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PREPARATION OF LITHIUM
ISOTOPIC STANDARDS

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PREPARATION OF LITHIUM ISOTOPIC STANDARDS

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ABSTRACT

A method has been developed for the precise preparation of lithium isotope standards in the range of 20 to 97 at % lithium-6. Standards were prepared by carefully weighing and mixing isotopically pure lithium-6 and lithium-7 sulfates. A statistical limit of error (0.95) of $\leq \pm 0.0025$ at % lithium-6 was achieved.

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SUMMARY

Five reference lithium isotope standards were prepared from isotopically pure lithium-6 and lithium-7 sulfates. Final concentrations were: 20, 44.3, 66.5, 93, and 97 at % lithium-6. Maximum limit of error (0.95) for the calculated isotopic contents of the final standards was ± 0.0025 at % lithium-6.

Details of the preparation and computations are included to verify the validity of the standards and to serve as a ready reference to the procedures involved should other high-precision standards be desired.

INTRODUCTION

Reference lithium isotope standards in a wide range of isotopic concentrations were desired by the Plant Laboratory of the Oak Ridge Y-12 Plant^(a) for calibration of mass spectrometers. The objective was to provide several intermediate-assay levels in the range of 20 to 97 at % lithium-6, spaced to complement the existing standards. An accuracy of $\pm 0.01\%$ in isotopic content was desired. Lithium-6 and lithium-7 sulfates of high chemical and isotopic purity were available. It seemed practical to achieve the desired certainty of isotopic content by carefully mixing precisely weighed quantities of the lithium salts.

Factors affecting the quality and value of the final isotopic standards prepared in this way include: purity of the starting materials, handling techniques, certainty of weighings, adequate mixing, and computation of isotopic content. All these factors must be carefully controlled to produce reference standards having a high degree of confidence.

(a) Operated by the Union Carbide Corporation's Nuclear Division for the US Atomic Energy Commission.

PREPARATION OF LITHIUM ISOTOPIC STANDARDS

PURITY OF LITHIUM SULFATES

Isotopic purity of the lithium salts was achieved by separation in a Calutron (large-scale electromagnetic mass spectrometer). Chemical purification of the isolated isotopes was accomplished by chromatographic separations in ion-exchange columns utilizing both anion and cation exchange resins.⁽¹⁾ Conversion to the sulfate form provided a chemically and isotopically pure lithium compound in a stable and weighable form.

Isotopic contents of the final sulfate salts were determined by repeated mass spectrometer measurements by two laboratories. Statistical evaluation of 67 measurements on the lithium-6 sulfate showed an isotopic content of 99.999292 ± 0.000072 at % lithium-6; evaluation of 64 measurements on the lithium-7 sulfate gave an isotopic content of 99.99868 ± 0.00042 at % lithium-7. All limits of error were calculated at the 95% confidence level.

Chemical purity of the lithium sulfate salts was checked by spark-source mass spectrometry. A total of 72 elements were sought, most of which were absent or less than background and not detected. Elements found and their concentrations are listed in Table 1. The amount of impurities found was $< 0.02\%$ by weight and approximately the same in both the lithium-6 and lithium-7 materials.

HANDLING TECHNIQUES

To avoid contamination chemically and isotopically, but especially isotopically, great care was used in handling materials and equipment. Individual stainless steel weighing scoops were made for each of the two lithium sulfates. New stainless steel scoop-type spatulas, nitric acid leached, were used in transferring material while weighing. Storage of the final mixes was in four-ounce polyethylene bottles leached several days in 1:1 hydrochloric acid followed by a demineralized water leach.

The two pure lithium sulfates were handled only in hydrochloric acid-leached platinum dishes. Each material was handled separately in such procedures as weighing, ignition, and desiccator storage. The lithium sulfates contained in the platinum dishes were ignited at 775°C for 2 1/2 hours to constant weight to remove any residual moisture. Final ignition was no more than two days before use, with storage in a desiccator kept dry with

Table 1
ANALYSIS OF LITHIUM SULFATE MATERIALS

Element Found	Element Concentration ($\mu\text{g/g}$)(1)	
	Lithium-6	Lithium-7
Aluminum	8	3
Arsenic	2	3
Boron	6	5
Bromine	ND(2)	2
Calcium	60	50
Chromium	30	20
Copper	9	7
Iron	30	10
Nickel	10	10
Selenium	5	10
Strontium	ND	3
Vanadium	20	10
Total	180	133

(1) Data obtained by visual estimation of line densities on a photographic plate and should be correct within a factor of two.

