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**Characterization of Three
Fibrous Insulations for
Potential HTGR Application**

J. E. Selle

P407

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Structural Materials Program
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CHARACTERIZATION OF THREE FIBROUS INSULATIONS FOR
POTENTIAL HTGR APPLICATION

J. E. Selle

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CHARACTERIZATION OF THREE FIBROUS INSULATIONS FOR
POTENTIAL HTGR APPLICATION

J. E. Selle*

ABSTRACT

Three fibrous insulation materials manufactured by Babcock and Wilcox were characterized for possible use as HTGR core thermal insulations. These materials (Unifelt 3000 board, Unifelt 2600 board, and Kaowool blanket) were characterized by spectrographic analysis, x-ray diffractometry, scanning electron microscopy, ignition studies and bulk density measurements, resilience tests, helium permeation tests, and compressive stress-strain tests. Of these properties, resilience is the most important for determining acceptability of material for HTGR use. The arbitrary resilience acceptability criterion is 90% or greater after 20 h at 816°C (1500°F). Based on the resilience test, the Unifelt 3000 was acceptable, the Unifelt 2600 was marginal, and the Kaowool was unacceptable.

INTRODUCTION

As part of the HTGR Structural Ceramics Program, work was initiated on the characterization of fibrous insulations for potential use as reactor core thermal insulations. To avoid duplication of work at General Atomic Company, discussions were held and it was agreed that we would investigate three fibrous insulations manufactured by Babcock and Wilcox (B&W): Unifelt 3000 board, Unifelt 2600 board, and Kaowool blanket. General Atomic has investigated insulation materials produced by other vendors.^{1,2}

Unifelt 3000 board is formed from a slurry of SAFFIL (trademark of Imperial Chemical Industries, Ltd.) alumina fiber, Kaowool aluminum silicate fiber, and an organic binder. Its maximum use limit is 1650°C (3000°F). Unifelt 2600 is similar but has a higher percentage of the Kaowool aluminum silicate fiber and a maximum use limit of 1425°C

*Rockwell International, Rocky Flats Plant, P.O. Box 464, Golden, Colorado 80401.

(2600°F). Both Unifelt 3000 and Unifelt 2600 contain an organic binder that must be baked out prior to use as described later. The as-received Unifelt boards are stiff, easy to handle, and easy to cut to size. However, after ignition of the organic binder, handling is more difficult because the material tends to delaminate across the thickness of the board. It also becomes light, with lower tensile strength, and care must be exercised in handling. Kaowool, a needled blanket of aluminum silicate fibers has good handling characteristics, no organic binder, and needs no baking prior to use.

PROCEDURE

To assess their potential for HTGR application, these materials were characterized by:

1. spectrographic analysis,
2. x-ray diffractometry,
3. scanning electron microscopy (SEM),
4. ignition studies and bulk density measurements,
5. resilience tests,
6. helium permeation tests, and
7. compressive stress-strain tests.

Procedures for spectrographic analysis, x-ray diffractometry, and SEM are reasonably standard and need not be repeated here. However, to establish a reference for comparison with other materials, the procedures for the remaining tests are described.

Ignition Studies and Bulk Density Measurements

Rectangular blocks [76 mm (3 in.) long × 63–76 mm (2.5–3 in.) wide × nominal thickness ~63 mm (2.5 in.)] were used for all except the helium permeation tests. Dimensions were measured to the nearest 0.1 mm with a vernier caliper, and the weight of each sample was measured to the nearest 0.01 g.

The blocks were placed in a preheated pot-type furnace with an air atmosphere and held for a predetermined time at 400 or 500°C (752 or

932°F). After blocks were removed from the furnace, the dimensions and weight were again recorded.

Resilience Tests

Specimens for these tests were 76 mm (3 in.) long $\times$ 63 mm (2.5 in.) wide $\times$ nominal thickness. The thickness was measured to the nearest 0.1 mm while under a stress of 167 Pa (3.5 lb/ft²), which was obtained by placing a 76 $\times$ 63 $\times$ 6.35 mm (3 $\times$ 2.5 $\times$ 0.25 in.) aluminum plate on top of the specimen. The specimens (without the aluminum plate) were then placed in stainless steel holders and compressed to 75% of the thickness as measured above, heated for 20 h at 816°C (1500°F) in helium, and cooled in helium. After the specimens were released from the holders, the thickness was again measured to the nearest 0.1 mm while under the 167-Pa stress.

Eight specimens of each material were tested at 816°C (1500°F), and two specimens of Unifelt 3000 and two of Unifelt 2600 were tested at 982°C (1800°F).

