6. The possible structures of the reaction products have been discussed.

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A Modified Procedure for Obtaining "Total Residue at 160°C." in Crude Glycerine

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Introduction: The procedure for obtaining "Total Residue at 160° C." as outlined in the Journal of Industrial and Engineering Chemistry, 3,682 (1911), has undergone no essential changes. In the light of newer theoretical knowledge and with the aid of more highly-developed chemical tools, the author has made a critical investigation into this determination. The result was a much shortened and simplified procedure.

Apparatus: A specially designed oven was assembled which made use of the radiant, penetrating heat of the commercial infrared drying lamps. The parts of this oven, which are all inexpensive and easily obtained, are listed as follows:

(A) Enamelware dish of approximately ten inches diameter and four inches depth. A small hole is bored through the side, flush with the bottom of the dish, to admit a laboratory thermometer.

(B) Infrared drying lamp, 250-watt, with built-in reflector. (General Electric, Model R-40, 250-watt.)

(C) Reflector of thin gauge aluminum, about nine inches across, which screws on a standard electric light socket. This is used primarily to shield the oven from drafts.

(D) A burette stand and clamp for holding and adjusting the height of the lamp above the samples.

Procedure: The preparation of the samples follows the official method. The total percentage of Na_2O to methyl orange indicator is found by titration of a 10 per cent solution of crude glycerin in water. A ten-gm. sample of crude glycerin is weighed into a 100 c.c. volumetric flask, sufficient standard hydrochloric acid or sodium hydroxide is added to adjust the percentage of Na_2O to 0.2 per cent, and the volume is made up to 100 c.c. with distilled water.

Aliquot portions of 10 c.c. each are pipetted into tared evaporating dishes, 70 m.m. in diameter and 15 m.m. in depth. These are placed in pairs adjacent to the center of the bottom of the enamelware dish. The lamp with aluminum reflector attached is adjusted four inches above the evaporating dishes and with its axis in line with the center of the enamelware dish. The thermometer is inserted through the hole in the side of the latter until the bulb rests in the middle of the dish.

The lamp is switched on. Temperature is disregarded until fumes are no longer observed. This usually takes about one hour. The temperature is then regulated to 160° C. by adjusting the height of the lamp. The samples are baked at this temperature for one-half hour and then are placed in a dessicator preparatory to weighing. The acetylizable on the residue is subsequently obtained in the regular manner.

Experimental: These results were obtained on the standard sample of crude glycerin issued by the A.O.C.S., February 1, 1929.

Per cent total residue at 160° C. (modified procedure)

10.35
10.20
10.29
10.27
10.25% average (10.28% A.O.C.S. analysis)

Per cent acetin in residue as glycerol

0.46
0.36
0.54
0.56
0.48% average (0.48% A.O.C.S. analysis)

Ten grams of C. P. glycerol were dissolved in distilled water and made up to a volume of 100 c.c.; 10 c.c. portions were run by the modified procedure as outlined above.

Sample	Added material	Increase in weight after drying
1.	none	less than 0.0001 gm.
2.	none	less than 0.0001 gm.
3.	0.0792 gm. NaCl	0.0792 gm.
4.	0.0624 gm. NaCl	0.0624 gm.

Summary: Evidence has been presented that by means of the infrared drying lamp "Total Residue at 160° C." may be obtained in one drying. Analytical results on a standard A.O.C.S. sample of crude glycerin agree well with the established analysis for both "Total Residue" and "Acetin on Residue." Pure glycerol even in the presence of sodium chloride is completely evolved without a trace under the infrared lamp at 160° C. The equipment and technic are much simplified. Work time on the determination is reduced by two hours and elapsed time by eight hours.

With these points in mind the author respectfully recommends to the Glycerin Analysis Committee that further work be done on this procedure with a view toward substituting it for the present one.