A semi-quantitative x-ray diffraction technique for estimation of smectite, illite, and kaolinite
by Roger W E Hopper

A thesis submitted in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE in Soils
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Abstract:
Two studies are reported.

I. An assessment of the major sources of error in the X-ray diffraction procedure was conducted using a nested design and ANOVA for peak area and clay mineral composition. Clay separation, slide preparation and slide positioning were significant sources of error.

II. Modifications of the factor method for semi-quantitative characterization of clay mineral composition by X-ray diffraction analysis were tested. Samples used in the study were from early Tertiary aged sediments of the Fort Union Formation and associated soils in Southeastern Montana. Estimates of the total CEC of the clay-sized fraction were based on X-ray diffraction results. The accuracy of estimation for each modification was tested by linear regression comparing these estimates with measured CEC values. Variation in measured CEC explained 90% of the variation in estimated CEC, 92% of the variation in smectite composition, and 82% of the variation in kaolinite composition. Percent illite was compared with illite content estimated by total K analysis. Variation in measured illite content accounted for 74% of the variation in estimated illite content.

A modification of the factor method is presented that provides relatively fast and reasonably accurate estimations of percent smectite, illite, and kaolinite for material that does not contain significant portions of vermiculite or chlorite.

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Date October 28, 1981

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by

ROGER W E HOPPER

A thesis submitted in partial fulfillment
of the requirements for the degree

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ABSTRACT

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I. An assessment of the major sources of error in the X-ray diffraction procedure was conducted using a nested design and ANOVA for peak area and clay mineral composition. Clay separation, slide preparation and slide positioning were significant sources of error.

II. Modifications of the factor method for semi-quantitative characterization of clay mineral composition by X-ray diffraction analysis were tested. Samples used in the study were from early Tertiary aged sediments of the Fort Union Formation and associated soils in Southeastern Montana. Estimates of the total CEC of the clay-sized fraction were based on X-ray diffraction results. The accuracy of estimation for each modification was tested by linear regression comparing these estimates with measured CEC values. Variation in measured CEC explained 90% of the variation in estimated CEC, 92% of the variation in smectite composition, and 82% of the variation in kaolinite composition. Percent illite was compared with illite content estimated by total K analysis. Variation in measured illite content accounted for 74% of the variation in estimated illite content.

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INTRODUCTION

X-ray diffraction methods are central to studies of the clay fraction of soils with the exception of those soils suspected to contain large amounts of amorphous material. A study of the components of the clay fraction of soil must begin with the proper identification of the minerals present. Quantitative estimations of the clay mineral components have applications to many disciplines.

Several methods have been employed to make quantitative estimations of clay minerals in soil investigations. The factor method used in making the investigations reported here has the advantages of being relatively rapid and precise. However, estimations obtained are relative values. Consequently, the method has been referred to as semi-quantitative. Few investigations report tests of the accuracy of quantitative estimations. McNeal [31] used a combination of chemical and X-ray diffraction methods to make quantitative estimations of the mineralogy of arid and semi-arid land soils.

The objective of this study was to derive a relatively fast and reasonably accurate method of determining clay mineral composition of soils and associated parent material for application to large numbers of samples. Ten modifications of the factor method for semi-quantitative characterization of clay mineral composition by X-ray diffraction analysis were tested for accuracy and precision. Appropriate factors were determined. Estimations of relative mineral composition and cation exchange capacity derived from X-ray diffraction results were tested against cation exchange capacity values obtained by chemical methods. Estimates of the percent illite derived from X-ray diffraction analysis were tested against percent illite values obtained from total potassium analysis.

In addition an attempt was made to assess the major sources of error in the method of X-ray diffraction analysis used in this study, and to identify the diffraction maxima that may be measured with the greatest precision.

LITERATURE REVIEW

Quantitative applications are firmly based on sound theoretical considerations. However, almost every procedural step in X-ray diffraction methods may be considered as a potential source of error [16,43,32,39]. In this review of pertinent literature, an attempt is made to briefly survey the major procedural steps in making quantitative clay mineral estimations by X-ray diffraction methods. These steps may be identified as: (1) sample dispersion and particle size segregation, (2) sample preparation and presentation, and (3) quantitative estimation of clay mineral components.

Sample Disperson and Particle Size Segregation

Day [7], Kunze [27], Kittrick and Hope [23], Jackson [18] and Watson [53] provide reviews of procedures applicable to sample dispersion. It is generally accepted that cementing agents and free oxides and salts should be removed to some extent to aid in dispersion. Apparent disagreement does exist, however, as to the severity of the pretreatment required.

The procedures described by Jackson [18] are generally rigorous. It has been demonstrated that less severe pretreatments are adequate to obtain satisfactory sample dispersion and X-ray diffraction results [23]. Several authors have found that sample pretreatment can seriously affect apparent clay mineral composition. Harward, Theisen, and Evans [17] compared the effects of several different dispersion methods. Generally, they found that, although iron removal enhanced dispersion, it also resulted in significant differences in apparent clay mineral composition. More rigorous iron removal and dispersion treatments generally resulted in a greater number of clay minerals identified, however, this was also dependent upon the soil itself.

The choice of the proper combination of pretreatment and dispersion methods remains to the discretion of the investigator. A combination of methods might be most worthwhile. While it is important to identify the maximum number of mineral components present, it is also advantageous to use those methods which retain the real mineral assemblages as they exist *in situ* [17].

Use of ultrasonic vibrations to obtain sample dispersion may eliminate need for drastic pretreatment. Olmstead [37] found that sonic vibrations could be used in conjunction with chemical treatments to obtain stable dispersed suspensions of soil colloids. However, his work was largely overlooked for almost thirty years. Recently Edwards and Bremner [9,8] found, using soils having a wide range of characteristics, that sample dispersion could be obtained with most soils using only distilled water, thus reducing both the time involved in treating the sample and the possibility of destruction of natural mineral structure. This work has been corroborated by Genrich and Bremner [12]. Vladimirov [52] suggested the value of ultrasonic dispersion methods in studying highly calcareous soils where chemical treatments disallow a particle size investigation of carbonate salts. Emerson [10] found that sodium hexametaphosphate improved dispersion of soils particularly high in organic matter or soluble salts. In most cases it has been reported that abrasion of minerals is lower using ultrasonic methods than with either shaker or mixer methods of mechanical dispersion except in the case of biotite [9].

Particle size segregation may be obtained by settling or centrifugation [18]. Tanner and Jackson [48], in considering settling and centrifugation techniques, have published nomographs by which the sedimentation of particles having a particular effective radius may be predicted according to time temperature, particle density and centrifuge

speed. Procedures employing density gradient centrifugation and heavy liquid techniques have not received much attention in clay mineralogy as yet. Towe [51] suggested these latter techniques while critically considering the use of the less-than-2-micron particle size fraction in making typical clay mineral studies. Towe seriously questioned the ability of current sedimentation and centrifuge techniques to yield accurate representative samples of this size fraction based on inherent differences in particle density and settling times.

Sample Preparation and Presentation

Preferential orientation is rather easily obtained because of the shape of most layer-silicate minerals. Orientation results in the enhancement of basal (001) diffraction maxima and thus permits greater sensitivity to small amounts of the mineral components present [27].

The length of the specimen irradiated and the depth to which the X-ray beam penetrates are functions of the angle at which the X-ray beam intersects with the sample (θ). The length of the irradiated specimen (L) may be calculated by the relationship:

$$L = \alpha R/2\sin\theta \ ,$$

where α represents the divergence slit width (in radians) and R represents the radius of the goniometer [38]. The irradiated specimen length increases rapidly at smaller angles of θ. This results in a maximum d-spacing that may accurately be measured and a minimum sample length. It is interesting to note that the majority of studies reported in the literature use CuKα radiation in conjunction with a divergence slit width of 1° to study soil clay minerals. According to the values reported by Parrish [38] the maximum d-spacing accurately measured under these conditions is 5.2A, a value well below even the relatively small c dimension of the kaolinite minerals (approximately 7A).

Cullity [6, pp. 269-272] described the effective depth of X-ray penetration in terms of the fraction (Gx) of the total diffracted intensity contributed by a surface layer of a certain thickness (x) by the relationship:

$$Gx = (1 - e^{-2\mu x/\sin\theta}),$$

where μ represents an appropriate mass absorption coefficient. Gibbs [13] used this equation to calculate penetration values. The need for a uniform sample in which no particle segregation has occurred is imperative.

In a comparison of several techniques Gibbs [13] found that particle segregation was best avoided by smear-on-glass techniques as described by Theisen and Harward [50] and suction-on-ceramic tile techniques described by Kinter and Diamond [22]. Centrifuge methods for deposition on either ceramic tiles or glass were found to cause particle size segregation and thus bias estimates toward the finer grained smectite minerals.

An additional consideration in preparing oriented samples is the degree of orientation that is actually achieved. Departure from the preferred orientation can cause a reduction in the peak intensity. Taylor and Norrish [49], using the suction-on-ceramic tile method reported significant variations in the degree of preferred orientation between specific minerals. They also found that variations in the degree of orientation for specific minerals may vary between duplicates. Quakernaat [41] reported relatively low absolute orientation for all minerals studied using a suction-on-plastic membrane technique. Schultz [44], in a study of kaolinite-illite mixtures using a smear-on-glass technique, found that the degree of preferred orientation in pure kaolinite samples was greater than the orientation of either kaolinite or illite in mixed samples. However, for any one mixture,

the preferred orientation of the kaolinite and illite was about the same. He concluded that the effect of orientation was eliminated within a single slide.

At present no clear advantage is held by either the smear-on-glass or the suction-on-ceramic tile techniques in comparison with each other [50].

Quantitative Estimation of Clay Mineral Components

Quantitative X-ray diffraction methods fall into three basic approaches described here as: (1) the Theoretical Method, (2) the Standard Clay Mixture Methods, and (3) the Factor Method.

The Theoretical Method. The work reported by Alexander and Klug and their associates [1,24,25] form the theoretical basis for current quantitative methods. In its simplest form, the method of Alexander and Klug [1] reduces to:

$$I_p/I_{o,p} = w_p ,$$

where I_p equals the diffraction intensity of the P component in a multiphase mixture, $I_{o,p}$ equals the diffraction intensity of the P component in pure form (the external standard), and w_p equals the weight fraction of the P component in the mixture. This method assumes that the mass absorption coefficient of the P component (μ^*_p) is equal to the mass absorption coefficient of the matrix containing the rest of the components of the mixture (μ^*_m). This assumption is not strictly true and can lead to large errors. Leroux, Lennox, and Kay [28] attempted to correct for this by extending Eq. 1 to include a ratio of the mass absorption coefficient of the P component to the average mass absorption coefficient of the mixture ($\bar{\mu}^*_m$):

$$I_p/I_{o,p} = w_p(\mu^*_p/\bar{\mu}^*_m) .$$

Tabulated values of μ_p^* for several minerals are available [3]. Assuming an investigator has previously determined $I_{o,p}$, the application of this technique requires only a measurement of I_p and $\bar{\mu}_m^*$. Williams [57] provided an improved method for determining the average mass absorption coefficient.

The Standard Clay Mixtures Method. Methods using mixtures of standard clays have been applied through two basic avenues for quantification: (1) the calibration curve approach and (2) the empirical factor approach.

The calibration curve approach uses mixtures of known weighed amounts of standard clay minerals to calibrate the method. Probably the most extensive use of this technique was that of Willis, Pennington, and Jackson [58]. They used 141 standard clay mixtures containing either 2, 3, 4, 5, or 6 components based on their conception of the weathering sequence of clay size material. Talvenheimo and White [47] used a diffractometer in developing a standard clay mixture method for multiphase system containing kaolinite, illite, and bentonite. With this technique they reported 5 to 10% accuracy.

Internal standards have been employed in the calibration curve approach. Compounds such as MgO, LiF, and CaF_2 having low absorption coefficients and high symmetry are normally used [3] so that small amounts may be incorporated in the sample to be measured without disrupting the desired degree of orientation. The internal standard method of quantitative analysis is based upon the ratio of the integrated intensity of a component in a clay mixture with the integrated intensity of an internal standard added to the mixture in a constant amount. Calibration curves are normally prepared using synthetic mixtures of standard clay samples together with a constant amount of the internal standard. The use of an internal standard circumvents the need to know mass absorption

coefficients or crystal lattice parameters. Because of this advantage the method has been applied to the study of soil clays by several investigators. Many of these investigations were done using photographic techniques on random powder mounts [55,19]. In a more recent study, Glenn and Handy [15] applied the internal standard method using a diffractometer.

Orientation problems have been approached by Quakernaat [41] by the use of molybdenite as an orientation indicator. Compensating for deviations from preferred orientation, he set up quantity intervals using standard mineral mixtures. In determining quantities of kaolinite, illite, and smectite, he claimed an accuracy of about 7 percent. Estimations of chlorite, vermiculite, and pyrophyllite were within about 10 percent accuracy.

The empirical factor approach is best exemplified by the work of Schultz [44,45]. Basically this method uses standard clay minerals in binary combinations to obtain ratios of integrated diffraction intensities for two minerals. These ratios were than applied to a multiphase mixture to characterize the peak intensities to obtain relative clay mineral compositions. As stated previously, Schultz recognized that such factors not only resulted from characteristics of the composition and lattice structure of the minerals, but from orientation effects as well. Schultz reported that in 50/50 mixtures by weight of several kaolinites to Fithian Illite, the ratio of the integrated intensities was approximately 1/1. He found no consistent ratio for chlorite minerals. In a similar study Moore [33] reported the accuracy to be within 2 percent of the actual values.