Helium Permeation

The apparatus for measuring the helium permeation rate is shown in Fig. 1. The doughnut-shaped specimen is 165 mm (6.5 in.) in diameter with a 51-mm (2-in.) hole in the center. The specimen holder is adjustable so that any desired compressed density can be obtained. Helium flowed into the center hole and escaped radially through the side of the specimen. The helium flow rate was measured as a function of pressure difference at each compressed density.

Compressive Stress-Strain

Specimens measuring approximately 76 $\times$ 76 mm (3 $\times$ 3 in.) or 63.5 $\times$ 76 mm (2.5 $\times$ 3 in.) were placed in an Instron machine and loaded in compression. A 0.2-lb load was applied, the crosshead was adjusted to zero, and the thickness of the specimen was measured. The predetermined load was applied and held for 300 s, and the crosshead movement and the

final thickness were measured. Tests were run sequentially from lower load to higher load on the same specimen. Between tests the load was

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Fig. 1. Schematic of Helium Permeation Test Apparatus.

removed for 300 s to allow the specimen to recover from the prior deformation before applying the new load.

RESULTS

Spectrographic Analysis

Results from the spectrographic analyses are given in Table 1. Silicon, aluminum, and oxygen are the primary constituents and are not included in these analyses. Nominal compositions for each material are given in Table 2.

X-Ray Diffractometry

X-ray diffractometry results and d -spacings for the various peaks are given in Appendix A. Unifelt 3000 contained a small amount of glassy phase, and crystalline phases included α - Al_2O_3 (corundum), δ - Al_2O_3 , Al_2SiO_5 , $\text{Na}_6\text{Al}_4\text{Si}_4\text{O}_{17}$, and MoSi_2 . A few other peaks from undetermined phases were

Table 1. Spectrographic Analysis of Impurities
in Fibrous Insulations

Impurity	Unifelt 3000 (ppm)	Unifelt 2600 (ppm)	Kaowool (ppm)
Ag	<0.005	<0.005	<0.005
B	<0.01	<0.01	<0.01
Ba	0.05	0.1	0.08
Be	<0.005	<0.005	<0.005
Bi	<0.02	<0.02	<0.02
Ca	0.07	0.1	0.09
Cd	<0.05	<0.05	<0.05
Co	<0.02	<0.02	<0.02
Cr	0.05	0.1	0.02
Cs	<0.1	<0.1	<0.1
Cu	<0.01	0.1	<0.01
Fe	0.3	0.7	0.5
Hg	<0.05	<0.05	<0.05
In	<0.05	<0.05	<0.05
K	<0.02	0.5	<0.02
Li	0.005	0.01	0.007
Mg	0.17	0.2	0.15
Mn	0.01	0.01	0.01
Mo	<0.01	<0.01	<0.01
Na	0.05	0.3	0.1
Nb	<0.05	<0.05	<0.05
Ni	<0.02	0.02	<0.02
Pb	<0.02	<0.02	<0.02
Rb	<0.02	<0.02	<0.02
Sn	<0.02	<0.02	<0.02
Sr	<0.02	0.05	0.05
Ta	<0.05	<0.05	<0.05
Ti	0.6	1	1
V	0.02	0.02	0.02
W	<0.05	<0.05	<0.05
Zn	<0.05	<0.05	<0.05
Zr	0.02	0.02	0.02

Table 2. Nominal Compositions^a (%) of Fibrous Insulations

	Unifelt 3000 Board	Unifelt 2600 Board	Kaowool
Al ₂ O ₃	71.7	55.7	45
SiO ₂	22.8	38.7	52
Inorganics	0.3	0.5	~3.3 ^b
Weight loss on ignition	5.5	5.5	

^aSupplied by Babcock and Wilcox.

^b1.3% Fe₂O₃, 1.7% TiO₂, 0.2% Na₂O, 0.1% CaO, 0.08% B₂O₃, trace MgO.

also observed. Unifelt 2600 contained a considerable amount of glassy phase and crystalline phases included α -Al₂O₃ (corundum), δ -Al₂O₃, SiO₂, Al₂SiO₅, Na₆Al₄Si₄O₁₇, and LiOH. Other peaks were observed but could not be identified. Kaowool was predominantly glassy with a few very minor peaks. No crystalline phases could be identified with certainty.

Scanning Electron Microscopy

Scanning electron microscopy was used to examine the structure of these fibrous insulations in the as-received condition and after ignition of the organic binder. There was some indication of the organic binder in the as-received material but not in the ignited material. A small amount of flaky material was seen in the Unifelt 2600 and Unifelt 3000, but differences in composition between these particulates and the fibers could not be detected by the nondispersive detector.

