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BASELINE STUDIES OF THE CLAY MINERALS SOCIETY SOURCE CLAYS: CHEMICAL ANALYSES OF MAJOR ELEMENTS

AHMET R. MERMUT[1†] AND ANGEL FAZ CANO[2]

[1] University of Saskatchewan, Department of Soil Science, 51 Campus Drive, Saskatoon, Saskatchewan, Canada S7N 5A8
[2] Area de Edafologia y Quimica Agricola, Departamento de Produccion Agraria, Universidad Politécnica de Cartagena, Paseo Alfonso XII, 52, 30.203 Cartagena, Murcia, Spain

INTRODUCTION

Chemical analysis is an essential step to establish the nature of minerals (Newman, 1987). The techniques used in rock and mineral analyses are generally valid for the analyses of clays. Additional information from other analytical techniques, which are mentioned here, is needed for accurate interpretation of the chemical analysis results of major elements (Gabis, 1979). In traditional chemical analyses, the aim is to obtain accurate analyses for all elements present in the sample, in such a way that the sum of elements expressed as oxides, including hydration and structural water, approaches the sample weight as closely as possible.

The following elements are essential for the calculation of structural formulae of most clay minerals and silicates: Si, Al, Fe^{3+}, Fe^{2+}, Mg, Ti, Mn (in special cases), P, Ca, Na, K and H_2O evolved below 105°C (H_2O^-) and between 105–1000°C (H_2O^+). For some minerals, additional determinations, such as for F and Li, may be needed for the calculation of the composition of clay minerals. Methods used to determine the chemical composition, for both major and minor elements, are described elsewhere (Jackson, 1979; Lim and Jackson, 1982; Laird *et al.*, 1989; Amonette and Zelazny, 1994). For minor or trace elements of the Source Clays, see Elzea Kogel and Lewis (2001).

Despite the progress made in science, and the increased accuracy which can be obtained from very sophisticated instruments, total major element analysis is often performed by standard methodologies. One problem with standard methodologies involving wet analyses is that the concentration of these elements is high and a very high rate of dilution is needed to bring the concentration to a level that can be determined by the instruments available.

Neutron activation analysis may be utilized to determine accurately the oxygen content, which is the neglected element in most standard silicate analyses. Methods for determining Fe^{2+}, H and F are critically reviewed in Newman (1987).

Clay minerals are seldom monomineralic. Therefore, before attempting to perform any analyses, it is essential that the sample be examined by X-ray diffraction to identify possible impurities in the sample. Some poorly crystalline phases can be removed by selective dissolution techniques (Newman, 1987; Amonette and Zelazny, 1994).

INSTRUMENTATION AND METHOD

The Source Clay samples were prepared as described by Costanzo (2001). For the total analyses, although the samples appear homogeneous, the clays were suspended one additional time to remove particles of >2 μm using the method described by Jackson (1979). Note that with impurities present in clays, unless their quantity and chemical compositions are known, it is not possible to calculate accurately the apparent structural formulae. Details of the calculation of the structural formulae are given in Newman (1987).

The samples were saturated with NH_4^+ prior to total elemental analysis, following the general procedure of Brindley and Ertem (1971). This procedure involved washing the homoionic sample three times with a solution of 1 M NH_4Cl and then with a 50% (v/v) mixture of ethanol and distilled water until the supernatant solutions were free of Cl^- by the $AgNO_3$ method. The samples were oven-dried overnight at 105°C. The elemental composition of the source clays was determined following digestion with analytical-grade aqua regia and boric acid in a microwave oven (Sawhney and Stilwell, 1994). The Na and K contents were analyzed by flame emission spectrometry. All other elements, including Si, Al, Fe, Mg, Mn and Ti were determined by atomic absorption spectrometry. Four different individuals analyzed the samples using exactly the same procedures and chemicals. Four replicate analyses by each analyst were averaged and recorded as a single analysis. These four sets of analyses were averaged again to obtain a single value. The mean values, standard deviation (SD), and standard error (SE) were calculated.

RESULTS

The analytical data (overall averages) are presented in Table 1 with SE for the selected eight Source Clay samples. The average data from each analyst are given in Tables 2 to 7. Examination of the results shows that the spread in the data is not large. The high degree of precision is due to the use of the same methods, pro-

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Table 1. Summary of the elemental composition of the source clay minerals.

