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**LOS ALAMOS SCIENTIFIC LABORATORY  
OF THE UNIVERSITY OF CALIFORNIA ○ LOS ALAMOS NEW MEXICO**

RECOVERY OF LITHIUM-6 DEUTERIDE AND CONVERSION  
TO LITHIUM CHLORIDE

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Per EMS 11-21-79  
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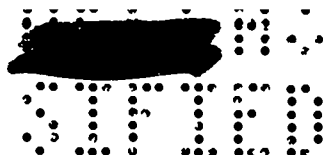
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ABSTRACT

Residues produced from the fabrication of lithium-6 deuteride shapes were collected and hydrolyzed and the lithium converted to the chloride. The chloride was concentrated by evaporation to  $\text{LiCl} \cdot 1.2\text{H}_2\text{O}$  for shipment to the Y-12 Plant of the Union Carbide Nuclear Company.

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## Introduction

When lithium-6 deuteride shapes were manufactured as part of the weapons program at the Los Alamos Scientific Laboratory, it was necessary to recover and recycle the lithium-6 in the residues. Dust collecting equipment was required for the machining operation and a small chemical plant was needed to hydrolyze the lithium-6 in the residues and convert it to the chloride for shipment to the Y-12 Plant of the Union Carbide Nuclear Company. No attempt was made to recover the deuterium.

The problem was somewhat complicated in that lithium deuteride of two different isotopic compositions had to be handled and kept separate. Some of the shapes contained added constituents that required the residues to be worked up individually and chemically purified before conversion to the chloride.

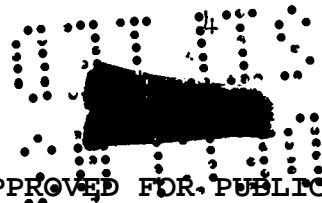
The details of the equipment and procedures for carrying out this assignment are presented in this report.

## Collection of Residues

The majority of the residues from fabrication of lithium-6 deuteride shapes were produced during the following operations: (1) grinding of the feed, (2) pressing into the desired shape, (3) machining to tolerance, and (4) chemical analysis.

Residues from the feed grinding operation were collected by the group (CMB-6) responsible for the grinding. These were exceedingly fine dust and were packaged in an inert atmosphere in bottles which were stored until ready for hydrolysis.

Residues from pressing consisted of small amounts which impregnated the graphite dies. The dies were machined to a depth sufficient to cut out all the lithium and these machine turnings were stored.





Residues produced in the machining operations by the Shops Department varied from fine dusts, resulting from finish cuts, to pieces as large as 3 in. in length, broken off during the rough cutting. This material had to be collected efficiently, not only because of its value but also because of the health and fire hazards associated with the dust.

A lucite box was built around the tool area of each machine. This box was designed to enclose the work, but still allow freedom in the use of the machine. The enclosures were left partially open to allow freedom in the use of the machine, as well as to allow a large flow of air over the tool, which removed the dust and reduced the sparking and explosion hazard. Each box was connected to one of three separate collector systems. Figure 1 illustrates the enclosure and connections installed on one of the milling machines and Fig. 2 shows the installation applied to the lathes.

Adequate air flow and immediate removal of residues were assured by connecting only one or two machines to each collecting system.

Air flow was provided by a 3 h.p. centrifugal blower delivering 500 to 1000 c.f.m. at the pressure drop range maintained (1 to 3 in. of water).

Each collecting system was composed of a cyclone-type separator, an ultra filter, and blower. Figure 3 is a photograph of one of the collecting systems and a cross-section sketch is shown in Fig. 4. Approximately 80 to 90 percent of the residues were removed from the air stream by the 36 in. diameter cyclone separator, which discharged its solids into a 4 or 10 liter glass bottle attached by the standard-taper top. The remaining fine powder was removed by passing the air stream through a 24 by 24 by 12 in. ultra filter, employing fiber-glass mat filter elements.

The air discharged from the blower impinged on a piece of filter paper which continually wetted by a phenolphthalein solution. Any hole or leak in the filter was made immediately apparent by the appearance of bright red spots on the moistened paper.