Measurements of the fiber diameters taken from the photomicrographs are summarized in Table 3. The Unifelt 3000 structure was fairly uniform, and the fibers were randomly oriented. Fiber diameters ranged from 3.6 to

Table 3. Summary of Fiber Size Distribution
for Fibrous Insulations

Material	Number of Fibers Measured	Fiber Diameter (μm)		
		Range	Mean	Standard Deviation
Unifelt 3000	21	3.6-14.1	6.2	2.24
Unifelt 2600	24	2.3-9.6	5.0	1.94
Kaowool	11	1.4-7.5	4.6	1.94

14.1 μm , with very few fibers in the upper end of this range. Similar results were found with the Unifelt 2600, where fiber diameters varied from 2.3 to 9.6 μm . Kaowool fiber diameters ranged from 1.4 to 7.5 μm . The fibers were much less randomly oriented, and the material had a higher number of flaky particulates.

Ignition Tests and Density Measurements

The organic binder used in the manufacture of the Unifelt boards burns off after heating for several hours at 400°C, resulting in a weight loss and subsequent density decrease. Typical results are summarized in Table 4. Stability in the lateral dimensions was good, with no measurable changes observed. However, expansion across the thickness occurred, further decreasing the density.

Ignition temperatures of 400 or 500°C produced little difference in final density or weight loss, although the specimens ignited at 400°C were often off-white to gray, suggesting incomplete ignition. Because of this, all subsequent specimens were ignited at 500°C.

Table 4. Results from Ignition Tests and Density Measurements

Ignition Temperature (°C)	Density [Mg/m ³ (lb/ft ³)]		Weight Loss on Ignition (%)
	Initial	Final	
<u>Unifelt 3000</u>			
400	0.119 (7.4)	0.085 (5.3)	6.4
500	0.119 (7.4)	0.088 (5.5)	6.3
<u>Unifelt 2600</u>			
400	0.146 (9.1)	0.112 (7.0)	7.3
500	0.147 (9.2)	0.122 (7.6)	7.2
<u>Kaowool</u>			
500	0.128 (8.0)	0.127 (7.9)	0.2

Resilience Tests

Data obtained from the resilience tests are summarized in Table 5. According to the arbitrary criterion of 90% resilience or greater after 20 h at 816°C (1500°F) that has been established for acceptance, Unifelt 3000 is acceptable, Unifelt 2600 is a marginal, and the Kaowool is definitely unacceptable.

Table 5. Resilience Test Data for Fibrous Insulations

Material	Resilience, %			Specimens
	Mean	Range	Standard Deviation	
Unifelt 3000 ^a	97.3	95.5–100.4	1.92	8
Unifelt 2600 ^a	88.3	85.7–88.8	1.19	8
Kaowool ^a	76.2	75.6–77.2	0.45	8
Unifelt 3000 ^b	87.1	85.4–88.8	2.40	2
Unifelt 2600 ^b	76.5	75.6–77.4	1.27	2

^aAfter 20 h at 816°C (1500°F).

^bAfter 20 h at 982°C (1800°F).

Helium Permeation

Data obtained from the helium permeation tests are summarized in Table 6 and Fig. 2. In Table 6, the flow rate at 6.895-kPa (1-psi) pressure is given at various compressed densities. This pressure is the

Table 6. Helium Permeation as a Function of Compressed Density

Compressed Density (Mg/m ³) (lb/ft ³)	Helium Permeation ^a [10 ⁻⁴ m ³ /s (scfm)]		
	Unifelt 3000	Unifelt 2600	Kaowool
0.128 (8.0)	5.95 (1.26)		
0.144 (9.0)	5.66 (1.20)	6.09 (1.29)	6.28 (1.33)
0.160 (10.0)	5.43 (1.15)	5.80 (1.23)	6.11 (1.30)
0.176 (11.0)	5.14 (1.09)	5.50 (1.16)	5.92 (1.26)
0.192 (12.0)	4.86 (1.03)	5.31 (1.12)	5.78 (1.22)

^aAt 6.895 kPa (1 psi).

Fig. 2. Helium Permeation at 6.895 kPa (1 psi) as a Function of Compressed Density.

net pressure after the line losses are subtracted from the total recorded pressure. Line losses were determined by measuring the pressure difference Δp (~ 100 mm H₂O) at a flow rate of about 4.7×10^{-4} m³/s (1 scfm). A pressure of 100 mm H₂O equals 0.986 kPa (0.143 psi). Therefore, plots of the flow rate as a function of Δp were extrapolated to 7.881 kPa (1.143 psi) to obtain the flow rate at a net Δp of 6.895 kPa (1 psi).

