The precipitation should be made in solutions just alkaline to phenolphthalein. Ammonium hydroxide is used instead of sodium hydroxide or potassium hydroxide since it is volatile and in case some ammonium salt remains in the precipitate after washing, it will be volatilized during ignition of the precipitate.

The method has been used, with excellent results, in some alum investigations. It is applicable under any conditions in which the ammonium hydroxide method can be used.

Summary

1. Aluminum may be determined quantitatively as lithium aluminate.
2. Lithium aluminate precipitate is filtered and washed much more rapidly than aluminum hydroxide.
3. More concordant results are obtained by this process than by the ammonium hydroxide method.

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AN OVERLOOKED SOURCE OF ERROR IN THE FERROCYANIDE TITRATION OF ZINC

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The ferrocyanide method of determining zinc proposed by Fahlberg,[1] and modified by Low[2] and Demorest,[3] includes the addition of hydrogen sulfide or hydrogen sulfide water just before the titration.

Hydrogen sulfide is added to precipitate copper and to reduce any bromine, chlorine or chlorate which might be present in the zinc solution. The fact that ferricyanide is also reduced by hydrogen sulfide has apparently been overlooked.

When a solution of ferrocyanide stands, a small amount of ferricyanide is formed. According to Kolthoff,[4] this change is hastened by light. Even in freshly prepared solutions of ferrocyanide there is usually a little ferricyanide. During the zinc titration, if hydrogen sulfide is present, this ferricyanide is reduced to ferrocyanide, which reacts with the zinc. If no hydrogen sulfide is present, there is no reaction between

[1] Fahlberg, *Z. anal. Chem.*, **13**, 379 (1874).

[2] Low, "Technical Methods of Ore Analysis," 9th ed., John Wiley and Sons, Inc., New York, **1922**, p. 252.

[3] Lord and Demorest, "Metallurgical Analysis," 5th ed., McGraw-Hill Book Co., Inc., New York, **1924**, p. 203.

[4] Kolthoff and Furman, "Volumetric Analysis," John Wiley and Sons, Inc., New York, **1928**, Vol. I, p. 239.

the ferricyanide and the zinc. Therefore a solution of ferrocyanide containing appreciable amounts of ferricyanide exhibits a different zinc titer in the presence of hydrogen sulfide than in its absence.

The accepted method of standardizing solutions of ferrocyanide for use in zinc determination is to titrate in the absence of hydrogen sulfide. Scott[5] even goes so far as to state that its presence is not necessary. The author's experience is to the contrary.

A solution of ferrocyanide made in March 1924, and stored in an ordinary clear glass bottle in a cupboard, was standardized from time to time with the results shown in Table I.

TABLE I

Date of standardization	Zinc value per cc.
March, 1924	0.00532
Dec., 1924	.00531
Oct., 1930	.00530
Sept., 1931	.00524

These values were all obtained with zinc oxide as the primary standard. The titrations were carried on in the presence of hydrogen sulfide under the same conditions of temperature, volume and acidity. At the time the last standardization was made duplicate samples of zinc oxide were titrated in the same manner but without the addition of hydrogen sulfide. The results of these titrations are listed in Table II.

The presence of ferricyanide in this solution was shown by its oxidizing action on potassium iodide. A fresh solution of ferrocyanide was prepared and standardized in the same manner. The results are given in Table II.

TABLE II

ZnO, g.	H_2S	Old ferrocyanide, cc.	New ferrocyanide, cc.
0.2490	None	39.65	39.33
.2490	None	39.60	39.30
.2490	Present	38.20	39.18
.2490	Present	38.18	39.15

It is therefore recommended that hydrogen sulfide be passed into the zinc solution during standardization and that the solution smell distinctly of the gas when the end-point is reached.

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[5] Scott, "Standard Methods of Chemical Analysis," D. Van Nostrand Co., New York, 1927, 4th ed., p. 606B.