The major problem shared by the methods employing mixtures of standard clays is the difficulty faced in obtaining mineral standards that are comparable to the clays naturally occurring in soils. Gibbs [14], however, has reported a technique in which he

obtained standard minerals directly from the samples to be studied. Coupling the approach of Schultz as described above together with an internal standard method, he avoided the problems of absorption and crystallinity differences between the standards and the unknowns.

The Factor Method. The factor method incorporates the use of an empirical multiplication factor by which measured peak intensities or integrated intensities are characterized. These factors may be derived experimentally as in the case of studies reported by Weaver [54] and Freas [11], or by calculations based on chemical and crystal lattice parameters [4,42]. The method has several advantages in that it is relatively rapid and generally has good precision. Any of the diffraction maxima may be used in the calculations along with a careful and reasonable choice of multiplication factors.

The method as outlined by Johns, Grim, and Bradley [20] is probably the most often cited of all quantitative procedures. Basically it uses illite somewhat like an internal standard. The integrated intensities of the diffraction maxima of the other minerals are then related to the integrated intensity of the illite peak by appropriate multiplication factors. They used two illite peaks for comparison purposes. The 10A illite peak was multiplied by 4 to allow direct comparison with the 17A peak of smectite. The 3.3A peak was compared directly with the 3.5A maximum for chlorite and kaolinite. Heat treatments were used to discern minerals which occur concurrently in a peak. An apparently arbitrary correction for quartz was applied with the 3.3A peak of illite.

Similar applications of this method have been reported by several authors and differ from the method of Johns *et al.* by either the method used to determine peak intensity

or integrated intensity, in the multiplication factors used, and/or in the diffraction maxima being measured. Weaver [54] used a factor of 2.5 in comparing the 7A peak with the 10A peak for the determination of kaolinite. Freas [11], on the other hand, in comparing all minerals present to the (001) diffraction maximum of kaolinite at 7A, used factors of 3, 3, and 1 for comparison with the (001) reflections of illite, chlorite, and smectite, respectively. Biscayne [2] in comparing all minerals to the 17A peak of montmorillonite used a factor of 4 for the 10A peak of illite and a factor of 2 for a comparison with the 7A peak. The relative composition of chlorite and kaolinite was further discerned by using the doublet occurring near 3.5A. Meade [29] assumed that smectite, kaolinite, and illite reflected X-rays at the same intensity. In addition, he used different intensity factors for Type A chlorite (x2) and Type B chlorite (x 1.5) at 7A for comparison with the 10A peak of illite. The method of Keller and Richards [21] is closely similar to that of Johns *et al.* with the exception that a factor of 3 was used to compare the 17A peak to the 10A peak. Neiheisel and Weaver [34] used a factor of 2 for comparing the 17A and 7A peaks with the 10A peak.

MATERIALS AND METHODS

MAIN STUDY--QUANTIFICATION

Samples

The fifty samples used in this study were obtained from the Decker Coal Company, Decker, Montana. The material consists of early Tertiary aged sediments of the Fort Union Formation, together with soil formed on this moderately indurated material.

The samples were chosen on the basis of clay mineral composition estimated from preliminary X-ray diffraction analysis in an effort to obtain a wide range of clay mineral composition. The description of those samples used in this study appeared in Table 1.

Sample Preparation

The samples were first ground to pass a 2mm sieve. Sample dispersion was obtained by using a probe-type ultrasound machine (120 volts, 4 amps, 60 cycles) manufactured by Blackstone Ultrasonics, Inc. Ten grams of each sample were placed in 50 ml of 0.01% Na_2CO_3 and subjected to ultrasound for 2 minutes. Excessive heating of the samples was experienced using longer periods of dispersion.

Stock clay-sized ($< 2\mu$) particle suspensions were prepared for each sample by five washings using 0.01% Na_2CO_3, centrifuging at 500 RPM according to the nomographs of Tanner and Jackson [48], and saving the supernatant from each wash. Four 25 ml sub-samples were removed from these stock suspensions and prepared for X-ray diffraction analysis as described below. The remaining stock suspensions were saturated with calcium by centrifuge washing three times with N $CaCl_2$. Excess salt was removed by simple dialysis until a test for chloride was negative. Upon completion of dialysis the samples were air dried and hand ground with an agate mortar and pestle to pass a 60 mesh sieve.

Table 1. Sample Identification and Description

Sample No.	Lab. Ident. No.	Description (Depth in Feet)	Sample No.	Lab. Ident. No.	Description (Depth in Feet)
1	44518	0 - 2	26	45257	54 - 60
2	44519	2 - 5	27	45258	60 - 65
3	44520	5 - 11	28	45261	75 - 77
4	44521	11 - 15	29	45262	129 - 135
5	44522	15 - 20	30	45263	135 - 140
6	44523	20 - 23	31	45264	140 - 145
7	44524	23 - 27	32	1235-4	55 - 60
8	44525	27 - 29	33	1235-5	60 - 66
9	44528	40 - 45	34	1235-6	73 - 78
10	44529	45 - 50	35	1235-8	103 - 107
11	44530	50 - 55	36	1235-9	107 - 117
12	44531	55 - 60	37	1235-10	117 - 126
13	44532	60 - 65	38	1237-1	5 - 10
14	45245	0 - 2	39	1237-2	10 - 20
15	45246	2 - 5	40	1237-3	20 - 25
16	45247	5 - 11	41	1237-4	25 - 35
17	45248	11 - 15	42	1237-5	35 - 45
18	45249	15 - 20	43	1237-6	45 - 50
19	45250	20 - 25	44	1237-8	60 - 70
20	45251	25 - 28	45	1237-10	98.8 - 105.3
21	45252	28 - 34	46	1237-11	105.3 - 110.0
22	45253	34 - 40	47	1237-12	111.8 - 120
23	45254	40 - 44	48	1237-13	120 - 130
24	45255	44 - 49	49	1256-2	42 - 52
25	45256	49 - 54	50	1256-12	138 - 142

Total Potassium Determination

Duplicate 0.0500 g clay samples were weighed. The HF-$HClO_4$ decomposition method was employed as suggested by Pratt [40]. The extract was diluted to 100 ml. so that the resulting solution contained 0.5% Sr as $SrCl_2$. The concentration of potassium ions in solution was determined by atomic absorption. The results were reported in terms of illite, expressed as a percentage of the total clay fraction as calculated assuming 8.3%

[30] and 5.1% [54] elemental potassium per unit cell illite. The resulting estimations of the percent illite were tested against the percent illite estimated by X-ray diffraction analysis using linear regression methods.

Cation Exchange Capacity Determination

Free carbonates were removed from the dry Ca-saturated clay samples by a modification of the method described by Jackson [18]. The modification involved four centrifuge washings with normal sodium acetate buffer (pH 5.0) without heating the sample. Following carbonate removal air-dried Ca-saturated clay samples were prepared by the method previously described.

The cation exchange capabilities of the clay samples were determined by a Ca//Mg exchange system. Duplicate 0.050 g samples were centrifuge washed four times using 10 ml aliquots of *N* $MgCl_2$, saving the supernatant following each wash. The resulting extract was diluted to 50 ml so that the resulting solution additionally contained 0.5% Sr as $SrCl_2$. The concentration of Ca in the extract was determined by atomic absorption and the results reported in terms of meq/100 gm of clay.

X-ray Diffraction Analysis

One subsample of each clay sample was saturated with Mg by centrifuge washing three times with 25 ml aliquots of *N* $MgCl_2$. Excess salt was removed by washing twice with distilled water. A second subsample was saturated similarly with potassium using *N* KCl. Excess salt was removed by washing once with distilled water followed by a second wash with 50% ethanol. Subsamples were duplicated for each clay sample.

Parallel oriented samples were prepared by the paste method of Theisen and Harward [50]. The Mg-saturated samples were ethylene glycol solvated by the condensation method described by Kunze [26]. K-saturated samples were heated to both 350°C and 550°C for three hour periods.

X-ray diffraction analysis was carried out on a General Electric XRD-5 diffractometer using Ni filtered CuKα radiation at 45Kv and 18ma with beam and detector slit widths of 1° and 0.2°, respectively. Medium range collimating assemblies were used for both the incident and reflected beams. Scanning speed of the goniometer was 2° 2θ per minute and the chart speed was 1 inch per minute, giving a 2° 2θ per inch diffractogram scale for all samples. All Mg-saturated, ethylene glycol solvated samples were scanned through a 2θ range of 2°-30°. A 2°-15° 2θ range was used for K-saturated samples for both heat treatments.

The criteria used to identify the clay minerals present in the samples were taken from [18], [56], and [5] and are as follows:

Mineral Group	Identification Characteristics
Smectite	d(001) maximum at approximately 17A under Mg-saturation and glycol solvation. K-saturation together with heat treatments cause progressive collapse of interlayer space resulting in a d(001) maximum at approximately 10A for the K-saturated, 550°C heat treatment.

Vermiculite	d(001) maximum at approximately 14A under Mg-saturation and ethylene glycol solvation. Total collapse of the interlayer space and a consequent d(001) maximum of approximately 10A result from K-saturation together with heat treatments.
Chlorite	d(001) maximum at approximately 14A for all treatments. d(002) maximum may or may not be present in the K-saturated, 550°C heat treatment.
Illite	d(001) maximum at approximately 10A for all treatments.
Kaolinite	d(001) maximum at approximately 7A and a d(002) maximum at 3.5A for all treatments except K-saturated, 550°C heat treated samples. On heating to approximately 550°C the mineral reported here as kaolinite becomes amorphous to X-rays due to the collapse of crystalline structure.
Quartz	a diffraction maximum at approximately 3.3A and coincides with an accompanying d(003) maximum of illite.

Peak intensities of the d(001) reflections were measured to a hand-drawn background line. The areas under the peaks were estimated by multiplying the peak height by the width of the peak at half the peak height [36], as illustrated in Fig. 1.

Characterization of the minerals followed the factor method of Johns, Grim, and Bradley [20] as modified by Wilding [59]. Modifications in this method involved both the peaks and factors used to characterize the minerals considered. First order basal reflections were used in the characterization of all clay minerals. The 3.3A reflection was used to characterize quartz. Often the 14A peak of the Mg-saturated, ethylene glycolated slide appears as a shoulder on the high angle side of the 17A peak. In such cases the low angle side of the 14A peak was estimated, as in Fig. 1, and the area calculated. The area of the 17A peak was then corrected by subtracting the area of the 14A peak from the area of the 17A peak.

Ten modifications were tested involving different factors for smectite and kaolinite. Computer programs were used to complete the characterization. The following computations were used to calculate characterized peak areas:

Modification I.

17A Mg-sat. E.G./4 = Smec. Peak Area
(14A Mg-sat. E.G. minus 14A K-sat. 350°C)/2
= Verm. Peak Area
14A K-sat. 350°C/2 = Chlor. Peak Area
10A Mg-sat. E.G./1 = Ill. Peak Area
(7A Mg-sat. E.G. minus 7A K-sat. 550°C)/4
= Kaol. Peak Area
(3.3A Mg-sat. E.G. minus 3/4(10A Mg-sat. E.G.)/4
= Quar. Peak Area

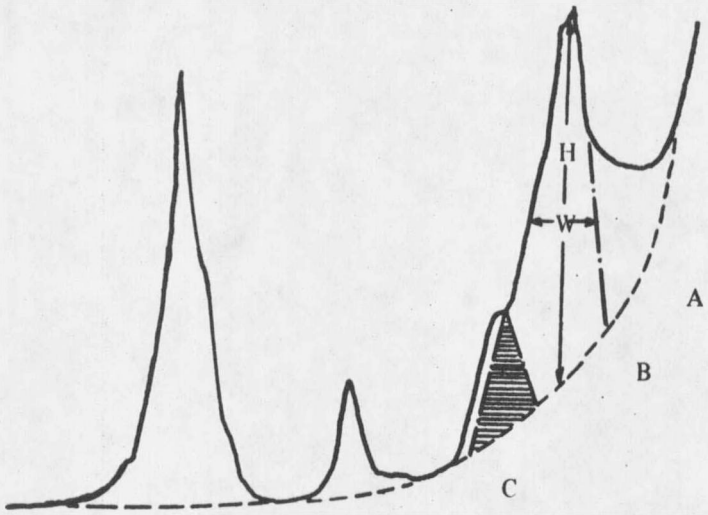

Figure 1. Peak Area Determination

A = Hand drawn background line
B = Hand drawn line estimating the extent of the peak
C = Area of peak overlap
H = Maximum height of peak measured from A
W = Peak width measured at H/2

Peak Area = H × W

Modifications II, III, IV, and V were similar to Modification I except that the (7A Mg-sat. E.G. minus 7A K-sat. 550°C) peak area was divided by 3, 2.5, 2, and 1, respectively, for kaolinite estimates. Modifications VI, VII, VIII, IX, and X were similar to Modifications I-V except that the 17A Mg-sat. E.G. peak area was divided by 5 for smectite estimates.

For each modification tested, the characterized peak areas were totaled and the relative percent composition of each mineral calculated according:

Smec. Pk. + Verm. Pk. Area + Chlor. Pk. Area

+ Kaol. Pk. Area + Quar. Pk. Area = Total Peak Area

Percent Smectite = Smec. Pk. Area/Total Pk. Area × 100
Percent Vermiculite = Verm. Pk. Area/Total Pk. Area × 100
Percent Chlorite = Chlor. Pk. Area/Total Pk. Area × 100
Percent Illite = Ill. Pk. Area/Total Pk. Area × 100
Percent Kaolinite = Kaol. Pk. Area/Total Pk. Area × 100
Percent Quartz = Quar. Pk. Area/Total Pk. Area × 100

Cation Exchange Capacity Estimation

Cation exchange capacity values were estimated for the $< 2\mu$ particle size fraction of each sample by multiplying the percent mineral compositions estimated by each modification with cation exchange capacity values reported by McNeal [31] for the minerals considered:

Smectite	100 meq/100 g
Illite	25 meq/100 g
Kaolinite	8 meq/100 g
Chlorite	25 meq/100 g
Vermiculite	175 meq/100 g
Quartz	2 meq/100 g

The resulting estimated cation exchange capacity values were tested against the cation exchange capacity values determined by the laboratory procedure previously described, using linear regression methods.