(2) Not detected.

magnesium perchlorate. The ignition muffle was platinum lined to maintain purity, and each desiccator was especially cleaned before use.

Moisture pickup was minimized by performing weighing operations in a low-humidity room. With the temperature range maintained to $\pm 1^{\circ}\text{C}$ and a relative humidity of 6 to 10%, no detectable moisture absorption was observed in two experiments that involved the exposure of one gram of lithium sulfate on a balance pan for times of up to 17 minutes, a period that was more than adequate for weighing and handling the material.

WEIGHING APPARATUS

Weighing Requirements

A limiting factor in the accuracy of the standard mixes was the ability to achieve an accurate weight. Approximately one gram of mixed lithium sulfates was desired in each standard. For the 97 at % Li-6 standard, this one gram of mixed sulfates consisted of approximately 970 milligrams of lithium-6 sulfate and only 30 milligrams of lithium-7 sulfate. To achieve the desired accuracy of 0.01 at % in the final standards, weighings must be certain to ± 0.1 milligram, and preferably less.

Choice of Balance

Readily available balances with the desired sensitivity included a Cahn Gram Electrobalance, Model G, and a conventional double-pan semimicro balance. Tests with the Cahn balance showed that the desired sensitivity could be reached with counterweights and a sensitive scale; however, the capacity of the Cahn balance was only 1.2 grams. This low limit required the use of an extra-lightweight scoop to prevent overloading of the weighing mechanism. A suitable scoop was made from two-mil (51- μm)-thick aluminum foil. Simulated weighing experiments soon showed that the Cahn balance pans were too small and were unstable with the scoop and counterweights in place. Consequently, the risk of spillage was great and the Cahn balance was considered inconvenient to use in this application.

An Ainsworth, Type FDJ, semimicro balance of 20-gram capacity was set up in a low-humidity room. This light projection balance was the most sensitive conventional balance readily available and, from prior usage, was believed to be adequate for the purpose. It was soon found that the balance must be protected from strong air currents by placing a deflector at the room air intake. Even small air currents, such as the breath of a person, affected a tiny beam rider used for zeroing the balance. This secondary rider was subsequently removed to avoid any unnoticed changes in setting.

A series of tests for sensitivity, stability, and accuracy were performed on the Ainsworth balance to confirm the expected suitability for weighing standards.

Balance Testing and Calibration

The Ainsworth FDJ balance was checked for sensitivity, accuracy of beam-rider calibration, equality of beam-arm length, and reproducibility. Sensitivity was found to be 117 divisions on the light projection scale per milligram weight or 0.0085 milligram (8.5 μg) per scale

division. A one-milligram change in the beam-rider setting represented 1.004 milligrams of true weight. Tests showed that the left balance beam arm was longer than the right beam arm by one part in a million. Strouts, et al⁽²⁾ state that less than three parts in a 100,000 is insignificant for normal work. Reproducibility weighings of a two-gram stainless steel object gave a limit of error at the 95% confidence level for a single weighing of ± 0.0025 milligram. Details of testing and calibration are given in Appendix A.

WEIGHING TECHNIQUES

The weighing scoops (5 centimeters in length) were fashioned from a five-mil (127- μ m) polished stainless steel sheet. The design, as seen in Figure 1, was tapered with a small end which fit into the mouth of a four-ounce plastic bottle for easy transfer of the weighed crystals. Easy grasp for forceps was provided by a protrusion from one side of the scoop. Each scoop was provided with a stainless steel counterweight of mass within one milligram of that of the scoop. The scoops were scrupulously cleaned and handled only by forceps. The lithium-6 and lithium-7 scoops were stored in individual plastic vials to avoid cross contamination.

