Scapolite, 20 kV results

	PSU4-294		PSU62-1703		PSU63-1805	
	reported	probe	reported	probe	reported	probe
SiO_2	55.37	55.77	54.06	54.60	54.84	55.53
TiO_2	0.01	0.02	0.01	0.0	0.02	0.01
Al_2O_3	22.86	23.36	21.62	22.19	22.81	23.23
Fe_2O_3	0.01		0.27		0.10	
FeO		0.01		0.15		0.04
MnO	0.00	0.00	0.04	0.0	0.00	0.00
MgO	0.34	0.00	0.10		0.08	0.00
CaO	7.48	7.29	9.02	6.81	8.33	6.93
Na_2O	9.22	7.87	8.78	7.52	8.83	7.90
K_2O	0.24	0.13	1.04	1.04	1.06	0.93
P_2O_5	0.05	0.00	0.02	0.0		0.01
H_2O^+	0.35		0.16		0.26	
H_2O^-	0.03					
CO_2	1.90		2.12		1.50	
SO_3	0.18	0.21	0.66	0.68	0.05	0.77
Cl	2.22	2.80	2.57	2.82	2.57	2.84
F	0.00	0.0		0.0		0.01
SrO	0.08	0.18		0.42		0.08
BaO	0.12	0.07		0.0		0.02
total	100.46	97.73	100.48 *	96.25	100.45	98.31
O=F+Cl	0.50	0.63	0.61	0.64	0.56	0.65
	99.96	97.10	99.87	95.61	99.89	97.67

* includes 0.01 Li_2O

Scapolite, Moro Goro Tanzania

SiO_2	56.89	Si	8.2576]	12.0000
TiO_2	0.00	Al	3.7424]	
Al_2O_3	21.87			
FeO	0.13	Fe	.0157	
MnO	0.00			
MgO	0.00	Na	2.5319 ┐	
CaO	6.59	K	0.26955]	3.8304
SrO	0.04	Ca	1.0257]	
BaO	0.00	Sr	0.0034 ┘	
Na_2O	8.99			
K_2O	1.45	Cl	.6768 ┐	
F	0.00	CO_3	.1544]	0.9332
Cl	2.75	SO_3	.1020 ┘	
SO_3	0.94			
CO_2	0.78			
total	100.48			
$-O \equiv F+Cl$	0.62			
total	99.81			

April 7, 1980

Orogenic Studies Laboratory

J. A. Speer
OSL
1056 Derring Hall
VA Tech
Blacksburg, VA 24061

February 9, 1981

Dr. C. O. Ingamells
AMAX R&D Laboratory
5920 McIntyre Street
Golden, CO 80501

Dear Dr. Ingamells:

I would like to take advantage of your offer in the October, 1980 Geostandards Newsletter for some 62-1703 scapolite as a Cl-microprobe standard. We have been using a synthetic Ba chlorapatite which is almost gone. We explored the possibility of using sodalite but found that sodium vaporized under our operating conditions of 15KV and 10 nanoamps with a focused and 10x10 micron rastered beam. Apparently no one else encountered this problem or compensated for Na-loss for the sodalites reported in your article.

I appreciate your help in obtaining a Cl-standard.

Sincerely yours,

J. Alexander Speer,
Research Associate

AMAX
EXTRACTIVE RESEARCH & DEVELOPMENT, INC.
6950 McINTYRE STREET • GOLDEN, COLORADO 80401 • (303) 279-7636

February 16, 1981

J. Alexander Speer
Research Associate
Virginia Polytechnic Institute
Orogenic Studies Laboratory
4044 Derring Hall
Blacksburg, Virginia 24061

Dear Dr. Speer:

Thanks for your interest in my microprobe standards.

Under separate cover I am sending a selection of chlorine-bearing samples. A summary of your findings as to the homogeneity and suitability of these materials in microprobe analysis will be appreciated.

Analyses are enclosed.

Sincerely,

C. O. Ingamells

COI/act
enc.

