

Proceedings of the Iowa Academy of Science

Volume 60 | Annual Issue

Article 39

1953

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Recommended Citation

Resnick, James D.; Kiser, Robert W.; and Dalton, J. Lester (1953) "Analysis of Lithium," *Proceedings of the Iowa Academy of Science*, 60(1), 315-318.

Available at: <https://scholarworks.uni.edu/pias/vol60/iss1/39>

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Analysis of Lithium

By JAMES D. RESNICK¹, ROBERT W. KISER², AND J. LESTER DALTON

The technology of lithium and its compounds was accorded little consideration a few years ago. Immediately following World War II fundamental knowledge of lithium began to be greatly expanded and its importance in industry has risen. Lithium and its compounds are used in porcelain enamels, and glazes; in air conditioning units; in greases, cosmetics and the like, just to cite a few examples. Lithium is the lightest of all the metals having an atomic weight of 6.94. The lithium ion has the smallest ionic radius of the alkalis and is approximately the same size as the magnesium ion. Its chemical properties frequently parallel those attributed to alkaline earths; this is the property which sets lithium apart from the other alkalis.

In examining various quantitative methods suggested for lithium, an attempt was made to find one that used a minimum of equipment, was easily adaptable for general and industrial use and gave good quantitative results. Perhaps the best known of all the quantitative procedures is that employed by Gooch (1). This is an amyl alcohol extraction based upon the solubilities of the various alkali metals in the solvent. There are several other methods of analyses depending upon the solubility of the chloride of lithium in an organic solvent, most notable of which are: the acetone separation as suggested by Brown and Reedy (2); the Kallmann (3) method using n-butyl alcohol and a perchlorate separation of lithium from potassium and sodium; the n-hexanol method suggested by Axilrod and E. Caley (4); and Alexander Sinka's (5) proposal using dioxane. A more recent paper by T. Shigematsu and H. Kimura (6) presents a method for the complexing of the lithium ion using Nickel uranyl acetate. In 1938 O. Procke and R. Uzel (7) presented a precipitation using a reagent composed of ferric chloride, potassium hydroxide and potassium metaperiodate. All the aforementioned procedures are lengthy and some of them are quite involved. In many cases correction factors must be calculated for each analyses based on the volume of the solvent. In 1943 Rogers and E. Caley (8) proposed a very simple and quick method for the determination of lithium. Their reagent was simply KIO_4 , in

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a strong alkaline solution of potassium hydroxide. The precipitation of LiIO_4 may be accomplished by allowing the mixture to stand at room temperature for one hour or in a water bath at $60\text{-}70^\circ\text{C}$ for twenty minutes. As with other methods of analysis, metals other than the alkalis and ammonia must first be removed. The iodine content which is constant in composition is then titrated.

In the metal analysis of a grease, the usual method is to ash it; then, from the ash weight the oxide of the metal determines its metal percent. Lithium and other alkali greases when subjected to this type of analysis do not respond in the same manner as do the other greases. At the temperatures necessary to ash the grease the lithium and the other alkali metals react with crucibles of varying composition and give only negative results. A platinum crucible may be employed if chlorine is definitely known to be absent (although the oxide is extremely alkaline and if allowed to become too hot will react to a small degree). Our method allowed a separation of other metallic constituents from lithium.

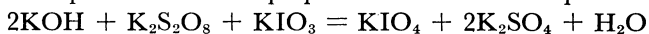
Procedure:

We accurately weighed in a 250 ml beaker 2-5 grams of the grease to be analyzed. 100 ml of 1:1 HCl was added and slowly heated on a hot plate.* The solution was allowed to simmer until the last particles of the grease were broken down and then allowed to digest thoroughly for twenty minutes longer. The solution was cooled almost to room temperature and filtered. The filtrate was made basic to methyl orange by the addition of a saturated solution of potassium hydroxide. We then added 3-4 ml of the KOH in excess and the solution was heated to boiling. The solution was filtered while hot into a 250 ml erlenmeyer flask.

The reagent may be prepared as stated by Rogers and Caley (8) or, to alleviate the use of the more expensive KIO_4 , by making use of the reaction between potassium iodate, potassium hydroxide and potassium persulfate. The reagent was prepared in the following proportions:

150 ml H_2O
24.0 gm KOH
9.33 gm KIO_3
11.75 gm $\text{K}_2\text{S}_2\text{O}_8$

The water and the hydroxide are first mixed thoroughly to complete solution and cooled. The potassium iodate is added, then the potassium persulfate. The preparation follows the equation:



These proportions give a solution two-thirds the concentration of the

*A short stem funnel with its stem inverted may be placed in the solution with the stem upward and act as a "percolator" for the solution.

KIO_4 recommended by Rogers and Caley (8). Dilution produces a clearer solution. This solution may be stored for a month or longer without loss of sensitivity. Carbonate precipitates may be avoided in the reagent by ordinary protection from carbon dioxide of the air.

When the solution in the flask had cooled to room temperature an excess of the lithium reagent was added—approximately 20 ml—and warmed to about 70°C . The solution was then filtered immediately in a Gooch crucible. Rogers and Caley state: "The precipitate formed by this reagent is a mixture of lithium periodates . . . it is apparently useless for the gravimetric determination of lithium." The attempted conversion of the precipitate to the sulfate and a subsequent weighing however, fails. When precipitated in and washed with a strongly alkaline solution (in which the lithium periodate is insoluble only), considerable alkali is retained in the precipitate. The amount varies greatly and it is even possible to eliminate all but a small trace of it under certain conditions, yet it still interferes with the final weighing as the sulfate since it forms a double salt with the lithium. Some typical results are shown in Table I.

Table 1

Vol of solution	Conc. of Standard LiNO_3	Wt. of Li (actual)	Wt. of Li (found)
2 ml	5%	0.0106	0.0205
2 ml	5%	0.0106	0.0135
2 ml	5%	0.0106	0.0170
2 ml	5%	0.0106	0.0204

When we checked the sulfates for potassium, each gave a precipitate immediately with a sodium cobaltinitrite reagent.

Failing to achieve our goal in this direction, we deviated slightly. After precipitation of the lithium as the periodate the precipitate was dissolved in 5 ml of concentrated HCl. Perchloric acid was then added to remove the potassium (less than 5 ml need be used) and the solution was filtered into a tared 50 cc beaker. The precipitate was washed twice with small amounts of perchloric acid. We then added 1 gm of solid ammonium sulfate and boiled the filtrate to dryness. The salt was baked to complete removal of the SO_3 fumes, cooled and weighed as lithium sulfate.

Table 2

Vol of solution	Conc. of Standard LiNO_3	Wt. Li_2SO_4 (actual)	Wt. Li_2SO_4 (found)
1 ml	5%	0.0436	0.0438
1 ml	5%	0.0436	0.0432
2 ml	5%	0.0011	0.0014

A sample of grease* was then broken down and the lithium content analyzed. Results: Wt. of Li_2SO_4 from 2 gm. sample were: 0.0222, 0.0226, 0.0227.

The method of grease break down for the analysis of metallic constituents is very rapid and simple. Ordinary apparatus and reagents found in any laboratory are sufficient for the procedure. The time of the procedure for quantitative studies is shorter than that provided by other standard methods of analysis.

Further study of the complete removal of the lithium content from the grease by precipitation of the periodate in the large volumes of solution used is now being carried out on grease standards.

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