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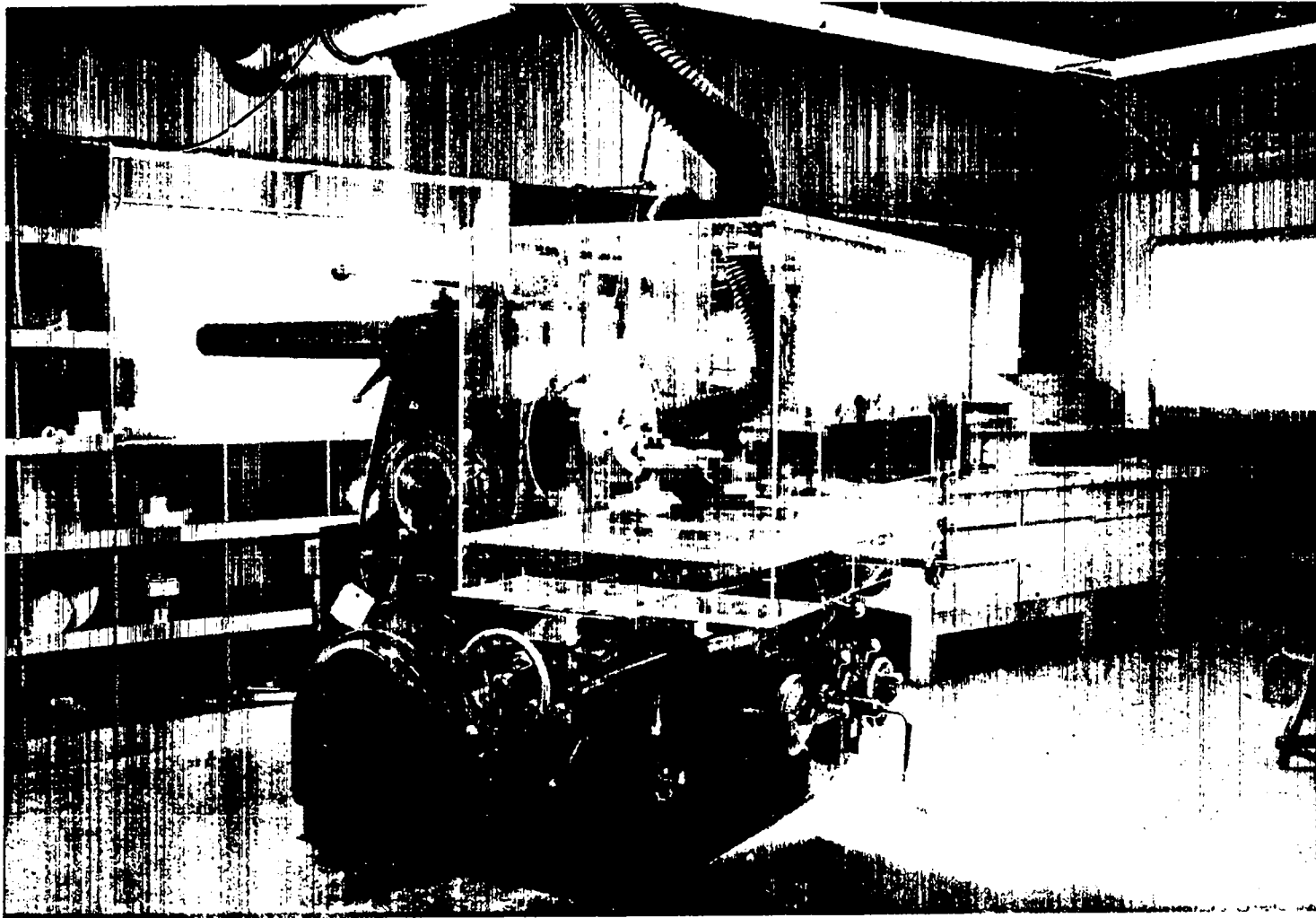


Fig. 1 Enclosed Milling Machine for Lithium Deuteride



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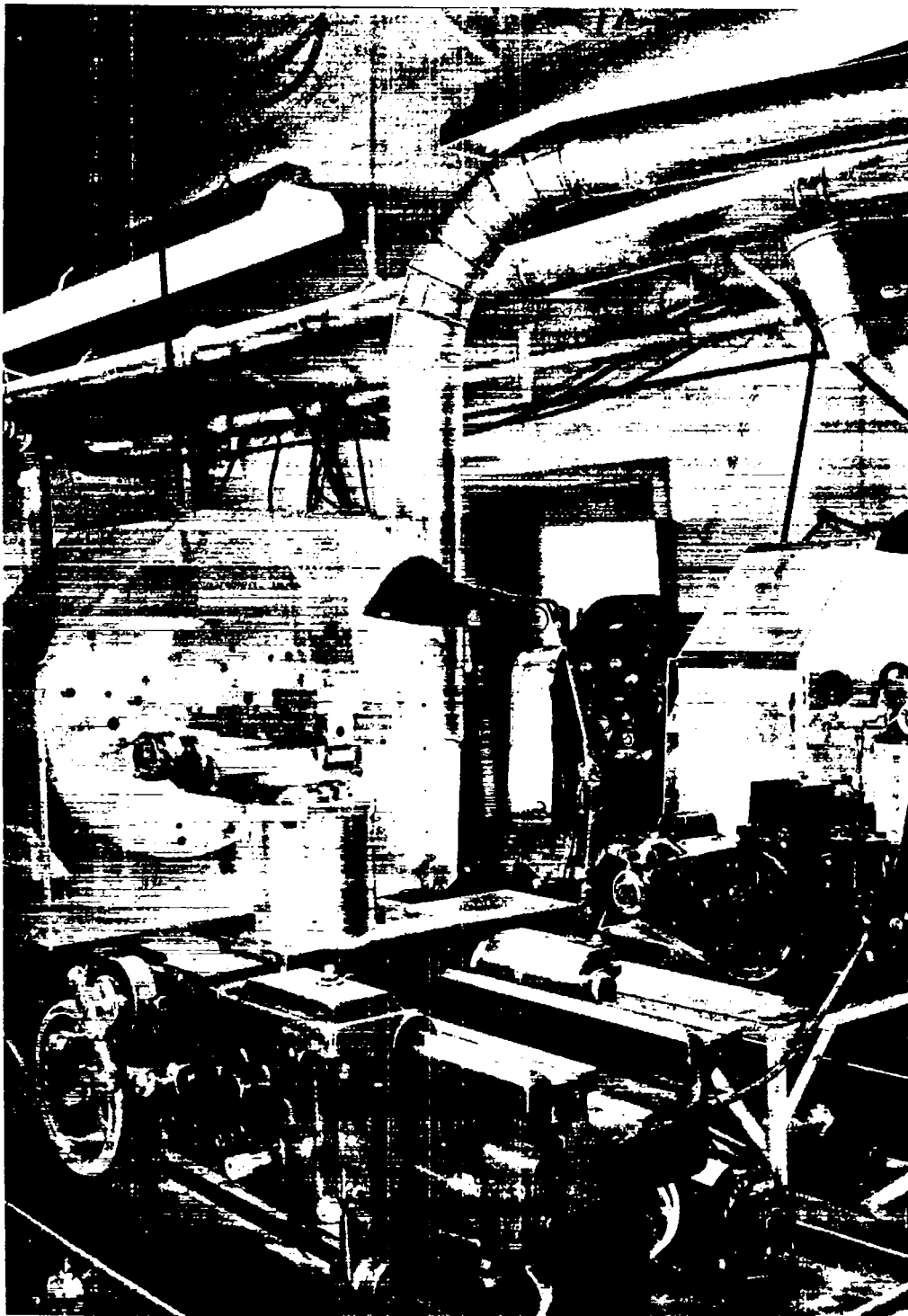