Scapolite PSU 4-294 (Gib Lake Scapolite)

	Analysis by C. O. Ingamells	Analysis by J. Muysson
SiO_2	55.37	55.44
TiO_2	.01	
Al_2O_3	22.86	22.89
Fe_2O_3	.01	.00
MnO	.00	.00
MgO	.34	.30
CaO	7.48	7.53 (uncorrected for Sr)
Na_2O	9.22	9.36
K_2O	.24	.22
P_2O_5	.05	.05
H_2O+	.35	.22
H_2O-	.03	.03
CO_2	1.90	1.85
SO_3	.18	.18
Cl	2.22	2.30
F		.00
SrO	.08	
BaO	.12	
TOTAL	100.46	100.37
O = Cl	.50	.52
	99.96	99.85

Reference: C. O. Ingamells and J. Gittins, Can. Mineral. 9, 214, 1967

Calculation of J. Muysson's analysis according to principles outlined in this paper gives a meionite $Si_{11.64}Al_{10.36}Ca_7H_{.36}(C,S,Cl)_2$ and a marialite $Si_{17.63}Al_{6.36}Na_{7.76}H_{.44}Cl_{1.62}$, with a calcite residue. Homogeneity of this mineral at the microprobe sampling level is not established. The two bulk analyses (by Muysson and Ingamells) were performed by totally different methods, quite independently: agreement for most constituents may induce some confidence in the values reported.

Microscopically, about 10% of the grains show fine brown dust, i.e. incipient alteration. If microprobe analysis does not show too great a grain-to-grain variation, this may still be useful.

Scapolite	PSU 63-1805		Meionite	Marialite
	%			
SiO_2	54.84	Si	11.69	17.73
Al_2O_3	22.81	Al	10.31 — 22.00	6.17 — 24.00
CO_2	1.50	C	1.61	
SO_3	.05	S	.03	
		Si	.36 — 2.00	
Fe_2O_3	.10	Fe^{+++}		.03
TiO_2	.02	Ti		.01
MgO	.08	Mg		.05
MnO	.00	Mn		
CaO	8.33	Ca	7.00 — 7.00	
Na_2O	8.83	Na		8.30
K_2O	1.06	K		
H_2O+	.26	H	.31	.60 — 8.99
Cl	2.57	Cl		1.88
		O + Cl	50.00	50.22
TOTAL	100.45			
O = Cl	.56			
	99.89			

Reference: C. O. Ingamells and J. Gittins. Can. Mineral. 9, 214, 1967

Analysis by E. Martinec, with N. H. Suhr and C. O. Ingamells

White, coarsely crystalline scapolite from calcareous gneiss, Lot 32, Con. XVII, Monmouth Township, Ontario, Canada. ON7 of Shaw, J. Petrology, 1, 261, 1960.

Homogeneity at the microprobe level of sampling not established

Pennsylvania State University PSU 62-1703 Scapolite

SiO_2	54.06	
Al_2O_3	21.62	
TiO_2	.01	
Fe_2O_3	.27	
MnO	.04	
MgO	.10	
CaO	9.02	
Li_2O	.01	
Na_2O	8.78	
K_2O	1.04	
H_2O+	.16	
P_2O_5	.02	
Cl	2.57	
CO_2	2.12	
SO_3	.66	
	100.48	
O = Cl	.61	
	99.87	
less O	.05	(oxygen deficiency)
	99.82	

Analyst C. O. Ingamells

University of Minnesota Rock Analysis Laboratory R-2208 Biotite

SiO_2	33.09
Al_2O_3	17.65 (includes ZrO_2 etc.)
TiO_2	1.30
Fe_2O_3	2.42
FeO	29.22
MnO	.04
MgO	2.83
CaO	.10
BaO	.09
Na_2O	.13
K_2O	9.04
Rb_2O	.10
H_2O+	2.92
H_2O-	.04
Cl	1.11
F	.23
	100.31
O = F,Cl	.34
	99.97

Analysis by Eileen H. Oslund

October 25, 1982

Dr. C. O. Ingamells
AMAX R&D Laboratory
5920 McIntyre Street
Golden, CO 80401

Dear Dr. Ingamells:

I appreciate your sending us the Cl-bearing minerals for microprobe standards. Enclosed is a reprint summarizing our work on the Cl-biotite and a copy of the individual analyses. In addition, I have sent you a copy of the individual analyses for the French reference biotites knowing of your probable interest. Each analysis represents a different grain.

Work on the scapolites is not going as well as evidenced by the summary of our results thus far on the enclosed copy.

Best regards,

J. Alexander Speer

JAS/mgs

Enclosure

Microprobe Column

C.O. INGAMELLS

970 Xenon Street
Denver, Wheat Ridge, Colorado 80215, USA

Dr. E. Jarosewich (Smithsonian Institution) has submitted data on several minerals offered as potential microprobe standards via this column. Sigma ratios ($S/\sqrt{N}$) for various elements are reported as follows, with comments below the table.