Static eliminating devices, such as polonium sources, are useful with delicate balances in a dry atmosphere. Two of these units were mounted on a small stand, one unit on either side

Figure 1. STAINLESS STEEL WEIGHING SCOOP. (Scale is in Centimeters)

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of the balance pan. Both balance pans were so surrounded. The sources available were old and not as effective as desired, although some radiation was still emitted.

The balance rest point was determined by a method of swings often used with undamped balances. Details are given in Appendix B.

An approximate amount of each isotopic lithium sulfate needed for each standard was calculated as a guide in weighing. While not exact, these values gave standards in the concentration ranges of interest. (Calculation details are given in Appendix B.)

The weighing procedure was straightforward. A weighing scoop in the left pan was balanced against its counterbalance, with any difference in weight being made up by the beam rider. Fractional weights from a National Bureau of Standards (NBS) calibrated set were added to the right pan plus any beam-rider changes necessary to equal the weight of the lithium sulfate to be weighed. Lithium sulfate was carefully added to the scoop until the weight was within 0.1 milligram of the calculated value. Data for the balance rest point were recorded.

Transfer of the dry salt was performed by inserting the tip of the weighing scoop into the mouth of a specially cleaned four-ounce plastic bottle and lightly tapping the scoop to dislodge any adhering particles. The empty scoop was weighed back as the tare weight, again carefully recording the rest-point data. No particles could be seen on the polished scoop surface after any of the transfers were made. Note was made of the temperature, relative humidity, and barometric pressure during the weighing period for later buoyancy corrections.

MIXING OF SALTS

Once both the weighed lithium-6 and lithium-7 fractions of a mix were transferred to a plastic bottle, a homogeneous mixture was produced by dissolving the salts in 50 milliliters of demineralized water. No lithium was detected by emission spectrographic analysis of the water (< 0.01 mg Li/l).

CORRECTION OF THE LITHIUM SULFATE WEIGHTS

Corrections were applied to the apparent weights of the lithium sulfate portions to obtain, as closely as possible, the true mass of the weighed lithium sulfate. The tantalum and aluminum weights used were corrected to true mass. Buoyancy corrections for the lithium sulfate were made, based upon the densities of the lithium sulfate material and the weights used. Weight equivalent of any changes in the beam-rider settings and rest point were added to the apparent mass of the lithium sulfate. Details of these corrections are given in Appendix C.

ISOTOPIC VALUES OF THE PREPARED STANDARDS

From the corrected weights of lithium sulfates taken and the isotopic content of the starting materials, isotopic values were calculated for the final mixes. All known errors in weighing operations and isotopic purity were included in a statistical evaluation of the results. The computed lithium-6 content of each standard mix and the corresponding limit of error at

the 95% confidence level are reported in Table 2. The degree to which each lithium-6 isotopic value was known was equal to or less than two parts in 20,000, or 0.01% relative. Details of the calculations are given in Appendix D.

Table 2
COMPUTED VALUES OF LITHIUM ISOTOPE STANDARDS

Nominal Lithium-6 Content (at %)	Corrected Weight of Lithium-6 Sulfate Used (mg)	Corrected Weight of Lithium-7 Sulfate Used (mg)	Computed Lithium-6 Content (at %)	Limit of Error (0.95) on Lithium-6 Content (at %)
20.0	197.141	802.963	20.0050	± 0.0022
44.3	438.740	561.620	44.3109	± 0.0019
66.5	661.377	339.195	66.5098	± 0.0019
93.0	929.213	71.391	92.9853	± 0.0024
97.0	969.809	30.482	97.0058	± 0.0025

APPENDIX A

BALANCE TESTING AND CALIBRATION

Balance Sensitivity

Balance sensitivity was determined by observing the change in the rest point caused by the addition of a known weight to one side of the balance. A rest-point change of 47 divisions of the light projection scale was produced by a beam-rider change of 0.4 milligram. From this information, the balance sensitivity was calculated as 117 scale divisions per milligram or 0.0085 milligram per scale division.

