

A. O. C. S. GLYCERIN ANALYSIS COMMITTEE 1947-1948

Sample "B"—Salt Crude Glycerin

Member	1		3				5		6		7	
	A	A	A	B	C	D	A	B	A	A	B	
Laboratory Analysis												
% Ash.....	8.99	8.84	9.18	9.05	9.02	9.30	9.11	9.2	9.11	9.20	8.52	
% Total Alkalinity as Na ₂ O.....	0.80	0.72	0.79	0.77	0.78	0.80	0.82	0.86	
% Free Caustic Alkalinity, as Na ₂ O.....	0.005	0.012	0.02	0.01	0.02	0.02	0.01	0.025	
% Carbonate Alkalinity as Na ₂ O.....	0.035	0.046	0.02	0.08	0.02	0.06	0.06	0.055	
% NaCl.....	6.90	6.97	7.02	7.12	6.99	7.36	7.12	7.13	7.17	6.95	6.83	
% Alkalinity Comb. with or Equiv. to Organic Acids.....	0.76	0.67	0.75	0.68	0.74	0.73	0.75	0.78	
% Total Residue at 160°C.....	10.48	9.52	10.86	10.86	11.08	10.98	11.04	11.08	11.64	10.63	
% Organic Residue at 160°C.....	1.49	0.68	1.68	1.81	1.78	1.87	1.84	1.97	2.44	2.11	
% Total Acetylatable as Glycerol.....	83.44	83.39	84.04	83.80	84.06	83.85	83.8	83.2	83.65	83.98	84.47	
% Acetylatable in Residue.....	0.76	0.43	0.57	0.37	0.46	0.40	0.4	0.3	0.43	0.64	0.80	
% Glycerol Corrected.....	82.68	82.96	83.47	83.43	83.60	83.45	83.4	82.9	83.22	83.14	83.67	
% Moisture by Fischer Method.....	5.07	4.99	5.07	5.02	
% Glycerin by Periodic Acid.....	85.03	85.1	81.91	83.6	83.7	84.94	83.26	

Member	8		10					Average	Standard Deviation	Number of Analyses
	A	A	A	B	C	D	E			
Laboratory Analysis										
% Ash.....	9.33	9.20	9.10	9.37	9.05	9.08	9.13	9.10	0.19	18
% Total Alkalinity as Na ₂ O.....	0.81	0.80	0.78	0.72	0.78	0.74	0.79	0.78	0.04	15
% Free Caustic Alkalinity, as Na ₂ O.....	0.02	0.031	0.016	0.015	0.023	0.03	0.02	0.018	0.007	15
% Carbonate Alkalinity as Na ₂ O.....	0.05	0.18	0.09	0.05	0.055	0.01	0.04	0.057	0.039	15
% NaCl.....	7.04	6.95	6.85	7.12	7.04	6.80	7.09	7.03	0.14	18
% Alkalinity Comb. with or Equiv. to Organic Acids.....	0.74	0.59	0.67	0.66	0.71	0.70	0.73	0.71	0.05	15
% Total Residue at 160°C.....	10.65	9.95	9.50	11.02	9.81	9.16	10.56	10.52	0.66	17
% Organic Residue at 160°C.....	1.32	0.75	0.40	1.65	0.76	0.08	1.43	1.42	0.64	17
% Total Acetylatable as Glycerol.....	83.5	82.98	83.70	83.16	83.20	82.88	83.44	83.59	0.41	18
% Acetylatable in Residue.....	0.5	0.61	0.20	1.07	0.00	0.00	0.23	0.47	0.27	18
% Glycerol Corrected.....	83.0	82.37	83.50	82.09	83.20	82.88	83.21	83.12	0.41	18
% Moisture by Fischer Method.....	5.55	5.30	5.15	5.16	0.18	7
% Glycerin by Periodic Acid.....	84.3	83.62	84.35	83.98	0.93	10

size sample has been selected. When the sample contains less than 10% glycerol, the range of the glycerol content of the sample tested may be extended to between 0.1000 and 0.1650 gram.

$$\text{Glycerol, gram} = (S - B) \times N \times 0.09209$$

The excess periodic acid is determined as follows:

Transfer the blank and sample, after the final titration, to 500-ml. volumetric flasks and fill to the mark with distilled water. Mix thoroughly and pipet a 50-ml. portion of each into 250-ml. wide mouth Erlenmeyer flasks. Add 50 ml. of distilled water, 20 ml. of 15% KI solution, 5 ml. of HCl (sp. gr. 1.19) and mix thoroughly by shaking. Titrate the iodine with 0.1 N Na₂S₂O₃ soln. using starch indicator. The sample titration \times 100 divided by the blank titration must be more than 80%."

Acetic Anhydride-Pyridine Method

The acetic anhydride-pyridine method of analysis (2) was tested on samples "B," "C," and "E." The results were not encouraging and the committee voted against further consideration of this procedure.

Standard Crude Glycerin

About 1929, the A. O. C. S. Glycerin Analysis Committee, under the leadership of A. K. Church of Lever Brothers Company, prepared a standard crude glycerin of accepted analysis for use by the industry. The usefulness of this sample for checking purposes was so apparent that the number of samples in the office of the Secretary became depleted to the vanishing point a number of years ago. It was thought advisable to prepare and standardize a fresh supply.

Sample "B" represents a fair quality salt crude glycerin which was allowed to settle for several months to eliminate the difficulty in sampling caused by formation of a slimy deposit on the bottom of the container. Every precaution was taken to insure each bottle being representative of the entire lot. All of the analyses usually made in order to evaluate quality of crude glycerin were made on this sample.