Fig. 2      Enclosed Lithium Deuteride Lathe

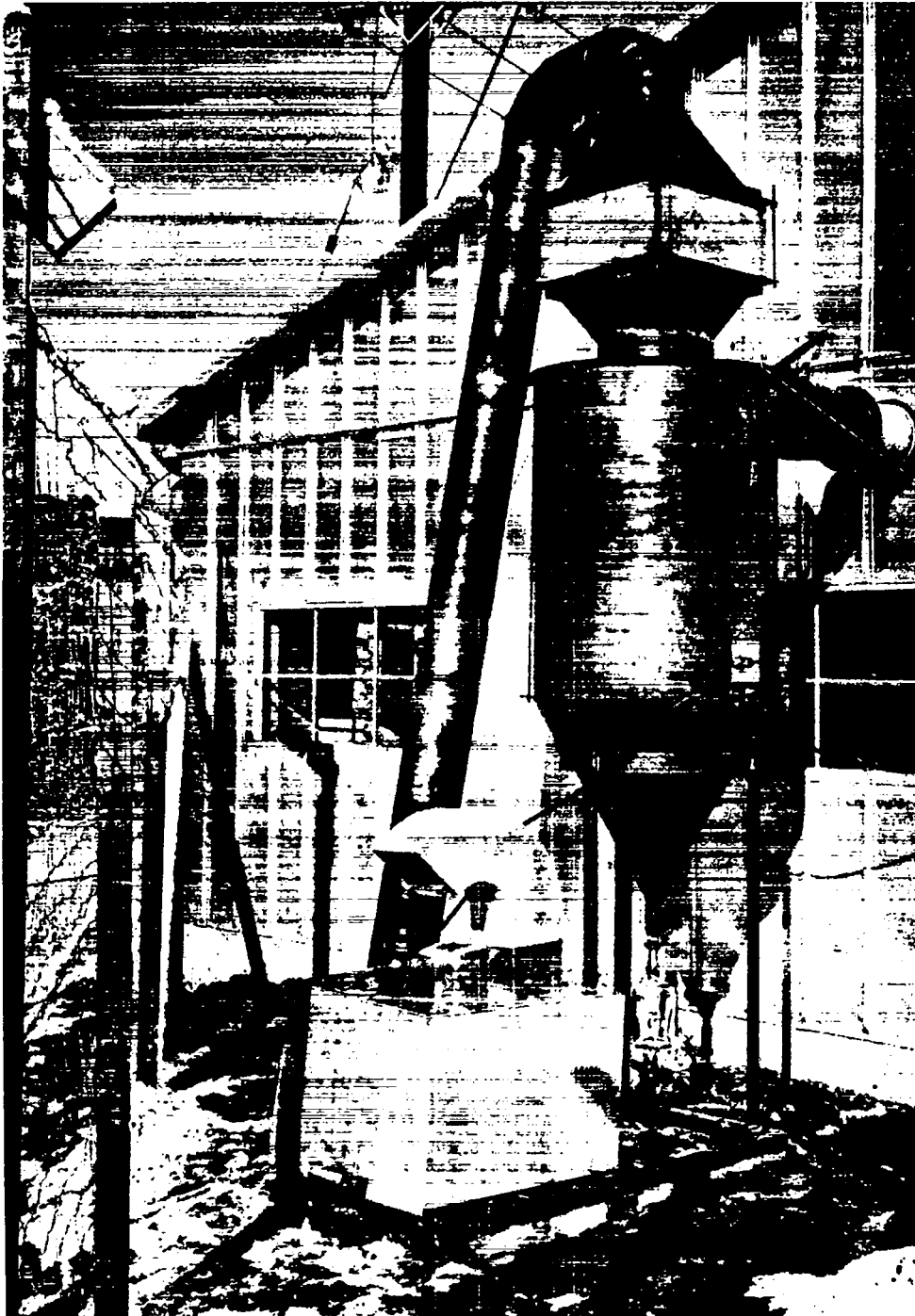


Fig. 3 Photograph of Cyclone and Filter for Collection of Lithium Deuteride Dust

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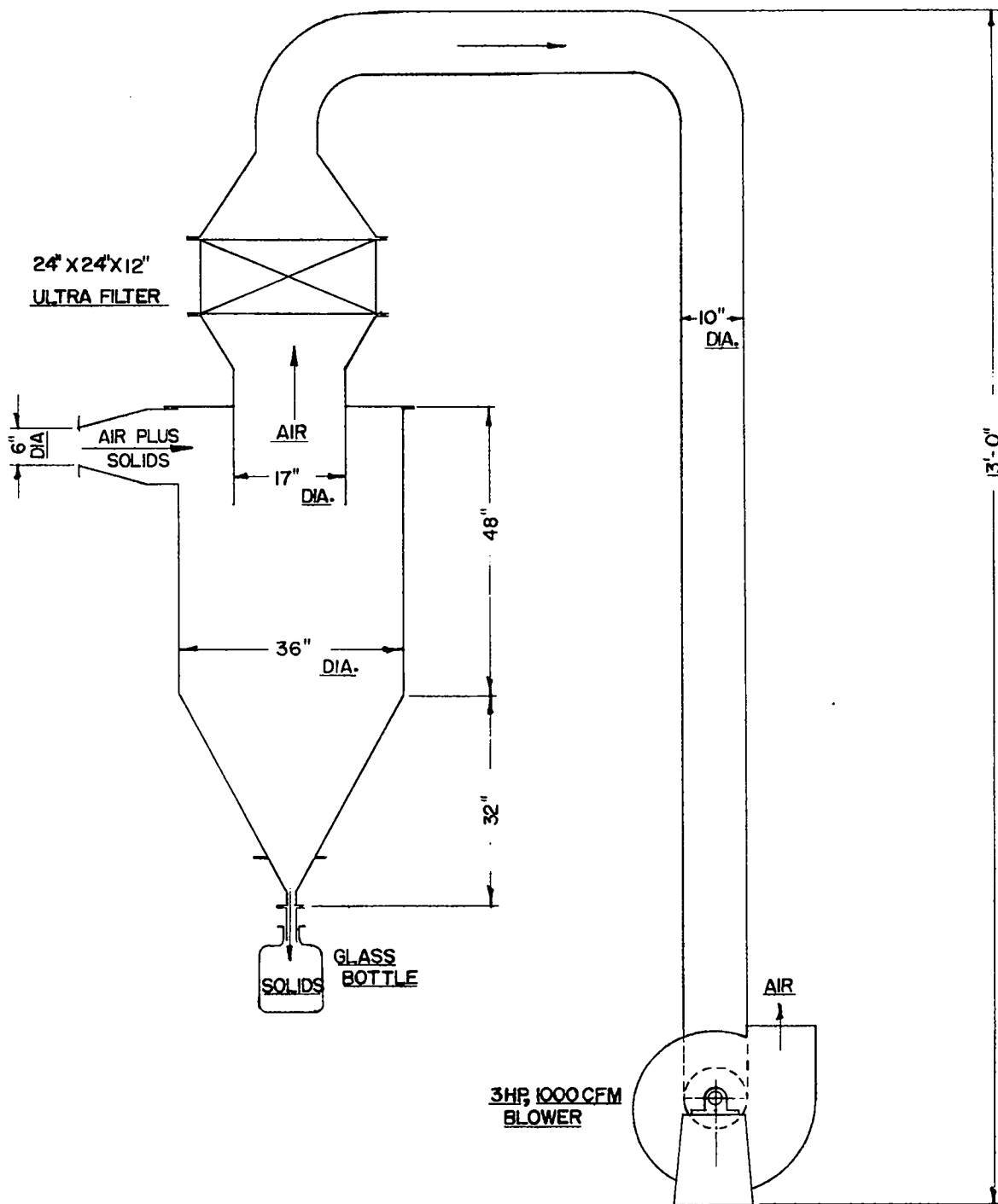


Fig. 4 Sketch of Cyclone and Filter for Collection of Lithium Deuteride Dust

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### Hydrolysis and Conversion to Chloride

The basic steps in the hydrolysis of the lithium deuteride and conversion to chloride are shown in the accompanying flowsheet (Fig. 5).

Hydrolysis. The hazardous step in the operation was the hydrolysis, since the evolution of heat was occasionally sufficient to ignite the evolved hydrogen. Finely dispersed lithium deuteride or hydride would occasionally flash with considerable vigor also.

Because of this hazard, the very fine dusts such as those from the grinding operation were carefully piled into 500 to 1000 g. lots on a stainless steel tray in a hood which had a high air flow. These were then ignited and they burned slowly. Experience had shown that the first lithium oxide formed during ignition created a layer over the pile and prevented the fire from getting out of hand.

Most of the residues from machining had been exposed to sufficient moisture in the air during collection so that the explosion hazard was diminished. Therefore, these powders were not burned prior to hydrolysis.

Hydrolysis was, nevertheless, a delicate operation. The powder was fed at a slow, uniform rate into a turbo-mixer which rapidly dispersed the powder into a large volume of water. A sketch of the turbo-mixer for this hydrolysis is shown in Fig. 6.

The powder residues were placed in the hopper, whence they flowed at a controlled rate by means of a vibratory feeder into the introductory tube of the turbo-mixer. The lower end, which projected into the solution when the stirrer was at rest, was positioned in such a manner that the vortex created by stirring emptied it of water and, at the same time, maintained a water seal on the outer surface of the tube. The stirrer forced air downward through the tube and outward into the solution, thus providing a strong downdraft within the tube which carried the powder down into the liquid. This prevented ignition of the dust and escape of air-borne particles.

This operation was carried out in a well-ventilated hood to prevent the accumulation of an explosive hydrogen-oxygen mixture.