% Oxide SE	KGa-1	STx-1	KGa-2	SWy-2	PFl-1	SAz-1	Syn-1	SHCa-1
SiO_2	43.36	70.03	43.49	61.46	60.35	59.65	49.77	46.66
SE	0.39	0.12	0.19	1.29	0.34	0.16	0.32	0.33
Al_2O_3	38.58	17.86	38.14	22.05	11.13	19.98	38.56	0.86
SE	0.24	0.08	0.14	0.19	0.04	0.09	0.03	0.01
Fe_2O_3	0.35	1.20	1.15	4.37	3.74	1.77	0.12	0.32
SE	0.02	0.03	0.02	0.06	0.03	0.03	0.02	0.01
TiO_2	1.67	0.26	1.91	0.09	0.48	0.25	0.03	0.04
SE	0.01	0.00	0.03	0.00	0.01	0.01	0.00	0.00
MgO	0.04	3.79	0.04	2.94	10.58	6.73	0.18	20.01
SE	0.01	0.01	0.01	0.02	0.03	0.03	0.00	0.43
CaO	0.04	1.73	0.03	1.18	1.89	3.15	0.05	14.01
SE	0.01	0.01	0.01	0.03	0.04	0.04	0.00	0.22
Na_2O	0.05	0.31	0.06	1.47	0.05	0.06	0.08	1.35
SE	0.01	0.02	0.01	0.03	0.01	0.01	0.01	0.02
K_2O	0.00	0.07	0.02	0.20	0.87	0.19	0.00	0.14
SE	0.00	0.01	0.00	0.01	0.02	0.01	0.00	0.00
P_2O_5	0.37	0.01	0.32	0.00	0.92	0.01	0.00	0.00
SE	0.03	0.00	0.00	0.00	0.03	0.00	0.00	0.00
Ignition loss 110–550°C	13.60	0.99	13.30	0.61	7.87	4.58	8.65	0.84
Ignition loss 550–1000°C	1.45	3.64	1.73	5.15	1.65	3.59	2.18	15.47
Total	99.51	99.91	100.19	99.52	99.53	99.95	99.61	99.70

cedures and standards. The difference between the individuals was probably related to human error; *e.g.* analyst 2 produced less consistent results. Gabis (1979) discussed the reasons for a possible high spread in analytical data and attributed variations to storage conditions, expression of results either on the basis of the sample being processed wet or dried at 110°C, application of different procedures to fuse the sample, and the techniques used to determine the elements.

As commonly observed, the SE for Si is relatively large, because phyllosilicates contain large amounts of Si and this element is also present in the impurities.

Table 2. Al content of the clays.

Sample	% Moisture	Sample weight	Dry weight	Al ppm in solution (100 mL)	% Al in dry sample	% Al_2O_3 in sample	Mean and SD
KGa-1	0.404	0.101	0.100	204.600	20.441	38.622	38.576
KGa-1	0.404	0.100	0.100	200.000	20.061	37.905	
KGa-1	0.404	0.100	0.100	204.600	20.523	38.777	0.237
KGa-1	0.404	0.100	0.100	206.400	20.641	39.001	
STx-1	10.113	0.101	0.091	85.100	9.392	17.746	17.864
STx-1	10.113	0.101	0.091	85.900	9.481	17.913	
STx-1	10.113	0.101	0.091	86.600	9.567	18.077	0.083
STx-1	10.113	0.101	0.090	84.800	9.378	17.719	
KGa-2	0.500	0.101	0.100	201.800	20.140	38.055	38.140
KGa-2	0.500	0.101	0.100	200.200	20.001	37.790	
KGa-2	0.500	0.100	0.100	202.000	20.241	38.244	0.144
KGa-2	0.500	0.100	0.100	203.200	20.361	38.471	
SWy-2	7.387	0.101	0.093	106.300	11.409	21.558	22.051
SWy-2	7.387	0.101	0.093	110.800	11.892	22.470	
SWy-2	7.387	0.100	0.093	108.200	11.671	22.053	0.188
SWy-2	7.387	0.101	0.093	109.100	11.710	22.125	
PFl-1	8.162	0.101	0.093	54.300	5.860	11.072	11.134
PFl-1	8.162	0.101	0.093	55.200	5.957	11.256	
PFl-1	8.162	0.100	0.092	54.200	5.890	11.129	0.043
PFl-1	8.162	0.100	0.092	53.900	5.863	11.078	
SAz-1	13.800	0.101	0.087	92.400	10.634	20.093	19.978
SAz-1	13.800	0.101	0.087	91.500	10.541	19.917	
SAz-1	13.800	0.100	0.086	92.200	10.664	20.149	0.090
SAz-1	13.800	0.100	0.086	90.300	10.455	19.754	
Syn-1	3.338	0.101	0.097	198.780	20.401	38.547	38.560
Syn-1	3.338	0.101	0.097	199.000	20.424	38.590	
Syn-1	3.338	0.101	0.097	198.800	20.444	38.628	0.033
Syn-1	3.338	0.101	0.097	198.000	20.362	38.473	
SHCa	5.022	0.101	0.096	4.200	0.439	0.830	0.863
SHCa	5.022	0.100	0.095	4.300	0.452	0.855	
SHCa	5.022	0.100	0.095	4.400	0.463	0.874	0.014
SHCa	5.022	0.100	0.095	4.500	0.473	0.893	