Compressive Stress-Strain

Results from the compressive stress-strain measurements are summarized in Tables 7 to 9 and Figs. 3 to 5. Figure 3 shows similar results with Unifelt 3000 on samples run before and after the resilience tests. Some unaccountable scatter exists in the data. Table 7 indicates some permanent deformation of the material which may account for some of the deviations, but the problem has not been resolved.

Table 7. Compressive Properties of Ignited^a
Unifelt 3000 Fibrous Insulation Board

Starting Thickness [mm (in.)]	Amount of Compression [mm (in.)]	Compressive Stress [kPa (psi)]	Compression (%)
<u>As Ignited</u>			
84.1 (3.31)	41.3 (1.62)	6.06 (0.88)	49.0
69.8 (2.75)	30.2 (1.19)	9.03 (1.31)	43.3
70.6 (2.78)	33.8 (1.33)	11.67 (1.69)	47.8
59.5 (2.34)	28.9 (1.14)	14.41 (2.09)	48.6
<u>Resilience Tested at 816°C (1500°F)</u>			
76.2 (3.00)	32.5 (1.28)	6.89 (1.00)	42.5
69.8 (2.75)	33.0 (1.30)	10.20 (1.48)	47.2
68.1 (2.68)	33.0 (1.30)	13.30 (1.93)	48.5
57.9 (2.28)	24.9 (0.98)	16.48 (2.39)	43.0
<u>Resilience Tested at 982°C (1800°F)</u>			
69.8 (2.75)	29.0 (1.14)	7.38 (1.07)	41.4
70.1 (2.76)	34.0 (1.34)	10.89 (1.58)	48.5
70.6 (2.78)	36.3 (1.43)	14.62 (2.12)	51.5
70.1 (2.76)	48.8 (1.92)	18.20 (2.64)	69.6

^aIgnited 4 h at 500°C.

Table 8. Compressive Properties of Ignited^a
Unifelt 2600 Fibrous Insulation Board

Starting Thickness [mm (in.)]	Amount of Compression [mm (in.)]	Compressive Stress [kPa (psi)]	Compression (%)
<u>As Ignited</u>			
63.5 (2.50)	14.7 (0.58)	6.14 (0.89)	23.4
59.2 (2.33)	19.3 (0.76)	9.17 (1.33)	32.4
65.8 (2.59)	23.4 (0.92)	12.13 (1.76)	35.4
66.5 (2.62)	26.4 (1.04)	16.13 (2.34)	39.4
<u>Resilience Tested at 816°C (1500°F)</u>			
66.8 (2.63)	22.4 (0.88)	6.76 (0.98)	33.5
69.3 (2.73)	28.2 (1.11)	10.20 (1.48)	42.1
67.8 (2.67)	29.2 (1.15)	13.37 (1.94)	42.0
68.1 (2.68)	30.2 (1.19)	16.54 (2.40)	44.5
<u>Resilience Tested at 982°C (1800°F)</u>			
54.6 (2.15)	11.4 (0.45)	7.03 (1.02)	21.0
55.1 (2.17)	15.0 (0.59)	10.48 (1.52)	27.2
58.9 (2.32)	21.3 (0.84)	13.86 (2.01)	36.2
58.7 (2.31)	23.1 (0.91)	17.37 (2.52)	39.4

^aIgnited 4 h at 500°C.

Table 9. Compressive Properties of Ignited^a
Kaowool Fibrous Insulation Blanket

Starting Thickness [mm (in.)]	Amount of Compression [mm (in.)]	Compressive Stress [kPa (psi)]	Compression (%)
<u>As Ignited</u>			
63.2 (2.49)	5.1 (0.20)	5.93 (0.86)	8.0
62.5 (2.46)	14.2 (0.56)	8.96 (1.30)	22.8
62.5 (2.46)	16.2 (0.64)	11.58 (1.68)	26.0
62.5 (2.46)	10.7 (0.42)	14.55 (2.11)	17.1
<u>Resilience Tested at 816°C (1500°F)</u>			
46.7 (1.84)	4.3 (0.17)	7.51 (1.09)	7.6
46.5 (1.83)	4.8 (0.19)	11.31 (1.64)	10.4
46.5 (1.83)	5.8 (0.23)	14.48 (2.10)	12.5
46.7 (1.84)	6.8 (0.27)	17.79 (2.58)	14.7

^aIgnited 4 h at 500°C.