Statistical Methods

Correlations were computed using the *Bivariate Correlation Analysis* routine, subprogram *Scattergram* and the *Multiple Regression* routine, subprogram *Regression* of the *Statistical Package for the Social Sciences* [35]. For each modification, linear regression models were developed for the following:

dependent	vs.	independent
MCEC		ECEC
ECEC		MCEC
ESM		MCEC
ECDIF		ESM
EIL		MIL8
EIL		MIL5
ILDIF8		EIL
EKA		MCEC
ECDIF		EKA

where,

MCEC = Cation exchange capacity determined by chemical means: measured CEC.
ECEC = Cation exchange capacity estimated from X-ray diffraction results.
MIL8 = Relative Illite content of the clay fraction as determined by total potassium analysis assuming 8.3% K per unit cell of Illite; measured illite content.
MIL5 = Relative Illite content of the clay fraction as determined by total potassium analysis assuming 5.1% K per unit cell of Illite; measured illite content.
EIL = Percent Illite content of the clay fraction estimated from X-ray diffraction results.
ESM = Percent Smectite content of the clay fraction estimated from X-ray diffraction results.

EKA = Percent Kaolinite content of the clay fraction estimated from X-ray diffraction results.
ECDIF = Difference in CEC of ECEC minus MCEC
ILDIF8 = Difference in the percent Illite content given by EIL minus MIL8.

The estimating accuracy and precision of a modification in the factor method was primarily based on comparisons of the slope, the y-intercept, the correlation coefficient (r), the coefficient of determination (r^2), and the standard error of the estimate (SEE) for the linear regression models listed above.

PRELIMINARY STUDY–SOURCES OF ERROR IN LABORATORY TECHNIQUE

Three soils were chosen from samples obtained from the Decker Coal Company, Decker, Montana, and the Coal Mine Reclamation Program, Montana State University, on the basis of their relative smectite content:

	Ident. No.	Description
Soil A (High)	44523	20-30 feet
Soil B (None)	44531	55-60 feet
Soil C (low to moderate)	15	Colstrip–C.M. Watershed #1

From each soil, three 10 g samples were taken and clay size ($< 2\mu$) material separated by the method previously described. From the resulting stock clay suspensions three Mg-saturated slides and three K-saturated slides were prepared. The Mg-saturated slides were ethylene glycolated and scanned through a range of 2°-30°, 2θ. K-saturated slides underwent successive heat treatments of 350°C and 550°C and were scanned through a range of 2°-15°, 2θ following each heat treatment. Each slide was positioned to create three arbitrary fields on the slide corresponding to center, slightly left of center, and

slightly right of center. Three readings were made at each position. The design results in 81 samples from each soil and 243 samples for the whole experiment.

Characterization and relative clay mineral composition were determined by Modification No. 1 described previously in Materials and Methods–Main Study.

One-way analysis of variance for the nested design [46] was computed over all soils tested considering peak area (measured in square inches), and relative clay mineral composition.

In the analysis of peak area the following peaks were considered:

Mg-E.G. treatment: 17A, 14A, 10A, 7A, 3.5A, 3.3A

K-350°C treatment: 14A, 7A

K-550°C treatment: 7A

These peaks have been used in the past by numerous investigators in making quantitative estimations of clay mineral composition.

RESULTS AND DISCUSSION

The results herein reported and discussed were derived from two separate studies. The two studies are reported separately under the headings: I. Preliminary Study–Sources of Error in Laboratory Technique, and II. Main Study–Quantification.

I. PRELIMINARY STUDY–SOURCES OF ERROR IN LABORATORY TECHNIQUE

Peak area measurements (in^2) and percent clay mineral composition data appear in Tables 15 and 16, respectively. Analysis of variance was conducted according to the model of ANOVA table appearing in Table 14 by computer. Computations were conducted by Dr. Erwin Smith. Complete analysis of variance statistics for peak area appear in Tables 17-24. Complete analysis of variance statistics for relative percent composition appear in Tables 25-30.

Percent of the total variance for the main effects was calculated from variance components[1] for all analyses and appear in Table 2.

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[1] Variance Components were calculated for the Analysis over all soils by:

$$S^2_{B \subset A} = MS_{soils} - MS_{clay\ sep./bcdn}$$

$$S^2_{C \subset B} = MS_{clay\ sep.} - MS_{slide/cdn}$$

$$S^2_{D \subset C} = MS_{slide} - MS_{field/slide/dn}$$

$$S^2_{N \subset D} = MS_{field/slide} - MS_{read./field/n}$$

$$S^2 = MS_{read./field}$$

$$\text{Total Variance} = S^2 + S^2_{N \subset D} + S^2_{D \subset C} + S^2_{C \subset B} + S^2_{B \subset A}$$

Table 2. Percent of Total Variance for Main Effects on Peak Area for All Soils Studied

Treatment Peak	Mg EG 17A	Mg EG 14A	Mg EG 10A	Mg EG 7A	Mg EG 3.5A	Mg EG 3.3A	K350 14A	K350 7A
Soil	90.38*	42.46*	81.79*	65.62*	65.98*	62.26*	11.78*	45.74*
Clay Sep./Soil	0.00	7.52*	1.59	2.89	0.00	6.33	0.00	25.06*
Slide/Clay Sep.	0.00	0.00	0.00	14.96*	14.20*	9.66*	0.00	2.45
Field/Slide	8.69*	36.49*	11.98*	13.04*	13.36*	18.22*	75.88*	26.30*
Read/Field	0.92	13.57	4.64	3.49	6.47	3.53	12.34	0.45

*Significant for $\alpha = 0.05$ for variance components within columns.

The percent of the variance as it appears in the tables is a useful statistic, although the magnitude of the values can be somewhat misleading. For this reason asterisks have been used to indicate main effects significant at the 5% level based on F-test values (Tables 17-30).

Significant Main Effects in Determining Peak Area Over All Soils Tested

The X-ray diffraction method used was shown to be sensitive to the variation in mineralogy of the clay size fraction of the soils used when considering peak area. As may be seen in Table 2, soil was shown to be the biggest source of variation. This was expected since the soils were chosen on their apparent clay mineral composition (high smectite, low to moderate smectite, and no smectite).

Positioning of the slide (field/slide) was found to have a significant effect in determining peak area for all peaks considered in this study. This points to a definite need to be consistent in the positioning of the samples in the diffractometer. It may be further inferred that, in attempting to use different treatments, requiring removal and repositioning of the same or a different slide, a significant source of variation may be incurred in repositioning. This effect is not simply the effect of variations in the thickness of the specimen, but also the effect of the length of the specimen exposed to irradiation. Positioning of the slide significantly off center has the effect of shortening the effective length of the specimen irradiated by allowing a substantial portion of the incident X-ray beam to miss the sample [39].

For Mg-saturated, ethylene glycol solvated samples, between slide (slide/clay separation) variations were found to have significant effects in determining peak area for the

7A, 3.5A and 3.3A peaks. This points to possible clay mineral segregation during the centrifuge washing technique following Mg-saturation, difference in preferred orientation, variation of clay film thickness, or a combination of these effects. This seems to be especially important when considering minerals in the high 2θ angle region such as kaolinite and quartz, since the peaks showing significant variation are the first order peaks for these minerals. While these peaks also represent high order reflections for other minerals (chlorite, vermiculite, and illite) similar variation was not recognized in the first order reflections for these minerals.

For these samples, variations due to the clay separation procedure were found to be significant in determining peak area for the 14A and 7A peaks. Again this points to clay mineral segregation, in this instance during the clay separation procedure. The effect seems to be significant in considering chlorite and kaolinite. Vermiculite also exhibits a first order reflection at 14A but was not recognized in many samples.

Sources of Error in Determining Clay Mineral Composition Between Soils

The X-ray diffraction method used in this study was found to be sensitive to the variation in mineralogy of the clay size fraction of the soils.

As may be seen in Table 3, the soil was found to be a significant source of variation. Soil was the major source of variation for smectite, illite, and kaolinite. This was anticipated since the soils were chosen on their apparent clay mineral composition.

The positioning of the slide was also found to have a significant effect in determining the relative clay mineral composition for each of the minerals tested.

Table 3. Percent of Total Variance for Main Effects on the Determination of Clay Mineral Composition Over All Soils Studied

	Smectite	Illite	Kaolinite
Soil	98.56*	94.10*	87.31*
Clay Sep./Soil	0.09	1.44*	2.20*
Slide/Sep.	0.00	0.00	0.00
Field/Slide	1.12*	2.58*	8.19*
Read./Field	0.23	1.89	2.30
Coef. of Var.	6.10	8.33	6.80

* - significant at $\alpha = 0.05$

The clay separation was found to be significant in determining the relative clay mineral composition for all the minerals considered in this study except smectite. The crystallite size of smectite is relatively small. Variation in temperature, time, and centrifuge speed not compensated for during the centrifugation procedure would have a greater effect on the amounts of clay minerals having larger particle size than smectite.

II. MAIN STUDY--QUANTIFICATION

The accuracy and precision of each modification of the factor method tested in this study were determined by linear regression analysis. In this way measured parameters were compared with related estimated parameters. Complete peak area measurements appear in Table 31.

Cation Exchange Capacity Estimation

Linear regression models were developed to determine the relationship of the cation exchange capacity of the clay-sized fraction as estimated from X-ray diffraction results (ECEC) to the CEC of the same clay fraction as determined by chemical methods (MCEC) for each of the modifications tested. Regression models of MCEC as estimated from levels of ECEC also permit an assessment of the variability of MCEC for any level of ECEC. Table 4 contains the linear models and associated statistics developed for the ten modifications tested in this study.

Table 4. Linear Regression Models of the Measured CEC on Estimated CEC

	Slope	Intercept	r	r^2	SEE
Mod. I	0.683	3.66	0.927*	0.859	6.52
Mod. II	0.684	4.43	0.936*	0.877	6.10
Mod. III	0.688	4.97	0.939*	0.881	6.01
Mod. IV	0.697	5.50	0.941*	0.886	5.87
Mod. V	0.750	7.11	0.949*	0.900	5.51
Mod. VI	0.733	3.01	0.932*	0.869	6.29
Mod. VII	0.739	3.82	0.935*	0.875	6.15
Mod. VIII	0.741	4.43	0.937*	0.878	6.07
Mod. IX	0.750	5.02	0.940*	0.883	5.94
Mod. X	0.815	6.64	0.946*	0.895	5.63

* - significant at $\alpha = 0.05$

Based on the interdependence between MCEC and ECEC as measured by the correlation coefficient (r), the coefficient of determination (r^2), and the dispersion of MCEC about the regression line as measured by the standard error of the estimate (SEE), Modifications IV, V, IX, and X were chosen as the best modifications tested. Complete data tables for these four modifications appear in Tables 32-35.

It may be assumed that if a modification accurately accounted for the CEC as measured by chemical methods, the regression model developed would be of the form MCEC = ECEC, where the regression line passes through the origin and has a slope of 1.0. The modification providing a linear relationship with a slope and y-intercept closest to these values may be assumed to provide the most accurate assessment of the suite of clay minerals present in the clay-sized fraction.

Statistical results for these four modifications appear in Table 5. Confidence limits have been applied for consideration of both the inherent accuracy and precision of these modifications.

Table 5. Selected Linear Regression Models of Measured CEC on Estimated CEC

Mod. No.	Slope	Intercept	r	r^2	SEE
IV	0.697±0.072	5.50±1.66	0.94*	0.89	5.87
V	0.750±0.068	7.11±1.56	0.95*	0.90	5.51
IX	0.750±0.073	5.02±1.68	0.94*	0.85	5.94
X	0.815±0.069	6.64±1.59	0.95*	0.90	5.63

* - significant at $\alpha = 0.05$

A value for the y-intercept greater than 0.0 is evidence of a failure of the modification to completely account for the CEC of the clay-sized fraction as measured by chemical methods and/or the error inherent in the methods employed in obtaining values for both MCEC and ECEC. Since the values of the y-intercepts do not significantly differ from each other for the four models considered here it might be assumed that the error involved in estimating CEC is constant for all of the modifications discussed. The positive intercepts could be caused by one or more of several things. A few erroneously high measured CEC values (MCEC) on samples dominated by low CEC clays would have this effect. Another possible explanation is that the CEC of the clay minerals occurring in samples varied from those assumed in estimating the CEC of the clay fraction. Alternatively, the positive intercepts may indicate the presence of additional minor, low CEC constituents of the clay-sized fraction, such as feldspars.

Smectite, when present in a suite of clays, significantly affects both the estimated and measured values of CEC. Overestimation of smectite would tend to reduce the slope of these graphs to some value less than 1.0. A comparison of the slopes obtained for the regression models of MCEC on ECEC for the four modifications discussed here reveals that all of the modifications apparently overestimate smectite, since all of the slopes are significantly less than 1.0 ($\alpha = 0.05$). Of these four modifications, Modification X has the highest value for the slope of the model, coupled with favorable values for the y-intercept, r, r^2, and SEE.