Table 3. Fe and Ti contents of the clays.

Sample	Fe ppm in solution	% Fe in dry sample	% Fe_2O_3	Mean and SD	Ti ppm in solution	% Ti in dry sample	% TiO_2	Mean and SD
KGa-1	2.500	0.250	0.357	0.351	10.000	0.999	1.666	1.674
KGa-1	2.500	0.251	0.359		10.100	1.013	1.690	
KGa-1	2.700	0.271	0.387	0.018	10.000	1.003	1.673	0.005
KGa-1	2.100	0.210	0.300		10.000	1.000	1.668	
STx-1	7.900	0.872	1.247	1.200	1.400	0.155	0.258	0.263
STx-1	7.600	0.839	1.199		1.400	0.155	0.258	
STx-1	7.800	0.862	1.232	0.028	1.400	0.155	0.258	0.005
STx-1	7.100	0.785	1.123		1.500	0.166	0.277	
KGa-2	7.900	0.788	1.127	1.148	11.900	1.188	1.981	1.906
KGa-2	7.900	0.789	1.128		11.400	1.139	1.900	
KGa-2	7.800	0.782	1.117	0.023	11.400	1.142	1.905	0.029
KGa-2	8.500	0.852	1.218		11.000	1.102	1.839	
SWy-2	28.700	3.080	4.404	4.371	0.500	0.054	0.090	0.094
SWy-2	28.400	3.048	4.358		0.500	0.054	0.090	
SWy-2	27.400	2.956	4.226	0.056	0.600	0.065	0.108	0.005
SWy-2	29.300	3.145	4.496		0.500	0.054	0.090	
PFl-1	24.600	2.655	3.796	3.740	2.600	0.281	0.468	0.479
PFl-1	23.800	2.568	3.672		2.700	0.291	0.486	
PFl-1	23.800	2.586	3.698	0.032	2.700	0.293	0.489	0.005
PFl-1	24.400	2.654	3.795		2.600	0.283	0.472	
SAz-1	10.600	1.220	1.744	1.770	1.300	0.150	0.250	0.250
SAz-1	10.400	1.198	1.713		1.200	0.138	0.231	
SAz-1	10.800	1.249	1.786	0.027	1.400	0.162	0.270	0.008
SAz-1	11.100	1.285	1.837		1.300	0.151	0.251	
Syn-1	0.600	0.062	0.088	0.125	0.200	0.021	0.034	0.026
Syn-1	0.700	0.072	0.103		0.100	0.010	0.017	
Syn-1	1.200	0.123	0.176	0.019	0.200	0.021	0.034	0.005
Syn-1	0.900	0.093	0.132		0.100	0.010	0.017	
SHCa	2.200	0.230	0.329	0.324	0.200	0.021	0.035	0.039
SHCa	2.300	0.242	0.346		0.200	0.021	0.035	
SHCa	2.100	0.221	0.316	0.009	0.300	0.032	0.053	0.004
SHCa	2.030	0.213	0.305		0.200	0.021	0.035	

Table 4. Mg and Ca contents of the clays.

