Parent Material Contribution to Jemez River Basin, NM Soils Using Quantitative XRD and XRF

S. Mercer Meding* and Craig Rasmussen

The University of Arizona, Department of Soil, Water & Environmental Science, * smmeding@cals.arizona.edu

INTRODUCTION

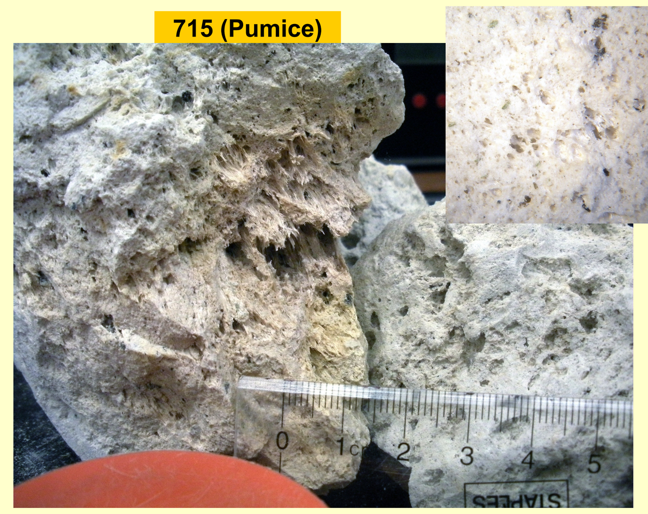
The Jemez River Basin lies within the Valles Caldera National Preserve in New Mexico. The geology of this area includes examples of rhyolite, pumice, and tuffs with some zeolitized rock units. Soils in the Jemez River Basin are part of the critical zone which extends from vegetation heights down to the groundwater depths. Recently, this area was established as part of the University of Arizona led Jemez River Basin-Santa Catalina Mountains Critical Zone Observatory. In order to better understand soil development and processes in the basin, soil and parent material samples were collected near the top of the Redondo resurgent dome and analyzed for mineralogy and chemistry. In order to determine the contribution of individual parent material types to soil sample mineralogy, rock samples were used as standards within quantitative mineralogical software (Rockjock; Eberl, 2009). Rock and soil samples were analyzed for mineralogy using x-ray diffraction. Rock mineralogy was quantitatively determined using a combination of Rockjock (Eberl, 2009) and Rietveld analysis. X-ray fluorescence (XRF) analysis of both rock and soil samples provided elemental compositions. Quantitative elemental ratios were then used to constrain and/or confirm the quantitative mineralogy results.

OBJECTIVES

- Examine soils with mineral quantification software for determining relative parent material contribution to soil horizons while adapting the use of collected parent material rocks as substitutes for mineral phases.
- Compare the quality of quantification using parent material phase substitutes to the use of standard reference mineral phases.
- Compare the quantification of mineral phases in parent material rocks using the Rockjock and Rietveld methods.
- Compare elemental data provided by XRF analysis to quantitative mineralogy results.

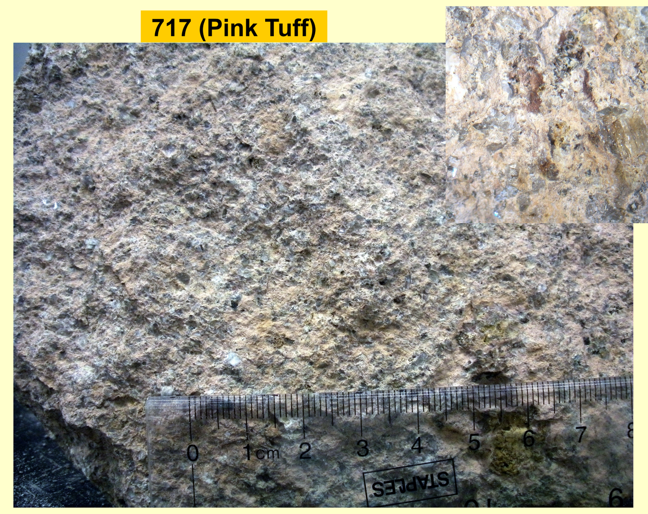
PARENT MATERIAL ROCKS USED IN STUDY

715 (Pumice)



