Analytical Results for Prepared UIUC F⁷LiNaK

Chemical Fuel Cycle Technologies Division

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1 INTRODUCTION

Source materials including 275 g of ${}^7\text{LiF-NaF-KF}$ (46.5-11.5-42.0 mol %) and 10 g each of several reagent salts were prepared at Argonne for shipment to the University of Illinois at Urbana Champaign. The F^7LiNaK was packaged in a single jar and reagent salts were placed in small vials. The mass of each shipped salt is listed in Table 1. This report contains details of the preparation procedure and results of elemental composition measurements using inductively coupled plasma-mass spectrometry (ICP-MS) and inductively coupled plasma-optical emission spectrometry (ICP-OES) and phase analyses using X-ray diffraction.

Table 1: Masses of F^7LiNaK , LiF , NaF , KF , and CeF_3 shipped

Salt	Mass (g)
F^7LiNaK	275.7
${}^7\text{LiF}$	10.02
NaF	10.03
KF	10.08
CeF_3	10.19

2 Reagent Preparation

All activities related to salt preparation and packaging were performed in an argon atmosphere glovebox (<5 ppm O_2 and <1 ppm H_2O). The reagents were placed in graphite crucibles and heated at 300°C for three hours and then at 700°C for three hours to remove residual water prior to mixing. The ${}^7\text{LiF}$ and NaF were baked as single batches and the KF was baked in two batches (KF-1 and KF-2). The weight losses of the reagents were measured after six hours of total baking time and are listed in Table 2. Certificates of analysis for the reagents were obtained from the manufacturers and are attached in the appendix.

Table 2: Percent weight loss of reagents measured after drying

Salt	Percent Loss by Weight
${}^7\text{LiF}$	0.29%
NaF	0.27%
KF-1	0.36%
KF-2	0.49%

A single batch of F^7LiNaK was prepared by combining appropriate masses of the dried reagent salts to achieve a composition of 46.7-11.3-42.0 mol % LiF-NaF-KF in a large graphite crucible. The target and batched masses of dried reagents are listed in Table 3. The added amounts of each reagent were weighed using a balance accurate to 10 mg. The batch was mechanically mixed and then melted at approximately 700 °C for 6 hours. The crucible was left in the furnace to slowly cool. The salt ingot was then recovered from the inverted crucible in one solid piece. The ingot was broken up into chunks using a chisel and then crushed by using a stainless-steel mortar and pestle. The recovered salt was weighed and placed in a Mason jar, which was then sealed and stored in the glovebox. Weighed amounts of dried reagent powders and placed in plastic vials. All salt containers were vacuum sealed in plastic prior to shipping.

Table 3: Target and batched masses of reagents used in $FLiNaK$ production

	7LiF	NaF	KF	Total
Target Mass (g)	84.714	33.913	171.373	289.99
Batched Mass (g)	84.70	33.92	170.90	289.52
Target Composition (mol %)	46.5	11.5	42.0	100
Batched Composition (mol %)	46.55	11.52	41.93	100

3 Elemental Composition Analyses

Approximately 5 g of the retained F^7LiNaK was used in analyses performed at Argonne. The elemental composition of the mixture was measured by dissolving three samples (179.5 mg, 184.9 mg, and 156.2 mg) in high-purity acid. The concentrations of the major cations were measured by using ICP-OES and the concentrations of impurities were measured by using ICP-MS. The ICP-OES results and salt compositions are given in Table 4, where the fluoride contents were calculated by stoichiometry. Values measured using the ICP-OES technique are typically accurate to $\pm 5\%$. The measured compositions are consistent across the three samples and with the as-batched and target compositions within the measurement uncertainty.

Table 4: Major Cation Analyses by ICP-OES and Calculated Salt Composition

	Concentration, wt %			Composition, mol %		
	Li	Na	K	LiF	NaF	KF
Sample 1	7.27	6.18	38.7	45.4	11.7	42.9
Sample 2	7.38	6.19	37.6	46.3	11.7	41.9
Sample 3	7.46	6.27	37.8	46.4	11.8	41.8
Average	7.37	6.21	38.0	46.1	11.7	42.2
1 s	0.01	0.05	0.6	0.6	0.1	0.6

Aliquots of the solutions used for ICP-OES analyses were further diluted and used to measure the amounts of trace contaminants in each F^7LiNaK sample by using ICP-MS. These results are shown in Table 5. Values measured using the ICP-MS technique are typically accurate to $\pm 10\%$. The concentrations of impurities measured in the salt were all less than 100 ppm, which is consistent with certified reagent purities.

Table 5: Results of Trace Element Analyses by using ICP-MS (ppm)

	Be	Mg	Ca	Cr	Mn	Fe	Ni	Rb	Cs
Sample 1	< 0.05	< 0.33	28	1.23	0.82	< 14	0.42	62	< 0.02
Sample 2	< 0.05	< 0.32	25	0.41	0.66	< 14	0.23	55	< 0.01
Sample 3	< 0.06	< 0.38	28	0.44	0.55	< 16	0.28	56	< 0.01
Average	BDL	BDL	27	0.7	0.7	BDL	0.3	57	BDL
1 s	—	—	2	0.5	0.1	—	0.1	4	—

< indicates the concentration was below the reported detection limit.