Beam Rider Calibration

The balance beam rider, covering ten milligrams in 200 divisions, was calibrated at the 5 and 10-milligram marks by balancing against 5 and 10-milligram weights from a National Bureau of Standards (NBS) calibrated set. The mass of the NBS weight, corrected to the true mass, plus the change in rest point from zero gave the weight represented by the beam-rider settings. At the 5-milligram rider setting, an NBS weight of a true mass of 5.017 milligrams on the balance pan produced a change in rest point of $-1/4$ division (-0.002 mg), a net observed weight of 5.015 milligrams true mass for a 5-milligram rider setting. Similarly, at the 10-milligram rider setting, an NBS weight of 10.014 milligram true mass produced a $3\ 3/4$ -division (0.032-mg) rest-point change for a net observed weight of 10.046 milligrams. The average of these results showed a one-milligram beam-rider setting to be equivalent to 1.004 milligrams of true mass.

Equality of the Beam Arm Lengths

Accuracy of weighing with a two-pan, equal-arm balance requires that the beam arms be of equal length. The ratio of the balance beam arm lengths is calculated from the formula given by Strouts, et al: (2)

$$\frac{L}{R} = 1 + \frac{w^1 - w}{2M} \quad (A-1)$$

where:

- L represents the length of the left beam arm,
- R the length of the right beam arm,
- w the weight added to the left pan with a weight, M, to bring to equilibrium with a weight, M^1 , in the right pan, and
- w^1 the weight added to the right pan with weight M to bring to equilibrium with weight M^1 in the left pan.

The rest point of the balance was determined and a 20-gram weight placed on each of the balance pans. The rest-point deflection was determined (w), the weights were reversed, and

the deflection from zero again recorded (w^1). The conditions of test gave a zero rest point of + 7 1/4 divisions, a rest point of + 4 1/2 divisions with M in the left pan, and a rest point of + 15 1/2 divisions with M in the right pan. From this, $w = + 2 \frac{3}{4}$ divisions and $w^1 = + 8 \frac{1}{4}$ -division change in the rest point. Values for Equation A-1 were:

$$M = 20.00001 \text{ grams (NBS calibrated weight)}$$

$$w^1 - w = 8 \frac{1}{4} - 2 \frac{3}{4} \text{ divisions,}$$

$$= 5 \frac{1}{2} \times 0.0085 \text{ milligram} = 0.047 \text{ milligram.}$$

Substituting in Equation A-1:

$$\frac{L}{R} = 1 + \frac{0.000047}{2 \times 20.00001} = 1 + 0.0000011 = 1.000001.$$

From this calculated ratio, the left beam arm was longer than the right beam arm by one part per million.

Balance Reproducibility

Balance reproducibility was checked over a three-week period, before and on the days the standards were being weighed. A check weight (a stainless steel washer) of approximately 2 grams was used; a weight that was near that of the weighing pan plus the lithium sulfate in the final weighings. After determining the "zero weight" rest point of the balance, the check weight was weighed, always using the same 2-gram weight from a set of weights and placing the beam rider at the same beam mark. Observed weights were corrected for the change in rest point from the zero weight position. Statistical analysis of 26 repetitive weighings of the check weight showed no difference between days, and a limit of error at the 95% confidence level for a single weighing (zero plus the check-weight readings) of ± 0.0025 milligram. Results are summarized in Table A-1.

Table A-1
BALANCE REPRODUCIBILITY
(Corrected Repetitive Weighings of
Stainless Steel Object, Grams)

2.036387	2.036364	2.036387
2.036387	2.036398	2.036392
2.036383	2.036394	2.036385
2.036379	2.036411	2.036379
2.036366	2.036385	2.036366
2.036368	2.036366	2.036396
2.036374	2.036362	2.036379
2.036396	2.036379	2.036374
2.036379	2.036370	2.03638 (mean)

NOTE: The same two-gram, 20-milligram, and 10-milligram weights and balance-beam rider setting of 6.400 milligrams were used for all weighings. Weights were corrected for rest-point change from the balance zero-weight rest point.