Several members volunteered to submit this sample to other laboratories in their organizations which perform such analyses regularly. This cooperation enabled the participation of up to 18 skilled analysts in the more important tests, or nine more than the

A. O. C. S. GLYCERIN ANALYSIS COMMITTEE 1947-1948

Sample "C"—Commercial C. P. Glycerin

Member	1	2	3	4	5	6	7	8	9	10	Average	Standard Deviation	Number of Analyses
Laboratory Analysis													
% Glycerin—Periodic Acid Method.....	96.12	95.65	96.01	96.0	95.6	96.71	96.64	96.9	95.56	96.80	96.20	0.50	10
% Glycerin from Sp. Gr.....	95.63	95.88	95.86	96.09	95.85	95.85	95.90	95.8	95.70	96.00	95.86	0.13	10
Dichromate.....	95.5	95.25	95.38	2
Gross Acetin.....	94.57	94.4	95.07	93.96	94.50	0.40	4
% Moisture.....	4.26	4.10	4.33	4.0	4.18	4.08	4.2	4.60	4.19	4.22	0.16	9

Sample "D"—Salt Plus Added Glycerin in Water Solution

Laboratory Analysis													
% Glycerin—Periodic Acid Method.....	0.178	0.171	0.17	0.17	0.17	0.17	0.17	0.185	0.179	0.174	0.005	9
Dichromate.....	0.17	0.17	0.17	2
Gross Acetin.....	0.15	0.15	1
Potassium Permanganate.....	0.18	0.18	1
% NaCl.....	16.33	16.77	16.85	17.0	16.81	16.63	16.5	16.93	16.82	16.74	0.20	9

A. O. C. S. GLYCERIN ANALYSIS COMMITTEE 1947-1948
 Sample "E"—Saponification Crude Glycerin

Member	1	2	3	5	6	7	8	9	10	Average	Standard Deviation	Number of Analyses
Laboratory Analysis												
% Ash.....	1.07	0.99	1.10	1.3	1.13	1.14	1.12	1.20	1.18	1.14	0.082	9
% Total Alkalinity as Na ₂ O.....	0.090	0.054	0.05	0.12	0.02	0.03	0.050	0.08	0.06	0.031	8
% Free Acidity.....	0.035	0.034	0.03	0.015	0.03	0.04	0.03	0.057	0.04	0.035	0.011	9
% NaCl.....	0.022	0.058	0.12	0.12	0.11	0.09	0.15	0.16	0.12	0.106	0.041	9
% Alkalinity Comb. with or Equiv. to Organic Acids.....	0.125	0.142	0.14	0.103	0.16	0.16	0.20	0.19	0.14	0.15	0.029	9
% Total Residue at 160°C.....	1.44	1.74	1.60	1.80	2.05	2.30	1.91	1.98	1.62	1.83	0.25	9
% Organic Residue at 160°C.....	0.38	0.75	0.50	0.53	0.92	1.16	0.79	0.78	0.71	0.72	0.22	9
% Total Acetyltable as Glycerol.....	87.96	88.58	88.66	87.8	88.14	88.34	87.6	87.99	88.13	88.14	0.33	9
% Acetyltable in Residue.....	1.13	0.12	0.34	0.3	0.28	0.56	0.2	1.26	0.37	0.51	0.39	9
% Glycerol Corrected.....	86.83	88.46	88.32	87.5	87.86	87.78	87.4	86.73	87.76	87.63	0.56	9
% Moisture by Fischer Method.....	9.45	9.33	9.51	9.23	9.7	9.90	9.99	9.59	0.27	7
% Glycerin by Periodic Acid.....	89.06	90.9	89.75	88.2	89.56	87.90	89.1	88.53	89.93	89.21	0.88	9

present membership of the committee. The arithmetical mean, or average, has been computed for each analysis from the data reported, also the stand-

$$\text{ard deviation } \left(\sqrt{\frac{\sum d^2}{N}} \right)$$

This value is a measure of the reproducibility of the test by analysts of the same degree of skill, in this case in different laboratories. In any one laboratory with a single analyst, the standard deviation would be expected to be considerably smaller. Particularly important are the standard deviations for "% Total Acetyltable as Glycerol" and "% Glycerol Corrected" which, by coincidence are both 0.41. This value is almost identical with that calculated from data given in previous reports of the Glycerin Analysis Committee (3), which was 0.4.

These analyses are not advanced by the committee as absolute and final. They represent accepted values, obtained by our official methods, in laboratories regularly engaged in such work, by analysts of such skill and experience that they are typical of what may be expected on crude glycerin from which the error in sampling caused by non-homogeneity has been eliminated.

The Glycerin Analysis Committee recommends that sample "B" be accepted by the American Oil

Chemists' Society as a standard crude glycerin for distribution by the Secretary. We further recommend that a certificate of analysis be included with each bottle sold, showing the average values for the first 12 items in the table, with a reference to the appropriate A. O. C. S. method for each analysis. About 250 4-oz. bottles of this sample are available.

Future Work

The committee has planned a program of further study of the acetin and periodic acid methods on crude and distilled glycerins in the hope that the latter procedure may be improved to such an extent that it may eventually become our official method for the chemical analysis of glycerin.

REFERENCES

1. J. A. O. C. S. 24, 18-23 (Jan. 1947).
2. Oil & Soap 23, 48-50 (Feb. 1946).
3. Oil & Soap 11, 211-21 (Oct. 1934).

GLYCERIN ANALYSIS COMMITTEE—1947

H. C. BENNETT	W. D. POHLE
E. L. BOLEY	J. B. SEGUR
W. C. CLARK	B. S. VAN ZILE
E. R. LUCKOW	L. K. WHYTE
L. B. PARSONS	J. T. R. ANDREWS, chairman

The 1948 committee includes the same personnel as above with the exception of Mr. Van Zile.