

INORGANIC SYNTHESSES

CHAPTER I

1. PURIFICATION OF LITHIUM CARBONATE

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Since lithium salts obtainable by purchase, even of so-called "c.p." or "reagent" quality, frequently contain impurities totaling about 1 per cent, it is desirable to have a rapid method for obtaining such salts in a reasonably pure state. The following simple procedure for the purification of c.p. or reagent grade lithium carbonate provides such a method, since the resulting pure carbonate may be readily converted by treatment with the proper pure acid into practically any lithium salt desired. The procedure is based upon the fact that lithium carbonate, in contrast to the salts that contaminate it, is much less soluble in hot than in cold water.‡ In other words, simple recrystallization is employed, but the process is carried out in the reverse direction.

Procedure

Twenty-five grams of lithium carbonate is dissolved in 2 l. of water in a 3-l. beaker at room temperature. This operation requires about half an hour if stirred by a motor stirrer. Suspended impurities and any small residue of undissolved carbonate are then removed by filtering the solution, preferably through a large folded filter. The

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‡ At 20°C., 1.33 g. of lithium carbonate is soluble in 100 g. of water; and at 100°C., the solubility drops to 0.72 g.

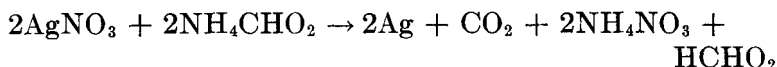
filtrate, in an *unscratched* beaker, is heated gradually almost to the boiling point, vigorous stirring being employed continuously to prevent adhesion of the precipitated lithium carbonate to the walls of the vessel. When precipitation is complete, the hot mixture is filtered at once, preferably through a sintered-glass filtering crucible or funnel, and the carbonate is washed two or three times with small volumes of boiling water. The salt is dried at 110°C.

The yield, based on the amount of lithium carbonate taken, is about 40 per cent. The filtrate from the second filtration may, after cooling, be again saturated with lithium carbonate to give a second crop almost as pure as the first. In this way, the total yield can be considerably increased.

For special purposes, where a product of higher purity is required, a double recrystallization from hot water may be employed; but in this case, of course, the yield is considerably reduced.

From a crude lithium carbonate containing 0.78 per cent SO_4 and 0.54 per cent foreign alkali (calculated as Na) three lots of purified lithium carbonate containing 0.03, 0.08, and 0.07 per cent foreign alkali and only traces of sulfate were obtained. The third lot was obtained by crystallizing lithium carbonate from the filtrate from a previous treatment.

2. PURIFICATION OF SILVER RESIDUES



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Procedure

The dried silver residue is treated with aqua regia (**Care! Hood!**). After action has ceased the solution is filtered through glass wool and the residue is washed to

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