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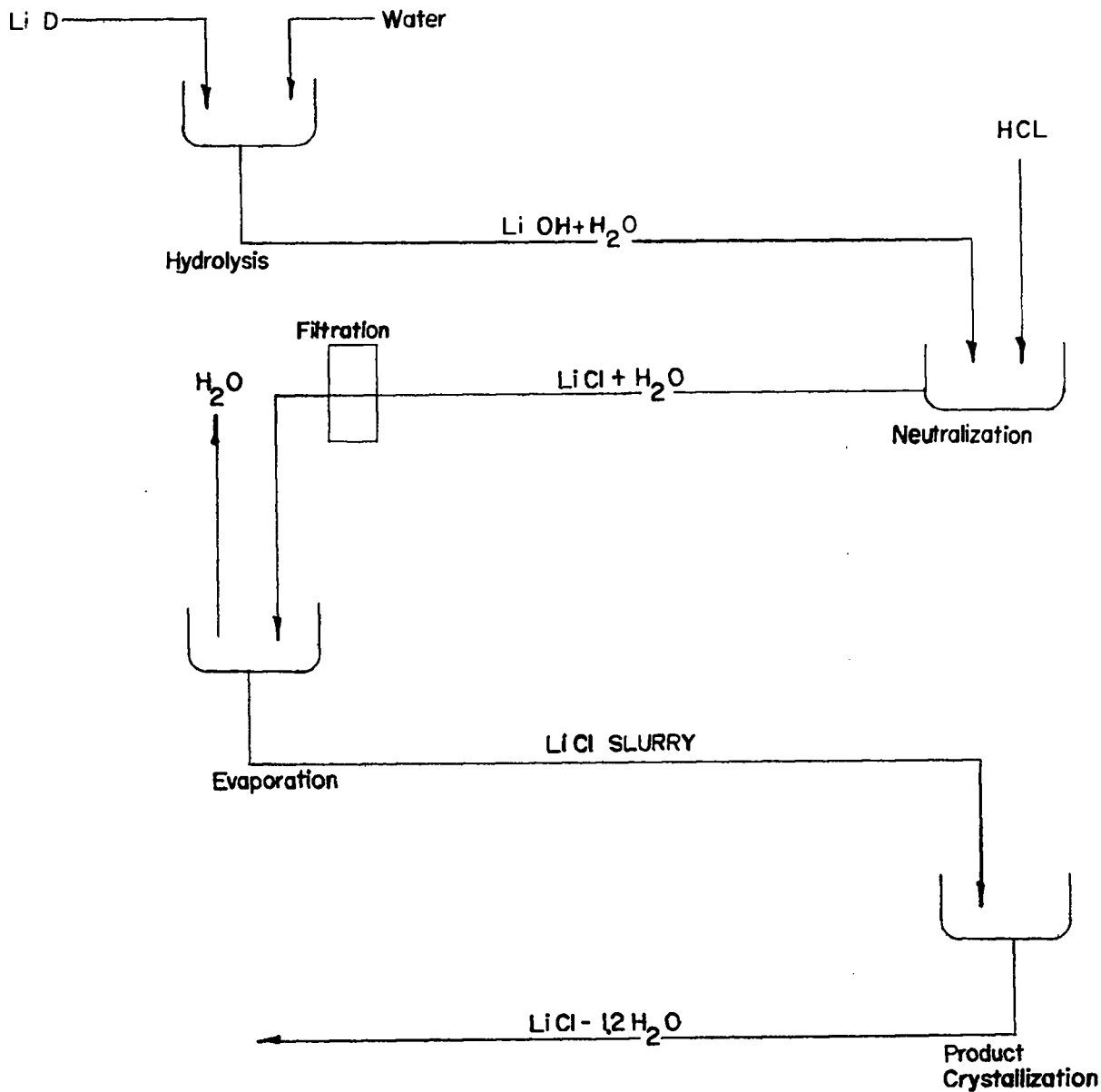


Fig. 5 Flowsheet for Conversion of Lithium from Deuteride to Chloride

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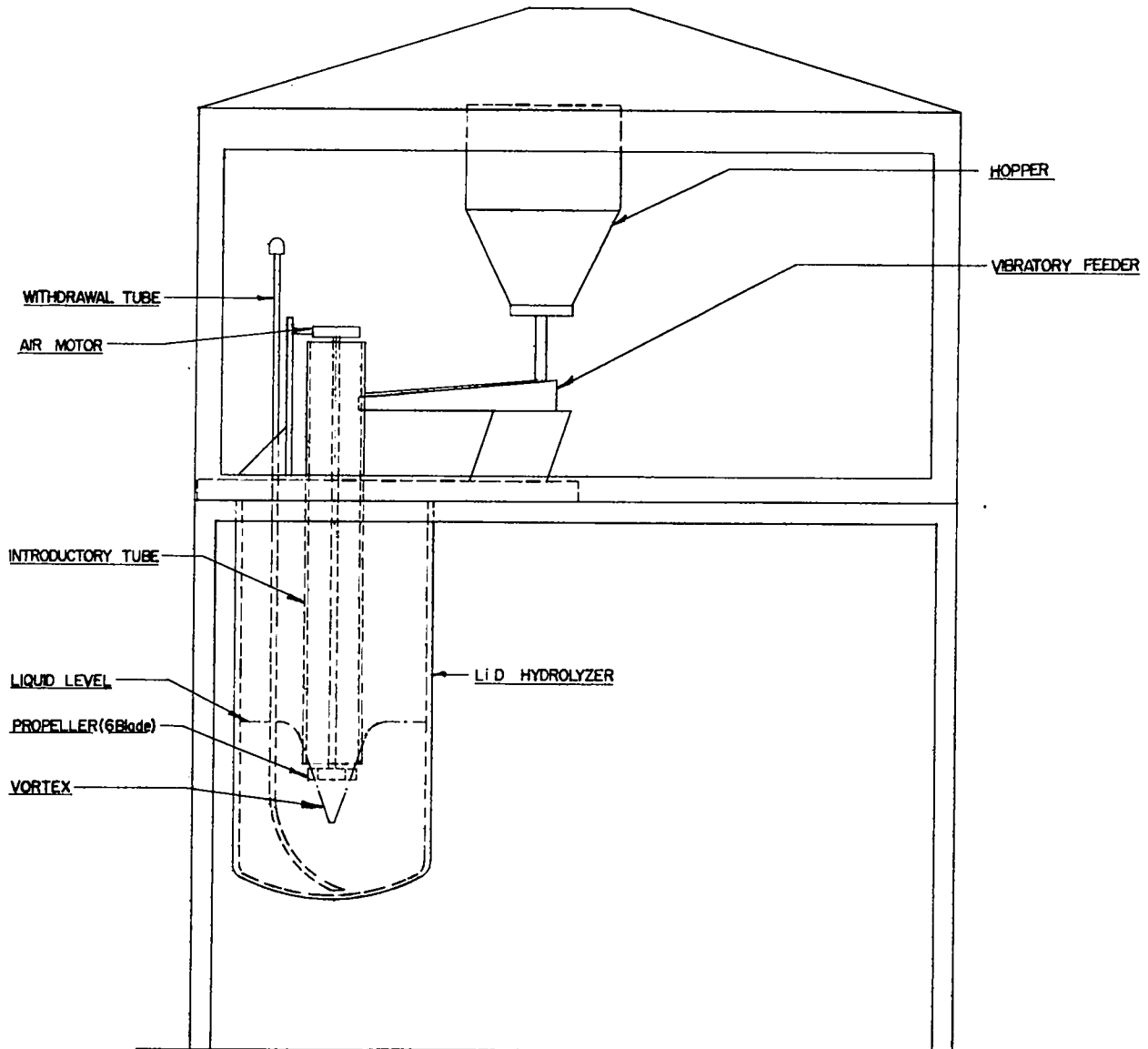