Fig. 3. Compressive Properties of Unifelt 3000.

Fig. 4. Compressive Properties of Unifelt 2600.

Fig. 5. Compressive Properties of Kaowool.

The data for Unifelt 2600 are fairly consistent and indicate that after the 816°C resilience test the material deforms more under a given load than the ignited material, while after the 982°C resilience test the material deforms less.

Kaowool tested after ignition at 500°C gave rather erratic results, but after resilience testing very consistent data were obtained.

Data taken from the figures at 10.34 kPa (1.5 psi) are summarized in Table 10, which gives the amount of compression obtained at 10.34 kPa (1.5 psi) for the various materials and conditions. This table supports the observations made above.

Table 10. Compression Obtained at 10.34 kPa (1.5 psi) on Fibrous Insulations

Condition	Compression, %		
	Unifelt 3000	Unifelt 2600	Kaowool
Ignited at 500°C	46.5	33.0	24.5
Ignited at 500°C plus 816°C re- silience test	46.5	39.5	9.5
Ignited at 500°C plus 982°C re- silience test	46.5	28.0	

CONCLUSIONS

Three B&W fibrous insulations (Unifelt 3000 board, Unifelt 2600 board, and Kaowool blanket) have been characterized for consideration as thermal insulation for potential HTGR application.

Based on the resilience test, the Unifelt 3000 is acceptable, the Unifelt 2600 is marginal, and the Kaowool is unacceptable. Since the Unifelt 2600 is close to the acceptable criterion for resilience (88.3 vs 90%), it may be useful, particularly in view of the fact that its other properties are similar to those for Unifelt 3000. It is a little easier to handle, has less tendency for delamination, and costs much less (\$13 vs \$31/ft²). Since maintenance of acceptable resilience is critical for fibrous insulations in HTGR application, aging effects on this characteristic must be known. Aging tests are being conducted on both Unifelt insulations. Specimens for these tests were compressed in the same manner as for the 20-h resilience tests and will be aged for periods of 3, 6, 12, and 24 months at 816°C (1500°F), after which resilience measurements will be made. Results of these tests will be reported later.

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APPENDIX A

X-Ray Diffractometry Results

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Fig. A.1. Results of X-Ray Diffractometry Tests on Unifelt 3000. Points represent average of ten measurement.

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Fig. A.2. Results of X-Ray Diffractometry Tests on Unifelt 2600. Points represent average of ten measurement.

ORNL-DWG 80-12145

Fig. A.3. Results of X-Ray Diffractometry Tests on Koawool. Points represent average of 20 measurement.

Table A.1. d -Spacings for Peaks in Fig. A.1 — Urifelt 3000

2 θ	d Spacing (Å)	Intensity (Counts/s)	Al ₂ SiO ₅		Na ₆ Al ₄ Si ₄ O ₁₇		δ -Al ₂ O ₃		α -Al ₂ O ₃ Corundum		MoSi ₂	
			d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1
15.96	5.5483											
16.35	5.4168		5.36	16								
16.65	5.3199	14										
16.88	5.2479											
17.61	5.0320						5.02	30				
17.70	5.0066											
19.64	4.5162	3	4.58	2			4.55	30				
19.74	4.4936											
21.02	4.2227	5			4.21	100	4.07	20				
23.80	3.7354		3.74	6							3.91	10
25.80	3.4502	80							3.479	75		
26.19	3.3997	15	3.42	100								
26.48	3.3631	20	3.37	65								
27.95	3.1895		3.20	2								
30.43	2.9350										2.94	20
30.78	2.9024		2.896	12								
31.20	2.8643		2.853	2			2.87	40				
31.38	2.848	5										
31.54	2.8341	10										
31.70	2.8202											
31.83	2.8090	10										
32.47	2.7551											
33.03	2.7096	50					2.73	80				
33.44	2.6773	5	2.679	30								
34.31	2.6114											
34.81	2.5750	5			2.58	95	2.58	30				
35.38	2.5348	90	2.541	40					2.552	90		
36.70	2.4466											
36.91	2.4332	30	2.421	14			2.43	60				
37.42	2.4012											
38.01	2.3653	45							2.379	40		
38.74	2.3224		2.320	2								
39.18	2.2973	20										
39.58	2.2750		2.285	6			2.28	40				
39.76	2.2651											

Table A.1. (Continued)

