following infectivity LD<sub>50</sub>s were obtained: no cortisone, 1:30, 0 hr, 1:65; 4 hr, 1:230; 7 hr, 1:220; 16 hr, 1:205; 20 hr, 1:70; 24 hr prior, 1:70; and 24 hr after, 1:190.

The results presented here demonstrate quite readily that cortisone greatly increases the susceptibility of white mice to murine typhus. In Table 1, evidence is presented concerning the effect of cortisone on infection in other strains of mice as compared with white mice. Cortisone-treated white mice were found to be 7 times as susceptible as untreated mice. The susceptibility of NHF<sub>8</sub> mice was increased more than 40 times. Cortisone treatment of the C strain, which was routinely more susceptible than the white mice, showed an increase of only a little more than 4 times that of the untreated mice. The effects of cortisone on the dba mice seemed to be approximately equal to those on white mice when 1.25 or 2.5 mg was administered. Although the titration with white mice is not complete, 5 mg appeared to have a greater effect on the dba mice than on the white mice. With the 1:5120 inoculum, the average day of death (ADD) for white mice was 9.25, whereas for the dba it was 5.0.

Jackson and Smadel (9) reported that 0.1 to 1.25mg cortisone administered over 2.5 days had no effect on the susceptibility of mice to toxins of Rickettsia tsutsugamushi, Rickettsia prowazeki, or Salmonella typhosa. We have found that there is little or no effect on the susceptibility to toxin of R. mooseri where 1.25 to 2.5 mg of cortisone was administered 4 to 12 hr prior to titration. White mice receiving 2.5 mg 4 hr prior to toxic titration gave a toxic  $LD_{50}$  of 1 : 80, while controls gave 1:50. When 1.25 mg was given 12 hr prior to toxic titration, the following toxic  $LD_{50}$ s were obtained: white mice, untreated and treated, respectively, 1:90, 1:65; dba mice, untreated and treated, respectively, 1:90, 1:145.

Cortisone treatment of mice seems, therefore, to enhance the susceptibility of this animal to infection with R. mooseri, and the presence of smaller numbers of rickettsiae is indicated by death of mice following injection of infectious material. With the use of cortisone-treated animals, it seems possible that a more rapid diagnostic method could be developed and that experimental typhus infections could be more readily studied.

Smaller numbers of rickettsiae caused death of NHF<sub>8</sub> and dba mice than of white mice after cortisone treatment. This would indicate that the use of these strains of mice would be valuable in studies using R. mooseri.

### **References** and Notes

- L. Thomas, Ann. N. Y. Acad. Sci. 56, 799 (1953).
   N. H. Topping and N. J. Shear, Bull. Natl. Inst. Health, No. 183, 33, 13.
- 3. Committee on Standardized Nomenclature for Inbred
- Committee of Strains of Mice, Cancer Research 12, 602 (1952).
   Albino mice obtained from Maple Grove Rabbitry, Spring-field, Mo.; C strain and NHF<sub>8</sub> from J. K. Frenkel, Kansas Medical School, Kansas City, Kan.; and dba mice from New York and New York. Rockland Farms, N.Y. 5. Merck brand Cortone.
- 6. L. J. Reed and R. Muench, Am. J. Hygiene 27, 493 (1938). 7. E. B. Jackson and J. E. Smadel, Bact. Proc. 1950, 92.

Received December 7, 1953.

# Preparation of Isotopic Lithium Metal by Thermochemical Reduction<sup>1</sup>

## P. S. Baker, F. R. Duncan, and H. B. Greene Stable Isotope Research and Production Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee

In conjunction with the portion of the program of the Stable Isotope Research and Production Division of the Oak Ridge National Laboratory dealing with the measurements of physical properties of isotopes and their compounds (1), small quantities of lithium metal were needed. Since the commercial techniques apparently are suitable only for quantities of 10 lb of salt or more and, thus, could not be used for the small isotopic amounts of the order of 0.5 to several grams, it became necessary to investigate other possible smallscale methods.

Preliminary work with miniature electrolytic cells of our own design showed them to be usable; but, both because of the holdup and because of the longer time required to carry out reduction, an electrolytic procedure was still somewhat less satisfactory than had been desired. Hence, the work turned toward thermochemical possibilities.

Earlier investigators had tried various reducing agents under different conditions, as is indicated in Table 1; but, with the exception of some of the

TABLE 1. Previous investigations.

Year	Investigator	Thermochemical reaction
1857	Troost (2)	Na + LiCl
1896	Warren (3)	Mg + LiOH
1913	Hackspill (4)	Ca + LiCl
1943	National Research	
	Corp. (5)	$Fe + Li_2CO_3$
1944	National Research	
	Corp.	$FeSi_{6} + CaO + LiOH(or Li_{2}CO_{3})$
1947	Kroll and	· · · · · · · · · · · · · · · · · · ·
	Schlechten (6)	$Al(or Si) + CaO + Li_2O$
1947	Stauffer (7)	$\operatorname{FeSi}_{6}(\operatorname{or} \operatorname{Al-Si} \operatorname{alloy}) + \operatorname{Li}_{2}O$

vacuum metallurgical processes involving the reactions of oxy-compounds of lithium with aluminum, silicon, or ferrosilicon, the procedures were not very satisfactory. We found that the FeSi<sub>6</sub>-LiOH-CaO reaction of the National Research Corporation gave a satisfactory product, but we objected to the necessary high vacuum and high temperature.