Fig. 6 Lithium Deuteride Hydrolysis Equipment



Neutralization. The lithium hydroxide solution was neutralized with concentrated hydrochloric acid, batchwise, in the same tank in which hydrolysis took place. The progress of the neutralization was followed with a pH meter. After the desired pH value of between 6 and 8 was attained, the lithium chloride solution was transferred through a stainless steel filter element into a glass-lined, steam-jacketed evaporator.

Evaporation. Evaporation was carried to a point (157°C. at 590 mm. mercury) such that the solution was liquid while boiling, but solidified to a slush upon cooling. The liquid remaining after cooling to 20°C. was decanted through a filter and further evaporated to a 162°C. end point.

Crystallization. The boiling solution was dumped from the evaporator into a crystallizer (Fig. 7), which consisted of a paddle-stirred tank in a water-cooled medium. This achieved rapid and reasonably uniform cooling, and prevented the formation of large massive crystals.

The consistency of the final combined product from the two evaporations was approximately that of wet sand and presented no handling, packaging, or shipping problems.

#### Sampling of Product

The molten evaporator bottoms from the initial concentration were weighed and an aliquot was withdrawn before crystallization occurred. The product from the secondary evaporation was combined with that from the first without additional sampling. The samples were analyzed by Group CMB-1, by adding sulfuric acid to excess, heating to red heat, and driving off hydrogen chloride, water, and sulphur trioxide, leaving only anhydrous lithium sulfate. This was weighed and the total lithium content of the batch calculated.

Analysis of the crystallized product of the first evaporation disclosed a mean composition of 56.78 percent lithium chloride. The final product, as shipped, was composed of 65 percent lithium chloride, corresponding to an empirical formula of  $\text{LiCl} \cdot 1.2\text{H}_2\text{O}$ .

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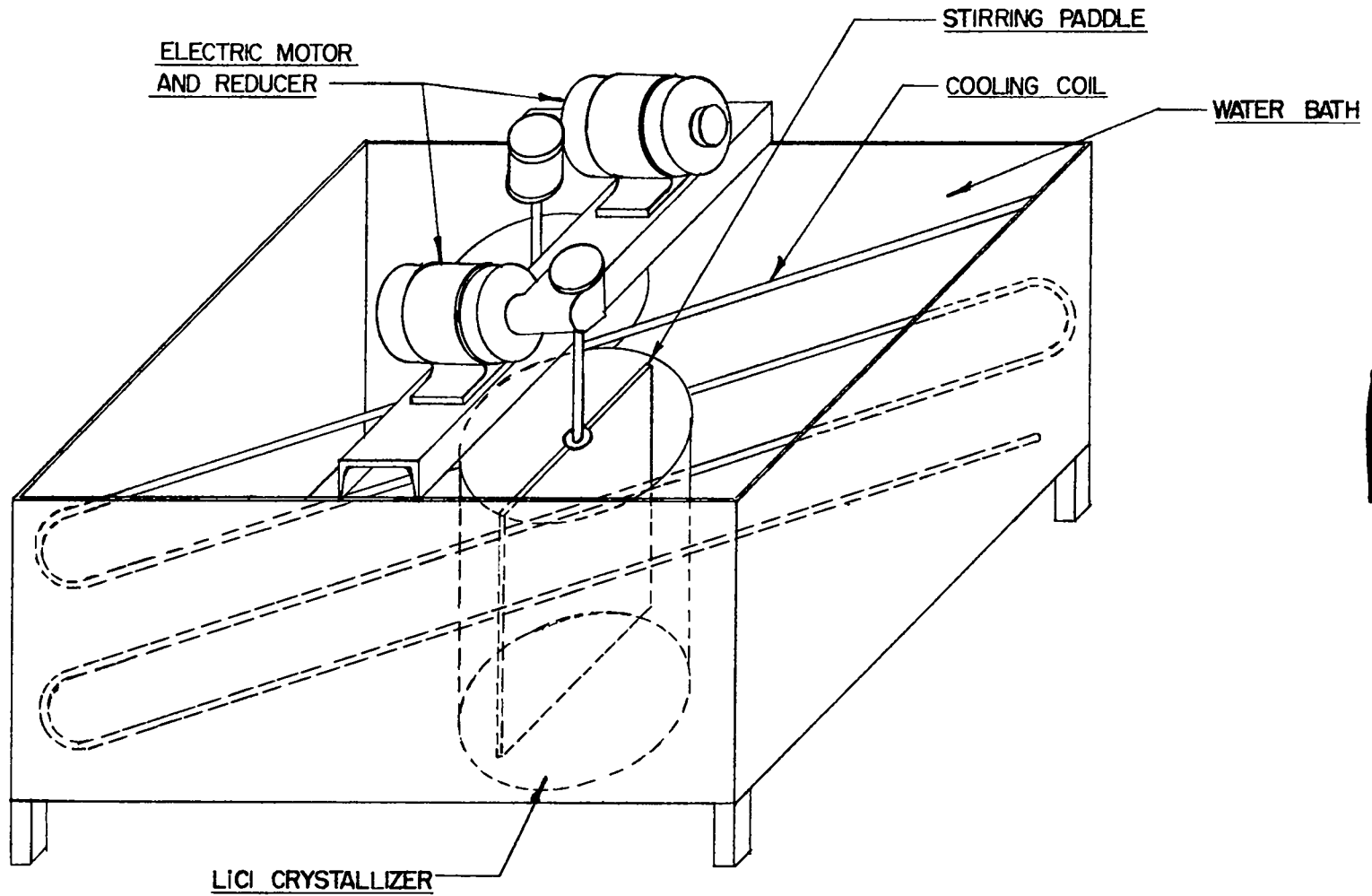


