Menhaden is from 175 to 185 and Sardine from 180 to 195. Since the low melting point fats or stearine are non-drying, their iodine is low. Thus the iodine value of a pressed oil to some extent indicates the amount of destearinization and drying value of the oils.

From pressed oils are made various kinds of processed and refined oils, such as alkali refined oils, which process removes the free fatty acids, color and largely deodorizes the oil. Blown oils are made to any selected viscosity. Deodorized oil is made from refined and specially processed oil.

Generally speaking, the following uses are made of the different grades of processed and refined fish oils: From pressed oil is made stack paint, where heat resistance is required. In combination with Linseed or China Wood Oil it is used for barn paints, and largely in industrial maintenance paints which are exposed to sulphur fumes, salt air, etc. It is recommended in rosin and oil driers for the reason that fish oil is less apt to separate than vegetable oils.

Alkali refined oil as a kettle oil bodies rapidly, holds its color well, deodorizes easily with heat and is used in combination with other oils for mixed paints. It gives good washing and brushing properties to flat wall paints. The combination of Wood Oil and Fish Oil makes an excellent exterior cement paint, is a very good vehicle for aluminum paint—its flexibility and waterproofness are especially good, and its leafing properties with aluminum are excellent.

Blown oils are used as a base for cold cut paint oils where varnish fires are not available. These oils are clear and free from haze. They are used to make colored asbestos roof coatings and are added to red, green and brown Linseed Oil paints to prevent chalking, blistering and peeling. Some blown oils of heavy body are prepared with approximately 25% light mineral oils in order to give the finished paint a fuller body than could be acquired by the use of pure blown oil cut back in the kettle with thinner.

Truly deodorized refined oils are used successfully for white enamel, flat wall paints, interior and exterior mixed paints and undercoatings. The preparation of deodorized oils in the film trades is distinctly new and just emerging from the experimental stages.

In 1929 the amount of fish oils of all kinds used was about 180,000,000 lbs., split up among the following industries:

Paint and varnish	5.6%
Linoleum, oilcloth, etc.	5.4%
Edible fat	8.0%
Soap	69.4%
Miscellaneous	11.6%
Total	100.0%

In the Paint and Varnish trade:

	(lbs.)
Linseed	340,000,000
Wood Oil	88,300,000
Fish Oil (3% of Linseed)	10,600,000
Soya	5,800,000
Perilla	2,800,000

In Oil cloth and Linoleum:

	(lbs.)
Linseed	112,850,000
Fish Oil (9% of Linseed)	10,140,000
Wood Oil	6,000,000
Soya Bean Oil	3,200,000

The following are approximate specifications for various grades of Fish Oil:

Light Pressed Fish Oil—

Iodine	175-185
Saponification Value	190-193
Acid Number	5 maximum
Color	Amber

Low Cold Test Oil: All figures are the same except that a cold test of two hours at 32° and an iodine value of 185-190.

24° Cold Test Oil: Has the same specifications except iodine of 190-195 and a cold test clear two hours at 24°.

The same grades of oil can be obtained Alkali Refined, if desired.

REPORT OF THE GLYCERIN ANALYSIS COMMITTEE—SOAP SECTION, AMERICAN OIL CHEMISTS' SOCIETY

Early in 1934 Mr. O. C. Ames, 4906 Davenport Street, Omaha, Nebraska, reported to the Society that he had devised a modification of the acetin method which he believed would yield results within 0.25% of the actual glycerin content. On being assured of our willingness to cooperate, the details of his modified method were disclosed as follows:

"Follow the Standard International Acetin Method but with these modifications: After acetylization is complete allow the reaction mixture to come nearly to room temperature before addition of water, then add the required 50 cc at a temperature of not much over 50° C. and, by giving the stand, flask and all a rotary rocking motion, bring to solution as quickly as possible and immediately on solution, surround the flask with a small ice and water bath and chill thoroughly. A liter beaker is convenient.

A rather large bath of ice and water should be at hand in which the supply of distilled water and the wash bottle have been supported and well chilled.

Support a liter conical in the bath and as soon as the triacetin solution is thoroughly cold, filter at once into this flask, using a rapid filter paper, and wash with the chilled water, rinsing the acetylization flask well and bringing the solution up to a volume of about 300 cc.

Proceed at once to the neutralization. Two burets, one 25 cc and one 100 cc, the latter with a very free flowing tip, make the most satisfactory apparatus. The approximate amount of alkali should be calculated. Ex.: If 1.3 gm. sample of glycerin has been taken and 7.5 cc of anhydride has been used, about 100 cc of normal alkali will be needed, assuming about 85% true glycerol content. The use of chilled alkali is probably not practical. The large buret should be able to discharge 90 or 95 cc in not more than 20 seconds.