The work of Jellinek and Czerwinski (8) dealing with the equilibria of the system Ba-LiCl-Li-BaCl<sub>2</sub> suggested barium as a possible reducing agent for obtaining lithium from lithium chloride. Some exploratory work in our laboratory showed that the reaction was suitable; and, subsequently, the following procedure for producing and handling lithium metal was developed.

<sup>1</sup>This paper is based on work performed for the AEC by Carbide and Carbon Chemicals Co., a division of Union Carbide and Carbon Corp., at the Oak Ridge National Laboratory.

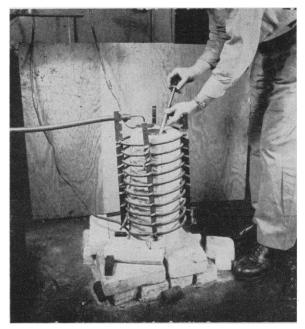


FIG. 1. Pipetting molten lithium.

Lithium chloride and barium metal-the latter in quantity about 80 percent of the stoichiometric requirement-are placed in a container that is enclosed in a heavy-walled stainless steel system. As a precautionary safety measure to prevent any atmospheric oxidation, the system is placed under 5 to 10 lb positive argon pressure by alternately evacuating it and charging it with argon. The system is then heated by induction heating, using a 20-kw, 220-kc Megatherm (9) unit, to a temperature of about 850° C, where it is held for 20 min. The reactor is cooled slowly to about 300° C, the cover is removed, and the molten lithium metal, which is resting on top of a solid BaCl<sub>2</sub>-LiCl salt mixture, is poured into mineral oil.

The metal produced in this manner contains 5 to 8 percent impurities, mostly barium. The barium content may be reduced to a much lower value by reheating the metal with an excess of fresh lithium chloride to about 650° C, and holding this temperature for 15 min before cooling again to 300° C and pouring into oil.

In cases where small quantities of metal are involved, the metal may be pipetted from the surface of the salt after the "cleanup" step by remelting the salt and then using a stainless steel pipet which can be operated either by a plunger or by suction through a plastic tube (Fig. 1). By appropriate choice of the amount of cleanup salt, the barium content may be reduced to about 0.1 percent and the total impurities to about 0.5 percent. Table 2 shows the effects of various treatments upon the final purity of the metal.

Yields of lithium metal amounting to 85 percent, based on barium, are common for metal of 92 to 95 percent purity. Where the purity is 98 to 99 percent, the yield decreases to about 60 percent in the larger batches and to less in smaller batches; much of the loss is attributable to the difficulty in removing metal that is physically entrapped in the solid salt. (This latter yield may be improved significantly by remelting the salt, stirring to coalesce the metal, and then removing with a pipet whatever metal floats to the top.)

Treatment	Impurities (%)	
Reduction of LiCl with stoichiometric		
amount of barium	$\sim 12$	
Reduction of LiCl with 80% of stoichio		
metric amount of barium	~ 8	
Cleanup, 1.5 g fresh LiCl per gram of 92% metal	~ 4	
Cleanup, 4 g fresh LiCl per gram of 92%		
metal	1.5	
Cleanup, 50 g fresh LiCl per gram of	•	
92% metal	0.5	

Once the metal has been poured into mineral oil, it may be remelted by heating the oil to about 190° C. and then it may be cast into any desired shapes or sizes without particular difficulty. Under oil in tightly closed containers, the lithium metal retains a mirrorlike appearance for years.

### **References** and Notes

- 1. P. S. Baker, F. R. Duncan, and H. B. Greene, Science 118, 778 (1953).

- 778 (1953).
  2. L. Troost, Ann. chim. et phys. 51, 103, 112 (1857).
  3. H. N. Warren, Chem. News 74, 6 (1896).
  4. L. Hackspill, Ann. chim. et phys. 28, 613, 630 (1913).
  5. New Processes for Lithium Production, OSRD Report #3758 (June, 1944).
  6. W. J. Kroll and A. W. Schlechten, Metals Technol. 14, T.P. No. 2179 (1947).
  7. P. A. Stanfer, U.S. Potent No. 2 424 512 (to Natl Res.
- R. A. Stauffer, U.S. Patent No. 2,424,512 (to Natl. Res. Corp.) July 22, 1947.
   Federal Telephone and Radio Corp., Model M 120 A.
- 139, 233 (1924)
- 8. K. Jellinek and J. Czerwinski, Z. anorg. u. allgem. Chem.

Received January 8, 1954.

## Quaternary Ammonium Compounds as Molluscacides

A. Vallejo-Freire, O. Fonseca Ribeiro, and I. Fonseca Ribeiro

Instituto Butantan and Faculdade de Medicina Veterinaria da Universidade de São Paulo, São Paulo, Brazil

In a screening study of the killing power of chemicals on fish Poecilia vivipara and Lebistes reticulatus, we observed that some quaternary ammonium compounds which kill them within 1 hr at a concentration of 5 ppm or less also kill Australorbis sp. snails at the same concentration within 24 hr, and that the limit of active concentration is almost the same for both fish and molluscs, especially when the fish used are no larger than 3.0 cm. These facts were observed with cetylpyridinium chloride, cetyltrimethylammonium bromide, cetyldimethylammonium bromide, dimethyl-