THE ANALYSIS OF SODIUM PEROXIDE.

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SHORTLY after sodium peroxide became an article of commerce, I had occasion to make a full analysis of a sample of it, which led to some observations of interest. As I am not aware that a really accurate, rapid method for the assay of this article has been published, I venture to bring the results of my experiments before the Society.

The sample of the peroxide which I examined was taken from a full 14-lb. canister fresh from the makers' works. A portion of it was transferred to a stoppered tube, and all the weighings were made by taking some from the tube, which was immediately re-stoppered and the loss of weight ascertained.

Estimation of Soda (Na_2O).

About 0.8 gramme was accurately weighed, dissolved in water, boiled to decompose the peroxide, cooled, tinted with methyl orange, and titrated with normal sulphuric acid. A few drops more of the acid were then added, the solution evaporated to dryness in a tared platinum dish, and the residue weighed after ignition. From the weight obtained there was deducted the weight of the alumina and iron oxide found by another experiment, and the difference was calculated to Na_2O . Two experiments gave :

Na_2O by titration, 78.60 and 78.65. Mean	...	...	78.63
Na_2O weighed as Na_2SO_4 , 78.72 and 78.69. Mean	...	...	78.70
General Mean	...	...	78.66

Estimation of Alumina and Iron Oxide.

About 2 grammes were taken, dissolved in water, acidified with hydrochloric acid, and precipitated by ammonia in slight excess :

Weight of ignited ppt.	...	...	Per Cent.
			0.49

Estimation of Oxygen existing as Peroxide.

First Method tried.—From 0.2 to 0.25 gramme of the sample was weighed into a small dry tube closed at one end, which was then placed in the flask attached to a Lunge nitrometer. The flask contained a small volume of potassium permanganate solution acidified with sulphuric acid, and after the whole had acquired the temperature of the room, the mercury was levelled, and then the peroxide and the permanganate solution were brought into contact.

Weight taken in grammes.	Oxygen obtained in c.c., reduced to 0° and 760 mm. (dry).	Oxygen obtained in grammes.	Oxygen obtained per cent., assuming half came from the KMnO_4 .
0.1974	29.78	0.0426	10.79
0.2434	37.28	0.0533	10.95
0.2457	38.56	0.0551	11.22
0.2447	38.47	0.0550	11.24

Evidently the decomposition was only partially effected in accordance with the equation :

Second Method tried.—About 0.1 gramme was dissolved in cold water previously mixed with some dilute sulphuric acid, and the solution was titrated with decinormal permanganate. Three experiments gave 18.4, 18.3, and 17.8 per cent. of oxygen.

There was a slight loss of oxygen at the moment of contact between the peroxide and the acidified water. A larger quantity, 1·0019 grammes, was dissolved in acidified water, diluted to 1 litre, and 100 c.c. titrated. Two experiments gave only 17·45 per cent. of oxygen.

Third Method tried.—An attempt to decompose the peroxide in the nitrometer with water alone was not successful, decomposition being much too slow and incomplete. But a trace of cobalt nitrate previously added to the water was found to be all that was required to effect rapid and complete decomposition. The peroxide was weighed into the small tube, and into the flask were placed about 5 c.c. of water and one small drop of cobalt nitrate solution. Three experiments were made, and gave the following results :

Weight taken in grammes.	Oxygen obtained in c.c., reduced to 0° and 760 mm. (dry).	Oxygen obtained in grammes.	Oxygen obtained per cent.
0·2499	32·40	0·0463	18·54
0·2472	31·91	0·0456	18·46
0·2537	32·94	0·0471	18·57

The mean of these results—viz., 18·52 per cent.—was accepted. They show that decomposition by water in the nitrometer flask in the presence of a milligramme or two of cobalt sesquioxide is the most accurate, as well as the simplest, method for the assay of sodium peroxide. It would be more accurate to add precipitated cobalt sesquioxide than cobalt nitrate, as obviously a trace of oxygen is used up in oxidizing the cobaltous oxide. My result may, therefore, be a trifle too low ; but, taking it as practically correct, the composition of the sample works out as follows :

Constituents found.				Calculated composition.			
			Per cent.				Per cent.
Na ₂ O	...	...	78·66	Na ₂ O ₂	...	...	90·41
O	...	...	18·52	NaOH	...	...	8·73
Al ₂ O ₃ and Fe ₂ O ₃	...	...	0·49	Al ₂ O ₃ and Fe ₂ O ₃	...	...	0·49
							99·63

This analysis does not take into account a very little carbonate, a trace of chloride, a minute trace of sulphate, and a few particles of unoxidized sodium which the sample contained. No other impurity was detected.

DISCUSSION.

Dr. Dupré said that the analysis of sodium peroxide was a very unsatisfactory operation to perform. All the processes he had tried were almost impossible to conduct without loss of oxygen. He had not, however, tried the cobalt process, but thought it would be a sensible improvement.