

ART. XLIX.—*The Separation of Magnesium from Lithium by Means of Ammonium Carbonate in Alcoholic Solution*;
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(Contributions from the Kent Chemical Laboratory of Yale Univ.—cclxvii.)

THE following work was undertaken to determine the conditions under which lithium may be separated quantitatively from magnesium by means of ammonium carbonate in alcoholic solution. It has been shown* that by the agency of such a solution magnesium may be separated quantitatively from sodium and potassium and† that very small amounts of magnesium may be detected qualitatively in presence of limited amounts (100 mgm.) of lithium. In the quantitative procedure, the solution containing the salts of magnesium, sodium, and potassium is brought to a volume of about 50^{cm}³, an equal amount of absolute alcohol is added, precipitation is made by addition of 50^{cm}³ of a saturated ammoniacal solution of ammonium carbonate in 50 per cent alcohol, and the mixture is allowed to stand half an hour after stirring vigorously for five minutes. If the amount of alkali salt present is small, the precipitate may be collected without further treatment upon asbestos in the perforated crucible, washed with the precipitant, dried, ignited, and weighed as magnesium oxide. When the amount of alkali salt is large the precipitate may be freed from included alkali salt by decanting the supernatant liquid through the prepared asbestos filter, dissolving the precipitate in hydrochloric acid and reprecipitating by ammonium magnesium carbonate as at first. This second precipitate, collected upon the filter used in the decantation, leaves upon ignition a practically pure magnesium oxide. The essential condition of this process is that precipitation shall be brought about by a considerable excess of ammonium carbonate in a 50 per cent alcoholic solution.

In the work to be described, it was convenient to make use of 93 per cent alcohol instead of absolute alcohol in making up the solution to be treated and the precipitant, but the proportions of solution and precipitant were so adjusted that the liquid at the time of precipitation should be 50 per cent alcohol.

The alcoholic solution of ammonium carbonate was made by mixing 1500^{cm}³ of the saturated aqueous solution of that reagent with 360^{cm}³ of concentrated ammonium hydroxide and 1900^{cm}³ of 93 per cent alcohol, settling out the precipitated ammonium carbonate, and filtering off the clear solution. In carrying out

* Gooch and Eddy, this Journal (4), xxv, 444, 1908.

† A System of Qualitative Analysis for the Common Elements, Noyes and Bray: reprint from The Technology Quarterly, xxii, p. 472.

the determinations, portions of the magnesium chloride solution (15cm^3 – 20cm^3) were measured out and carefully weighed, lithium chloride was introduced in the amounts stated, 25cm^3 of 93 per cent alcohol and 50cm^3 of the precipitating mixture were added, the mixture was stirred vigorously (for perhaps a minute) and set aside for five hours or more. The supernatant liquid was decanted upon asbestos in the perforated crucible (weighed), the precipitate was stirred up with more of the precipitating mixture and allowed to settle, and the liquid was again decanted upon the same asbestos filter. In the cases in which a single precipitation was employed, the precipitate was simply transferred to the filter and washed with the precipitating mixture, and ignited to constant weight. When two precipitations were made, in order to remove possible inclusions of lithium salt from the precipitated ammonium magnesium carbonate, the precipitate was dissolved in the least necessary amount of hydrochloric acid, the solution was diluted to a volume of 10cm^3 , 13cm^3 – 15cm^3 of 93 per cent alcohol and 50cm^3 of the precipitant were added, and the precipitate was transferred, washed, and ignited as described.

The following tables show the results obtained with solutions of magnesium chloride with and without the addition of lithium chloride in varying amount. The mean of the three determinations in which the solution contained no lithium salt was taken to fix the value of the magnesium solution and by comparison with this standard the errors of the single determinations were computed.

TABLE I.
Standardization of the Magnesium Solution.

Solution MgCl_2 gram.	MgO found gram.	MgO found per gram. of solution gram.	Average MgO per gram. of solution gram.
20.4071	0.2788	0.013662*	0.013651
20.3956	0.2779	0.013626*	
15.4271	0.2108	0.013664†	

* One precipitation.

† Two precipitations: washings made with precipitant saturated with precipitate.

It will be seen that while the separation of 0.2 gram. of magnesium oxide from lithium amounting to 0.5 gram. of the chloride or 0.083 gram. of the element may be reasonably good in a single precipitation, the ammonium magnesium carbonate only once precipitated is largely contaminated, presumably

TABLE II.

Solution of MgCl ₂	<i>Separation of Magnesium from Lithium.</i>					Number of precipita- tions
	LiCl \rightleftharpoons Li taken		MgO taken	MgO found	Error	
	gram.	gram.	gram.	gram.	gram.	
14.8191	0.5	0.083	0.2023	0.2028	+0.0005	1
15.1409	1.0	0.166	0.2067	0.3191	+0.1124	1
20.7421	0.5	0.083	0.2831	0.2834	+0.0003	2
20.1706	0.5	0.083	0.2753	0.2761	+0.0008	2
19.9744	1.25	0.207	0.2727	0.2730	+0.0003	2
20.3998	1.25	0.207	0.2785	0.2783	-0.0001	2
19.9291	2.10	0.347	0.2718	0.2730	+0.0012	2

with lithium carbonate, when the amounts of lithium are considerably greater. Two precipitations made in the manner described will yield, however, a very fair separation of the magnesium (about 0.3 gram. of the oxide) from lithium chloride equivalent to 0.2 gram.-0.3 gram of the element.

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