With vigorous rotation of the flask add the most, 90% or better, of the neutralizing alkali rapidly, buret wide open. Proceed to the end point with the small buret and with suitable caution.

The balance of the analysis per standard method."

Mr. Ames points out that triacetin hydrolyzes slowly in acetic acid solution and that this hydrolysis is greatly affected by the temperature of the solution. Furthermore he states that the neutralization of acetic acid with NaOH is practically instantaneous, while the saponification of triacetin proceeds more slowly. From these considerations he concludes that slow neutralization is not necessarily careful neutralization and that rapid addition of 90-95% of the alkali with vigorous agitation is least favorable to accidental saponification of triacetin during neutralization.

Samples of "A.O.C.S. Standard C. P. Glycerin—1933," which were available from our work last year[1], were distributed to members of the Committee with the request that it be analyzed as follows:

A. Specific gravity by pycnometer method, at one of the temperatures covered by the Bosart and Snoddy Table.

B. % Apparent glycerol from specific gravity.

C. % Apparent glycerol by International Acetin Method (uncorrected for

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[1] Oil & Soap, Vol. II, No. 3—p. 53 (March, 1934).

acetylizable impurities in the organic residue at 160° C.).

D. % Apparent glycerol by Mr. Ames' modification of the I.A.M.

Nine members of the Committee contributed experimental data which have been tabulated. One member obtained slightly lower results by the Ames modification than by the I.A.M.; one noted little difference by the two methods; one reported approximately 0.9% higher by the Ames procedure (which checked glycerol from specific gravity within the limit of accuracy of the latter determination); and six showed apparent glycerol by the Ames modification to be higher than that by the I.A.M. by differences ranging from 0.1% to 1.2%. It will be noted, however, that this last difference is due to the low figure obtained by the regular method which was the lowest reported by any laboratory.

The average difference for all laboratories is 0.33% in favor of Mr. Ames' modified procedure. This value is 0.69% below the average % glycerol from specific gravity which is believed to be an accurate index of the true glycerol content of this highly purified material. Incidentally the values for apparent glycerol obtained from specific gravity, 91.02%, and by I.A.M., 90.00% are in close agreement with the corresponding averages obtained on the same sample last year which were, respectively, 91.05% and 90.10%.

"A. O. C. S. STANDARD C. P. GLYCERIN—1933"

Laboratory	A-Specific Gravity	% Apparent Glycerol B-From Sp. Gr.	C-By I.A.M.	D-Ames
1.	1.23853 (25°/25° C.)	91.01	89.80	90.10
			(av. of 2)	(av. of 2)
2.	1.23864 (25°/25° C.)	91.05	89.37	90.60
3.	1.23844 (25°/25° C.)	90.98	90.08	90.60
			90.02	90.27
			90.10	90.41
4.	1.2382 (25°/25° C.)	90.89	89.73	89.56
			90.10	90.13
			89.80	89.90
			89.56	89.82
5.	1.2418 (15.5°/15.5° C.)	91.00	90.07	90.99
6.	1.2422 15°/15° C.	91.04	89.98	90.42
	1.2421	91.00	89.84	90.27
			89.97	90.02
			89.91	90.52
				89.35
7.	1.24020 (20°/20° C.)	91.00	[1]90.22	[1]90.58
			[1]90.30	[1]90.88
			[2]90.30	[2]90.44
			[2]90.34	[2]90.66
8.	1.23885 (25°/25° C.)	91.13	90.16	90.28
9.	1.2421 (15.5°/15.5° C.)	91.09	90.42	90.08
			90.62	90.39
			90.50	90.21
Average (of all laboratories)		= 91.02	90.00	90.33

[1] Analyst A.
[2] Analyst B.

Mr. Doherty, Mr. Hoyt and Mr. Sheely all encountered difficulty with the end point while neutralizing in the Ames procedure. Mr. P. C. Clark, working with Mr. Hoyt, comments as follows:

"The end point while neutralizing the acetic acid under Ames modification is very indefinite. It disappears very soon, whereas the end point in the standard I.A.M. method remains at least 15 minutes. The neutralization of the ice-cold solution seems to throw the phenolphthalein out of solution as the end point is approached, necessitating the addition of more indicator at the end point. It seems possible to this analyst that the higher result is due to an uncertain neutral point or possibly to unhydrolyzed acetic anhydride. False end points such as occur in this method always raise doubt as to the final results."

