interference of sulfates in the presence of other bases, but it is believed that with the bases usually found in cement materials, where there is a great excess of calcium, any sulfate in the perchlorate precipitate will be present as $2\text{CaSO}_4 \cdot \text{H}_2\text{O}$, or if not all combined with calcium, the rest will be combined as anhydrous sodium sulfate whose molecular weight, 142, is so close to the weight of $\text{CaSO}_4 + \frac{1}{2}\text{H}_2\text{O}$, 145, that no serious error is introduced by correcting for a small amount of sulfate in the way indicated.

SUMMARY

It is believed that for the determination of potassium alone in silicate materials which are free from appreciable amounts of sulfur compounds the method here given is the simplest and most accurate yet devised. It has the following advantages:

1—It is not necessary to have the material finely ground.

2—There is no chance for loss of potassium by volatilization, as is the case in other methods where the salts, usually chlorides, are heated to decompose the silicate or to remove ammonium compounds.

3—All separations of other elements by precipitation which might cause loss of potassium salts by adsorption, formation of double salts, etc., are avoided.

4—The only time-consuming operations are the evaporations, which may be carried on with very little attention, especially on an electrically heated sand bath, as there is absolutely no tendency to spatter when a moderate heat is used.

5—The potassium compound which is filtered and weighed is, under the conditions of the method, the most insoluble of any used in the determination of potassium, and is absolutely constant in composition.

THE ALSATIAN POTASH INDUSTRY

Discussing the Alsatian potash situation before the Société de Chimie Industrielle, in December 1920, M. Matignon pointed out the remarkable progress made under adverse conditions since the war, and the wide possibilities of future development.

In 1913 Alsace produced 50,000 tons of potash (K_2O). In spite of transportation difficulties and strikes, the 1919 production reached 90,000 tons. The potash sold in 1920 will amount to 200,000 tons, or almost one-fifth of the world's pre-war production. It is hoped that in 5 yrs., with a sufficient operating force, the production may approach 600,000 tons.

The resumption of work since the war has involved the replacement of a German directing personnel by a French or Alsatian staff, and also the adoption of a more modern and economical mining system than was followed by the German operators.

The deposit should guarantee the world's 1913 consumption for 250 to 300 years. The purification of the Alsatian products is much simpler than that of those from the German deposits, owing to the low content of magnesium salts. Common 12 to 16 per cent sylvite (a mixture of common salt and potassium chloride) and sylvite containing 20 to 22 per cent pure potash, both of which are in demand for agriculture, are easily obtained from the crude product. From a sylvite brine, a 90 per cent KCl is obtained, which is easily converted to a 98 per cent chloride. Three factories are at present producing 300 tons of rich chloride daily. At the Reichland mine, from 1000 tons extracted, 600 tons are removed as common and rich sylvite, the remaining 400 tons passing through the factory and yielding 100 to 120 tons of chloride.

CENTRIFUGAL METHOD FOR DETERMINING POTASH

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The ever-increasing production and consumption of potash in this country and the efforts to discover and develop new sources make the need of an accurate, economical, and rapid method of analysis keenly felt. Also in potash producing and consuming factories it is desirable to exercise chemical control over operations, heretofore a difficult if not an impossible procedure.

The object of this article is to describe a method which can be depended upon to furnish the desired results, practically reducing the time element from hours to minutes and the cost for chemicals per determination from dollars to cents, and which in its simple form is capable of wide application.

OUTLINE OF METHOD

Five cc. of an approximately 1 per cent K_2O solution of the sample are transferred to a potash centrifuge tube containing 17 cc. of specially prepared sodium cobaltic nitrite solution. To a similar tube add 5 cc. of a standard 1 per cent potash (K_2O) solution. Centrifuge both at once in a Babcock milk test hand centrifuge at 1000 r. p. m. for 1 min. Observe each tube, tap gently with the finger to level the surface of the precipitate, and centrifuge again for 15 sec. Calculate results by the formula:

$$\frac{\text{Cc. to which sample is diluted} \times \text{reading of sample}}{\text{Gram of sample in above solution} \times \text{reading of standard}} = \text{per cent } \text{K}_2\text{O}$$

STANDARD POTASH (K_2O) SOLUTION—Dissolve 15.83 g. of highest purity potassium chloride in distilled water in a liter volumetric flask, add 8 or 10 drops of C. P. glacial acetic acid, dilute to the mark, and mix thoroughly.

SODIUM COBALDIC NITRITE—To 450 g. of C. P. sodium nitrite in a large wide-mouth bottle add 800 cc. of distilled water. Let stand over night, or longer, with occasional stirring. In a similar manner treat 250 g. of C. P. cobalt acetate with 800 cc. of distilled water. As soon as the sodium nitrite is all dissolved pour it into the cobalt acetate solution. Mix well and dilute to 2 liters. This solution keeps well for months.

To prepare the solution for use add 65 cc. of water and 5 cc. of glacial acetic acid to 100 cc. of stock solution, mix, and let stand over night before using. It does not keep well, and a new solution should be prepared every 3 or 4 days.

SODIUM HYDROXIDE SOLUTION—Prepare 500 cc. of 10 *N* solution. A saturated solution of sodium carbonate is sometimes used instead.

