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

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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. Knop[3] 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

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of iron with dichromate solution . . . increases the accuracy of the determination of iron.

b—"The diphenylamine method is much less dependent on the proportion of iron taken than are other methods.

c—"The titration of iron salts which have been reduced by stannous chloride is rapid and very accurate . . ."

2—*Standardization of Dichromate Solution.* It is not common practice in the published methods to standardize the dichromate solution. We have found it advisable to do this since our experience has shown that the dichromate solution prepared according to the A.O.C.S. and A.C.S. methods does not always represent the exact strength desired. This may be accounted for by the purity of the dichromate used, errors in weighing and in making up to volume, etc. Slight errors in the dichromate solution will appreciably affect the final results.

In preparing the standard dichromate solution the quantity of potassium dichromate used is such that 1 c.c. of the standardized solution is equivalent to approximately 0.01 grams of glycerol. The exact glycerol equivalent is determined by standardization of this solution against standard iron wire using diphenylamine as indicator.

3—*Use of Phosphoric-Sulfuric Acid Solution.* A few c.c. of a mixture of these acids are added to the standard ferrous ammonium sulphate solution prior to titration with the dichromate. As pointed out by Knop, the use of this acid mixture avoids interference with the sharpness of the end-point caused by the troublesome yellow color of ferric chloride.

4—*Method of Titration.* The sample, after the usual clarification procedure, is oxidized with the standard dichromate solution. The oxidized solution is made up to volume in a 300 c.c. volumetric flask. This is followed by filling a burette with this solution and titrating same against a measured amount of standardized ferrous ammonium sulphate, using diphenylamine as an indicator. The end point is an intense dark blue that remains unchanged on further addition of dichromate.

5—*Accuracy of Method.* Concordant results have been obtained when the Armour method was checked against the standard A.C.S[1] method on identical samples of spent lyes and on aqueous solutions containing known amounts of glycerol.

A sample was made in our laboratory by adding a known amount of C.P. glycerin to distilled water. This sample was checked by a very careful specific gravity determination. Calculating from the amounts of C.P. glycerin and water used, the sample contained 5.56 per cent glycerol. According to the specific gravity determinations, the figures showed:

Test No. 1—Specific gravity at 15.0/15.0°C—1.01333—5.51%.

Test No. 2—Specific gravity at 15.0/15.0°C—1.01337—5.53%.

Average—5.52%.

Using our dichromate method on this sample we obtained the following results:

Test No. 1—5.49%
Test No. 2—5.49%

Obviously, the results obtained with our method checked very closely with the known amounts of glycerol contained in the sample.

In order to preclude the possibility that the impurities commonly found in glycerin spent lyes did not interfere with the method, we have prepared and tested a sample of synthetic spent soap lye.

The synthetic spent lye was prepared using a known amount of C.P. glycerin, water, soap, salt, fatty acids, soda ash and free alkali in proportion usually found in this type of material. The per cent of glycerin added to this sample was 4.53 and on analysis using our method was found to be 4.47.

Further experimental work was done on various samples of glycerin spent lyes, checking this method against the standard A.C.S. method on identical samples. Below are listed results obtained using both methods:

	Armour Method % Glycerol	A. C. S. Method % Glycerol
Sample No. 1........	5.07%	5.01%
Sample No. 2........	7.31	7.37
Sample No. 3........	5.12	4.96
Sample No. 4........	4.92	5.01
Sample No. 5........	6.45	6.42

Cooperating with our Eastern laboratory, we have made determinations at various times on identical samples of glycerin spent lyes using both the Armour method and the A.C.S. method with the following results:

	Laboratory No. 1	Laboratory No. 2
Sample No. 1—		
A. C. S. method....	5.13%	5.11%
Armour method....	5.16	5.13
Sample No. 2—		
A. C. S. method....	3.70%	3.70%
Armour method....	3.67	3.68

The method in detail is given as follows:

Detailed Procedure

REAGENTS REQUIRED

(a) *Potassium Dichromate Solution*—Dissolve 76 g. of potassium dichromate in distilled water and make up the solution to 1 liter. Mix thoroughly and allow to stand over night before standardizing.

(b) *Ferrous Ammonium Sulfate Solution*—Dissolve 30 g. of ferrous ammonium sulfate in distilled water, add 100 c.c. of concentrated surfuric acid (Sp. Gr. 1.84), cool and dilute to 1 liter, mix thoroughly and allow solution to stand over night before standardizing.

(c) *Phosphoric-Sulfuric Acid*—Mix 150 c.c. of concentrated sulfuric acid (Sp. Gr. 1.84), and 150 c.c. of phosphoric acid (85 per cent syrupy) with 700 c.c. of distilled water.

(d) *Diphenlyamine Indicator Solution*—Dissolve 1 g. of diphenylamine in 100 c.c. of concentrated sulfuric acid (Sp. Gr. 1.84).

(e) *Lead Subacetate Solution*—Prepare saturated solution of lead subacetate.