2θ	d Spacing (Å)	Intensity (Counts/s)	Al ₂ SiO ₅		Na ₆ Al ₄ Si ₄ O ₁₇		δ-Al ₂ O ₃		α-Al ₂ O ₃ Corundum		MoSi ₂	
			d	I/I ₁	d	I/I ₁	d	I/I ₁	d	I/I ₁	d	I/I ₁
40.20	2.2413	20									2.24	50
40.63	2.2186											
40.88	2.2056		2.204	60								
41.13	2.1928	15							2.165	<1		
41.66	2.1661											
41.95	2.1518											
42.29	2.1353											
42.87	2.1077	5	2.111	20	2.103	8						
43.59	2.0746	140							2.085	100		
44.42	2.0377											
45.01	2.0123	15									2.01	60
45.63	1.9864	12					1.99	80				
45.84	1.9778											
46.30	1.9592								1.964	2		
46.53	1.9501						1.95	30				
46.75	1.9414	5									1.94	20
46.92	1.9348											
47.13	1.9267											
47.55	1.9106						1.91	20				
47.94	1.8960	15										
48.25	1.8845		1.867	6								
49.92	1.8253		1.830	2								
50.06	1.8205				1.821	16						
50.21	1.8154		1.817	2								
50.63	1.8014						1.80	20				
50.99	1.7895	12	1.786	4								
51.86	1.7615											
51.99	1.7574											
52.81	1.7320	57							1.740	45		
54.25	1.6894		1.682	12	1.669	4						
57.75	1.5951	120	1.597	8					1.601	80	1.59	25
58.84	1.5681		1.570	2								
59.19	1.5596		1.563	2								
60.11	1.5380	12					1.54	40	1.546	4		

Table A.1. (Continued)

2 θ	d Spacing (Å)	Intensity (Counts/s)	Al ₂ SiO ₅		Na ₆ Al ₄ Si ₄ O ₁₇		δ -Al ₂ O ₃		γ -Al ₂ O ₃ Corundum		MoSi ₂	
			d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1
60.57	1.5274											
60.69	1.5246											
60.88	1.5203											
61.00	1.5176	7	1.519	2								
61.19	1.5134						1.51	30	1.514	6		
61.57	1.5049	10							1.510	8		
62.16	1.4921						1.49	40				
62.57	1.4833	5			1.486	30						
62.70	1.4805	5										
62.88	1.4767											
63.02	1.4738											
63.16	1.4708											
63.33	1.4673											
63.74	1.4588										1.46	20
64.10	1.4515	10					1.45	30				
64.27	1.4481											
64.83	1.4369											
65.06	1.4324											
65.20	1.4297											
65.40	1.4258											
65.81	1.4179	4										
66.00	1.4142											
66.22	1.4101											
66.77	1.3998	50			1.404	4	1.40	60	1.404	30		
67.35	1.3891						1.39	100			1.39	25
67.61	1.3844	5										
67.69	1.3830	35										
67.79	1.3812	5										
68.45	1.3695	40							1.374	50		
69.07	1.3587											
69.26	1.3554											
70.01	1.3427								1.337	2		

Table A.1. (Continued)

2 θ	d Spacing (Å)	Intensity (Counts/s)	Al_2SiO_5		$\text{Na}_6\text{Al}_4\text{Si}_4\text{O}_{17}$		$\delta\text{-Al}_2\text{O}_3$		$\alpha\text{-Al}_2\text{O}_3$ Corundum		MoSi_2	
			d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1
73.10	1.2934										1.30	15
73.33	1.2899						1.29	20				
73.48	1.2877				1.287	10						
73.85	1.2821											
73.92	1.2811											
74.22	1.2766								1.276	4		
74.61	1.2709											
75.15	1.2631											
75.33	1.2606						1.26	10				
75.69	1.2555											
75.82	1.2536											
75.96	1.2517											
77.12	1.2357	19							1.239	16	1.24	100
77.36	1.2325	3			1.232	4			1.234	8		
77.49	1.2307	3									1.23	8
77.65	1.2286											
80.59	1.1910								1.1898	8		
80.76	1.1889											
80.97	1.1864	4										
81.11	1.1847											
81.21	1.1835											
81.38	1.1814											
82.09	1.1730											
82.75	1.1653								1.1600	<1		
83.90	1.1522				1.152	10						
84.62	1.1443	2							1.1470	6		
84.73	1.1431								1.1382	2		
86.57	1.1234								1.1255	6		
86.57	1.1234								1.1255	6		
86.70	1.1221	5							1.1246	4		

Table A.1. (Continued)