Graphical representation of this relationship for Modification X appears in Fig. 2. The regression model given by MCEC = 0.815 (ECEC) + 6.64 approximates the expected relationship given by ECEC = MCEC represented by the dashed line. The graphs are simul-

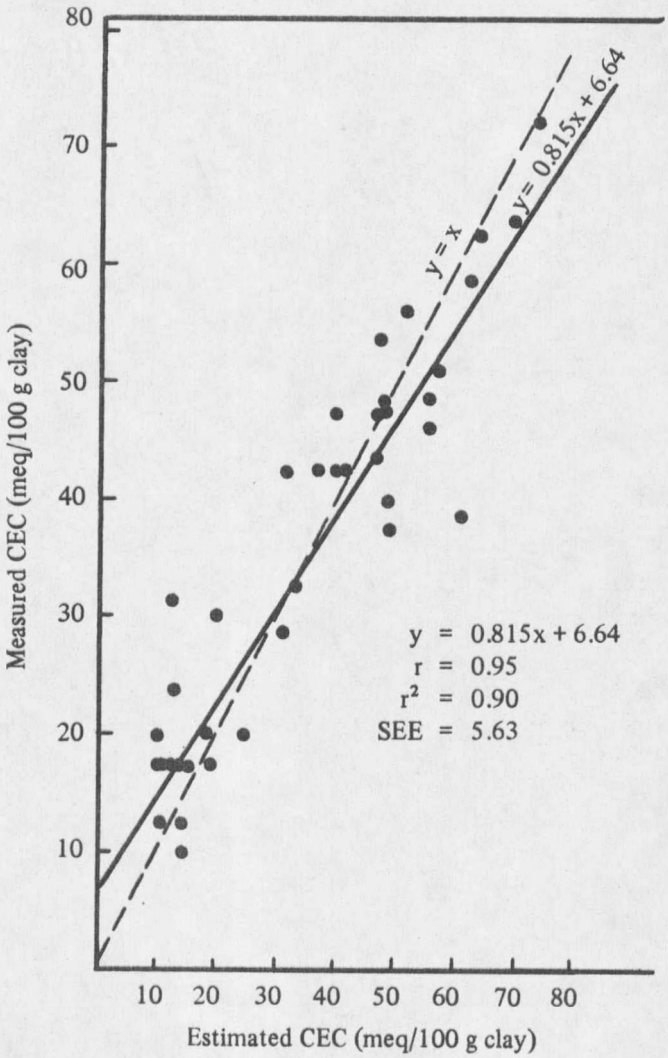

Figure 2. Linear Regression Model of Measured CEC on Estimated CEC for Modification X.

taneous at value of 36 meq/100 g clay. The model underestimates CEC of the clay fraction for values of MCEC less than this value and overestimates for values greater.

Regression models of ECEC as estimated from values of MCEC provide a measure of the ability of MCEC to predict estimated values of CEC. This approach also allows an assessment of the variability of the estimated CEC for various levels of the measured CEC as well as an indication of the error involved in applying the regression model at different levels of MCEC.

If a modification is making an accurate assessment of the clay minerals in a suite of clays, the regression model would be ECEC = MCEC. Values for the y-intercept that are less than 0.0 would reflect either a failure of the modification to entirely account for the actual CEC, or a use of inappropriate CEC values in estimating the total CEC of the clay fraction. Overestimation of the CEC could be attributed primarily to overestimation of the smectite minerals and would result in a slope of the regression line that is significantly greater than 1.0.

Statistical results for the four modifications discussed appear in Table 6.

Table 6. Selected Linear Regression Models of Estimated CEC on Measured CEC

Mod. No.	Slope	Intercept	r	r^2	SEE
IV	1.27±0.13	-2.40±2.25	0.94*	0.89	7.98
V	1.29±0.12	-4.98±1.97	0.95*	0.90	6.96
IX	1.18±0.12	-1.46±1.73	0.94*	0.88	7.18
X	1.10±0.11	-3.90±1.83	0.95*	0.90	6.48

* - significant at $\alpha = 0.05$

If, for any one value of CEC for the clay fraction one and only one suite of clay minerals exists, it might be assumed that the SEE for the regression models of MCEC on ECEC would equal the SEE for the regression models of ECEC on MCEC. Values of SEE for the regression models of ECEC on MCEC, for all the modifications discussed, are higher than those reported for the regression models of MCEC on ECEC. The variability of ECEC for any level of MCEC is greater than the variability of MCEC for any value of ECEC. This is because the clay mineral estimates exhibit greater variability than do values for measured CEC. This indicates that, in practice, due to the variability in X-ray diffraction results, one measured value of CEC does not represent a unique suite of clay minerals. As in the case of the regression models of MCEC on ECEC, the mean value for the slopes and intercepts indicate that these modifications tend to underestimate the CEC at lower levels of MCEC and overestimate the CEC when significant amounts of smectite are present.

If any of the values for the CEC of each mineral group varies significantly from the actual value it would be reflected in a slope different than 1.0. The slope obtained for the regression model testing Modification X does not significantly ($\alpha = 0.05$) differ from 1.0. The value for the y-intercept does significantly differ from 0.0. Since the y-intercepts of the models discussed do not significantly differ from each other, the difference between the y-intercepts of the expected model and the model actually obtained is probably due either to erroneously high measured CEC values or to the presence of a mineral group that was not considered in estimating the composition of the clay-sized fraction. This parallels the results of the regression models developed for MCEC on ECEC.

From Fig. 3 it may be seen that the regression model of Modification X for ECEC on MCEC given by ECEC = 1.10(MCEC) - 3.90, closely follows the expected relationship

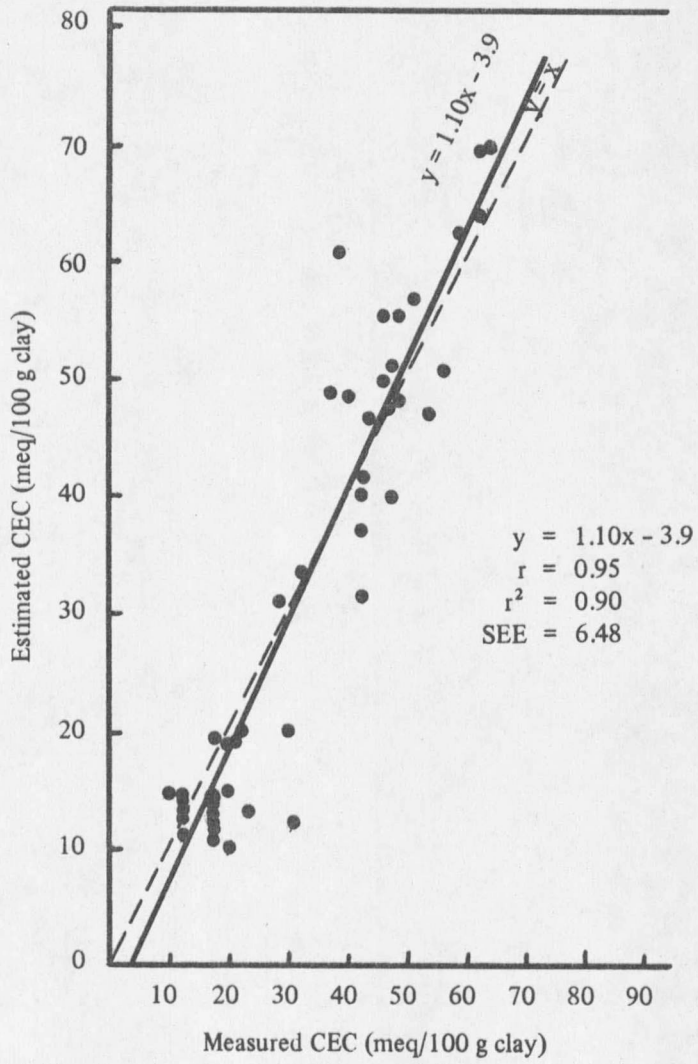

Figure 3. Linear Regression Model of Estimated CEC on Measured CEC for Modification X.

given by ECEC = MCEC represented by the dashed line. The graphs are seen to be simultaneous at a value of 39 meq/100 g. The model underestimates the CEC for values of MCEC less than this value and overestimates for values of MCEC greater than 39 meq/100 g. The percent error may be calculated by the following:

where, $\hat{y} = 1.10(\text{MCEC}) - 3.90$ = the predicted ECEC

and y = MCEC = the expected value of ECEC assuming ECEC = MCEC

then $\%\text{-error} = \frac{\hat{y} - y}{y} \times 100$, and the sign indicates underestimation (-) or overestimation (+).

From these calculations it was found that in the portion of the regression model corresponding to values of MCEC less than 20 meq/100 g the error is greater than 11%. It should be remembered, however, that in this portion of the graph the values of both MCEC and ECEC are themselves small and relatively small errors in terms of meq/100 g correspond to large errors when expressed as percent. At these low levels of CEC, illite and kaolinite typically dominate the exchange complex. Consequently, small differences in the ECEC could be the product of significant errors in estimating the amounts of these minerals.

The percent error involved in estimating CEC from this regression model in the area of the graph corresponding to values of MCEC greater than 30 meq/100 g is less than 5%. Consequently, Modification X appears to be most sensitive in determining the clay mineral composition for those clay-sized fractions containing significant amounts of smectite. Further, Modification X provides the greatest range in values over which it acceptably estimates clay mineral composition of the clay fraction.

Smectite Estimation

A more usable relationship is that of the estimated percent smectite composition (ESM) as determined by the measured CEC. These regression models present a direct predictive tool for the determination of smectite based on the measured CEC of the clay-sized fraction and permit a measure of the precision of the estimate (SEE).

Statistical results and confidence limits (α = 0.05) for the slope and y-intercept appear in Table 7.

Table 7. Selected Linear Regression Models of Estimated Smectite Content on Measured Cation Exchange Capacity

Mod. No.	Slope	Intercept	r	r^2	SEE
IV	1.49±0.13 *1.22±0.13	-21.78±2.27	0.96**	0.91	8.03
V	1.35±0.11 *1.21±0.11	-20.78±1.94	0.96**	0.92	6.87
IX	1.37±0.12 *1.21±0.12	-20.59±2.10	0.96**	0.91	7.42
X	1.23±0.10 *1.19±0.10	-19.41±1.78	0.96**	0.92	6.30

* - slopes of the expected regression model derived by substituting 100meq/100g at 100% smectite content
** - significant at α = 0.05

Expected models were derived from the regression models developed for each modification assuming a MCEC of 100 meq/100 g at 100% smectite. This expected model appears as a dashed line in Fig. 4 along with the graphical representation of the confidence interval for the regression model obtained (dotted line).

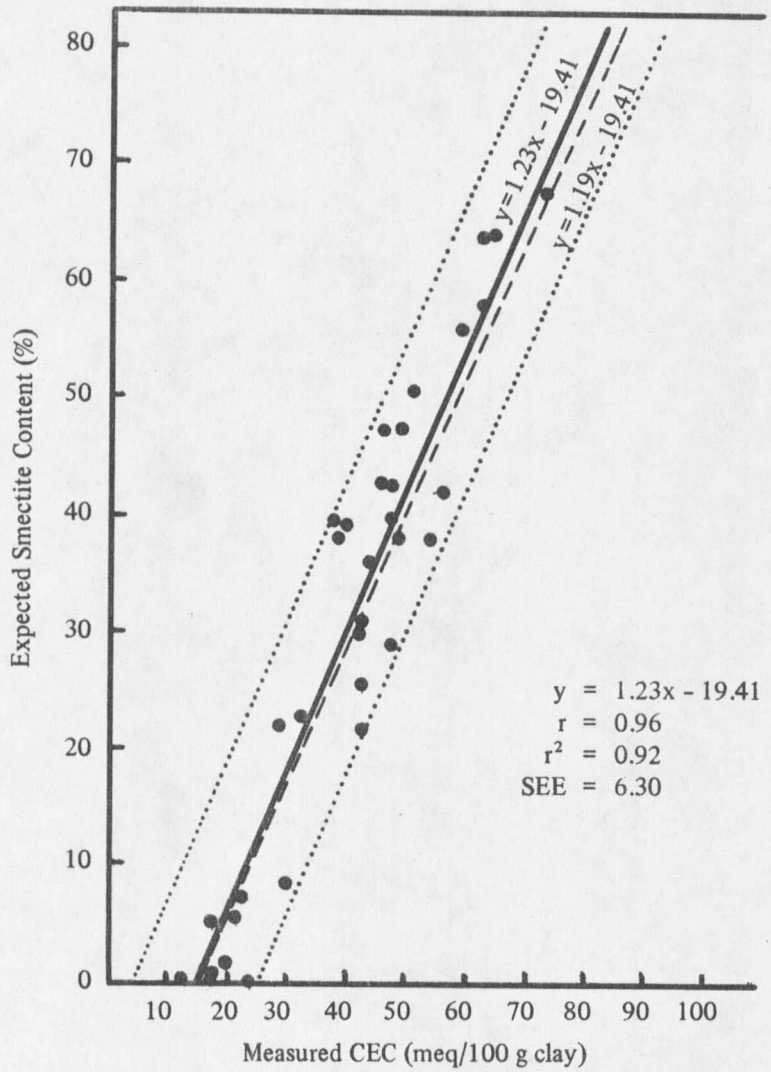

Figure 4. Linear Regression Model of Estimated Smectite Content on Measured Cation Exchange Capacity.

The y-intercepts obtained for these relationships did not significantly differ from each other and are related to the x-intercepts which correspond to the average CEC of the clay-sized fraction when no smectite is present. As may be seen from Table 7 the slopes of the expected regression models do not significantly differ. Consequently, the expected models are similar for all of the modifications tested.