4 X-ray Diffraction Analyses

Three portions of the salt (40 mg, 140 mg and 170 mg) were fixed to glass slides using double-sided tape and then covered with Kapton film for X-ray diffraction analysis. Figure 1 shows a comparison of the measured patterns to the combined pattern for LiF, NaF, and KF peaks. The locations of major peaks in the XRD patterns are generally consistent with the combined salt peaks. However, some discrepancies in peak locations are observed. Calibration was confirmed using a NIST silica reference material prior to sample measurements. The patterns were also compared with those of possible oxides and other contaminants (e.g., Li₂O, Na₂O, K₂O, RbF, Rb₂O, Fe₂O₃) to verify that the salt samples were not contaminated with water during production, sample preparation, or analyses. No contaminant peaks were observed in the XRD pattern. This indicates contaminant concentrations are <5 wt %. This finding is consistent with the results of ICP-MS analyses.

Figure 1: X-ray Diffraction Patterns for F⁷LiNaK Salt

5 Summary

Materials including 275 g of ${}^7\text{LiF-NaF-KF}$ (46.5-11.5-42.0 mol %) and 10 g each of several reagents were prepared at Argonne and shipped to UIUC to support experimental activities. Major cation compositions measured by using ICP-OES were consistent with the target eutectic composition and concentrations of trace elements measured by using ICP-MS were consistent with the certified purities of the reagents.

Appendix: Certificates of Analysis for Reagents

Certificate of Analysis

Product Name:

Lithium-⁷Li fluoride – 99 atom % ⁷Li, 99% (CP)

Product Number: 601497
Batch Number: MBBC0157V
Brand: ALDRICH
CAS Number: 17409-87-9
Formula: F⁷Li
Formula Weight: 26.01 g/mol
Quality Release Date: 19 SEP 2016

Test	Specification	Result
Appearance (Color)	White	White
Appearance (Form)	Powder	Powder
Water (by Karl Fischer)	≤ 0.5 %	0.2 %
IC	≥ 99.0 %	100.1 %
Enrichment Li-7	≥ 99.00	99.96

Greg Abernathy, QC Supervisor
Quality Assurance
Miamisburg, Ohio US

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Figure A.1: Certificate of Analysis for LiF Reagent

Certificate of Analysis

Product Name:

Sodium fluoride – anhydrous, powder, 99.99% trace metals basis

Product Number: 450022
Batch Number: MKCN8615
Brand: ALDRICH
CAS Number: 7681-49-4
MDL Number: MFCD00003524
Formula: FNa
Formula Weight: 41.99 g/mol
Quality Release Date: 02 FEB 2021

NaF

Test	Specification	Result
Appearance (Color)	White	White
Appearance (Form)	Powder	Powder
Gravimetric Analysis		45.9 %
% F With Lead Acetate		
ICP Major Analysis	Confirmed	Conforms
Confirms Sodium Component		
Purity	Meets Requirements	Meets Requirements
99.99% Based On Trace Metals Analysis		
Trace Metal Analysis	≤ 150.0 ppm	55.9 ppm
Boron (B)		4.0 ppm
Barium (Ba)		1.0 ppm
Calcium (Ca)		0.1 ppm
Chromium (Cr)		1.9 ppm
Cesium (Cs)		4.0 ppm
Iron (Fe)		18.3 ppm
Potassium (K)		16.4 ppm
Lithium (Li)		2.1 ppm
Magnesium (Mg)		0.2 ppm
Manganese (Mn)		0.3 ppm
Nickel (Ni)		1.4 ppm
Strontium (Sr)		4.9 ppm
Titanium (Ti)		0.6 ppm
Zinc (Zn)		0.3 ppm
Zirconium (Zr)		0.3 ppm

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Certificate of Analysis

Product Number: 450022
Batch Number: MKCN8615

Michael Grady, Manager
Quality Control
Milwaukee, WI US

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Version Number: 1

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Figure A.2: Certificate of Analysis for NaF Reagent

Certificate of Analysis

1 Reagent Lane

Fair Lawn, NJ 07410

201.796.7100 tel

201.796.1329 fax

Thermo Fisher Scientific's Quality System has been found to conform to Quality Management System
 Standard ISO9001:2015 by SAI Global Certificate Number CERT – 0120632

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Catalog Number	20135	Quality Test / Release Date	03/02/2020
Lot Number	B0149551		
Description	POTASSIUM FLUORIDE, ANHYDROUS		
Country of Origin	China	Suggested Retest Date	Mar/2025

N/A			
Result Name	Units	Specifications	Test Value
APPEARANCE		REPORT	WHITE POWDER, NOT CRYSTALS
ASSAY	%	>= 98.5	99.68
CHLORIDE	ppm	<= 500	<500
IDENTIFICATION	PASS/FAIL	= PASS TEST	PASS TEST
IRON (Fe)	ppm	<= 50	<50
SULFATE (SO4)	ppm	<= 500	<500

Julian Burton - Quality Control Manager – Fair Lawn

Note: The data listed is valid for all package sizes of this lot of this product, expressed as an extension of this catalog number listed above.
 If there are any questions with this certificate, please call at (800) 227-6701.
 *Based on suggested storage condition.

Figure A.3: Certificate of Analysis for KF Reagent

Product No.: 21118
Product: Cerium(III) fluoride, 99.9% (REO)
Lot No.: S03G025

CeF₃ >99.9 %

Total remaining
RE-Oxides <0.1 %

Retest date: June 3, 2025

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Figure A.4: Certificate of Analysis for CeF₃ Reagent

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