2 θ	d Spacing (Å)	Intensity (Counts/s)	Al ₂ SiO ₅		Na ₆ Al ₄ Si ₄ O ₁₇		δ -Al ₂ O ₃		α -Al ₂ O ₃ Corundum		MoSi ₂	
			d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1
86.90	1.1200										1.12	50
88.43	1.1045											
89.26	1.0964	5							1.0988	8		
89.37	1.0953											
89.49	1.0942											
89.65	1.0926											
89.72	1.0920											
91.10	1.0790								1.0831	4	1.08	13
91.37	1.0765	2							1.0781	8		
91.45	1.0758											
91.52	1.0751											
91.65	1.0740											
93.10	1.0610										1.05	30
95.54	1.0402	10							1.0426			
95.80	1.0381											
96.07	1.0359											
96.45	1.0328											
98.38	1.0177								1.0175	2		
99.52	1.0091										1.00	80
101.35	0.9956	12							0.9976	12		
101.44	0.9951											
101.61	0.9939											
101.70	0.9932											
101.83	0.9923											
102.31	0.9890								0.9819	4		
105.17	0.9698				0.975	6					0.978	25
108.25	0.9506										0.972	25
108.82	0.9472											
111.42	0.9323	2										
111.70	0.9307											
116.85	0.9041	3							0.9076	14		
118.05	0.8984								0.8991	8		
118.28	0.8973											
118.58	0.8959										0.895	25

Table A.2. *d*-Spacings for Peaks in Fig. A.2 — Unifelt 2600

2θ	<i>d</i> Spacing (Å)	Intensity (Counts/s)	Na ₆ Al ₄ Si ₄ O ₁₇		LiOH		δ-Al ₂ O ₃		Al ₂ SiO ₅		SiO ₂		α-Al ₂ O ₃	
			<i>d</i>	<i>I</i> / <i>I</i> ₁	<i>d</i>	<i>I</i> / <i>I</i> ₁	<i>d</i>	<i>I</i> / <i>I</i> ₁	<i>d</i>	<i>I</i> / <i>I</i> ₁	<i>d</i>	<i>I</i> / <i>I</i> ₁	<i>d</i>	<i>I</i> / <i>I</i> ₁
12.31	7.1840													
12.97	6.8198													
13.23	6.6864													
13.61	6.5006													
14.00	6.3203													
14.78	5.9885													
15.55	5.6936													
16.57	5.3454								5.35	70				
17.68	5.0122						5.02	30						
18.67	4.7486													
19.18	4.6235													
19.51	4.5460						4.55	30	4.56	30				
19.62	4.5208													
20.25	4.3815	3			4.37	90					4.37	80		
20.45	4.3391	3												
21.08	4.2108	5	4.21	100										
21.51	4.1276	2									4.118	100		
21.79	4.0752													
21.83	4.0678	2					4.07	20						
22.28	3.9867													
22.45	3.9569	3												
22.92	3.8768	2.5												
23.00	3.8635	3									3.862	60		
23.85	3.7277								3.73	50				
24.07	3.6941	2												
24.22	3.6116	6												
25.78	3.4528	6									3.479	75		
25.98	3.4254								3.41	90				
26.43	3.3694								3.36	100				
27.03	3.2959													

Table A.2. (Continued)

2θ	d Spacing (Å)	Intensity (Counts/s)	Na ₆ Al ₄ Si ₄ O ₁₇		LiOH		δ-Al ₂ O ₃		Al ₂ SiO ₅		SiO ₂		α-Al ₂ O ₃	
			d	I/I ₁	d	I/I ₁	d	I/I ₁	d	I/I ₁	d	I/I ₁	d	I/I ₁
27.35	3.2581													
27.85	3.2007								3.19	10				
28.35	3.1454													
29.70	3.0054										2.995	35		
30.98	2.8841						2.87	40	2.93	10				
31.85	2.8073								2.88	70				
32.27	2.7717	5												
32.78	2.7289	5			2.73	100	2.73	80						
32.93	2.7176													
33.03	2.7096	12												
33.30	2.6883								2.67	80				
34.57	2.5924													
34.81	2.5750	6	2.53	95			2.53	30					2.552	90
35.43	2.5314	6			2.53	70			2.53	90				
35.54	2.5238										2.523	25		
36.57	2.4550						2.43	60						
37.37	2.404	3							2.42	60				
37.55	2.3932	3											2.379	40
38.05	2.3629								2.37	10	2.325	20		
39.49	2.2800						2.28	40	2.28	60				
39.62	2.2728	3												
40.09	2.2472													
40.25	2.2387													
40.50	2.2254													
41.66	2.1661				2.17	40					2.152	2	2.165	1
42.71	2.1152		2.103	8							2.114	8		
43.57	2.0755	8							2.09	30	2.059	12	2.085	100
45.12	2.0077													
45.42	1.9951													
45.61	1.9873	15					1.59	80						