Expected models were derived from the regression models developed for each modification assuming a MCEC of 100 meq/100 g at 100% smectite. This expected model appears as a dashed line in Fig. 4 along with the graphical representation of the confidence interval for the regression model obtained (dotted line).

The y-intercepts obtained for these relationships did not significantly differ from each other and are related to the x-intercepts which correspond to the average CEC of the clay-sized fraction when no smectite is present. As may be seen from Table 7 the slopes of the expected regression models do not significantly differ. Consequently, the expected models are similar for all of the modifications tested.

Overestimation of the percent smectite composition would tend to increase the slopes of these relationships. Assuming that the values for the y-intercepts relate to a constant and true average of the CEC of the clay-sized fraction when no smectite is present, the preferable modification would be indicated by a regression model that closely estimated the expected regression model and did not significantly differ from it. It may be seen from Fig. 4 that this is true of the regression model developed for Modification X. The graph of the expected and derived regression models are nearly concurrent and the expected model lies well within the 95% confidence interval applied to the derived regression model for this modification. The coefficient of determination indicates that approxi-

mately 92% of the variation in the percent smectite composition was explained by variations in the measured CEC.

The error involved between the expected and the derived models was approximately 3% at MCEC = 100 meq/100 g clay. Less than 3% error is incurred in estimating the relative smectite content at lower values of measured CEC.

A limiting factor in applying this relationship to the prediction of levels of smectite content is the apparent low level of precision as indicated by an SEE = 6.30. The 95% confidence interval for any value of the percent smectite in a sample predicted from the measured CEC of the clay-sized fraction would be approximately ±12.6 percent smectite.

The difference between the measured CEC (MCEC) subtracted from the estimated CEC (ECEC) was used as a dependent variable (ECDIF) in testing relationships with ESM to assess any effect the apparent relative smectite composition might have on estimating accuracy. In the above discussions it was shown that accuracy is generally lower for estimates of the smectite content in samples containing low amounts of smectite when the error is expressed on a percentage basis. The regression models developed for ECDIF on ESM provide a direct indication of bias and an avenue for determining the significance of the apparent bias.

Statistical results for the four best modifications tested appear in Table 8. Confidence limits have been applied to the slope and y-intercept to facilitate the assessment of the significance of the apparent bias implied by the slope and intercept of the model.

Modifications providing accurate and unbiased estimates of the suite of clay minerals should reveal a relationship that is concurrent with the x-axis and is of the form ECDIF = 0, with $r = 0$, $r^2 = 0$, and SEE = 0.0. Regression models testing the modifications

Table 8. Selected Linear Regression Models of the Difference in Estimated and Measured Cation Exchange Capacity on Estimated Smectite Content

Mod. No.	Slope	Intercept	r	r^2	SEE
IV	0.248±0.067	-0.32±1.77	0.73*	0.53	6.28
V	0.217±0.067	-3.81±1.62	0.68*	0.46	5.72
IX	0.198±0.073	-0.63±1.79	0.62*	0.38	6.33
X	0.151±0.076	-3.83±1.66	0.50*	0.25	5.86

* - significant at $\alpha = 0.05$

that significantly differ from this perfect fit relationship indicate significant bias in making estimates of the clay mineral composition. Overestimation of the amount of smectite would tend to increase the slopes of these models and decrease the value of the y-intercept.

Of primary importance in analyzing these relationships is the slope and y-intercept of the regression models obtained. From Table 8 it may be seen that the slopes of all the regression models significantly differ from 0.0. Consequently, it may be assumed that bias is incurred in estimating the clay mineral composition by any of the four modifications discussed. However, the results indicate that Modification X provides the most accurate and relatively unbiased assessment of the clay mineral composition. Modification X has the lowest slope. Modifications IV and IX do include 0.0 in the 95% confidence interval. The y-intercept for Modification X is reasonably close to zero. In addition the r, r^2, and SEE values for Modification X are lowest of the modifications discussed.

These results tend to support those previously reported for the regression models developed for ECEC on MCEC, MCEC on ECEC and ESM on MCEC. From Fig. 5 it is observed that the graphs of the expected and derived models are simultaneous at a value

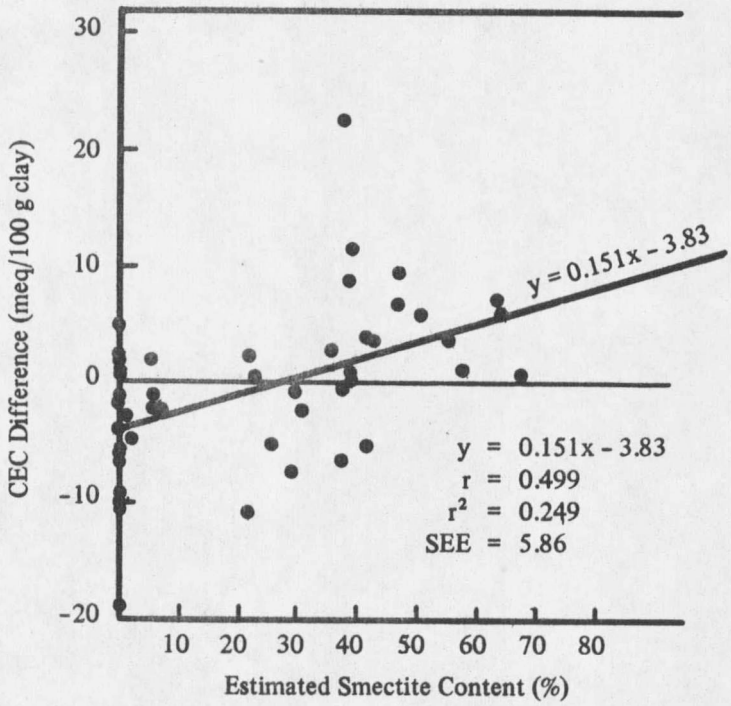

Figure 5. Linear Regression Model of the Difference in Estimated and Measured Cation Exchange Capacity on Estimated Smectite Content.

of approximately 25 percent smectite. The regression model indicates that Modification X tends to overestimate the amount of smectite present when it is greater than this amount and underestimate the amount of smectite when it is less than this amount. Modification X also tends to favor lower estimated amounts of illite. In the portion of the graph representing low smectite composition the suite of clay minerals would be dominated by illite and kaolinite. Consequently, Modification X should be expected to yield lower estimates of the CEC in samples dominated by illite.

Illite Estimation

As a further test of the accuracy of the mineral estimates, regression models were developed to examine the relationship of the relative composition of illite (EIL) in each sample with the percent illite based on total K analysis assuming 8.3% (MIL8) and 5.1% (MIL5) elemental K per unit cell of illite. Regression models of EIL as estimated from values of MIL8 and MIL5 provide a measure of the sensitivity of the chemical methods employed to account for variation in apparent percent composition of illite in the clay-sized fraction. They also allow an assessment of the variability of the estimated percent illite for various levels of MIL8 and MIL5 as well as the error involved in applying the regression model at various levels on MIL8 and MIL5.

It may be assumed that if a modification accurately estimated the percent illite and all of the K present in the clay-sized fraction was a component of the crystalline structure of illite then the expected regression model developed would be PIL = MIL8 or MIL5. This regression line would pass through the origin and have a slope of 1.0. The modification providing a linear relationship with a slope and y-intercept closest to these

values may be assumed to provide the most accurate assessment of the relative amount of illite present in the clay-sized fraction. Values for the y-intercept that are less than 0.0 would reflect a failure of the modification to entirely account for the relative amount of illite as estimated by total K analysis.

Models derived assuming 8.3% elemental K per unit cell illite. Statistical results for the four best modifications appear in Table 9. Confidence limits ($\alpha = 0.05$) have been applied for consideration of both the accuracy and the precision of these modifications.

Table 9. Selected Linear Regression Models of Estimated Illite Content on Measured Illite Content (Assuming 8.3% K per Unit Cell Illite)

Mod. No.	Slope	Intercept	r	r^2	SEE
IV	1.63±0.24	-11.12±1.73	0.87*	0.75	6.10
V	1.03±0.15	-4.83±1.10	0.87*	0.75	3.90
IX	1.58±0.23	-9.22±1.67	0.87*	0.76	5.92
X	0.99±0.15	-3.31±1.09	0.86*	0.74	3.87

* - significant at $\alpha = 0.05$

The y-intercepts of all the regression models (Table 9) are significantly less than 0.0. These negative intercepts support the conclusions made from the regression models developed for ECEC on MCEC, particularly that the inability of the modifications to completely explain the percent illite derived from total K analysis is due to the presence of a K-bearing mineral in the clay-sized fraction that caused an overestimation of the relative amount of illite. It may be seen, however, that there is a significant difference between the y-intercepts obtained for these models. While the y-intercepts of the regression models for Modifications IV and IX do not significantly differ from each other, they do significantly

differ from the intercepts obtained for the models testing Modifications V and X. The latter modifications do not significantly differ from each other. It should be noted that Modifications IV and IX tend to cause higher estimates of smectite and illite while Modifications V and X tend to favor higher estimates of kaolinite at the expense of the estimated smectite and illite contents.

If the modification is sensitive to the changes in illite content as determined by total potassium analysis and the K-content of the illite is 8.3%, the slope of the regression model testing that modification should not significantly differ from 1.0. In Figs. 6 and 7 it may be seen that the regression models closely parallel the expected models and differ in both cases by a relatively constant amount.

The constancy of the difference between the expected and derived regression model might also indicate the use of an inappropriate combination of coefficients in the characterization of the peak areas. As stated earlier it might be reasonable to expect such a relationship if the procedure used to estimate the mineral components of the clay-sized fraction failed to recognize a K-bearing mineral of minor, relatively constant, proportions.

Values for SEE indicate lower variability for EIL at any given level of MIL8 for Modifications V and X. SEE values were much higher for the regression models testing Modifications IV and IX.

Modification X seems to provide the most favorable combination of accuracy and precision for the estimation of illite of the four modifications discussed here. The regression model testing this modification is given by EIL = 0.99(MIL8) - 3.31. This closely follows the expected relationship given by EIL = MIL8. The coefficient of determination

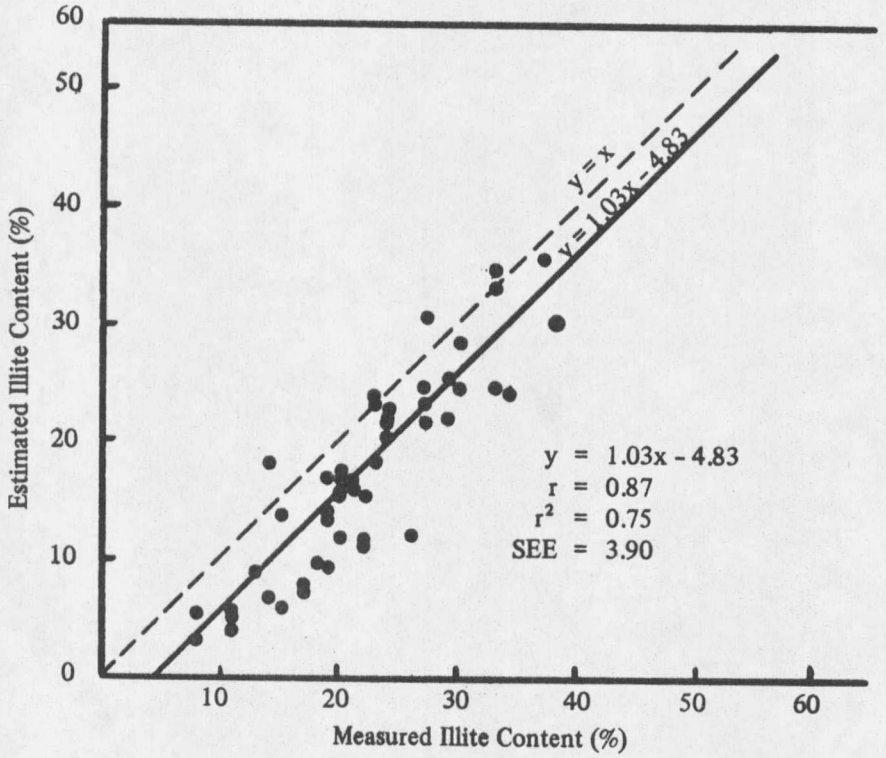

Figure 6. Linear Regession Model of Estimated Illite Content on Measured Illite Content (8.3% K) for Modification V.

