

many minutes are necessary to expel all of the volatile matter in the analysis of coals by the official method :

Time over the full flame in minutes	Total volatile matter
3. ....	38.65
4. ....	38.65
7. ....	38.65

To determine the number of minutes required over the full flame in the smoking off method the following experiment was made on Negro coal No. 21. The samples were carefully smoked off and then treated as above :

Time over the full flame in minutes	Total volatile matter	
1. ....	48.53	} Constant
3. ....	49.46	
4. ....	50.27	
5. ....	50.24	
6. ....	50.21	
7. ....	50.24 <sup>1</sup>	

The above results show that with either the official or the smoking off method the gases are all expelled after four minutes heating over the full flame of a Bunsen burner. They also show that no loss is entailed when the heating is continued for three minutes longer ; since no loss ensues by the seven minutes treatment prescribed by the Committee, it is well to maintain uniformity.

The official method is assumed to approach the conditions existing in a coke oven. However in actual practice the coal is charged in large quantities and the distillation for the most part begins slowly as in the smoking off method. The latter method is universally applicable and is therefore recommended for general adoption. A qualitative experiment will show in a few minutes whether or not a coal suffers mechanical loss by the official method. For the present we shall continue to use the official method in this laboratory wherever applicable, in order to facilitate direct comparison with coals from other sources, but in those cases where it entails large losses we shall employ the smoking off method followed by seven minutes over the full flame.

It is hoped that the Committee on Coal Analysis will recognize the demand and modify their method to include the non-coking coals which are causing so much difficulty. More data and a more complete treatment of this subject will appear in the Philippine Journal of Science under the title of "The Proximate Analysis of Philippine Coals."

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NOTES

*The Determination of Sulphurous Acid in Gelatine: A Manufacturer's Position with Regard to the Food and Drugs Act.*—In the deter-

<sup>1</sup> The average of four results the greatest variation of which is 0.06 per cent.

mination of sulphurous acid by the official method,<sup>1</sup> the substance to be examined is distilled with dilute phosphoric acid in a current of carbon dioxide, and the distillate collected in standardized iodine solution, the excess of which is afterward determined by titration with sodium thio-sulphate.

It is generally recognized that this method is inaccurate, for in many cases there appear to be substances other than sulphurous acid which reduce the iodine. Most chemists, therefore, instead of determining the sulphur dioxide by this indirect volumetric method, boil off the iodine, precipitate the resulting sulphuric acid with barium chloride, and determine it gravimetrically. The results by this modification are for the most part considerably lower than by the official method, but they must be looked upon with doubt until we know the nature of the iodine-reducing substances referred to.

Sulphurous acid and sulphites will probably be forbidden in food products under the Food and Drugs Act of June 30th, 1906. Recently one of our prospective customers reported that he had had our sample analyzed, and that his chemist had reported sulphurous acid present, in a sample of our gelatine which we knew to be free from sulphuric acid or sulphites. Further inquiry brought out the fact that the gravimetric method had been used, and that the amount reported was 0.009 per cent.

In order to secure some analyses free from any suspicion of bias, we submitted duplicate samples of several boilings or lots of gelatine to two analytical chemists of large experience and established reputation. Through the courtesy of the chief chemist in a large industrial laboratory where a specialty is made of sulphur dioxide determinations, we were able to have two samples analyzed there.

The results were as follows :

Sample No.	Chemist A.		Chemist B.		Industrial Laboratory.	
	per cent. Ash.	per cent. SO <sub>2</sub> <sup>2</sup>	per cent. Ash.	per cent. SO <sub>2</sub> <sup>2</sup>	per cent. SO <sub>2</sub> <sup>1</sup>	per cent. SO <sub>2</sub> <sup>2</sup>
1	2.78	0.012	2.31	0.04	0.009	0.0003
2	3.72	0.012	3.00	0.03	.....	.....
3	3.02	0.012	2.78	0.01	.....	.....
4	3.18	0.011	.....	.....	.....	.....
5	.....	.....	2.37	0.03	.....	.....
6	.....	.....	.....	.....	.....	0.0008

From the above table it is quite evident that grave differences may arise, not only in determinations by methods which are not above suspicion, but also in a determination so apparently simple as that of ash.

How shall we account for the apparent presence of sulphurous acid, or rather for the real presence of sulphuric acid ?

<sup>1</sup> Provisional methods for the analysis of foods, U. S. Dept. of Agriculture, Bureau of Chemistry, Bulletin No. 65 (1902).

<sup>2</sup> By gravimetric method.

<sup>3</sup> By volumetric (official) method.

The most likely explanation seems to be that some of the organic sulphur in the gelatine is carried over and is found as sulphuric acid. It is also probable that the quantity thus appearing in the distillate will vary with the time and intensity of heating, and the concentration of the solution distilled. The results of Chemist A seem to indicate a fairly constant source of error of this nature, and experiments are now being conducted to determine the probable amount of error and its cause.

C. Mentzel, in a most interesting article, "Zur Bestimmung der Schwefligen Saure im Fleisch,"<sup>1</sup> states that the *apparent* amount of sulphur dioxide in chopped meat is from 0.0014 to 0.0021 %, equivalent to from 0.0054 to 0.0084 % of sodium sulphite.

The results of nine analyses were :

	50 grm. Meat gave grms. BaSO <sub>4</sub>	Equivalent to Per cent. SO <sub>2</sub>	Equivalent to Per cent. (Na <sub>2</sub> SO <sub>3</sub> ·7H <sub>2</sub> O)
Highest.....	0.0039	0.0021	0.0084
Lowest.....	0.0025	0.0014	0.0054
Average.....	0.0030	0.0017	0.0067

When onions were added to the chopped meat, the percentage of apparent SO<sub>2</sub> was largely increased, probably owing to the presence of allyl sulphide.

Taken	grms. BaSO <sub>4</sub>	Equivalent to Per cent. SO <sub>2</sub>	Equivalent to Per cent. Na <sub>2</sub> SO <sub>3</sub> +7H <sub>2</sub> O
50 grms. Meat.....	0.0028	0.0015	0.0061
45 grms. Meat+5 g. Onions	0.0039	0.0021	0.0084
50 grms. Onions.....	0.0110	0.0060	0.0238
50 grms. Onions.....	0.0106	0.0058	0.0230

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LABORATORY OF NATIONAL GLUE  
AND GELATINE WORKS.

*Testing Contact Sulphuric Acid.*—The difficulty of transferring sulphuric acid containing free sulphuric anhydrides into water for the purpose of titrating the acid is overcome in the following manner :

The acid is weighed off in a small and narrow weighing bottle, and the bottle, with the stopper not removed, is held with the left hand upside down in a porcelain dish containing enough water to cover the stopper and opening of the bottle. By means of a pair of glass forceps held in the right hand, the stopper is gradually removed under the water, and the bottle, stopper and forceps rinsed with distilled water. The titration is then carried out. The transferring of the acid takes very little time, and there is absolutely no spurting.

A simple pair of glass forceps can be made by closing two pieces of glass tubing (each about six inches long) at one end and filling them with ground sealing wax. A piece of steel wire is bent to a narrow U

<sup>1</sup>Zeitschrift für Untersuchung der Nahrungs- und Genussmittel, 1906, Vol. 11, page 320.