Sample	Mg ppm in solution	% Mg in dry sample	% MgO	Mean and SD	Ca ppm in solution	% Ca in dry sample	% CaO	Mean and SD
KGa-1	0.100	0.010	0.017	0.042	0.500	0.050	0.070	0.042
KGa-1	0.200	0.020	0.033		0.100	0.010	0.014	
KGa-1	0.500	0.050	0.083	0.014	0.400	0.040	0.056	0.013
KGa-1	0.200	0.020	0.033		0.200	0.020	0.028	
STx-1	20.600	2.274	3.769	3.795	11.000	1.214	1.699	1.735
STx-1	20.800	2.296	3.806		11.300	1.247	1.745	
STx-1	20.900	2.309	3.828	0.014	11.300	1.248	1.747	0.012
STx-1	20.600	2.278	3.777		11.300	1.250	1.748	
KGa-2	0.400	0.040	0.066	0.041	0.300	0.030	0.042	0.028
KGa-2	0.100	0.010	0.017		0.100	0.010	0.014	
KGa-2	0.300	0.030	0.050	0.011	0.200	0.020	0.028	0.006
KGa-2	0.200	0.020	0.033		0.200	0.020	0.028	
SWy-2	16.500	1.771	2.936	2.940	8.300	0.891	1.246	1.177
SWy-2	16.700	1.792	2.972		7.600	0.816	1.141	
SWy-2	16.200	1.747	2.897	0.016	7.900	0.852	1.192	0.027
SWy-2	16.600	1.782	2.954		7.500	0.805	1.126	
PFl-1	59.000	6.367	10.556	10.582	11.800	1.273	1.782	1.887
PFl-1	59.500	6.421	10.645		12.900	1.392	1.948	
PFl-1	59.000	6.412	10.630	0.035	12.300	1.337	1.870	0.040
PFl-1	58.200	6.331	10.496		12.800	1.392	1.948	
SAz-1	35.500	4.086	6.773	6.731	20.200	2.325	3.253	3.145
SAz-1	35.400	4.078	6.761		19.200	2.212	3.095	
SAz-1	35.200	4.071	6.750	0.030	19.400	2.244	3.140	0.037
SAz-1	34.600	4.006	6.641		19.100	2.211	3.094	
Syn-1	1.100	0.113	0.187	0.179	0.300	0.031	0.043	0.047
Syn-1	1.000	0.103	0.170		0.400	0.041	0.057	
Syn-1	1.000	0.103	0.170	0.005	0.300	0.031	0.043	0.004
Syn-1	1.100	0.113	0.188		0.300	0.031	0.043	
SHCa	119.800	12.526	20.776	20.009	92.600	9.682	13.547	14.009
SHCa	110.400	11.612	19.251		93.200	9.803	13.716	
SHCa	110.600	11.633	19.286	0.427	97.200	10.224	14.305	0.223
SHCa	119.000	12.504	20.730		98.400	10.340	14.467	

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Table 5. Na and K contents of the clays.

Sample	Na ppm in solution	% Na in dry sample	% Na_2O	Mean and SD	K ppm in solution	% K in dry sample	% K_2O	Mean and SD
KGa-1	0.470	0.047	0.063	0.050	0.000	0.000	0.000	0.000
KGa-1	0.250	0.025	0.034		0.000	0.000	0.000	
KGa-1	0.380	0.038	0.051	0.006	0.000	0.000	0.000	0.000
KGa-1	0.370	0.037	0.050		0.000	0.000	0.000	
STx-1	1.850	0.204	0.275	0.309	0.500	0.055	0.066	0.073
STx-1	2.100	0.232	0.312		0.500	0.055	0.066	
STx-1	1.940	0.214	0.289	0.018	0.500	0.055	0.067	0.007
STx-1	2.400	0.265	0.358		0.700	0.077	0.093	
KGa-2	0.450	0.045	0.061	0.064	0.200	0.020	0.024	0.024
KGa-2	0.370	0.037	0.050		0.300	0.030	0.036	
KGa-2	0.570	0.057	0.077	0.006	0.100	0.010	0.012	0.005
KGa-2	0.500	0.050	0.068		0.200	0.020	0.024	
SWy-2	10.670	1.145	1.544	1.471	1.600	0.172	0.207	0.197
SWy-2	9.670	1.038	1.399		1.400	0.150	0.181	
SWy-2	10.370	1.119	1.508	0.033	1.500	0.162	0.195	0.006
SWy-2	9.910	1.064	1.434		1.600	0.172	0.207	
PFl-1	0.340	0.037	0.049	0.054	6.800	0.734	0.884	0.874
PFl-1	0.570	0.062	0.083		7.000	0.755	0.910	
PFl-1	0.210	0.023	0.031	0.011	6.600	0.717	0.864	0.015
PFl-1	0.370	0.040	0.054		6.400	0.696	0.839	
SAz-1	0.340	0.039	0.053	0.063	1.200	0.138	0.166	0.188
SAz-1	0.580	0.067	0.090		1.300	0.150	0.180	
SAz-1	0.470	0.054	0.073	0.012	1.500	0.173	0.209	0.009
SAz-1	0.240	0.028	0.037		1.400	0.162	0.195	
Syn-1	0.710	0.073	0.098	0.083	0.000	0.000	0.000	0.000
Syn-1	0.520	0.053	0.072		0.000	0.000	0.000	
Syn-1	0.610	0.063	0.085	0.006	0.000	0.000	0.000	0.000
Syn-1	0.570	0.059	0.079		0.000	0.000	0.000	
SHCa	9.240	0.966	1.302	1.350	1.100	0.115	0.139	0.139
SHCa	9.470	0.996	1.343		1.100	0.116	0.139	
SHCa	9.970	1.049	1.414	0.023	1.100	0.116	0.139	0.000
SHCa	9.470	0.995	1.341		1.100	0.116	0.139	