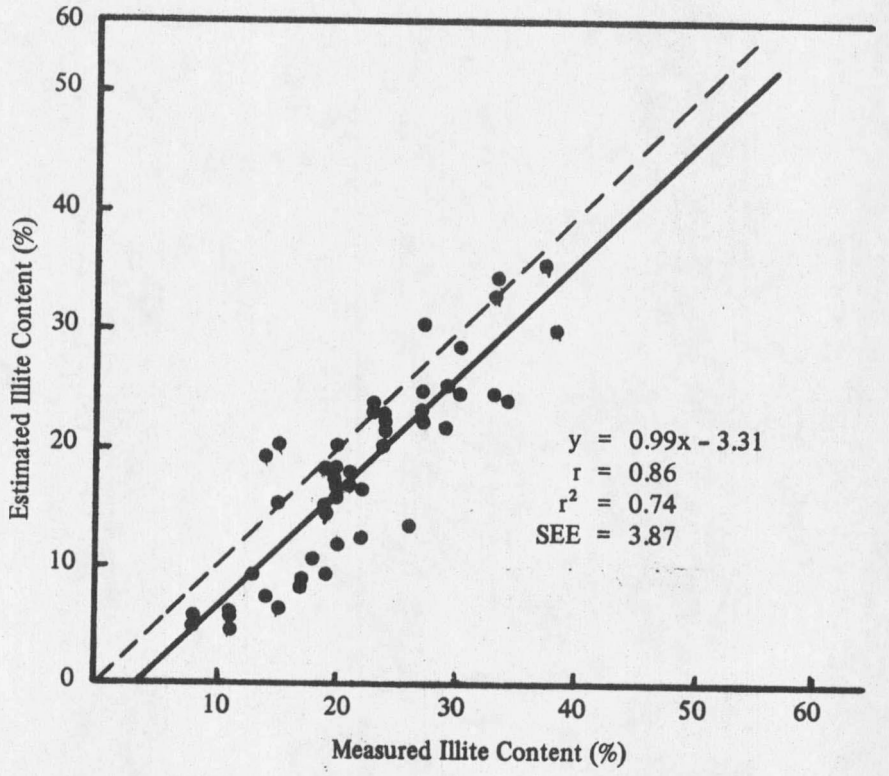

Figure 7. Linear Regression Model of Estimated Illite Content on Measured Illite Content (8.3% K) for Modification X.

indicates that 75% of the variation in the EIL values was explained by the variation of the MIL8 values. This is considered further evidence of the presence of additional K-bearing minerals in the clay-sized fraction.

The percent error was dependent on the relative amount of illite present, with more error, on a percent basis, being incurred on the estimation of low amounts of illite. The percent error was greatly reduced, to about 10%, in considering estimates of larger amounts of illite. This parallels the findings reported by several authors and might be anticipated from the parallel nature of the expected and derived models.

Models derived assuming 5.1% elemental K per unit cell illite. The effect of assuming 5.1% K per unit cell of illite is to increase the measured relative composition of illite in all samples compared to values derived assuming 8.3% elemental K. Modifications IV and IX favor higher estimates of smectite and illite. Based on the regression models developed for EIL on MIL8, it was anticipated that the estimated illite composition derived by these two modifications might more closely relate to the measured illite composition assuming 5.1% K per unit cell illite rather than 8.3% K.

Table 10 contains the statistical results for the four best modifications tested.

Table 10. Selected Linear Regression Models of Estimated Illite Content on Measured Illite Content (Assuming 5.1% K per Unit Cell Illite)

Mod. No.	Slope	Intercept	r	r^2	SEE
IV	1.00±0.04	-11.12±0.50	0.87*	0.75	1.75
V	0.63±0.03	-4.83±0.40	0.87*	0.75	1.40
IX	0.97±0.04	-9.22±0.47	0.87*	0.76	1.67
X	0.61±0.03	-3.31±0.38	0.86*	0.74	1.35

* - significant at $\alpha = 0.05$

The slopes of the regression models testing Modifications IV and IX do not differ from the perfect fit slope of 1.0. However, these modifications more seriously underestimated relative illite content in comparison with measured values assuming 5.1% K per unit cell illite than did Modifications V and X in similar comparisons assuming 8.3% K. These results indicate that for the samples used in this study the 8.3% value is a more accurate measure for the relative K-content per unit cell illite than is 5.1%. Consequently, the discussion and conclusions drawn based on the models of EIL derived from MIL8 appear to be more appropriate.

To assess the bias in estimating the relative illite composition that is related to the estimated percent composition of illite, the difference between MIL8 subtracted from EIL was used as a dependent variable (ILDIF8) in developing regression models with EIL. Statistical results for the four best modifications tested appear in Table 11.

Table 11. Selected Linear Regression Models of the Difference in Estimated and Measured Illite Contents on Estimated Illite Content (Assuming 5.1% K per Unit Cell Illite)

Mod. No.	Slope	Intercept	r	r^2	SEE
IV	0.519±0.075	-10.30±0.98	0.89*	0.80	3.48
V	0.237±0.116	-8.61±0.97	0.51*	0.26	3.43
IX	0.501±0.077	-9.46±0.98	0.88*	0.78	3.45
X	0.219±0.124	-7.83±1.00	0.45*	0.45	3.54

* - significant at $\alpha = 0.05$

Modifications providing accurate and unbiased estimates of the suite of clay minerals should reveal a relationship that is concurrent with the x-axis and is of the form ILDIF8 = 0, with r = 0, r^2 = 0, and SEE = 0. Underestimation of the relative amount of

illite present would tend to decrease the value of the y-intercept. A positive slope for the regression line indicates that the modification provides biased estimates of the percent illite composition such that, above some point at which the derived and expected models are simultaneous, the amount of overestimation is directly proportional to an increase in the apparent relative composition of illite. Conversely, the amount of underestimation is directly proportional to a decrease in the percent illite composition. In addition, statistically significant values for the r and r^2 further indicate that the bias inherent in the modification is significant.

From Table 11 it may be seen that the slope and correlation coefficients of all the regression models discussed significantly differ from 0.0. It follows that bias is incurred in estimating the relative composition of illite by any of the modifications discussed here. It may be anticipated, from the results of the regression models developed for EIL on MIL8 (Table 9) that the y-intercepts will significantly differ from 0.0. This was found to be the case for all four modifications discussed.

Modifications X and V combine the lowest slope with a value for the y-intercept that is in line with the results obtained for EIL on MIL8. The r and r^2 values are the lowest of the modifications discussed. The SEE is slightly higher for Modification X, but it is not felt that this indicates a significant increase in variability of the estimates compared to the other modifications.

The regression model as shown in Fig. 8 indicates that Modification X underestimates the relative amount of illite present below a value of 35.8% illite (EIL). This corresponds to the upper range of the percent illite composition as estimated by Modification X. Consistent apparent underestimation of EIL coupled with a low, yet significant cor-

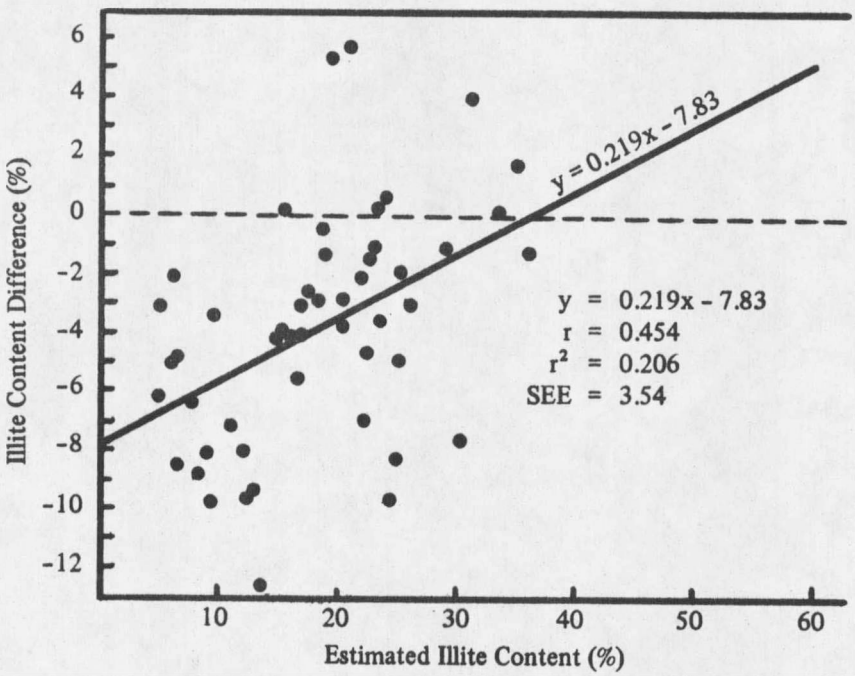

Figure 8. Linear Regression Model of the Difference in Estimated and Measured Illite Content on Estimated Illite Content (assuming 8.3% K per unit cell Illite).

relation coefficient is interpreted to indicate a consistent overestimation of MIL8. This could be due to the presence of an additional, but unmeasured, K-bearing mineral that composes a small, relatively constant portion of the clay-sized fraction. However, a definite conclusion based on the results of this study concerning the apparent underestimation of illite is not possible. When the results of these regression models are considered in conjunction with the results previously reported here, it is felt that they indicate Modification X is more responsive to the amount of illite in a sample.

Kaolinite Estimation

Measurements of the kaolinite content such as those made for illite content were not made during this study. As a result the accuracy and precision of a modification's ability to estimate the relative composition of kaolinite was inferred from linear regression models of the estimated kaolinite content (EKA) on the measured CEC (MCEC). Statistical results and confidence limits (α = 0.05) for the slope and intercept appear in Table 12 for the four best modifications tested.

Table 12. Selected Linear Regression Models of Estimated Kaolinite Content on Measured Cation Exchange Capacity

Mod. No.	Slope	Intercept	r	r^2	SEE
IV	-1.05±0.17	68.45±2.87	-0.88*	0.77	9.98
V	-1.22±0.16	87.39±2.77	-0.91*	0.83	9.79
IX	-1.02±0.16	68.36±2.74	-0.88*	0.77	9.70
X	-1.17±0.16	86.96±2.68	-0.91*	0.82	9.49

* - significant at α = 0.05

In general, samples exhibiting low values for measured CEC will possess high kaolinite contents and low smectite content. The reverse is assumed to be true at high values for measured CEC. As a result the slopes of the regression models of EKA on MCEC should be negative. As may be seen in Table 12 the slopes of the regression models testing the four modifications discussed exhibit this negative relationship.

Kaolinite typically exhibits a relatively small CEC. In the presence of measureable amounts of smectite, kaolinite is expected to compose a relatively small portion of the total CEC of the clay-sized fraction. Further, it is assumed that relatively large variations in the estimated kaolinite content would not affect the CEC of the clay fraction as much as a similar variation in smectite content. Consequently, it could be anticipated that the accuracy and precision of the modifications as measured by the regression models of EKA on MCEC would be lower than that indicated by similar models developed for smectite composition. The high values for the SEE obtained for all the modifications tested indicate that these modifications estimate kaolinite content with less precision than either smectite or illite. Modification X exhibited the lowest SEE (9.49).

The four modifications discussed exhibit significant correlation between the estimated kaolinite content and the measured CEC. The values of the intercepts and their confidence intervals indicate that all four modifications significantly underestimate kaolinite at 100% kaolinite content. Modifications V and X tend to favor higher estimates for kaolinite. These two modifications significantly differed from Modifications IV and IX but not from each other. Since regression models testing the estimation of smectite and illite content indicated that Modification X provided the best estimates of these minerals it was assumed that this modification provided adequate estimation of kaolinite content as

well. The regression model for Modification X is given by EKA = - 1.17 (MCEC) + 86.96, with r = - 0.91, r^2 = 0.82, and SEE = 9.49. This relation is graphically presented in Fig. 9.

Linear regression models of ECDIF as related to EKA were developed to assess the effect of the relative kaolinite composition has on the estimating accuracy. They appear in Table 13.

Table 13. Selected Linear Regression Models of the Difference in Estimated and Measured Cation Exchange Capacity on Estimated Kaolinite Content

Mod. No.	Slope	Intercept	r	r^2	SEE
IV	-0.318±0.106	17.28±1.81	-0.72*	0.52	8.69
V	-0.223±0.096	12.09±1.63	-0.67*	0.50	7.20
IX	-0.238±0.106	12.55±1.82	-0.60*	0.36	7.31
X	-0.150±0.096	6.64±1.66	-0.50*	0.25	6.03

* - significant at $\alpha = 0.05$

The expected regression model is concurrent with the x-axis and is of the form ECDIF = 0, with r = 0, r^2 = 0, and SEE = 0. A significant difference between this perfect fit model and the derived model indiates a significant bias in making estimates of the kaolinite content. Underestimation of the kaolinite content would tend to steepen the slopes of the regression models and increase the value of the y-intercept. Data in Table 13 show that the slopes and y-intercepts significantly differ from 0.0. The correlation coefficients indicate relationships significant at $\alpha = 0.05$. It may be assumed that bias is incurred in estimating the relatively kaolinite content. The results indicate that Modification X provides the most accurate and relatively unbiased assessment of the kaolinite composition. It combines

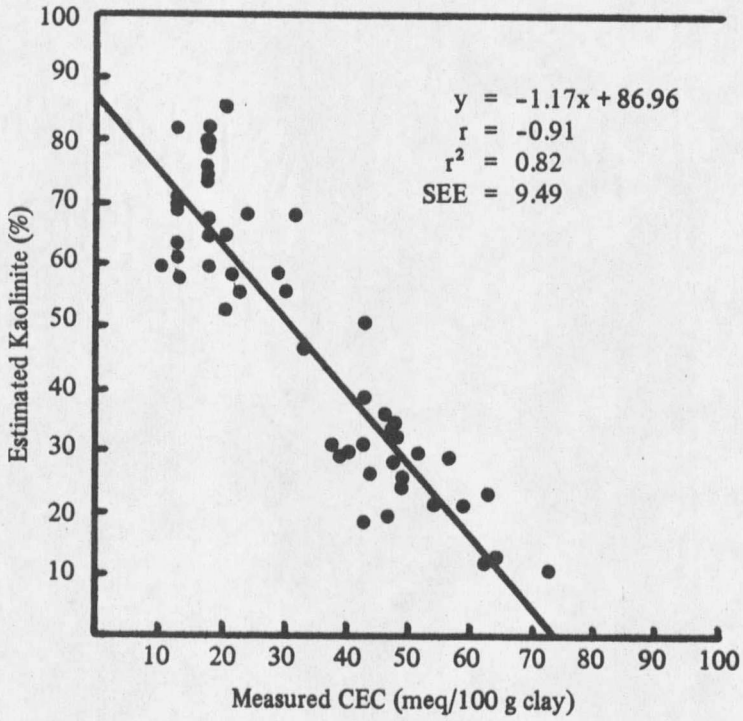

Figure 9. Linear Regression Model of Estimated Kaolinite Content on Measured Cation Exchange Capacity.

the lowest slope and y-intercept with the lowest value of r, r^2, and SEE for any of the modifications discussed.