HOMOGENEITY INDECES FOR 10 RANDOMLY SELECTED GRAINS

Index for Least Homogeneous Grain in Parantheses

	SiO_2	Al_2O_3	FeO	MgO	CaO	K_2O	Na_2O	TiO_2	P_2O_5	Cr_2O_3
Apatite					1.34 (2.26)				1.68 (1.68)	
Pyroxene	0.89 (1.11)		0.77 (1.30)	0.90 (1.14)	1.05 (1.39)					
Amphibole	1.08 (1.42)	1.38 (2.70)	1.04 (2.38)	1.10 (1.40)	1.21 (1.62)					
Diopside PSU 63-1827	1.05 (1.48)			1.04 (1.50)	0.98 (1.58)					
Orthoclase PSU OR-1A	1.12 (1.79)	1.00 (1.25)				1.05 (1.43)				
Biotite LP-6 (40-60)	1.48 (3.28)	1.46 (2.85)	1.89 (5.07)	1.64 (3.66)		1.50 (2.90)				
Biotite PSU 5-112	1.37 (2.26)	1.39 (2.53)	2.09 (6.95)	1.68 (3.78)		1.26 (2.40)				
Chromite R-2309		2.29 (-)	2.58 (-)	2.31 (-)						1.34 (2.66)
Chromite PSU-4-228		1.54 (-)	6.02 (-)	2.67 (-)						1.57 (2.88)
Microlite PSU-5-006					1.09 (1.99)		1.01 (1.35)			
Sphene	1.12 (1.63)				2.50 (3.46)			1.46 (2.81)		
Tremolite 62-1717	- (1.34)			- (1.11)	- (0.99)					

Sigma ratio for 10 grains = $\frac{\text{observed sigma for all grains}}{\text{sigma predicted from counting statistics}}$

Sigma ratio for least homogeneous grain = $\frac{\text{observed sigma for this particular grain}}{\text{sigma predicted from counting statistics}}$

Chromites contain two different types of grains.

R-2369 contains Mg and Al-rich and Mg and Al-poor grains.

Chromite 4-228 contains Mg rich and Mg poor grains.

Tremolite : Only two grains were available for analysis.

Microlite : Homogeneity for Ta and Nb was not performed.

Dr. J.A. Speer (Virginia Polytechnic Institute) has referred me to his article with T.N. Solberg describing a 16-element scheme for microprobe analysis of minerals in petrographic thin sections, together with numerous analyses of three biotites. Of special interest to me are the chlorine values in the Idaho biotite R2208. 22 microprobe vlaues vary from 1.06% to 1.30%, with an average of 1.24 and s.d of 0.06. The University of Minnesota value (gravimetric) on the bulk sample is 1.11%.

Dr. Speer has also provided analyses of three scapolites - PSU 4-294, PSU 62-1703 and PSU 63-1805. The probe values for chlorine are higher than the bulk chemical values :

	% Cl	
	Chemical	probe
PSU 4-294	2.22	2.80
PSU 62-1703	2.57	2.82
PSU 63-1805	2.57	2.84

Speer's probe reports 0.42 SrO in 62-1703; in the bulk analysis, SrO was not determined. The reported CaO value was uncorrected for Sr, contains much of the SrO, and must be considered too high.

I very much doubt that my chemical (bulk) values for Cl are in error, and draw attention to the fact that the ratio (Cl-probe)/(Cl chemical) is about 1.1 for 1703, 1805 and the Idaho biotite. The ratio for 4-294 is 1.26, and I concede the possibility of an error in the chemical analysis.

There is also the likelihood that chlorine in scapolite is often present, in part, in contaminating halite. This would not appear during microprobe analysis. Evidence of this possibility is given by Ingamells and Gittins (Canadian Mineralogist 9, Part 2, pp. 214-236, 1967). In the same paper, evidence is presented to imply that a reported low total (99.53%) is due to an erroneous chlorine determination on my part; so I have to leave any decision concerning the chlorine content of scapolites up to someone else.

I must add that microprobe determinations of chlorine in scapolite based on nonscapolite standards are, in my view, suspect. If the microprobe value for Cl in PSU 4-294, for example, is correct, the total of the analysis comes to 100.41 (an intolerable total) and there must be a gross error elsewhere in my analysis. This possibility I refuse to accept unless proof or explanation is forthcoming. Please note that contaminating halite would lead to lower, not higher, microprobe values for chlorine, and higher values for other constituents. Is it possible that chlorides are somehow introduced on the polished surfaces of samples prepared for microprobe analysis? It wouldn't take more than a fingerprint!