(f) *Stannous Chloride Solution*—Dissolve 6 g. of C.P. stannous chloride in water, add 60 g. of concentrated hydrochloric acid (Sp. Gr. 1.178—1.185) and dilute to 100 c.c. Add a few pieces of pure tin to the solution which will continue to be satisfactory for use so long as there is tin at the bottom of the reagent bottle.

(g) *Mercuric Chloride Solution*—Prepare a saturated solution of C.P. mercuric chloride.

(h) *Iron Wire*—B&S Gauge No. 30 for Standardizing. Iron content 99.8 per cent.

STANDARDIZATION PROCEDURE

Potassium Dichromate Solution—Weigh accurately three samples of 0.25 to 0.35 gms. of standard iron wire and place in 300 c.c. Erlenmeyer flasks. Add about 30 c.c. of conc. hydrochloric acid (Sp. Gr. 1.178—1.185), leave samples on a hot plate until wire is dissolved, remove and while hot reduce by adding stannous chloride drop by drop until the yellow color just disappears. Then dilute with 100 c.c. of cool (70° F.) distilled water and as soon as samples are at room temperature add 10 c.c. of a saturated solution of mercuric chloride. Rotate Erlenmeyer flasks a few times and allow to stand for a few minutes before proceeding further. Then to each of these solutions add 20 c.c. of phosphoric-sulfuric acid solution and five drops of diphenylamine (indicator). Dilute one part of the potassium dichromate solution to be standardized with 9 parts of water, add to a burette and titrate directly into the solutions in the Erlenmeyer flasks. Near the end point the green color of the solution deepens to a blue-green or grayish-blue and at this stage add the dichromate slowly. The end point is taken at the point where a drop causes an intense dark blue coloration that remains unchanged on further addition of dichromate.

CALCULATIONS

1 g. Fe = 0.8781 g. $K_2Cr_2O_7$

1 g. $K_2Cr_2O_7$ = 0.1341 g. Glycerol

1 g. Fe = 0.1178 g. Glycerol

$$\text{Glycerol equivalent per c.c. of } K_2Cr_2O_7 = \frac{\text{g. Fe} \times 0.1178 \times 0.998}{\text{titration} \times 0.1}$$

Ferrous Ammonium Sulfate Solution.—Fill a burette with the ferrous ammonium sulfate solution to be standardized and run 20 c.c. into a 375 c.c. porcelain casserole, then add 20 c.c. of phosphoric-sulfuric acid and five drops of diphenylamine (indicator). Dilute 1 part of the previously standardized potassium dichromate solution with 9 parts of water and titrate this solution into the ferrous ammonium sulfate solution. The end point is taken at the point when a drop causes an intense dark blue coloration that remains unchanged on further addition of dichromate.

CALCULATIONS

20 c.c. Ferrous Ammonium Sulphate $\Bumpeq$ x g. Glycerol

x g. Glyc. $\Bumpeq$ Titra. of $K_2Cr_2O_7$ × Glyc. equiv. per c.c. of $K_2Cr_2O_7$

Since the potassium dichromate solution is diluted 1 part to 9 parts of water for titrating, the above equation would read as follows:

Let T = Titration

Let V = Glycerol Equivalent per c.c. of $K_2Cr_2O_7$

20 c.c. F.A.S. = 0.1 VT

Then how many c.c. of F.A.S. = 0.01 gm. Glyc.

Let x = No. of c.c. of F.A.S.

$$\frac{x}{0.01} = \frac{20}{0.1\ VT}$$

$$x = \frac{0.2}{0.1\ VT}$$

$$x = \frac{2}{VT}$$

NOTES ON STANDARDIZATION

1—*The Standard Dichromate Solution,* if kept in a stoppered bottle, is very stable and shows practically no change in concentration over a period of several weeks. Therefore, when standardization is first made, sufficient solution can be made up to last several weeks and it is unnecessary to restandardize the solution from time to time as it is being used.

2—*The Ferrous Ammonium Sulfate Solution* is not as stable as the dichromate solution and should be standardized against the standard dichromate solution about every other day.

THE DETERMINATION OF PER CENT GLYCEROL IN SOAPS

Dissolve a 10 g. (± 0.01 g.) sample of the soap in a minimum amount of hot distilled water in a 250 c.c. beaker. (If starch is present, it will be necessary to remove the matter insoluble in water using the standard A.O.C.S. method.[2] Combine the alcohol and water solutions, evaporate off the alcohol, and proceed.) Add sufficient glacial acetic acid (50 per cent by volume) to split all of the soap and allow solution to remain on steam bath until there is a clear separation of the fatty acids. Cool, remove, and rinse the cake of fatty acids with distilled water. (Due to trouble encountered in the formation of silicic acid, heavily silicated soaps are not acidified but are treated directly with basic lead acetate solution.) To the acid water and washings, add a small amount of basic lead acetate solution, filter out any precipitate formed and test filtrate with lead acetate to be certain that all organic matter has been precipitated. Then add sulfuric acid (1:4) to the solution in the beaker to precipitate the excess lead. Filter the solution through a good quality filter paper into a 300 c.c. volumetric flask and wash the precipitate with small portions of cold (70° F.) distilled water. Test the filtrate with a small amount of sulfuric acid (1:4) to be certain that all of the excess lead has been precipitated. The filtrate should be clear with no precipitate present before proceeding further in the determination.