716 (Rhyolite)

717 (Pink Tuff)



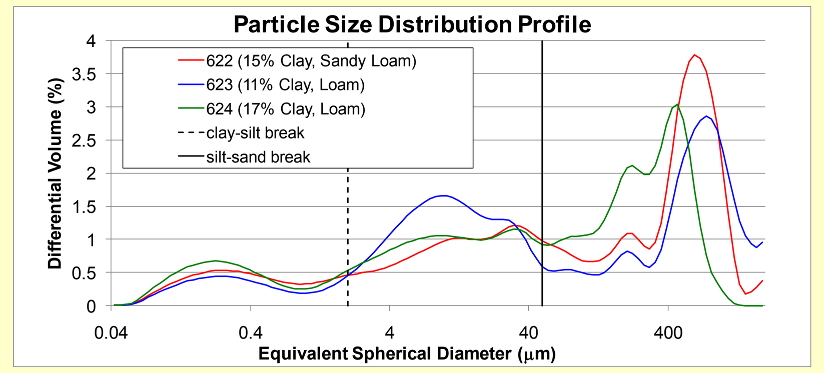
718 (White Tuff)

METHODS

SAMPLE PREPARATION: Samples were air-dried and sieved to 2 mm to remove coarse fragments. For preparation of mineralogical analysis by x-ray diffraction, sub-samples were pretreated to remove organic matter by oxidation with bleach at pH 9.5 (Jackson, 2005). Pre-treated samples were then ground in a McCrone micronizing mill with the addition of 20% corundum internal standard (Eberl, 2009). Samples were prepared as bottom packed random powder mounts and analyzed by x-ray diffraction from 5 to 65 degrees two-theta using Cu K-alpha radiation, a step size of 0.02 degrees two-theta and a count time of two seconds. All x-ray diffractions were carried out on a PANalytical X'Pert Pro x-ray diffractometer. Qualitative and quantitative data analysis of x-ray diffractograms was performed using the PANalytical X'Pert Highscore Plus software (v.2.1b, 2005) or run using Rockjock (v. 7 & 11, 2007 & 2009). Elemental analysis was performed on samples without pre-treatment to remove organic matter. Five grams of sample was ball milled in polypropylene vessels with zirconium grinding elements for fifteen minutes. Four grams of ground sample were mixed with 1g of Licowax C micropowder (Spectro) and pressed into pellets at a pressure of 22 tons for 60 seconds and analyzed using a Polarized Energy-Dispersive X-ray Fluorescence spectrometer (EDXRF – SPECTRO XEPOS, Kleve – Germany). Measurements were carried out under Helium atmosphere and four secondary targets (HOPG, Mo, $AlPO_4$ and Pd) were used to provide different excitation conditions at different voltage and current settings.

ADDITIONAL CHARACTERIZATION

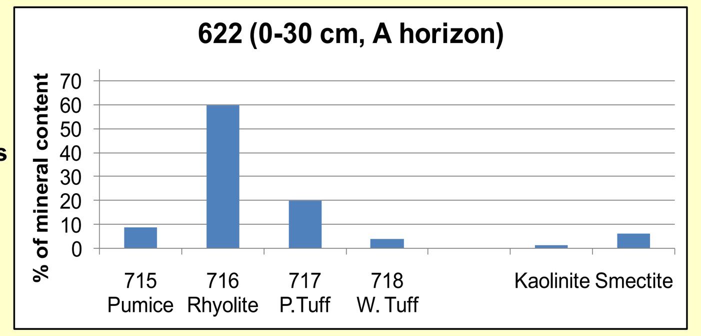
Soils were measured for particle size by laser diffraction (right). Soil pH in 1:1 H_2O was 4.98, 5.36, and 5.56 and EC was 35.1, 25.5, and 28.2 µS for samples 622 to 624 respectively. Dry soil colors were 7.5YR 4/2, 10YR 5/3, and 10 YR 6/3 for samples 622 to 624 respectively.



