

an article from chemically pure sulphuric acid which I had proven to contain no arsenic.

In conclusion I take pleasure in acknowledging my deep indebtedness to Dr. Charles E. Munroe for his many suggestions.

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## THE OCCURRENCE OF TRIMETHYLENE GLYCOL AS A BY-PRODUCT IN THE GLYCEROL MANUFACTURE.<sup>1</sup>

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**D**URING the past winter our attention was called to an unusual difficulty experienced by one of the soap-making firms in the neighborhood of Boston in obtaining their glycerol of the required commercial gravity. The information furnished in regard to it indicated the presence in the glycerol of some uncommon impurity, and a considerable quantity of the "light stuff" having been generously placed at our disposal by the soap company, we were enabled to investigate it. It was submitted to fractional distillation, first at diminished and then at ordinary pressure, and a liquid boiling between  $214^{\circ}$  and  $217^{\circ}$  at 760 mm. pressure was thus separated from it. This liquid was found to have a specific gravity of 1.056 at  $\frac{20}{4}$ , and gave the following results on analysis:

0.2293 gram substance gave 0.3998 gram carbon dioxide and 0.2158 gram water.

	Found.	Calculated for $C_3H_8O_3$ .
Carbon .....	47.52	47.37
Hydrogen .....	10.46	10.53

The substance is therefore trimethylene glycol, which has a boiling-point of  $214^{\circ}$  and a specific gravity at  $\frac{15.8}{0}$  of 1.0526. The isomeric propylene glycol boils at  $188^{\circ}$ – $189^{\circ}$  and has a specific gravity of 1.0403 at  $\frac{15.4}{0}$ . The "light stuff" contained a very considerable proportion, about thirty-eight per cent. of glycol.

The origin of the glycol is a matter of considerable interest. There is little doubt that it was produced by fermentation of the

<sup>1</sup> Read at the springfield meeting.

glycerol. For it has already been shown by Freund<sup>1</sup> that trimethylene glycol is, in fact, one of the principal fermentation products of that substance. It is moreover highly probable that the glycol was present in the fat before saponification by the alkali, as the fermentation can hardly have taken place in the soap lye, both on account of its saline character, and on account of the short time intervening between the saponification and the recovery of the glycerol. It had probably been produced in the fat by spontaneous saponification and subsequent fermentation of the glycerol.<sup>2</sup> Refuse house fat formed a considerable part of the soap stock.

The presence of the glycol in glycerol used for making nitroglycerol might be a source of danger, since it reacts with nitric acid with explosive violence. Its presence would be detected in the usual examination by a low specific gravity accompanied by a high oxidation equivalent as shown by the bichromate titration.

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## THE EVOLUTION METHOD FOR THE DETERMINATION OF SULPHUR IN WHITE CAST-IRON.<sup>3</sup>

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THIS method is based upon the assumption that when iron is dissolved in an acid any sulphur that may occur in the iron unites with the escaping hydrogen, forming hydrogen sulphide. In the usual mode of conducting the process the impure hydrogen evolved is led into a suitable absorbent and the sulphur finally determined, either by oxidation and precipitation as barium sulphate, or by a volumetric method. In applying the process Fresenius<sup>4</sup> directs that the iron be dissolved in *dilute* hydrochloric acid. Von Reis<sup>5</sup> and Blair<sup>6</sup> concur in this recommendation, as does also Dudley in

<sup>1</sup> Monatsh. Chem., 2, 638.

<sup>2</sup> Mr. E. Twitchell of Cincinnati informs us that he has found the glycol present in considerable quantity in the "tank liquor" separating from the fat before saponification.

<sup>3</sup> Read at the Springfield meeting.

<sup>4</sup> Quant. Analyse, 1877, 428.

<sup>5</sup> Stahl-eisen, 1894, 963.

<sup>6</sup> Chemical analysis of Iron, p. 54.