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JULY 1, 1965

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BATTELLE-NORTHWEST

RICHLAND, WASHINGTON

MONTHLY PROGRESS REPORT ON WORK UNDER C-38176

by

K. M. HARMON

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MONTHLY PROGRESS REPORT ON WORK ORDER C-38176

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by

K. M. HARMON

This document consists of 5 pages. [redacted]

Chemical Separations
Chemical Research
Chemistry Department

July 1, 1965

[redacted]

NOTICE: PRELIMINARY REPORT

This report contains information of a preliminary nature prepared in the course of work under Atomic Energy Commission Contract AT-(45-1)-1830. This information is subject to correction or modification upon the collection and evaluation of additional data.

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BNWL-CC-152

July 1, 1965

Mr. T. W. Evans
Senior Engineer
Chemistry and Metallurgy Operation
N-Reactor Department
General Electric Company
Richland, Washington

Dear Mr. Evans:

Attached is a copy of our June, 1965 monthly report concerning work done by the Chemistry Department under authorization of your Work Order No. C-38176.

Very truly yours,

ORIGINAL SIGNED BY
K. M. HARMON

Manager
Chemical Separations
Chemical Research

KM Harmon:ew

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MONTHLY PROGRESS REPORT
JUNE, 1965
W. O. Greenhalgh and W. H. Yunker

Examination of unirradiated lithium aluminate by infrared spectroscopy indicates that this technique will be a useful tool for studying the concentration of OH and OT in target materials. Absorption in the 1400-1600 cm^{-1} range, presumed due to bending (ν_2) of adsorbed H_2O, was found to disappear upon vacuum outgassing of the sample (4 hrs at 800° C), but a residual absorption remained near 3400 cm^{-1}, due to OH stretching. The aluminate was observed to be transparent in the region of 2100 cm^{-1}, that anticipated for the OT stretching frequency, so that it should be possible to detect any appreciable amounts of OT present in the lattice. Experiments are under way to determine approximate extinction coefficients. It is hoped that use of these techniques on irradiated material (aluminate and silicates) will provide a means for following hydrogen isotope exchange and concentration changes in the various target materials.

Solubility studies with unirradiated materials are completed, for the present. The results are summarized in Table I. The more successful solvents are being tried on irradiated materials. One sample of irradiated lithium silicate has been dissolved in sodium tetraborate at 900° C (~ 25 w/o lithium silicate). Most of the melt distilled to the cooler sections of the apparatus in the course of the treatment. Though all analyses are not yet in, it was noted that the atom ratio of T/He^4 in the non-condensable gas fraction was 1:1, and that twice as much tritium (probably as T_2O or TOH) was condensed in the liquid N_2 trap as passed through it.

The remains of the sample (including the part of the melt which distilled) are being analyzed for tritium. If none is found, further work will be done on the use of this dissolution-distillation technique for measuring the total tritium content of a sample.

Interest has focused on the production of "low temperature" lithium aluminate*, primarily because of the possibility of effecting a separation during the 30% volume increase which occurs during the transformation to "high temperature" aluminate (above 600° C).

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* Lehmann, Z. Anorgan. und Allgem. Chem. 313, 117 (1961).

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SUMMARY OF DISSOLUTION STUDIES

SOLVENTS FOR LITHIUM ALUMINATE AND LITHIUM SILICATE

Sample	Flux	Weight % Soluble	Time (hrs.)	Temp. (° C)	Melt Properties
$LiAlO_2$	Borax glass	85	2-1/2	750	Viscous glass
$LiAlO_2$	Potassium pyrosulfate	10	3	700	Low viscosity yellow melt; gas evolved initially.
$LiAlO_2$	Potassium Meta Phosphate glass	19	1-1/2	750	Low viscosity glass
$LiAlO_2$	50/50 wt. mixture Potassium carbonate Sodium carbonate	9	1	800	White melt; corrosion problems may be encountered
Li_2SiO_3	Borax glass	> 50	1-1/2	800	Viscous glass
Li_2SiO_3	50/50 wt. mixture Potassium carbonate Sodium carbonate	85	2-1/2	750	Low viscosity yellow melt; corrosion problems may be encountered

Non-solvents for Lithium Aluminate and Lithium Silicate

$LiAlO_2$	Li_2SiO_3
50% LiCl, $PbCl_2$ mixture	$K_2S_2O_7$
$CdCl_2$	$BaPO_3$
B_2O_3	
Na_2SO_4 with 10% eutectic LiCl, KCl	
$CaCl_2$	
$MgCl_2$	
$Na_4P_2O_7$	

Note: Powdered samples were used in all of the above studies