QUANTITATIVE ANALYSIS OF SOILS USING ROCKJOCK WITH PARENT MATERIAL ROCKS AS REFERENCE PHASES

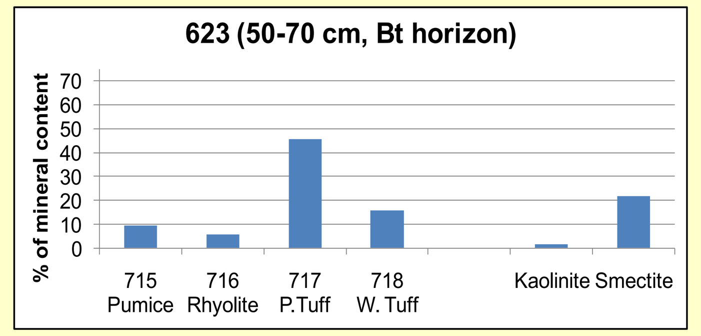
- PERFORMANCE

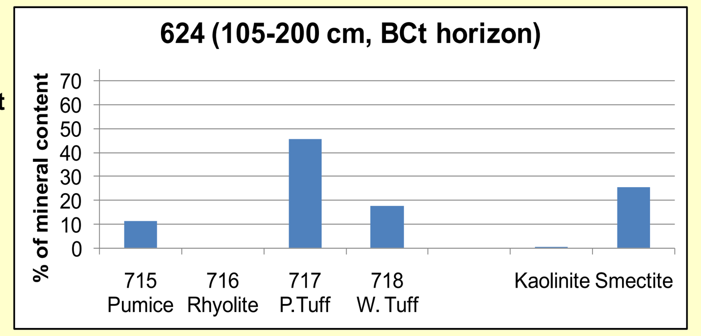
Profile fitting in Rockjock using parent materials as reference phases was quick and provided very acceptable degrees of fit. Degrees of fit were 0.1084, 0.0880, and 0.0962 for samples 622 to 623 respectively.



- PARENT MATERIAL CONTRIBUTION TO SOIL

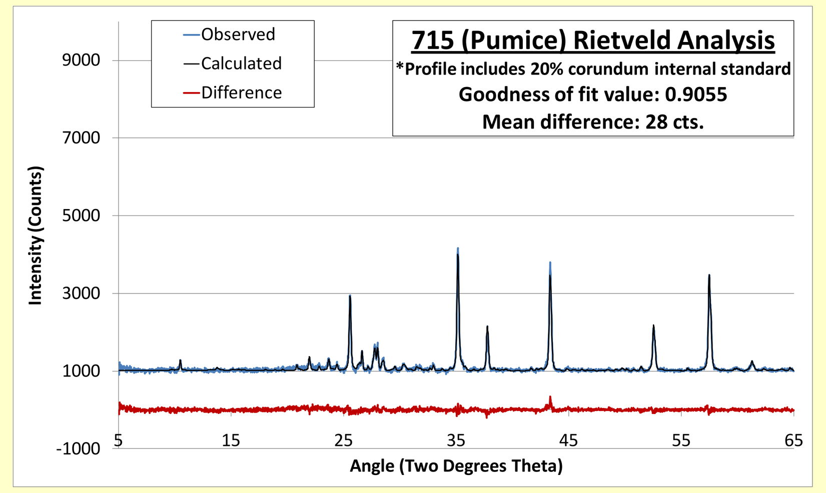
Relative parent material contributions to the three soil horizons examined, as inferred by the use of parent material reference phases in Rockjock quantification, showed a dominance of rhyolitic material present in the A horizon. Samples of tuff parent material appear to contribute more to soil development in the two lower horizons. Parent material contribution to untransformed soil material was shown to be 71 % in the A horizon, 82 % in the Bt horizon, and 70 % in the BCt horizon.