To the solution in the 300 c.c. volumetric flask add from a burette 25 c.c.** of standard dichromate solution, and then while carefully rotating the flask add slowly 25 c.c. of conc. sulfuric acid (Sp. Gr. 1.84). Place flask on a steam bath temperature about 85° to 90° C.) and allow solution to oxidize for 1½ hours. Then remove, cool to room temperature, and dilute exactly to the 300 c.c. mark. Fill a burette with this solution and titrate against the standard amount (approx. 20 c.c.) of standard ferrous ammonium sulfate to which has been added 20 c.c. of phosphoric-sulfuric acid and five drops of diphenylamine sulfate indicator. The end point is an intense dark blue color which shows up very definitely when titrating into a porcelain casserole. The percentage of glycerol is calculated as follows:

$$\frac{\text{Glyc. equiv. for c.c. of } K_2Cr_2O_7 - \left\{\dfrac{300 \times 0.01}{\text{c.c. titration}}\right\}}{\text{Wt. of sample}} \times 100 = \%\ \text{Glycerol}$$

Determination of Glycerol in the Presence of Sugar

Use procedure given above with the exception that the solution must be boiled at least 20 minutes using an air condenser to insure complete inversion of sugar. Determine the amount of standard dichromate solution required to oxidize both the sugar and glycerol. Determine also the per cent sugar using the standard A.O.C.S. method.[2]

Calculate the percentage of glycerol after deducting the amount of dichromate required by the sugar. If 1 c.c. potassium dichromate solution equals 0.0100 g. glycerol then 1 c.c. of potassium dichromate will equal 0.01142 g. invert sugar.

Determination of Glycerol in Spent Lyes

Follow exactly the above procedure for determination of glycerol in soaps with the following exceptions:

Weigh 25 g. (± 0.05 g.) of spent lye into a 250 c.c. beaker, add water to dilute, if necessary. Carefully add basic lead acetate solution until no more precipitate is thrown down. Filter the solution through a good quality filter paper into a 250 c.c. beaker and wash the precipitate 3 or 4 times with cold

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**The amount of dichromate solution to add depends on the approximate percentage of glycerol in the solution to be oxidized. Up to 6% glycerol add 25 c.c.; from 6% to 12% about 35 c.c.; from 12% to 20% about 50 c.c. It is imperative that an excess of dichromate be used. If there is a sufficient amount of dichromate present, the glycerol solution after oxidizing will have a reddish brown color and when there is an insufficient amount of dichromate, the solution will have a green color.

(70° F.) distilled water. Test the filtrate with basic lead acetate which should produce no precipitate and then add sulfuric acid (1:4) to precipitate the excess lead. Filter the solution into a 300 c.c. volumetric flask washing the precipitate with cold water and test filtrate with a small amount of acid to be certain that all excess lead has been precipitated. The filtrate is diluted to the 300 c.c. mark and a 25 c.c. aliquot of the filtrate is used for the glycerol determination.

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THE SOLVENT EXTRACTION OF SOY BEANS

By W. E. MEYERWEISSFLOG

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DURING the last couple of years a good deal of attention and work has been done on the continuous extraction of vegetable oil seeds by means of solvents.

The results obtained with this most modern method of oil production seem to deserve a short description of this process as well as some brief remarks about the experiences with it in practice.

Pressing.

It has not been long since pressing was the only possible method to get the fat content out of oleaginous substances in a satisfactory manner.

But in spite of the great progress made in the meantime in constructing oil presses, it had not yet been possible to reduce the remaining fat content in the press cake below 5 to 7 per cent.

This remaining oil content represents a loss which has been unavoidable so far. It is a practical loss because the oil left in the cake can not be sold at the high oil price but only at the much lower cake and meal price.

Especially in working poor fat containing oil seeds, as soy beans, for instance, where the residual fat content represents almost one-third of the whole oil content, the possibility of avoiding such a great loss logically is of greatest commercial interest.

Extraction in Kettles.

Towards the middle of the last century the solution of the old problem of extracting oil seeds by means of solvent so as to obtain as nearly as possible the complete oil content was accomplished for the first time. At that time the extraction was done with carbon bisulphide.

The extractors generally consisted of kettle-like containers or rotary cylinders. Their operation was somewhat complicated. They had to be charged first with the material to be extracted; second, with the proper amount of solvent and discharged and cleaned after extraction.

Initially, considerable work was done on extracting olive pulp by the batch process; later the process was applied to other seeds.

Continuous Extraction in General.

The deciding forward step in the development of the extraction process, however, has only been made within a very short time. Wide use of industrial continuous extraction processes is only about five years old.

Although the kettle extraction compared with the pressing method offered many advantages, it could but partly satisfy because of its complicated working system. The main efforts of the industry in regard to extraction on a large scale have been in the direction to automatize the extraction process and to develop it as a continuous working system.

In view of the enormous quanti-