Table A.2. (Continued)

2 θ	d Spacing (Å)	Intensity (Counts/s)	Na ₆ Al ₄ Si ₄ O ₁₇		LiOH		δ -Al ₂ O ₃		Al ₂ SiO ₅		SiO ₂		α -Al ₂ O ₃	
			d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1
45.70	1.9836								1.98	20				
46.28	1.9600								1.96	20			1.964	2
46.51	1.9509						1.95	30						
46.93	1.9344	5									1.930	2		
47.60	1.9087						1.91	20						
48.83	1.8635								1.868	50	1.863	6		
49.15	1.8521				1.85	50								
49.45	1.8416	1.5	1.821	16					1.829	60				
50.82	1.7951						1.80	20						
51.00	1.7892													
51.08	1.7862	8			1.78	75								
52.79	1.7326	5											1.740	45
52.92	1.7287													
53.50	1.7113								1.710	8				
54.17	1.6917													
54.59	1.6797													
54.67	1.6774													
55.58	1.6521				1.65	70					1.651	4		
56.57	1.6255				1.64	70								
56.85	1.6182										1.619	6		
57.66	1.5973	7											1.601	80
57.76	1.5948													
57.92	1.5908	7												
58.23	1.5823													
59.25	1.5582													
59.74	1.5466												1.546	4
59.90	1.5428						1.54	40			1.542	10		
60.66	1.5253						1.51	30			1.531	2	1.514	6
62.43	1.4863		1.486	30	1.49	70	1.49	40						
62.51	1.4845													

Table A.2. (Continued)

2 θ	d Spacing (Å)	Intensity (Counts/s)	Na ₆ Al ₄ Si ₄ O ₁₇		LiOH		δ -Al ₂ O ₃		Al ₂ SiO ₅		SiO ₂		α -Al ₂ O ₃	
			d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1
65.78	1.4184													
65.85	1.4171													
66.04	1.4135													
66.74	1.4004	6	1.404	4			1.40	60					1.404	30
67.28	1.3904													
67.45	1.3873													
67.58	1.3850	13					1.39	100						
67.70	1.3828				1.38	30								
68.10	1.3757												1.374	50
68.35	1.3712													
68.63	1.3663													
70.50	1.3346	2			1.34	30							1.337	2
71.69	1.3153													
73.00	1.2949	1.5												
73.23	1.2914													
73.32	1.2901						1.29	20						
74.07	1.2788	1.5	1.287	10										
74.46	1.2731	1.5			1.28	50							1.276	4
75.25	1.2617				1.26	60	1.26	10						
75.35	1.2603													
75.74	1.2547	2												
76.63	1.2424	1.5											1.239	16
77.18	1.2349	1	1.232	4									1.2343	8
78.32	1.2197	1			1.21	40								
79.63	1.2029	1.5												
79.99	1.1983													
81.03	1.1857												1.1898	8
81.94	1.1748													
84.35	1.1472	1	1.152	10	1.14	50							1.147	6
87.96	1.1092	1			1.12	40								

Table A.2. (Continued)

2 θ	d Spacing (Å)	Intensity (Counts/s)	$\text{Na}_6\text{Al}_4\text{Si}_4\text{O}_{17}$		LiOH		$\delta\text{-Al}_2\text{O}_3$		Al_2SiO_5		SiO_2		$\alpha\text{-Al}_2\text{O}_3$	
			d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1	d	I/I_1
88.22	1.1066													
89.12	1.0978	1			1.09	10								
90.16	1.0878													
90.43	1.0852													
90.61	1.0836													
92.13	1.0696				1.07	20								
92.71	1.0644													
94.67	1.0475													
95.41	1.0413	1			1.04	40								
95.98	1.0366	1												
96.46	1.0328	1												
98.17	1.0193													
98.65	1.0156													
99.35	1.0103													
99.96	1.0058													
100.55	1.0015													
101.16	0.9971	1			0.998	50							0.9976	12
101.48	0.9948	1												
101.74	0.9930													
102.74	0.9860													
103.40	0.9815													
103.74	0.9792													
104.36	0.9751		0.975	6										
105.43	0.9681	1												
106.76	0.9597	1												
107.66	0.9542													
107.74	0.9537													
108.05	0.9518													
109.08	0.9456													

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