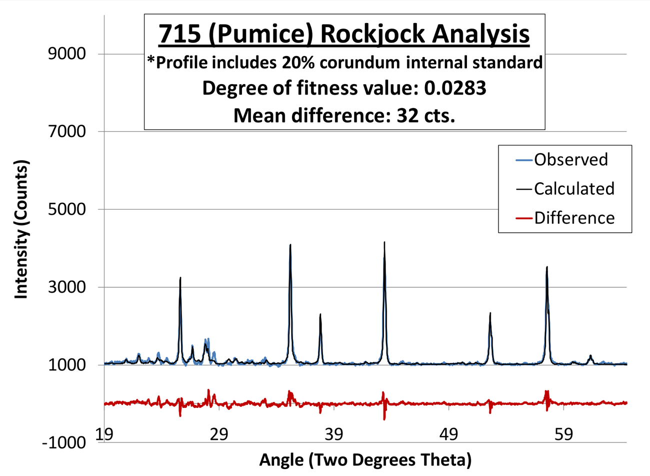


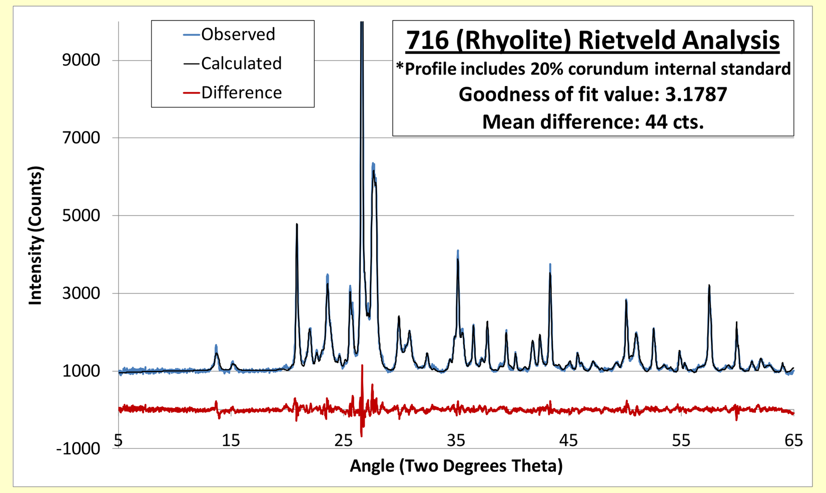


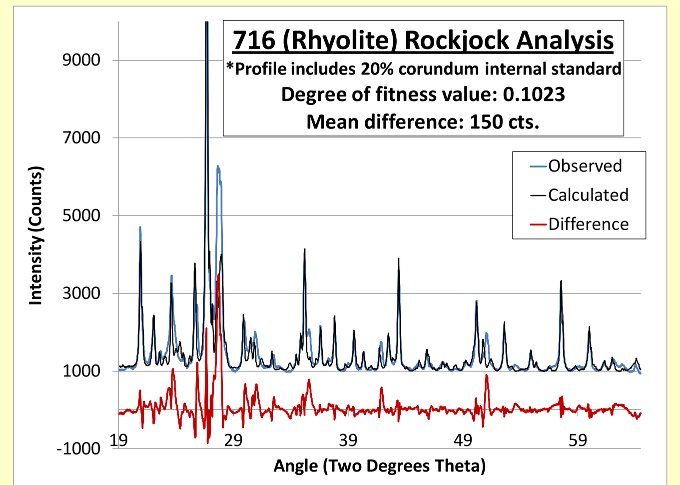
QUANTITATIVE ANALYSIS OF MINERAL PHASES IN PARENT MATERIAL ROCKS: ROCKJOCK VERSUS RIETVELD

