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MOUND LABORATORY-MONSANTO
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Mound Laboratory, Miamisburg, Ohio

July 13, 1966

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Mr. Lloyd B. Gnagey

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Attached is the analytical and development program planned by the Analytical Section in response to the SRD letter, COMW 66-190, from E. A. Stisser and A. W. Lundberg to D. L. Scott, dated March 7, 1966.

T. Ben Rhinehammer

T. Ben Rhinehammer

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DEVELOPMENT PROGRAM

1. Measurement of Protium and Hydroxide as a Function of Storage Conditions and Time

A study will be made of the protium and hydroxide content of Li(DT) salt under various packaging and storage conditions as a function of time in order to measure changes in the salt which occur during storage. Minimum criteria for storage and handling can be established.

2. Solubility of Lithium Salts in Non-Aqueous Solvents

Solubility of LiD, LiT and Li(DT) and their impurities, (LiO, Li(OH), etc.) in non-aqueous solvents will be determined using standard analytical techniques. This information is required for analytical methods development and studies on oxide formation.

3. Environmental Thermal Cycling

Measurements will be made of growth rates, helium-3 release rates and crystalline structure for fresh and aged Li(DT) salt which has been subjected to severe thermal cycling. Inter-effects of salt growth, annealing and pre-aging can be observed.

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4. Crystallography Study of Li(DT) Salts by Microscopy and X-Ray Diffraction

A microscopy study will be made: 1) to determine optical properties, crystallographic changes and refractive indices of various isotopic compositions of fresh and aged salt, 2) to determine the effect of radiation damage and helium-3 pressure upon crystallographic properties, grain size and grain boundaries, 3) to compare Li(DT) salt before and after heating to 375°C to expel helium-3, and 4) to estimate the amount and location of free lithium in aged salt. X-ray diffraction studies will include measurement of crystallographic changes, residual stress in the salt crystal and intensity levels over a range of temperatures and conditions with specially prepared and aged salts.

Analytical

Analytical capabilities will include analyses for lithium isotope ratio, total lithium, free lithium, hydrogen isotopes, hydroxide, oxide, carbon, carbonate, nitrogen and trace impurities (cations). Physical property analyses will include particle size, surface area and density measurements.

The accuracies requested in COMW 66-190 cannot be obtained in most cases by the usual wet chemistry or instrumental techniques. Generally the following relative accuracies will be provided.

Amount of Major Constituent	Accuracy
10 - 90%	±0.1%
0.1 - 10%	±1.0%
0.01 - 0.1%	±10%
0.001 - 0.01%	±50%

The latter two ranges can be reduced to ±1 and ±5% respectively, depending upon the components involved. A spark source mass spectrometer will be procured during FY-1967 and should be operational in FY-1968. Analyses down into the parts per billion range can be provided for most trace impurities found in Li(DT) salt.

In direct response to topics of Part II.

1. Analyses for all isotopic mixtures, spectrographic analysis of trace impurities, stoichiometry and free lithium analyses can be provided at the accuracy level given above. Analyses for

free hydrogen cannot be provided but can be determined by pulsed NMR at LRL.

2. Oxygen and hydroxide content of the salt will be provided. The form or oxygen distribution as lithium oxide or hydroxide will be supplied. The origin of the oxygen will be determined by analyses at various steps in the salt preparation: 1) starting materials, 2) reaction, and 3) handling and storage.
3. The origin and content of carbon and nitrogen can be provided at accuracy levels discussed above and location in the salt by techniques in (2) above.
4. Pulsed NMR and EPR equipment is not available at Mound Laboratory. A minimum of $30,000 would be required before studies could be initiated in this area. Optical studies are planned.

Special Li(DT) Salt Preparation

Ultra-high purity material cannot be supplied LRL before March 1, 1966 when the necessary dryboxes and equipment have been installed for preparing multiple-distilled lithium and salt under accurately-controlled conditions. Complete chemical analyses are scheduled

by that date pending completion of the analytical laboratory modifications for Li(DT) salt analyses. It is expected, however, that analyses for hydrogen isotopes, oxygen, nitrogen and hydroxide can be provided routinely by November 1, 1966.

Costs for High Purity Salt Preparation and Analyses

The manufacture of ultra-pure salt with complete chemical and physical property analyses to meet LRL requirements for various isotopic mixtures and specified compositions is estimated at $850 per salt batch. An average of four salt samples per month could be supplied at a cost of $40,000 per year.

The cost of current and planned Research and Development activities at Mound Laboratory, as outlined above, will be absorbed under research, development and surveillance programs connected with Li(DT) salt devices and weapon programs.

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COST CALCULATIONS FOR PREPARATION AND
ANALYSES OF SPECIAL Li(DT) SALT

Time Man-Hours	Procedure
8	Prepare lithium and gas mixture, gas analysis, reaction, lithium purity analysis
8	Grind salt, package, ship and prepare analytical samples
4	Oxygen and nitrogen analysis
4	(OH), C, CO_3 analyses
8	Impurity analyses - atomic absorption spectrophotometry and emission spectrograph
4	Gas analysis of salt for H, D, T
4	Physical property analysis - density, surface area, particle size distribution
4	Free lithium and lithium isotope ratio analyses
4	Administration and supervision
50	TOTAL

50 manhours x \$17/hr. = \$850/salt batch. Salary of \$10,000/year or \$5.00/hr. with 250% overhead equals \$17/hr. for cost figure.