APPENDIX B

WEIGHING TECHNIQUES

Obtaining the Balance Rest Point

A common method of swings was used to determine the balance rest point. While freely swinging, the scale readings were noted for the extremes of travel of the light pointer. Three positive swings and their two interspersed negative swings were so observed. The means of the three positive swings and the two negative swings were calculated separately. The rest point was the mean of the means of the positive and negative swings. For example, three positive readings of 12, 11 1/2, and 11 divisions and the corresponding two negative readings of 3 and 2 divisions gave a rest point of $(11 \frac{1}{2} - 2 \frac{1}{2}) \div 2$ or $+ 4 \frac{1}{2}$ divisions.

Calculating the Amounts of Lithium Sulfates Needed

An estimate of the amounts of each isotopic lithium sulfate to weigh for each mixture was calculated by assuming 100% isotopic purity for the lithium-6 and lithium-7 sulfates. Calculations were based on the molecular weights derived from the summation of the atomic weights. A table of isotopes compiled by R. L. Heath⁽³⁾ gave the atomic weights of the lithium-6 as 6.01512, lithium-7 as 7.01600, normal sulfur as 32.064, and oxygen as 15.9994. The molecular weight of lithium-6 sulfate, based on these atomic weights, was 108.09184; the weight for lithium-7 sulfate was 110.0936. For the 97 mol % lithium-6 sulfate the calculation was:

$$0.97 \times 108.092 = 104.849$$

$$0.03 \times 110.094 = \underline{3.303}$$

$$\text{Sum} = 108.152$$

$$\text{Weight fraction lithium-6 sulfate} = \frac{104.849}{108.152} = 0.9695.$$

$$\text{Weight fraction lithium-7 sulfate} = \frac{3.303}{108.152} = 0.0305.$$

Thus, for one gram of 97 mol or at % lithium-6 mixture, the approximate weights needed were 969.5 milligrams lithium-6 sulfate and 30.5 milligrams lithium-7 sulfate.

APPENDIX C

CALCULATIONS OF WEIGHT CORRECTIONS

For accurate weighings, corrections are made for the buoyancy or lifting effects of the air on the weights and the material being weighed. This correction gives a close estimate of the true mass of the material weighed.

The apparent mass versus brass values of the weights from the NBS calibrated set were corrected to true mass values using the NBS suggested factors. For the tantalum weights, the correction was -0.071 milligram per gram; for the aluminum weights, the correction was + 0.302 milligram per gram. Fractional weights in the set were tantalum for the 50 through 500-milligram sizes and aluminum for the 10 and 20-milligram weights.

Buoyancy corrections for the weights in air of lithium sulfate to true mass were made by the formula:

$$M = m + md_a \left(\frac{1}{d_m} - \frac{1}{d_w} \right), \quad (C-1)$$

where:

M represents the mass of material in vacuum, or true mass;

m the apparent mass of material weighed in air;

d_a the density of air at the conditions of weighing;

d_m the density of the material weighed, and

d_w the density of the weights used.

The average density of air under the weighing conditions of 726 to 742 millimeters of mercury pressure, 6 to 10% relative humidity, and a near-constant 25°C was taken at 0.00114 g/cm³. For the aluminum weights, the density was 2.7 g/cm³; for tantalum, 16.6 g/cm³. The handbook density for lithium sulfate was 2.221 g/cm³. Inserting these values into Equation C-1, the buoyancy correction factor obtained for the aluminum weights was:

$$d_a \left(\frac{1}{d_m} - \frac{1}{d_w} \right) = 0.00114 \left(\frac{1}{2.221} - \frac{1}{2.7} \right) = 91 \mu\text{g/g of the Al weights};$$

for the tantalum weights:

$$0.00114 \left(\frac{1}{2.221} - \frac{1}{16.6} \right) = 445 \mu\text{g/g of the Ta weights}.$$

Corrections for the beam rider were made by multiplying the calibrated value for the beam rider (1.004 mg/mg beam rider setting) by the beam-rider change from tare to gross weight.

The weight equivalent of a beam-rider change was added to or subtracted from the lithium sulfate weight, as indicated by the direction of change.

Changes in the rest point from the weighing scoop tare to the sample gross tare were multiplied by the factor of 0.0085 milligram per division to obtain the weight equivalent. This weight was added to the sample weight, following the sign of the rest-point change whether negative or positive.