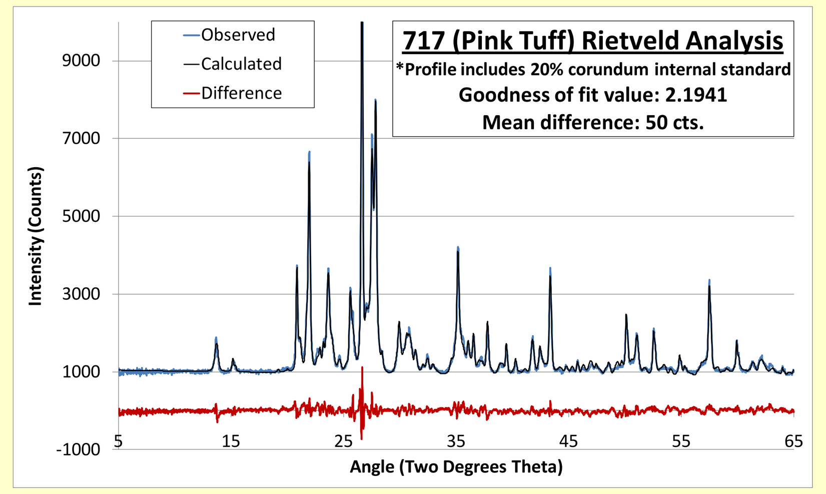
Both techniques provided good quantitative mineralogical results for the four parent material rocks analyzed as shown by the goodness of fit or degree of fitness values. The Rietveld method performed better based on the simulated profile matches. XRF elemental data from the four parent material rock samples was used to compare to estimated elemental concentrations from the two quantitative mineralogy methods. Estimated elemental concentrations were typically within 2 % of the measured values with the exception of the Rockjock Si estimations on samples 715 (3.1 % low) and 718 (4.5 % low). Rietveld elemental estimations were closer than Rockjock estimations for samples 715, 716, and 718.