Microprobe Column

C.O. INGAMELLS

AMAX R&D Laboratory, 5920 McIntyre Street,
Golden, Colorado 80401, USA

B. Robins, Universitetet I. Bergen, has examined six sodalites under the microprobe for sodium, aluminum, silicon and chlorine, comparing them to the homogeneous and well-analyzed PSU 4-296-8. Ten or more analyses of each provide an estimate of homogeneity at the microprobe level of sampling. One of these samples, PSU 4-296-2 appears satisfactory as a microprobe standard for Na, Al, Si and Cl. Comparison of Robins' averages with J. Muysson's analysis indicates that 4-296-2 may be a somewhat cleaner sample than 4-296-8:

	4-296-2 (B. Robins)	4-296-8 (J. Muysson)
Na_2O	24.0 %	23.9 %
Al_2O_3	32.0	31.5
SiO_2	37.6	36.7
Cl	7.0	7.0
	100.6	99.1
less O=Cl	.5	.5
	100.1	98.6

Chemical determination of chlorine in 4-296-2 gave 7.00 %; exactly the same as the microprobe value. The formula of 4-296-2 calculates to

$$Na_{3.87}\,Al_{3.02}\,Si_{3.02}\,Cl_{0.99}\,O_{12}$$

from Robins' analysis.

Our own examination of these sodalites shows that 4-296-2 contains small inclusions of magnetite and other opaque minerals, making up less than 1 % of the total. These should not seriously affect the usefulness of the sample as a probe standard.

I have read with interest the recent contribution of Jarosewich et al (1), and offer this column as a forum through which further information on their reference samples may be exchanged.

There seems to be a shortage of analyzed ore minerals, particularly sulfides, for use as microprobe standards. If anyone out there can suggest sources of such materials, I feel sure the microprobe community will be appreciative, especially within the mining industry.

Attention is drawn to a new column feature in *"American Laboratory"*, written by Stanley Rasberry (2) Deputy Chief of the Office of Standard Reference Materials, National Bureau of Standards. The column has thus far been devoted to statements concerning NBS SRM's, but there is an intention to include SRM's from other sources. Perhaps a few letters to him, c/o *American Laboratory*, 808 Kings Highway, Fairfield, Connecticut, U.S.A. 06430, might induce a column on probe standards. One might expect a wealth of information from this source, especially since Mr. Rasberry and his colleagues have been involved for many years in X-ray emission spectrometry and related techniques.

Dr. P.J. Potts, Director of Analytical Services, Department of Earth Sciences, The Open University, Walton Hall, Milton Keynes, England has reported on four samples as follows:

The diopside PSU 63-1827 appears homogeneous and useful as a microprobe standard, with probe values very close to the chemical values. In agreement with Chodos (Cal Tech), Chromite PSU 61-1436 is not entirely satisfactory; silicon and calcium reported in the bulk analysis are not present in chromite grains. The Beeson apatite appears to be a good standard for calcium and phosphorus, but probe and chemical values for fluorine and strontium are widely different. Surprisingly, Potts found PSU Or-1A orthoclase to be inhomogeneous with respect to potassium. This is the first evidence of possible inhomogeneity for this element in this sample; barium and sodium are known to be inhomogeneously distributed.

Several additional completely analyzed minerals are now available on request to responsible laboratories:

4-234	hornblende with 2.40 TiO_2 %
5-180	pyroxene with 18.93 CaO, 19.82 FeO, 1.47 Fe_2O_3
5-010	microlite with 0.32 UO_2, 1.63 UO_3

62-1703 scapolite with 2.57 Cl, 2.12 CO_2, 0.66 SO_3
R2208 biotite with 1.11 Cl
R2027 manganese dolomite with 23.31 MnO
R2469 grunerite with 27.16 FeO, 0.73 Fe_2O_3
4-190 hornblende with 21.52 FeO, 5.15 Fe_2O_3
4-206 tourmaline with 17.62 Fe_2O_3, 1.27 FeO
4-166 biotite with 3.20 TiO_2
R2535 amphibole with 6.28 Na_2O
4-222 riebeckite with 6.35 Na_2O

Although most of these have not been examined for homogeneity at the microprobe level of sampling, they appear reasonably clean under the microscope.

REFERENCES

(1) E. Jarosewich, J.A. Nelen and J.A. Norberg (1980) Reference samples for electron microprobe analysis, Geostandards Newsletter, 4: 43-47.

(2) S. Rasberry, editor (1980) Reference materials, American Laboratory, 12 : 109.