Mr. Sheely remarks that his results seemed to be more consistent by the I.A.M. and ascribes part of this to the fact that he was unable to obtain a very definite end point in the Ames modification when neutralizing the excess acetic acid. "Shortly before the end point was reached the solution became milky, making the end point very obscure. In several cases it seemed as though the end point had not yet been reached, but on continued shaking the solution slowly became strongly pink and on back titrating it was found that the end point had been passed by as much as one or two cubic centimeters." Such results were not reported for tabulation.

Messrs. Hoyt, Sheely and Clark agree that this poor end point is due to incomplete hydrolysis of acetic anhydride and in support of their belief cite an extract from the "Report of the Subcommittee on Glycerin Analysis," J.I.E.C. 3, 679-686 (1911). On page 683 under "Reagents Required" appears the following statement:

"The anhydride may be tested for strength by the following method: Into a weighed stoppered vessel, containing 10 to 20 cc of water, run about 2 cc of the anhydride, replace the stopper and weigh. Let stand with occasional shaking for several hours, to permit the hydrolysis of all the anhydride; then dilute to about 200 cc, add phenolphthalein and titrate with N/1 NaOH. This gives the total acidity due to free acetic acid and acid formed from the anhydride. It is worthy of note that in the presence of much free anhydride a compound is formed with phenolphthalein, soluble in alkali and acetic acid, but insoluble in neutral solutions. If a turbidity is noticed toward the end of the neutralization it is an indication that the anhydride is incompletely hydrolyzed and inasmuch as indicator is withdrawn from the solution, results may be incorrect."

Mr. Hoyt suggests that "if one of the difficulties with the I.A.M. is partial decomposition of triacetin in acetic acid solution, might not the addition of an excess of sodium acetate at the time the acetylated sample is being brought into solution tend to prevent this?" He ran two acetylations of the standard sample and, after cooling to room temperature, added 50 cc of water containing 25 gms. NaAc. $3H_2O$. The solutions were allowed to stand for two hours with occasional shaking. There were a few oily droplets at the bottom of the samples but none in the blank. On dilution to 300 cc, without filtering, these oily drops dissolved readily. The analyses were finished in the usual way by I.A.M. and showed 90.71% and 90.60% compared to 90.63% and 90.54% by I.A.M. not filtered. These values are very close to Mr. Hoyt's results by the Ames procedure and are about 0.3% higher than his analyses by I.A.M. (filtered).

The same idea of repressing hydrolysis of triacetin in acetic acid solution by addition of NaAc. $3H_2O$ to the acetylated samples occurred independently to Messrs. Reese and Andrews. Mr. Reese gave no figures but stated that his analyses "heavily buffered with NaAc. gave results in harmony with the regular method." Mr. R. C. Stillman, working with Mr. Andrews, using a procedure similar to Mr. Hoyt's but filtering after neutralization by the Ames method found an average of 90.62% apparent glycerol compared with 90.51% by I.A.M. and 90.23% by the Ames method.

Mr. Sheely ran one series of tests by the Ames method without filtering and found his results to be much less consistent than those which were filtered. He ascribes this result to more complete hydrolysis of the anhydride during the time consumed by filtration. He states that a better neutralization end point was obtained on the filtered samples. Mr. Sheely also applied Mr. Ames' procedure to two crude glycerins and obtained averages of 81.50% and 82.61% against 81.20% and 82.01% by I.A.M.

From our work this year it is fairly obvious that Mr. Ames has demonstrated successfully the truth of two of his conclusions:

1. Careful neutralization is not necessarily slow neutralization.
2. Hydrolysis of triacetin in acetic acid solution is a very likely source of error in the I.A.M. This hydrolysis may be reduced and perhaps prevented by chilling in ice water.

The Ames modification in general yields results higher than the I.A.M. yet still somewhat lower than actual glycerol content.

Certain features of the Ames procedure, notably the difficult end point in neutralization, require further investigation before it can be recommended as an improvement over the standard method.

The possibility of minimizing hydrolysis of triacetin in acetic acid solutions by addition of sodium acetate should not be overlooked in future work on glycerin analysis by the acetin method.

Your committee wishes to acknowledge its indebtedness to Mr. O. C. Ames for his valuable and stimulating contributions toward the solution of our problem.

Respectfully submitted,

GLYCERIN ANALYSIS
COMMITTEE,
J. T. R. Andrews, Chairman.
Ivorydale, Ohio.
October 1, 1934.