These results seem to corroborate those previously reported for estimates of smectite and illite composition. The graphs of the expected and derived models for Modification X (Fig. 10) are simultaneous at a value of approximately 44% kaolinite. This indicates that Modification X underestimates CEC of the clay fraction at kaolinite contents higher than 44% and overestimates CEC at kaolinite contents lower than this value. This could result from an underestimation of kaolinite and/or an overestimation of smectite. Previously it was shown that Modification X overestimates CEC and smectite at values of smectite composition greater than 25 percent. The results indicate that Modification X tends to underestimate kaolinite and overestimate smectite at values of relative kaolinite and smectite composition greater than 44% and 25%, respectively. An apparent overestimation of kaolinite and underestimation of smectite occurs at compositions below these values. Since regression models testing the accuracy of the illite estimations indicated a consistent error not directly involved with the X-ray diffraction pattern characterization process, it is inferred that Modification X overestimates smectite and underestimates kaolinite. These results indicate that further accuracy might be obtained by trying other modifications of the factor method that reduce the characterized peak area of the 17A peak while it increases the characterized peak area of the 7A peak.

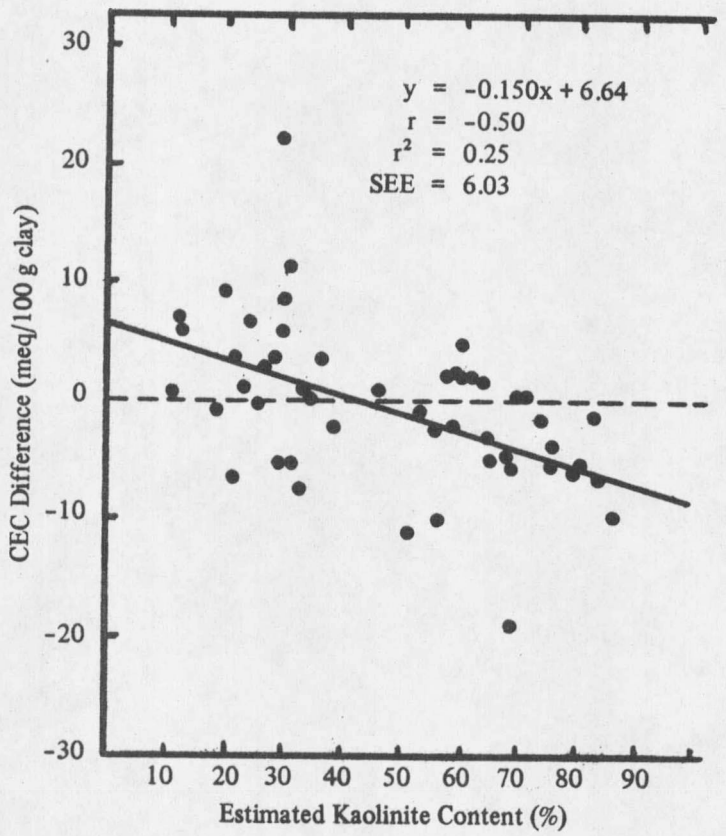

Figure 10. Linear Regression Model of the Difference in Estimated and Measured Cation Exchange Capacity on Estimated Kaolinite Content.

SUMMARY AND CONCLUSIONS

An assessment of the major sources of error in the X-ray diffraction procedure was conducted in a preliminary study using a one-way analysis of variance for a nested design for peak area and percent mineral composition. When considered over all soils tested, the X-ray diffraction method used was found to be sensitive to the variations in the mineralogy of the clay fraction of the soils used when considering peak area and relative mineral composition. The positioning of the slide was found to be significant in determining peak area and relative composition for all soils tested. Clay separation and between slide effects were found to be significant sources of variation in specific instances and seem to be particularly important in considering kaolinite content and those peaks associated with kaolinite determination. The results indicate the need for extreme care and consistency during all phases of sample preparation and presentation.

In the main study the accuracy of ten modifications of the factor method was investigated. The results indicate that the factor method may be applied in X-ray diffraction analysis to yield a relatively fast and reasonably accurate assessment of the dominant clay minerals composing the clay fraction of arid and semi-arid land soils and their parent material in southeastern Montana. The method seems particularly useful in making quantitative estimations of smectite, illite, and kaolinite.

Linear regression models indicated that Modification X is most responsive to the amount of smectite, illite and kaolinite in a sample. This modification yields accurate estimates for these clay minerals in material that does not contain significant amount of vermiculite and chlorite.

The following computations are used to calculate characterized peak areas for Modification X:

17A Mg-sat. E.G./5 = Smectite Peak Area
(14A Mg-sat. E.G. minus 14A K-sat. 350°C/2
= Vermiculite Peak Area
14A K-sat. 350°C/2 = Chlorite Peak Area
10A Mg-sat. E.G./1 = Illite Peak Area
(7A Mg-sat. E.G. minus 7A K-sat. 550°C)/4
= Kaolinite Peak Area
(3.3A Mg-sat. E.G. minus 3/4 (10A Mg-sat. E.G.))/4
= Quartz Peak Area

Characterized peak areas are totaled and the relative percent composition of each mineral calculated according to:

Smec. Pk. Area + Verm. Pk. Area + Chlor. Pk. Area
+ Kaol. Pk. Area + Quar. Pk. Area = Total Peak Area

Percent Smectite = Smec. Pk. Area/Total Pk. Area × 100
Percent Vermiculite = Verm. Pk. Area/Total Pk. Area × 100
Percent Chlorite = Chlor. Pk. Area/Total Pk. Area × 100
Percent Illite = Ill. Pk. Area/Total Pk. Area × 100
Percent Kaolinite = Kaol. Pk. Area/Total Pk. Area × 100
Percent Quartz = Quar. Pk. Area/Total Pk. Area × 100

Linear regression models involving CEC determined by chemical means (MCEC) and the CEC estimated from X-ray diffraction analysis (ECEC) showed that 90% of the variability of the predicted parameter could be accounted for by the measured parameter using estimates derived by Modification X. The variability of ECEC for any level of MCEC is greater than the variability of MCEC for any level of ECEC. This indicates that in practice, due to the variability in the X-ray results, one measured value of CEC does not represent a unique suite of clay minerals as determined by X-ray diffraction analysis.

Measurement of illite content by total K analysis was conducted assuming 8.3% and 5.1% elemental K per unit cell illite. The results of linear regression analysis indicated

that measurements made using the 8.3% K value more accurately depicted the estimated illite content.

Using estimates derived by Modification X predictive regression models for the estimation of the relative compositions of smectite, illite and kaolinite were developed based on values of measured CEC and the percent composition of illite as determined by total K analysis (MIL8):

$$\%\text{-smectite } (\pm 12.6\%) = 1.23(\text{MCEC}) - 19.41, \quad r^2 = 0.92$$

$$\%\text{-illite } (\pm 7.7\%) = 0.99(\text{MIL8}) - 3.31, \quad r^2 = 0.74$$

$$\%\text{-kaolinite } (\pm 19.0\%) = -1.17(\text{MCEC}) + 86.96, r^2 = 0.82$$

The relative amount of illite was frequently underestimated. The apparent underestimation could be caused by variability in the K per unit cell of illite resulting in overestimation of illite from total K analysis. This is supported by the moderate r^2 value. Improper factors employed in characterizing peak areas might have been responsible for the apparent underestimation, but, this is not supported by the slope of the regression. Alternatively, the y-intercept of this model could indicate either a threshold amount of illite that must be present to be recognized by the X-ray diffraction machine or could indicate the presence of an additional, K-bearing, nonmicaceous mineral such as feldspar in small constant amounts that was not considered in estimating the mineral composition of the clay-sized fraction of the samples studied.

Modification X tended to overestimate smectite and underestimate kaolinite at values of relative smectite and kaolinite composition greater than 25% and 44%, respectively. An apparent overestimation of kaolinite and underestimation of smectite occurs at compositions below these values. Since regression models testing the accuracy of the

illite estimations indicated a consistent error not directly involved with the X-ray diffraction pattern characterization process, it was inferred that Modification X overestimates smectite and underestimates kaolinite.

Significant bias caused by the apparent amount of the mineral present was found for all modifications tested in estimating smectite, illite and kaolinite. However, Modification X was shown to be least affected by the amount of mineral present. Based on a comparison of the SEE and r^2 values, the estimates of the relative illite composition appear to be less accurate than estimates of the relative smectite content. Illite and smectite are estimated with greater accuracy and precision than kaolinite.

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APPENDIX

Table 14. ANOVA Table (Over All Three Soils Tested)

Source of Variation	DF	SS	MS	F	Expected MS
$\bar{Y}_A - \bar{\bar{Y}}$ Among Soils	2	$\frac{\overset{a\ b\ c\ d\ n}{\Sigma(\Sigma\Sigma\Sigma\Sigma Y)^2}}{bcdn} - CT$	$\frac{SS_{Soils}}{2}$	$\frac{MS_{Soils}}{MS_{Clay\ Sep.}}$	$\sigma^2 + n\sigma^2_{D\subset C} + nd\sigma^2_{C\subset B} + ndc\sigma^2_{B\subset A} + ndcb\frac{\Sigma\sigma^2}{a-1}$
$\bar{Y}_B - \bar{Y}_A$ Among Clay Sep. within Soils	6	$\frac{\overset{a\ b\ \ c\ d\ n}{\Sigma\Sigma(\Sigma\Sigma\Sigma Y)^2}}{a\ b\ \ cdn} - \frac{\overset{a\ b\ c\ d\ n}{\Sigma(\Sigma\Sigma\Sigma\Sigma Y)^2}}{bcdn}$	$\frac{SS_{Clay\ Sep.}}{6}$	$\frac{MS_{Clay\ Sep.}}{MS_{Slide}}$	$\sigma^2 + n\sigma^2_{D\subset C} + nd\sigma^2_{C\subset B} + ndc\sigma^2_{B\subset A}$
$\bar{Y}_C - \bar{Y}_B$ Among Slides within Clay Sep.	18	$\frac{\overset{a\ b\ c\ d\ n}{\Sigma\Sigma\Sigma(\Sigma\Sigma Y)^2}}{dn} - \frac{\overset{a\ b\ \ c\ d\ n}{\Sigma\Sigma(\Sigma\Sigma\Sigma Y)^2}}{cdn}$	$\frac{SS_{Slide}}{18}$	$\frac{MS_{Slide}}{MS_{Field/Slide}}$	$\sigma^2 + n\sigma^2_{D\subset C} + nd\sigma^2_{C\subset B}$
$\bar{Y}_D - \bar{Y}_C$ Among Field/Slide within Slides	54	$\frac{\overset{a\ b\ c\ d\ \ n}{\Sigma\Sigma\Sigma\Sigma(\Sigma Y)^2}}{n} - \frac{\overset{a\ b\ c\ \ d\ n}{\Sigma\Sigma\Sigma(\Sigma\Sigma Y)^2}}{dn}$	$\frac{SS_{Field/Slide}}{54}$	$\frac{MS_{Field/Slide}}{MS_{Read/Field}}$	$\sigma^2 + n\sigma^2_{D\subset C}$
$\bar{Y} - \bar{Y}_D$ Within Field/Slide (Read/Field)	162	$\overset{a\ b\ c\ d\ n}{\Sigma\Sigma\Sigma\Sigma\Sigma Y^2} - \frac{\overset{a\ b\ c\ d\ \ n}{\Sigma\Sigma\Sigma\Sigma(\Sigma Y)^2}}{n}$	$\frac{SS_{Read/Field}}{162}$		σ^2
TOTAL	242	$\overset{a\ b\ c\ d\ n}{\Sigma\Sigma\Sigma\Sigma\Sigma Y^2} - CT$			

Soils = 3
Clay Sep. = 3
Slide = 3
Field/Slide = 3
Read/Slide = 3

$$C.T. = \frac{\overset{a\ b\ c\ d\ n}{(\Sigma\Sigma\Sigma\Sigma\Sigma Y)^2}}{abcdn}$$