Fig. 7 Lithium Chloride Crystallizer Equipment



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Certain of the lithium deuteride shapes had elements or detectors added to them prior to fabrication. These added elements were arsenic, thallium, germanium, scandium, or rhodium. Residues containing these, or combinations of them, were hydrolyzed in a special closed system so designed that the gases evolved during hydrolysis were scrubbed in sodium hypochlorite solution before being discharged into the air. The alkaline solutions resulting from this hydrolysis were filtered and purified by suitable chemical steps before being added to the regular system.

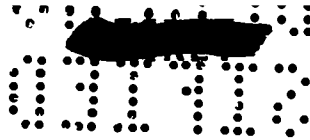
Material Balance

The material balance summary for the low enrichment (37 percent lithium-6) material handled is shown in Table 1. Material balances for the highly enriched (96 percent) material are similar.

TABLE 1  
LOW ENRICHMENT (37 PERCENT) LITHIUM-6  
MATERIAL SUMMARY

1. Material charged to recovery (by difference)	
Machining residues	33,161 g.
Grinding residues	3,446
Analytical residues	<u>165</u>
TOTAL	36,772 g.
2. Material shipped or on inventory	
Product shipped to ORNL	36,751 g.
Inventory: Samples, distillate	<u>44</u>
TOTAL	36,795 g.
3. Material gain in recovery operation	23 g.

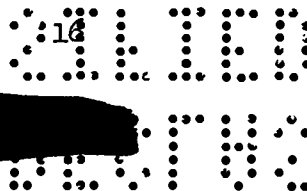
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APPENDIX

Fig. A-1 Boiling Point - Composition Diagram for Lithium Chloride - Water

Fig. A-2 Phase Diagram for Lithium Chloride - Water



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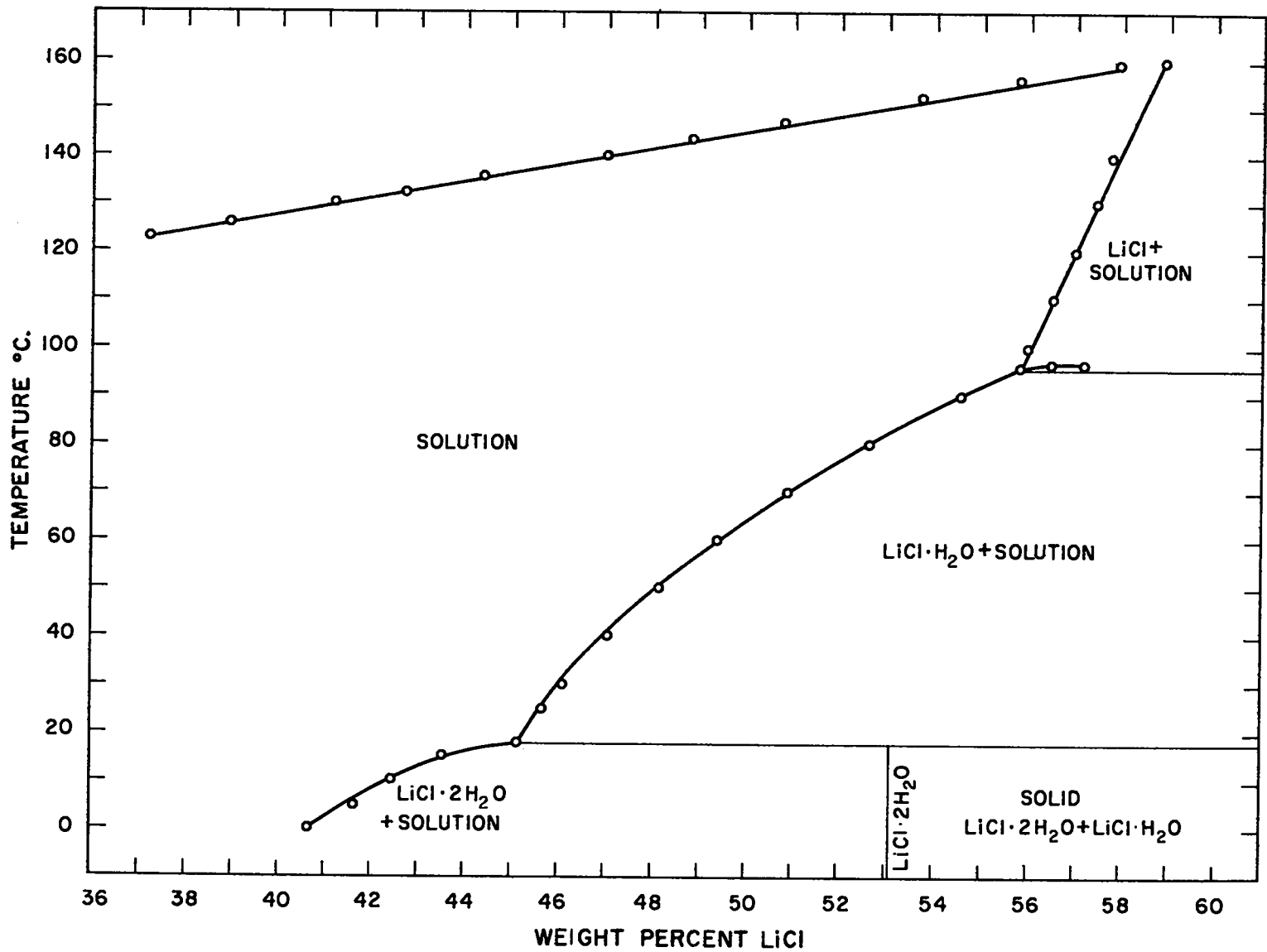