Analyzed Minerals for Electron Microprobe Standards

C.O. INGAMELLS

AMAX Metallurgical R & D Laboratory, Golden, Colo. U.S.A. 80401

Limited quantities of analyzed mineral samples are available, on request, to responsible persons who may find them useful in the calibration or control of microprobe analyses. These have been completely analyzed by primary methods, and have been examined for purity and homogeneity under the microscope. Most exist in too small amounts for other than microprobe work, and only a few milligrams will be supplied. Inquiries concerning mineral types not listed below are also welcome.

Engels' Amphibole : $(K,Na)_{0.59}(Na,Ca,Mn)_{2.00}(Mn,Mg,Fe^{2+},Fe^{3+},Ti,Al)_{5.00}(Al,Si)_{8.00}H_{1.67}O_{24.00}$

Denningite, PSU 61-1431 : $(Mn,Zn,Cd,Co,Mg,Ca)_{1.00}Te_2O_5$

Diopside, PSU 63-1827 : $(Ca_{0.993}Mg_{1.008})_{2.00}Si_{1.995}O_{6.000}$

Microlite, PSU 5-006 : $(Na,K,H)_{0.999}Ca_{1.009}(Si,Ta,Nb,Al)_{2.000}$

Lepidolite, PSU 60-1252 : $(Na,K,Rb,Cs,Li)_{2.00}(Li,Fe,Ti,Mn,Mg,Al)_{5.75}(Al,Si)_{7.99}(OH,F)_{4.01}O_{20.00}$

Elbaite, Donnay : $(Na_{1.69}Mn_{0.45}Ca_{0.42}{}^{*}\,0.38B_{0.05}K_{0.01})(Al_{4.78}Li_{3.74}Mn_{0.39}Fe^{++}{}_{0.09})$
$(Al_{18}B_9)(Si_{17.94}B_{0.06})O_{82.57}(OH)_{8.62}F_{1.81}$

Chromite, PSU 1436 : $(Fe^{++},Mg,Ca,Mn,Ni)_{1.00}(Cr,Al,Ti,Fe^{+++},V)_{1.98}$

Tremolite, PSU 62-1717 : $(Li,Na,K,Ca)_{2.00}(Mg,Mn,Fe)_{5.00}Si_{8.00}(OH,F)_{2.01}O_{22.00}$

Sphene : $(Ca,Fe,Mg,Mn,Na,K,Y,La,Ce,Pr,Nd,Sm,Eu,Gd,Tb,Dy,Ho,Er,Tm,Yb,Lu)_{0.99}$
$(Si,Ti,Zr,V,P,Fe,Al)_{1.00}Si_{1.03}(O,F)_{5.00}$

Ardennite, PSU 5-144 : $(Na,Ca,Zn,Cu,Ni,Co,Mn)_{1.00}Mn_{1.00}(Mn,Mg,Cr,Fe,Sn,Al)_{1.00}Al_{2.00}$
$(Al,V,As,Si)_{2.00}Si_{1.04}(OH,F)_{2.99}O_{11}$

Pyroxene, PSU Px-1 : $(Na,Ca,Sr,Fe^{++})_{8.00}(Fe^{++},Mn,Mg,Fe^{+++},Cr,Ti)_{8.00}(Al,Si)_{16.01}O_{48.00}$

Scapolite, PSU 63-1805 : $[Ca_{7.00}(C,S,Si)_{2.00}(Si,Al)_{22.00}H_{0.31}(O,Cl)_{50.00}]$
$[(Na,K,H,Mg,Ti,Fe^{+++})_{8.99}(Si,Al)_{24.00}(O,Cl)_{50.22}]$

Orthoclase, PSU Or-1A : $(K,Na,Rb,Ba,Sr)_{2.001}(Si,Al)_{7.99}O_{16.000}$

Biotite, PSU 5-110 : K_2O 10.00 $\pm$ 0.02 %. Of a very large number of biotites examined, this is the only one which has been shown to be perfectly homogeneous at the microprobe level of sampling. Purity 99.9+%. No inclusions. Trace apatite.

Sodalite, PSU 4-296-8 : 23.9% Na_2O and 6.82% Cl. Useful as a reference for chlorine and sodium. SiO_2, 36.7 %; Al_2O_3, 31.5 %.

Beeson Apatite : Completely analyzed for all constituents greater than 0.1 %, including all the rare earth elements.

Biotite LP-6 Bio 40-60# : This is available in 8-gram portions. Purity, 99.9+%, but grain-to-grain composition shows some variability. Very few inclusions, mostly apatite and rutile. Primarily a K-Ar standard.

The Nancy phlogopite Mica-Mg should also be mentioned as one of the very few minerals of high purity available in quantity and supplied with a complete and competent analysis.