Total corrected weight of the lithium sulfate was the sum of the apparent mass of the NBS fractional weights used + the true mass corrections for the NBS weights + the buoyancy correction for lithium sulfate + the beam-rider change weight equivalent + the rest-point change weight equivalent. Due regard was taken for the sign of each weight component in the summation. An example of weight corrections and summations for the 66.5 at % lithium-6 mix is given in Table C-1.

Table C-1
EXAMPLE OF WEIGHT CORRECTIONS FOR MIX WITH 66.5 ATOM PERCENT LITHIUM-6
(All Values in mg)

Material Weighed	Lithium-6 Sulfate	Lithium-7 Sulfate
NBS Weights Used		
Ta Weights	500 + 100 + 50	200 + 100
Al Weights	10	20 + 10
Apparent Mass of Weights Used	660.022	330.003
True Mass Corrections for Weights		
Ta Weights	- 0.046	- 0.021
Al Weights	0.003	0.009
Buoyancy Correction for Lithium Sulfate		
Ta Weights	0.289	0.134
Al Weights	0.001	0.003
Change in Beam Rider Setting	1.100	9.050
Weight Equivalent	1.104	9.086
Rest Point Change (gross-tare) Divisions	+ 1/2	+ 2 1/4
Weight Equivalent	0.004	- 0.019
Total Corrected Weight of Lithium Sulfate	661.377	339.195

APPENDIX D

CALCULATION OF THE ATOM PERCENT LITHIUM-6 IN THE FINAL MIXES

Account must be taken of the isotopic content of each of the lithium sulfate starting materials in order to compute the exact amount of each isotope present. Average molecular weight of the lithium-6 sulfate starting material was calculated from the known analyzed isotopic fraction and molecular weights of the lithium-6 and lithium-7 sulfates (see Calculation of the Amounts of Lithium Sulfates Needed, Appendix B).

$$0.99999292 \times 108.09184 = 108.091075$$

$$0.00000708 \times 110.0936 = \underline{0.000779}$$

$$\text{Average Molecular Weight of Lithium-6 Sulfate Starting Material} = 108.091854$$

Similarly, for the lithium-7 sulfate starting material with an isotopic content of 0.9999868 atom fraction lithium-7, the average molecular weight was 110.093574.

From these calculations, the mole fraction lithium-6 sulfate in the mixes was calculated using the formula:

$$f_6 = \frac{wm_6 + WM_6}{wm_6 + WM_6 + wm_7 + WM_7} \quad (D-1)$$

where:

f_6 represents the mole fraction ${}^6\text{Li}_2\text{SO}_4$ in the mix,

w milligrams (corrected) of lithium-6 sulfate material weighed,

W milligrams (corrected) of lithium-7 sulfate material weighed,

m_6 millimoles of ${}^6\text{Li}_2\text{SO}_4$ per milligram of lithium-6 sulfate starting material,

m_7 millimoles of ${}^7\text{Li}_2\text{SO}_4$ per milligram of lithium-6 sulfate starting material,

M_6 millimoles of ${}^6\text{Li}_2\text{SO}_4$ per milligram of lithium-7 sulfate starting material,

M_7 millimoles of ${}^7\text{Li}_2\text{SO}_4$ per milligram of lithium-7 sulfate starting material.

The values calculated for the constants for the starting materials in Equation D-1 were:

$$m_6 = \frac{1 \times 0.99999292}{108.091854} = 0.0092513255 ,$$

$$m_7 = \frac{1 \times 0.00000708}{108.091854} = 0.0000000655 ,$$

$$M_6 = \frac{1 \times 0.0000132}{110.093574} = 0.0000001199, \text{ and}$$

$$M_7 = \frac{1 \times 0.9999368}{110.093574} = 0.0090830624.$$

A greater-than-normal number of significant figures was carried for intermediate calculations; otherwise, the end result was adversely affected.

Purity factors for the two lithium sulfate starting materials are comparable. This purity factor would appear with the weight in both numerator and denominator of the mole fraction equation and would thus cancel out. Since the molecular form of the lithium-6 and lithium-7 material being mixed is the same, the mole fraction or mole percent is equivalent to the atom fraction or atom percent lithium-6 of the total lithium present.

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