Fig. A-1 Boiling Point - Composition Diagram for Lithium Chloride - Water

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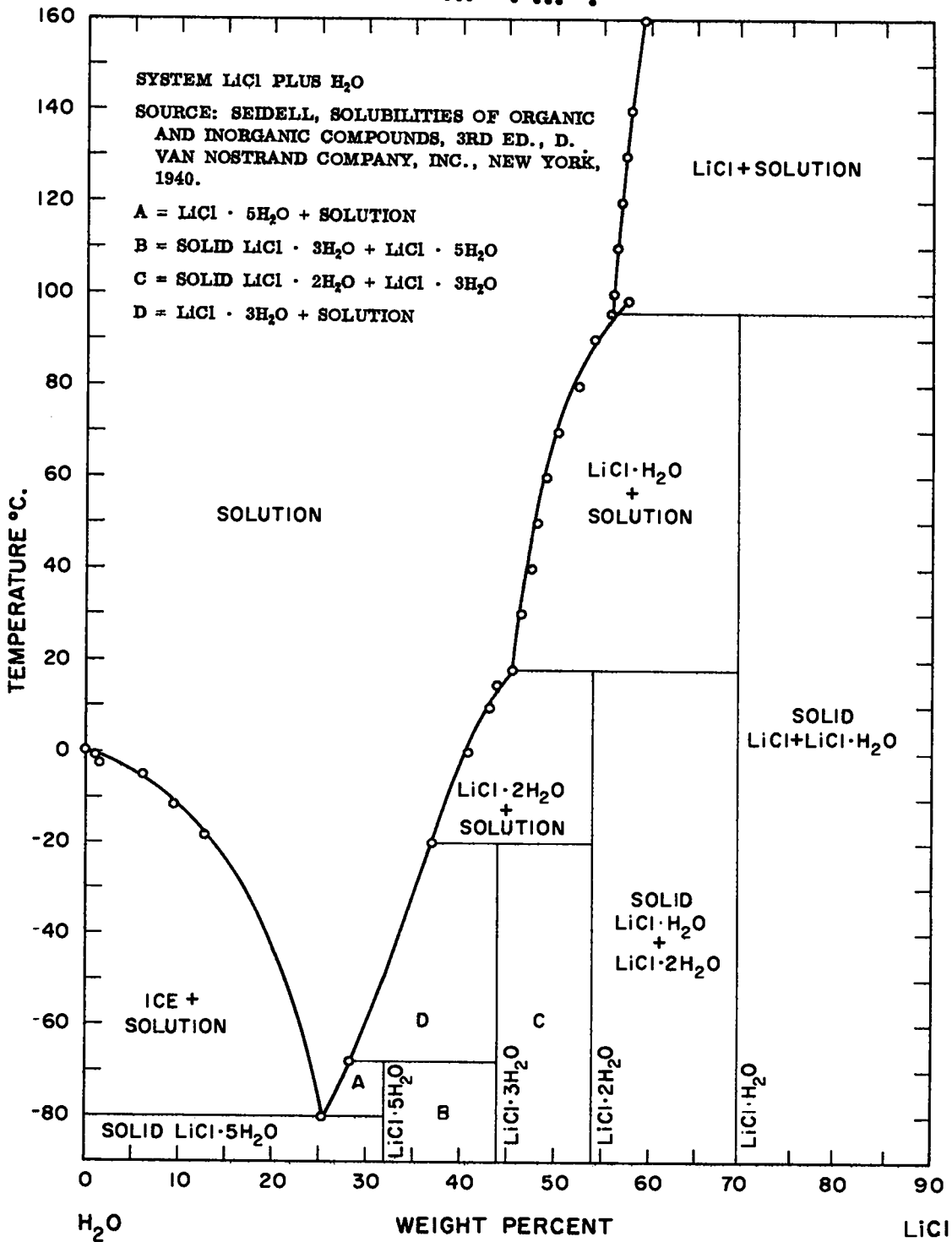


Fig. A-2 Phase Diagram for Lithium Chloride - Water

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