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W. J. Reese, Colgate-Palmolive-Peet Company, Kansas City, Kansas.
M. L. Sheely, Armour Soap Works, Chicago, Illinois.
J. T. R. Andrews (Chairman), Procter & Gamble Company, Ivorydale, Ohio.

Jack Harris Returns to Nuchar

The Industrial Chemical Sales Co., Inc., has been forced into a program of expansion of its sales service and development personnel through the amazingly rapid acceptance and demand for its activated carbon "Nuchar" and its precipitated chalk "Snowtop" in so many fields, and because of several new products which the Industrial Chemical Sales Company, Inc., now has ready for marketing.

And so John P. Harris has returned again to take charge of the Chicago office at 205 W. Wacker Drive. His constructive, aggressive leadership assures the same success in the new fields which has marked this company's previous sales expansion campaigns in the middle west under his direction.

Mr. Harris will be assisted by W. A. Welch, formerly chief chemist of the Industrial Chemical Sales Co., Inc., who is already well known for his development work.

Mr. Harris and Mr. Welch cordially invite all out-of-town OIL & SOAP chemists to make their offices, Suite No. 1511 of the Engineering Building, 205 W. Wacker Drive, their headquarters when visiting in Chicago.

A STUDY OF THE COMPOSITION OF AMERICAN TUNG OIL

By ROBERT S. McKINNEY and GEORGE S. JAMIESON

A CONTRIBUTION FROM THE OIL, FAT AND WAX LABORATORY, BUREAU OF CHEMISTRY AND SOILS, UNITED STATES DEPARTMENT OF AGRICULTURE

Tung oil is obtained from the seeds of two trees, Aleurites fordii, and A. montana, both indigenous to China. In comparatively recent years cultural experiments, chiefly with A. fordii, which is the principal source of the Chinese oil of commerce, have been undertaken in various more or less semi-tropical regions in Africa, Australia, New Zealand and North and South America. In numerous instances these plantings have been made with seed produced in Florida, the pioneer state in the attempt to establish a tung oil industry in the United States. Up to the present time more than 40,000 acres have been planted to tung trees in the States of Alabama, Florida, Georgia, Louisiana, Mississippi and Texas.

Since 1928 the seeds (commercially called nuts) not required for planting, produced in Florida, have been crushed for oil by the Alachua Tung Oil Company near Gainesville. Another mill at Bogalusa, Louisiana, is about to begin operation. Plans also are under way in several other places for the establishment of plants to handle the locally produced seed.

For those who desire a knowledge of the development and history of this new American industry, attention is called to publications*, which may be found in many libraries.

The present investigation was made on a sample of expressed Florida tung oil furnished by Henry A. Gardner of the National Paint, Varnish and Lacquer Association. As determined by the regular Hanus method, this oil gave an iodine number of 261, which is notably higher than the value (217) reported by Van Loon[1] as the "true" iodine value of tung oil. To calculate the unsaturated acids of tung oil from the thiocyanogen study it is essential to know the "true" iodine value of tung oil. Therefore, it was desirable to redetermine the "true" iodine value of tung oil. Although theoretically substitution is less likely to occur with the Hanus iodine bromide solution which we used than with the Wijs iodine chloride solution used by Van Loon, yet the following experiment was conducted to see if substitution could be the factor causing the high value which we obtained.

Several pairs of identical quantities of tung oil were subjected to the action of 25 cc. of Hanus reagent for one-half hour. To one of each pair potassium iodide and water were added, and the iodine absorbed was determined in the usual manner. About 70 cc. of water was added to the other one of each pair and the mixture extracted repeatedly with 35-cc. portions of chloroform until all the free halogen was removed, leaving the halogen acids in the aqueous layer. This procedure was repeated with blank determinations, using 25 cc. of the Hanus solution in the absence of tung oil. In the latter cases it was

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[1] Ir. J. Van Loon, Farben Ztg. 1767–69 (1930).

*Questions and Answers on Tung Oil Production in America. H. A. Gardner. Circ. 446, National Paint, Varnish and Lacquer Association.
Trade Promotion Series No. 133, entitled "Tung Oil," by C. C. Concannon. U. S. Department of Commerce.
Miss. State Department of Agriculture Bul. No. 1, "Tung Oil," by E. Squire Brooks.
Florida Agricultural Experiment Station Bul. No. 247, "Variations in the Tung Oil Tree," by H. Mowry (1932); and Bul. No. 221, "The Tung Oil Tree," by W. Newell, H. Mowry and R. M. Barnette.