POTASH CENTRIFUGE TUBES—These are manufactured specially for potash determinations. If a Babcock milk test bottle were inverted, the stem sealed and the bottom cut out, it would be nearly the same size and shape as a potash centrifuge tube, which has

comparatively a much smaller stem and finer graduations. They should be calibrated with mercury before using, in which case a long drawn capillary tube is useful in filling and emptying. It is also useful in washing out precipitates after a potash determination. Linings for the centrifuge tube shields can be made from large corks to prevent breakage of the potash tubes.

NOTES ON METHOD

The solution of the sample should contain approximately 1 g. of the K_2O per 100 cc. Before diluting to the mark, it should be rendered alkaline with sodium hydroxide and acidified with glacial acetic acid, using phenolphthalein as indicator. If it contains insoluble matter, filter through a dry paper, and centrifuge 5 cc. of the clear filtrate. Samples containing ammonium salts should be weighed and ignited before bringing into solution. The stem of the tube should be full of water before adding the nitrite solution. The temperature at which the determination is made is $22^\circ C.$ or above. The precipitate reading of the sample should not be over five small divisions above or ten below that of the standard, which should be about 10.5. A 4-tube head centrifuge allows three samples to be run with the one standard.

APPLICATIONS OF METHOD

The ease with which samples are prepared for analysis, the rapidity of obtaining reliable results, and the minimum costs of chemicals per sample make the method ideal. Samples that used to take the most time are now determined quickest. For instance, the K_2O in molasses is determined as follows: Transfer 26 g. more or less to a 100-cc. volumetric flask by aid of hot water, render alkaline with sodium hydroxide, acidify with acetic acid, cool, fill to the mark with water, and mix. Centrifuge 5 cc. and calculate the per cent K_2O . This requires 12 min. in all. Distillery and sugar factory wastes can be determined in from 4 to 8 min.

Altogether over 1700 determinations have been made in this laboratory by the method, about 60 of them being on 50-ton car shipments of crude potash.

The results obtained on twelve car samples by the centrifugal method, in comparison with those of a well-known public analyst and in the case of disputed samples those of an umpire chemist in New York, are given below:

Lab. No.	Centrifugal Method		Public Analyst Analysis		Umpire Analysis
	K_2O	Per cent Moisture	K_2O	Per cent Moisture	K_2O
85	32.80	..	32.68	5.63	...
88	35.62	..	34.12	3.95	...
89	35.54	..	33.74	4.11	35.65
97	28.75	0.98	26.36	1.11	...
101	30.87	1.45	30.92	1.47	...
102	30.57	1.30	29.76	1.58	...
103	29.55	1.59	28.10	2.20	...
104	30.02	1.36	28.38	1.53	29.38
111	36.77	1.65	37.28	1.32	...
112	40.78	0.73	39.44	1.01	...
P 1	41.51	0.83	41.30	..	...
P 2	41.39	0.82	40.80	..	...

The results given by the centrifugal method were those obtained the first time run, not averages of two or more determinations, and not over 25 min. were required for any result. In each case they were just one of several samples run during the day.

RAPID IODOMETRIC METHOD FOR DETERMINATION OF CHROMIUM IN CHROMITE

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The determination of chromium after oxidation to sodium chromate immediately suggests the rapid accurate method of iodometry. Because of the analogous action of the dichromate and ferric ions on the iodide ion, however, the analysis of chromite by this method presents a problem, and our purpose here is to present a method whereby an iodometric determination of chromium may be effected with its usual rapidity and accuracy without time-consuming, intermediate procedures for the elimination of iron.

Practically all the methods in use for the analysis of chromite prescribe that the ore be fused with sodium peroxide, or sodium peroxide and sodium carbonate, in a spun iron crucible. The methods differ after the extraction of the melt and are of two classes: first, those in which the iron is removed as ferric hydroxide by filtration, and the filtrate of sodium chromate is analyzed by any of the usual methods, including an iodometric method;¹ and second, those in which the chromium is determined in the presence of iron in an acid solution. The objection to the methods of the first class is that the filtration of a solution containing in suspension a voluminous precipitate of ferric hydroxide is a tedious operation and quite likely not to give quantitative results on the first filtration; three filtrations and subsequent reprecipitation are very often necessary.² In methods of the second class, the extract is acidified with either hydrochloric or sulfuric acid, a weighed excess of Mohr's salt or ferrous sulfate is added, and the excess titrated with standard permanganate or dichromate. The shortcomings or inconveniences of these methods are well known. In the case of the potassium permanganate, when the titration is carried out in a moderately small volume, the end-point is obscured by the bright blue-green color of the chromic ion; when larger volumes are used in order to overcome the above-mentioned difficulty, blank tests on the water are necessary. Furthermore, permanganate is rather unstable in solution, and frequent restandardizations are necessary. When dichromate is used, an outside indicator with its inconveniences is imperative. The use of an outside indicator is especially difficult in this analysis, owing to the high concentration of chromic ion, the color of which makes the end-point more difficult to determine.

An iodometric method has not been considered possible here on account of the presence of the ferric ion. A method has been outlined in which the interference of the ferric ion is claimed to have been removed by the addition of a solution of phosphoric acid, in the presence of which iron forms a very slightly ionized ferric acid phosphate.³ This method, however, has not been tried in the presence of large excesses of iron,

¹ Brunn, *Z. anal. Chem.*, **52**, 401.

² Schorlemmer, *Collegium*, **1918**, 145.

³ O. L. Barnebey, *J. Am. Chem. Soc.*, **39** (1917), 604.