyed weeds such as dandelions and had a very limited destructive effect upon grasses and various other monocotyledonous plants. One liter of solution was used per 50 square feet. The effect was noted in twenty-four hours and is to completely dry up the root system of the weed. Observations showed that the dandelion root first becomes very soft and mushy and then dries completely. This effect will be noted upon the root while the top is still green and apparently unaffected.

These mercury derivatives are specific for the removal of weeds having milk or latex containing roots, such as those belonging to the family of Compositae, which includes thistles, dandelions and sunflowers, or those belonging to the family Asclepiadaceae, which includes milkweeds. Those belonging to the genus Plantage, such as the common plaintain weed, are somewhat more resistant, but can be controlled without permanent injury to the grasses. It is our opinion that the root systems of the weeds which we have men-

tioned possess a specific affinity for this type of mercury derivative and that when these mercury compounds are sprayed upon the soil the root system of the weed specifically absorbs the compound thus resulting in the death of the weed. The root systems of grasses differ somewhat from those of the weeds and no not appear to absorb the compounds from the soil except when high concentrations are employed. As a result it is possible to spray a lawn with this solution and effectively control the weeds without injuring the grasses.

Observations extending over several months indicate that where an area is once treated new weeds do not appear. This suggests that the lawn could be treated easily in the spring and weed growth prevented. The tests reported upon above have been continued over a period of two years under various types of climatic and weather conditions.

When railroads or public rights of way are treated higher concentrations can, of course, be employed or the compounds could be dissolved in the tar distillates now employed so that permanent killing of the root system could be accomplished.

DISCUSSION

President Ainslie: Are there any questions?

Question: Have you done any work on crab grass?

Mr. Ralston: I think that crab grass would have to be considered as a grass.

Question: How about white clover?

Mr. Ralston: Clovers are less resistant than the grasses, although I think it is possible to treat in the presence of clover.

Question: I am curious to know whether you have any hypothesis as to how the mercury, or mercurycontaining compound gets at the root. Obviously the kerosene solution is not able to penetrate the particles of soil moistened with water down to any considerable depth, to contact with the root of the weed. Have you any explanation as to how that occurs?

Mr. Ralston: Of course one would have to assume a contact, and the explanation is that the root specifically absorbs the compound from the soil. In order to do that, one would have to postulate some means whereby it came in rather intimate contact—the spraying of course is done with a very fine spray. I cannot say exactly how it gets in; when the contact is very close, we explain it as a specific adsorption.

Question: You spoke of weed seed not growing. Would it have any influence on the grass seed growing?

Mr. Ralston: The grass will grow; it has no influence on grass. Question: Will not new seed of

grass be affected?

Mr. Ralston: No. As a matter of fact, in some of the earlier tests where we offered to treat areas and the grass was killed, they were replanted immediately with no effect upon the grass, but the weeds did not grow with the grass.

Question: What is there about it that makes it selective?

Mr. Ralston: That is a very complicated question.

Question: You may class the dandelion as a weed, and it may be so in this section of the world, but there are other sections in which it is known as the houseplant. I should like to know what makes it selective, why it kills a dandelion and not grass.

Mr. Ralston: I should say this, that a weed is characterized by a very rapidly growing root, and my observation is, although I know very little about plants, that a weed root is very much more porous, owing to the fact that it grows much faster than a grass root. I want to make this point clear, also, that this is not a qualitative distinction, it is quantitative. The adsorption in a weed root is so much faster and is with so much greater rapidity than it is in a grass root, that it is posible to treat one in the presence of the other if you limit the concentration so that essentially all of it will be adsorbed by the weed root. Then you get a killing effect upon the weed and an absence of effect upon the grass. If you over-treat it, the whole area dies. We found that out. We got some bad shocks when we started—the lawns did not look very well.

Question: Have you tried it on poison ivy?

Mr. Ralston: No, we have not. Mr. Harris: I hope you will forgive a pardonable skepticism (do not let me be misunderstood), it is rather a little pardonable curiosity on the part of a suburbanite, but have you any data as to the time interval between the initial spraying and let us say the initial attack on the weed?

Mr. Ralston: Yes. If you spray an area in the evening, by the next morning the weeds will be essentially gone, they will be starting to die, in a period of approximately 24 hours. Initial observations which were made in Iowa on lawns showed that it was over night.

Mr. Harris: If that is the case I would like to volunteer a trademark for it—"Suburbanite's Delight."

Mr. Ralston: If you will permit a personal observation—while this was under test, I felt very foolish going out and pulling my weeds with a weed puller. Nevertheless, one must realize that where you have a thing of this nature, you certainly have a lot of work in determining what concentration must be used, and in what amount, and we have found, for instance, that various weather conditions affected the results. If we spray and then there is a heavy rain, the results are not as good.

Question: Is the compound toxic to animals?

Mr. Ralston: Probably, yes. We have rubbed it on the skins of animals without toxic effect. However, I think if animals ate a lot of the weed roots which had been treated there would be a toxic effect. It, therefore, probably could not be recommended for places where cattle range or something of that sort. Its toxicity is not anywhere near what we thought it was at first, however. The tests were run on guinea pigs.

Question: Has it any effect on insect pests?

Mr. Ralston: Some observations have been made, yes. It kills worms and seems to kill essentially all the insects which bore under the ground. They come to the surface. That has been observed during the period of these tests.

Member: It seems to me, judging from this discussion, that oil chemists are more interested in lawns than they are in oil.

President Ainslie: I was feeling just that way.

RAPID DICHROMATE METHOD For the Determination of Glycerol In Soaps and Spent Lyes*

By E. RANDA

Armour and Company Soap Works, Chicago, III.

Introduction:

The Dichromate Method is commonly used for the determination of glycerol in soaps and spent lyes. However, there are many modifications of this method in use by various industrial laboratories and it is seldom that one finds two laboratories using exactly the same procedure. Several Armour and Company chemists have participated in the development of a reliable and comparatively rapid Dichromate Method which has been in use in

our laboratory for the last several years.

New Features of Method:

1—Indicator. The method employs diphenylamine as an internal indicator and differs in this respect from the standard published methods. The A.C.S.¹ method uses an external indicator, namely, potassium ferricyanide; the A.O.C.S.² method specifies starch solution as indicator to titrate the liberated iodine. The application of diphenylamine in our method has been suggested to us by the paper authored by J. Knop⁴ and entitled "Diphenylamine as Indicator in the Titration of Iron with Dichromate Solution." Our experience shows that the end point using this indicator is very sharp and definite and facilitates titration. These findings coincide with those of Knop who states in his paper that

a—"The use of diphenylamine as indicator in the titration