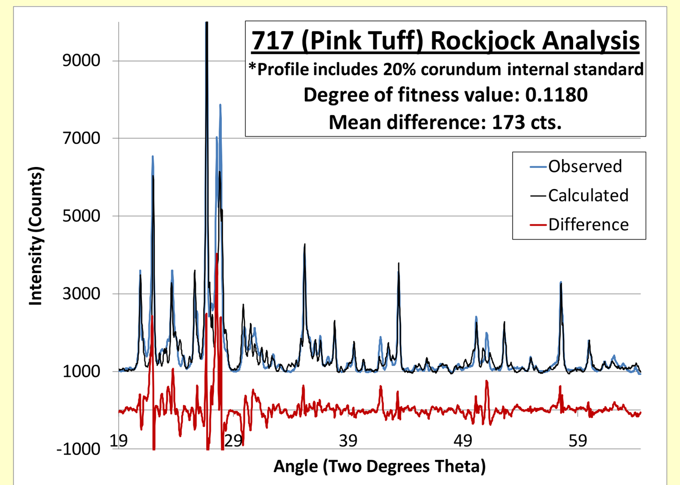


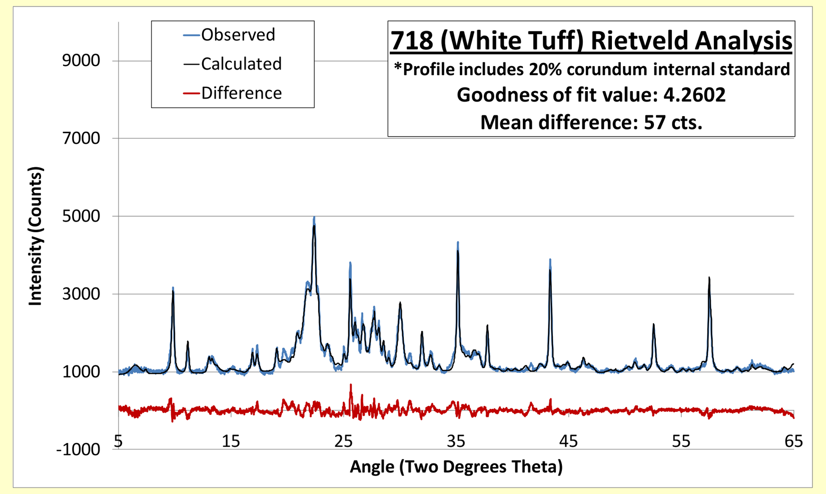


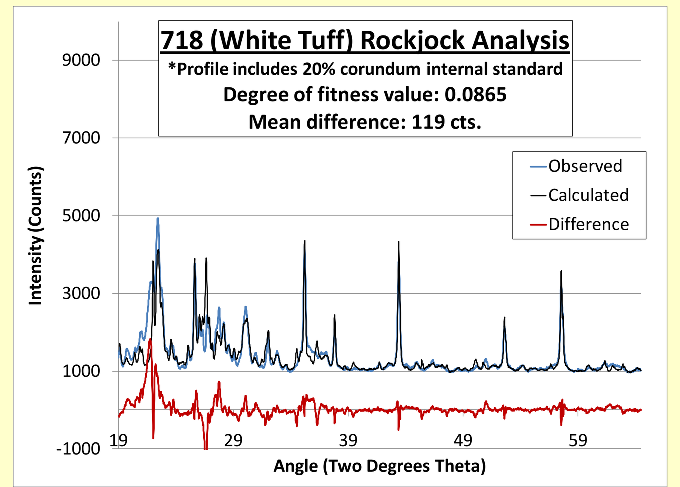












PARENT MATERIAL QUANTITATIVE MINERALOGY

Quantitative mineralogy of the parent material rocks was based on Rietveld analysis due to the methods comparison shown in the previous section. The selected quantitative results are shown in the table to the right. Rietveld analysis combined with a corundum internal standard allowed for amorphous content determination in the samples. The pumice sample was almost entirely amorphous. Amorphous content did not appear to effect elemental estimates, suggesting the amorphous chemistry was similar to combined rock mineral constituent chemistry.

715 Pumice		716 Rhyolite		717 Pink Tuff		718 White Tuff	
Mineral Phases (Percent of Mineral Content)							
Quartz	10.34	Quartz	28.27	Quartz	22.46	Quartz	4.53
Orthoclase	6.90	Sanidine	27.26	Cristobalite	6.08	Cristobalite	16.34
Anorthoclase	65.52	Albite	35.30	Sanidine	25.43	Sanidine	21.26
Tremolite	17.24	Oligoclase	9.17	Anorthoclase	22.21	Albite	0.39
				Oligoclase	23.82	Clinoptilolite	45.08
						Mordenite	12.40
Amorphous Content (Percent of Total Sample)							
Amorphous	92.75	Amorphous	0	Amorphous	0	Amorphous	36.50

SOIL QUANTITATIVE MINERALOGICAL COMPARISON BETWEEN ROCKJOCK WITH PARENT MATERIAL REFERENCES (PM) VERSUS ROCKJOCK WITH STANDARD MINERAL PHASES (STD) OR VERSUS THE RIETVELD METHOD

- CHEMISTRY COMPARISON

Major element concentrations estimated by the three quantitative mineralogy techniques used here are compared to measured values for all three soils to the right. Generally, the techniques combining the use of parent material references in Rockjock with Rietveld analysis of parent materials provides the best comparative estimates.

622 (0-30 cm, A horizon)	Measured Chemistry	Chemistry Estimated from Mineralogy		
Element	XRF	PM Phase Rockjock	Standard Phase Rockjock	Rietveld
	(%)	(%)	(%)	(%)
Si	34.6	34.4	35.4	37.1
Al	7.6	7.8	7.0	6.0
Fe	2.2	0.2	0.2	0.1
Mg	0.1	0.3	0.2	0.2
Ca	0.3	0.5	0.3	0.4
K	2.5	4.5	4.9	3.0
Na	3.4	2.9	2.1	3.0
Absolute Difference		5.2	7.4	7.1