Table 6. Pe and Si contents of the clays.

Sample	% P blank reduction	% P_2O_5	Mean and SD	Sample weight	Dry weight	Si ppm in solution (250 mL)	% Si in dry sample	% SiO_2	Mean and SE
KGa-1	0.133	0.305	0.373	0.101	0.101	81.370	20.143	43.092	43.356
KGa-1	0.193	0.442		0.101	0.100	84.900	21.121	42.370	
KGa-1	0.177	0.405	0.031	0.101	0.101	82.500	20.483	42.821	0.395
KGa-1	0.148	0.338		0.101	0.101	83.100	20.632	44.139	
STx-1	0.004	0.009	0.010	0.100	0.090	118.200	32.776	70.119	70.035
STx-1	0.005	0.011		0.101	0.091	119.100	32.765	70.094	
STx-1	0.004	0.009	0.001	0.101	0.091	118.300	32.577	69.692	0.118
STx-1	0.004	0.010		0.102	0.092	120.400	32.830	70.234	
KGa-2	0.139	0.318	0.322	0.101	0.100	81.300	20.305	43.439	43.490
KGa-2	0.139	0.318		0.100	0.100	80.300	20.176	43.163	
KGa-2	0.144	0.330	0.003	0.101	0.100	82.500	20.585	44.037	0.191
KGa-2	0.140	0.321		0.101	0.100	81.400	20.250	43.321	
SWy-2	0.001	0.002	0.003	0.101	0.094	103.900	27.687	59.231	61.460
SWy-2	0.002	0.004		0.102	0.094	114.700	30.445	65.130	
SWy-2	0.002	0.004	0.001	0.101	0.094	105.900	28.192	60.311	1.286
SWy-2	0.000	0.000		0.101	0.094	107.300	28.593	61.169	
PFl-1	0.365	0.837	0.920	0.101	0.093	104.100	28.085	60.083	60.347
PFl-1	0.406	0.931		0.101	0.093	105.300	28.269	60.476	
PFl-1	0.426	0.976	0.029	0.101	0.093	106.370	28.613	61.212	0.338
PFl-1	0.409	0.937		0.101	0.093	103.700	27.867	59.616	
SAz-1	0.003	0.008	0.008	0.102	0.087	97.500	27.859	59.600	59.655
SAz-1	0.004	0.010		0.101	0.087	98.050	28.100	60.114	
SAz-1	0.003	0.007	0.001	0.101	0.087	96.840	27.808	59.490	0.158
SAz-1	0.003	0.007		0.102	0.088	97.390	27.773	59.416	
Syn-1	0.000	0.000	0.000	0.100	0.097	90.700	23.435	50.134	49.773
Syn-1	0.000	0.000		0.101	0.097	91.700	23.599	50.485	
Syn-1	0.000	0.000	0.000	0.101	0.098	90.060	23.085	49.386	0.324
Syn-1	0.000	0.000		0.101	0.098	89.780	22.945	49.086	
SHCa	0.000	0.000	0.000	0.102	0.096	82.900	21.498	45.992	46.659
SHCa	0.000	0.000		0.102	0.096	85.100	22.069	47.212	
SHCa	0.000	0.000	0.000	0.101	0.096	82.900	21.583	46.174	0.335
SHCa	0.000	0.000		0.101	0.096	85.100	22.091	47.259	

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Table 7. Total oxide, ignition losses, and the total analyses components.