623 (50-70 cm, Bt horizon)	Measured Chemistry	Chemistry Estimated from Mineralogy		
Element	XRF	PM Phase Rockjock	Standard Phase Rockjock	Rietveld
	(%)	(%)	(%)	(%)
Si	30.8	33.1	32.3	33.7
Al	8.9	8.4	9.1	8.2
Fe	3.5	0.5	0.4	0.3
Mg	0.3	0.6	0.5	0.3
Ca	0.4	0.9	0.6	0.3
K	2.4	3.8	5.2	3.9
Na	2.8	2.2	2.1	3.0
Absolute Difference		8.6	8.5	8.7

624 (105-200 cm, BCt horizon)	Measured Chemistry	Chemistry Estimated from Mineralogy		
Element	XRF	PM Phase Rockjock	Standard Phase Rockjock	Rietveld
	(%)	(%)	(%)	(%)
Si	32.7	33.0	32.5	35.3
Al	8.2	8.4	8.8	6.8
Fe	2.8	0.6	0.4	0.4
Mg	0.2	0.7	0.6	0.4
Ca	0.4	1.0	0.6	0.5
K	2.5	3.8	5.1	3.0
Na	3.0	2.1	2.0	3.7
Absolute Difference		5.9	7.3	8.0

- MINERAL COMPARISON

Including all the previously identified mineral phases in the quantitative analysis of the three soils using either Rockjock or Rietveld did not provide accurate matches to the results obtained using the parent material reference method. In the case of Rockjock, relative contributions between feldspars were assigned differently, but otherwise matched well. The Rietveld software could not handle the abundance of similar phases and also lacked the availability of quality crystal data from the smectite group. Simplifying the mineral groupings (below right) greatly improved the mineral comparison with results obtained by Rockjock.

	622 (A horizon)			623 (Bt horizon)			624 (BCt horizon)		
Mineral Phase	Rockjock PM	Rockjock Std	Rietveld	Rockjock PM	Rockjock Std	Rietveld	Rockjock PM	Rockjock Std	Rietve
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
Quartz	22	29	34	14	9	14	12	9	15
Cristobalite	2	1	5	5	3	9	6	3	14
Orthoclase	1	23	5	1	22	4	1	23	3
Sanidine	22	0	5	17	0	12	15	0	5
Anorthoclase	10	25	20	16	29	16	18	27	19
Albite	21	4	17	2	3	26	0	3	26
Oligoclase	10	3	8	11	1	1	11	1	8
Clinoptilolite	2	5	1	7	6	2	8	6	0
Mordenite	0	0	2	2	1	3	2	2	5
Tremolite	2	1	2	2	0	3	2	1	4
Kaolinite	1	2	1	2	1	9	0	0	0
Smectite	6	7	0	22	24	0	25	24	1
Absolute Difference from Rj-PM		96	61		75	83		70	91

	622 (A horizon)			623 (Bt horizon)			624 (BCt horizon)		
Mineral Group	Rock jock PM	Rock jock Std	Riet-veld	Rock jock PM	Rock jock Std	Riet-veld	Rock jock PM	Rock jock Std	Riet-veld
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
SiO_2	24	30	39	19	12	23	18	13	29
Feldspar	64	55	55	47	55	58	45	54	62
Zeolite	2	5	4	9	8	6	10	8	5
Amphibole	2	1	2	2	0	3	2	1	4
Clay	7	9	1	23	25	9	26	25	1
Abs. Diff.		19	33		20	34		19	60

CONCLUSIONS

Using collected parent material samples as references in Rockjock to determine their relative contributions to local soils and to help improve quantitative mineralogy results was found to be very useful. Different parent material types were found to contribute differently to soils varied by depth/horizon. Continued use of this technique will be applied to soils collected from different locations and depths of the study site. Based on comparisons with measured elemental data, the quantitative mineralogical estimates were improved by using the parent material reference technique. Rietveld analysis proved to be more reliable than Rockjock for mineral quantification of rock samples. The Rietveld analysis had shortcomings when attempting to analyze soils with abundant similar mineral phases and for the estimation of smectite minerals.

REFERENCES

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