Sample	% Total oxide	Mean % total	Loss on ignition 550°C	Loss on ignition 550–1000°C	Total
KGa-1	84.193				
KGa-1	82.486	84.463	13.598	1.452	99.512
KGa-1	85.253				
KGa-1	85.558				
STx-1	95.188				
STx-1	95.405	95.282	0.993	3.638	99.913
STx-1	95.198				
STx-1	95.338				
KGa-2	85.113				
KGa-2	84.416	85.163	13.305	1.731	100.198
KGa-2	85.801				
KGa-2	85.323				
SWy-2	91.218				
SWy-2	97.745	93.764	0.612	5.146	99.522
SWy-2	92.494				
SWy-2	93.601				
PFl-1	89.527				
PFl-1	90.407	90.017	7.874	1.646	99.537
PFl-1	90.898				
PFl-1	89.234				
SAz-1	91.940				
SAz-1	92.111	91.789	4.576	3.592	99.957
SAz-1	91.873				
SAz-1	91.233				
Syn-1	89.132				
Syn-1	89.495	88.792	8.648	2.178	99.618
Syn-1	88.523				
Syn-1	88.018				
SHCa	82.939				
SHCa	82.898	83.392	0.839	15.472	99.703
SHCa	82.561				
SHCa	85.171				

The SE is quite large for Si in the SWy-2 sample only. Analytical data for the synthetic montmorillonite, Syn-1, suggest that the results are reliable, because the contents of Fe, Ti, Mg, Ca, Na, K and P are negligible. This sample appeared to be very 'fluffy' and homogeneous with no evidence of impurities. Both kaolinite samples have near ideal Si and Al contents. Considerable amounts of Ti, P and Fe suggest the presence of impurities in both samples. The KGA-2 sample contains more Ti and Fe than KGa-1. Many kaolinites contain titanite together with Fe-rich oxides as impurities, especially those formed in soils under tropical conditions.

The largest amounts of Mg and Ca were found in the SHCa-1 sample. Calcium can exist in exchange sites. Because the cation exchange capacity of hectorite is low, very large amounts of Ca indicate the presence of other minerals in this sample. Guggenheim and Koster van Groos (2001) describe the presence of dolomite and calcite in this sample. As the Mg content is greater than the Ca content, considerable amounts of Mg are likely in the silicate clay structure. The palygorskite (PFl-1) sample also contains small amounts of Ca (calcite) and P, indicating the presence of mineral impurities. Our experience with many palygorskites, including the source clay mineral sample from Florida, indicates that palygorskite commonly contains a small amount of P and other elements. The presence of K can be attributed to K-bearing minerals.

Table 8. Structural formulae of the Source Clay minerals.

	KGa-1	KGa-2	SWy-2	SAz-1	Syn-1
Si	3.85	3.84	7.89	7.86	6.50
Al	0.15	0.16	0.11	0.14	1.50
Σ tet.	4.00	4.00	8.00	8.00	8.00
Al	3.88	3.80	3.23	2.96	4.44
Ti	0.10	0.13		0.02	
Fe^{3+}	0.02	0.07	0.42	0.18	0.01
Mg			0.56	1.32	0.04
Σ oct.	4.00	4.00	4.21	4.48	4.49
Ca	0.005	0.005	0.52	0.88	0.007
Na	0.009	0.01	0.14	0.01	0.01
K		0.002	0.01	0.05	

An attempt was made to calculate the structural formulae of the Source Clay Minerals (Table 8). Owing to large amounts of impurities in STx-1, PFl-1 and SHCa, the calculation was not performed for these minerals. In Table 8, note that some discrepancies exist in comparison to idealized structural formulae, suggesting that there are also impurities in these samples.

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E-mail of corresponding author: mermut@skyway.usask.ca

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