Table 15. Peak Area Measurements (m^2) for the Preliminary Study

Sample No.	17A Mg-EG	10A Mg-EG	14A Mg-EG	14A K350	7A K350	7A MgEG	3.5A MgEG	3.3A MgEG
11111	5.10	0.21	0.19	0.50	1.35	0.94	0.46	0.50
11112	7.92	0.24	0.36	0.56	1.30	0.95	0.46	0.42
11113	5.00	0.18	0.36	0.43	1.31	0.93	0.46	0.44
11121	3.19	0.14	0.15	0.23	1.16	1.08	0.42	0.66
11122	3.24	0.28	0.25	0.22	1.16	1.08	0.44	0.66
11123	3.70	0.23	0.27	0.31	1.19	0.93	0.44	0.65
11131	4.23	0.16	0.29	0.36	1.41	0.75	0.40	0.55
11132	4.21	0.17	0.15	0.40	1.45	0.75	0.42	0.49
11133	4.13	0.14	0.28	0.32	1.49	0.75	0.40	0.61
11211	4.70	0.22	0.27	0.36	1.23	1.05	0.58	0.53
11212	4.70	0.23	0.23	0.45	1.24	1.05	0.57	0.44
11213	4.70	0.16	0.25	0.39	1.24	1.05	0.60	0.44
11221	4.05	0.27	0.26	0.24	0.90	0.95	0.46	0.61
11222	4.55	0.18	0.25	0.23	0.88	0.95	0.46	0.66
11223	4.63	0.23	0.19	0.17	0.91	0.90	0.46	0.72
11231	5.39	0.24	0.19	0.62	1.33	0.89	0.40	0.60
11232	6.12	0.18	0.28	0.74	1.31	0.87	0.40	0.59
11233	5.69	0.18	0.28	0.77	1.29	1.07	0.50	0.59
11311	5.00	0.24	0.28	0.47	1.30	1.19	0.66	0.63
11312	4.50	0.22	0.24	0.47	1.35	1.20	0.66	0.56
11313	5.05	0.20	0.28	0.43	1.35	1.21	0.68	0.60
11321	3.84	0.20	0.21	0.21	0.79	1.11	0.62	0.65
11322	4.37	0.22	0.15	0.30	0.79	1.13	0.60	0.63
11323	4.37	0.22	0.25	0.27	0.79	1.16	0.60	0.70
11331	6.53	0.25	0.35	0.78	1.27	1.25	0.60	0.60
11332	7.25	0.28	0.40	0.67	1.26	1.27	0.58	0.63
11333	6.47	0.26	0.40	0.76	1.25	1.27	0.60	0.70
12111	4.60	0.17	0.18	0.47	1.33	1.03	0.52	0.50
12112	4.14	0.18	0.21	0.58	1.38	1.04	0.50	0.50
12113	4.23	0.16	0.18	0.37	1.31	1.05	0.48	0.55
12121	3.78	0.14	0.18	0.27	1.28	0.97	0.54	0.60
12122	4.10	0.14	0.20	0.23	1.31	0.96	0.50	0.55
12123	3.36	0.16	0.24	0.41	1.31	1.00	0.52	0.55
12131	2.42	0.19	0.21	0.60	1.30	0.75	0.33	0.38
12132	2.42	0.15	0.20	0.51	1.30	0.77	0.32	0.34
12211	4.50	0.18	0.31	0.29	2.07	1.17	0.61	0.55
12212	4.79	0.25	0.28	0.45	2.08	1.16	0.62	0.55
12213	5.30	0.21	0.31	0.28	2.07	1.20	0.58	0.55
12221	4.85	0.18	0.14	0.21	1.19	1.09	0.61	0.60
12222	3.84	0.17	0.21	0.21	1.26	1.09	0.64	0.63
12223	4.23	0.18	0.21	0.13	1.25	1.15	0.60	0.65

Table 15 (continued)

Sample No.	17A Mg-EG	10A Mg-EG	14A Mg-EG	14A K350	7A K350	7A MgEG	3.5A MgEG	3.3A MgEG
12231	6.13	0.20	0.32	0.51	1.87	0.97	0.38	0.50
12232	6.13	0.23	0.28	0.39	1.86	0.94	0.40	0.49
12233	6.25	0.20	0.32	0.57	1.86	0.97	0.38	0.52
12311	4.55	0.20	0.30	0.35	1.29	0.96	0.52	0.55
12312	4.27	0.19	0.21	0.49	1.25	0.97	0.52	0.48
12313	4.19	0.17	0.24	0.36	1.22	0.99	0.48	0.55
12321	3.85	0.18	0.27	0.13	0.95	0.86	0.40	0.50
12322	3.47	0.15	0.16	0.12	0.97	0.93	0.44	0.50
12323	3.28	0.12	0.14	0.23	1.01	0.83	0.42	0.50
12331	6.46	0.13	0.25	0.54	1.38	0.87	0.48	0.50
12332	6.00	0.15	0.28	0.56	1.38	0.89	0.46	0.55
12333	6.25	0.18	0.32	0.45	1.37	0.94	0.44	0.55
13111	4.73	0.16	0.21	0.34	1.41	0.81	0.28	0.27
13112	4.67	0.14	0.23	0.39	1.45	0.77	0.33	0.27
13113	4.73	0.20	0.22	0.45	1.44	0.77	0.45	0.39
13121	3.11	0.09	0.13	0.16	1.24	0.83	0.40	0.48
13122	2.65	0.10	0.09	0.19	1.25	0.74	0.44	0.36
13123	2.29	0.08	0.11	0.19	1.23	0.70	0.42	0.40
13131	4.23	0.14	0.23	0.67	1.63	0.43	0.20	0.20
13132	4.50	0.12	0.13	0.47	1.59	0.49	0.25	0.24
13133	4.74	0.11	0.10	0.45	1.61	0.44	0.37	0.16
13211	4.41	0.14	0.28	0.52	1.26	1.03	0.52	0.57
13212	4.55	0.11	0.21	0.45	1.23	1.01	0.48	0.50
13213	4.50	0.13	0.26	0.39	1.26	1.01	0.48	0.54
13221	3.35	0.10	0.25	0.12	0.86	0.73	0.36	0.34
13222	2.88	0.09	0.06	0.15	0.86	0.73	0.36	0.32
13223	2.88	0.09	0.20	0.20	0.89	0.74	0.38	0.34
13231	5.40	0.09	0.06	0.64	1.46	0.60	0.29	0.30
13232	5.52	0.09	0.15	0.49	1.46	0.65	0.31	0.28
13233	5.58	0.13	0.08	0.64	1.39	0.61	0.31	0.28
13311	4.30	0.12	0.17	0.62	1.57	0.99	0.50	0.50
13312	4.16	0.12	0.16	0.45	1.65	0.90	0.53	0.54
13313	4.50	0.17	0.21	0.65	1.57	0.96	0.54	0.56
13321	4.05	0.12	0.18	0.25	1.53	1.09	0.64	0.48
13322	3.96	0.13	0.20	0.19	1.50	1.12	0.64	0.52
13323	3.87	0.14	0.19	0.17	1.45	1.10	0.66	0.56
13331	7.25	0.21	0.26	0.68	1.69	1.10	0.56	0.44
13332	7.97	0.16	0.28	0.60	1.70	1.14	0.55	0.50
13333	7.84	0.14	0.25	0.54	1.73	1.09	0.53	0.48
21111	0.00	0.40	0.15	0.51	0.71	1.11	0.47	0.47
21112	0.00	0.37	0.21	0.45	0.67	1.13	0.49	0.54

Table 15 (continued)

Sample No.	17A Mg-EG	10A Mg-EG	14A Mg-EG	14A K350	7A K350	7A MgEG	3.5A MgEG	3.3A MgEG
21113	0.00	0.33	0.15	0.41	0.69	1.16	0.59	0.47
21121	0.00	0.27	0.06	0.22	0.36	1.21	0.65	0.70
21122	0.00	0.28	0.10	0.22	0.44	1.19	0.72	0.68
21123	0.00	0.28	0.08	0.22	0.44	1.15	0.77	0.61
21131	0.00	0.36	0.15	0.50	0.64	1.19	0.53	0.58
21132	0.00	0.35	0.16	0.57	0.60	1.17	0.60	0.58
21133	0.00	0.45	0.17	0.62	0.59	1.15	0.51	0.58
21211	0.00	0.35	0.20	0.42	0.50	1.27	0.73	0.70
21212	0.00	0.39	0.24	0.41	0.44	1.31	0.69	0.61
21213	0.00	0.41	0.28	0.44	0.53	1.31	0.63	0.67
21221	0.00	0.51	0.17	0.25	0.25	1.74	0.99	0.89
21222	0.00	0.49	0.19	0.27	0.25	1.73	0.99	0.87
21223	0.00	0.48	0.15	0.23	0.25	1.71	0.85	1.00
21231	0.00	0.38	0.26	0.42	0.86	1.35	0.92	0.68
21232	0.00	0.49	0.16	0.47	0.89	1.32	0.71	0.68
21233	0.00	0.39	0.24	0.41	0.92	1.33	0.94	0.58
21311	0.00	0.40	0.14	0.52	0.69	2.04	1.13	0.96
21312	0.00	0.48	0.19	0.44	0.69	2.05	0.99	1.08
21313	0.00	0.52	0.17	0.44	0.80	2.07	1.03	1.06
21321	0.00	0.43	0.14	0.25	0.35	1.75	0.90	0.84
21322	0.00	0.44	0.20	0.31	0.32	1.73	0.93	0.92
21323	0.00	0.43	0.13	0.25	0.33	1.73	1.07	0.94
21331	0.00	0.89	0.29	0.65	0.92	2.16	1.29	1.04
21332	0.00	0.87	0.25	0.57	1.07	2.23	1.25	1.08
21333	0.00	0.71	0.32	0.47	0.91	2.22	1.05	1.08
22111	0.00	0.73	0.21	0.36	1.03	1.41	0.64	0.59
22112	0.00	0.44	0.21	0.39	0.97	1.39	0.51	0.68
22113	0.00	0.44	0.21	0.29	1.03	1.49	0.60	0.72
22121	0.00	0.49	0116	0.20	0.60	1.99	0.86	0.79
22122	0.00	0.51	0.14	0.25	0.62	2.04	0.71	0.96
22123	0.00	0.51	0.17	0.20	0.64	2.09	0.73	0.94
22131	0.00	0.47	0.41	0.43	1.02	1.53	0.65	0.59
22132	0.00	0.47	0.37	0.47	0.90	1.51	0.53	0.68
22133	0.00	0.49	0.30	0.44	1.01	1.43	0.51	0.72
22211	0.00	0.47	0.16	0.47	0.81	1.36	0.67	0.68
22212	0.00	0.40	0.21	0.39	0.85	1.56	0.66	0.76
22213	0.00	0.47	0.18	0.51	0.84	1.39	0.65	0.76
22221	0.00	0.55	0.17	0.25	0.57	2.09	1.03	1.10
22222	0.00	0.53	0.18	0.25	0.53	2.01	0.99	1.10
22223	0.00	0.52	0.20	0.28	0.48	2.05	0.96	1.06
22231	0.00	0.68	0.30	0.50	1.09	2.04	0.77	0.88

Table 15 (continued)

Sample No.	17A Mg-EG	10A Mg-EG	14A Mg-EG	14A K350	7A K350	7A MgEG	3.5A MgEG	3.3A MgEG
22232	0.00	0.77	0.25	0.56	1.09	2.09	0.81	0.84
22233	0.00	0.61	0.31	0.51	1.07	2.09	0.86	0.88
22311	0.00	0.53	0.21	0.46	0.79	1.73	0.81	1.00
22312	0.00	0.42	0.20	0.41	0.80	1.74	0.83	0.87
22313	0.00	0.44	0.17	0.47	0.80	1.68	1.17	1.02
22321	0.00	0.51	0.19	0.28	0.40	1.92	1.01	1.12
22322	0.00	0.64	0.20	0.26	0.40	1.91	1.04	1.16
22323	0.00	0.49	0.21	0.25	0.41	1.92	1.21	1.01
22331	0.00	0.46	0.36	0.38	1.10	1.77	0.74	0.73
22332	0.00	0.48	0.34	0.44	1.13	1.69	0.75	0.82
22333	0.00	0.50	0.26	0.44	1.10	1.67	0.75	0.88
23111	0.00	0.49	0.24	0.40	0.52	1.43	0.80	0.88
23112	0.00	0.49	0.19	0.35	0.54	1.47	0.91	0.82
23113	0.00	0.51	0.21	0.44	0.50	1.50	0.93	0.94
23121	0.00	0.49	0.17	0.31	0.25	1.66	0.94	1.12
23122	0.00	0.51	0.18	0.25	0.26	1.69	0.95	1.12
23123	0.00	0.50	0.17	0.29	0.25	1.63	0.99	1.00
23131	0.00	0.51	0.35	0.45	1.00	1.35	0.67	0.76
23132	0.00	0.48	0.31	0.45	1.03	1.33	0.67	0.78
23133	0.00	0.50	0.30	0.53	0.97	1.35	0.64	0.68
23211	0.00	0.51	0.29	0.45	0.68	1.53	0.73	0.70
23212	0.00	0.58	0.22	0.46	0.70	1.55	0.76	0.86
23213	0.00	0.60	0.27	0.39	0.68	1.50	0.76	0.77
23221	0.00	0.54	0.18	0.24	0.16	1.75	0.97	1.14
23222	0.00	0.58	0.19	0.21	0.21	1.73	1.01	1.16
23223	0.00	0.56	0.18	0.31	0.18	1.73	0.99	1.03
23231	0.00	0.43	0.28	0.44	0.73	1.43	1.07	1.14
23232	0.00	0.46	0.19	0.67	0.86	1.47	0.93	1.12
23233	0.00	0.47	0.19	0.65	0.84	1.45	1.26	1.14
23311	0.00	0.43	0.24	0.34	0.30	1.37	1.10	1.06
23312	0.00	0.40	0.25	0.29	0.30	1.15	0.97	1.04
23313	0.00	0.40	0.21	0.32	0.28	1.19	0.99	1.02
23321	0.00	0.32	0.11	0.26	0.18	1.26	1.04	0.84
23322	0.00	0.33	0.16	0.22	0.16	1.27	1.04	0.88
23323	0.00	0.33	0.11	0.21	0.15	1.07	0.89	0.86
23331	0.00	0.65	0.29	0.36	0.44	1.53	0.74	0.99
23332	0.00	0.63	0.27	0.47	0.47	1.53	0.71	0.75
23333	0.00	0.60	0.29	0.35	0.47	1.51	0.83	0.75
31111	0.86	0.43	0.45	0.34	1.23	1.70	0.83	0.82
31112	0.91	0.45	0.40	0.37	1.20	1.77	0.84	0.76
31113	1.03	0.45	0.50	0.37	1.20	1.45	0.83	0.78

Table 15 (continued)

Sample No.	17A Mg-EG	10A Mg-EG	14A Mg-EG	14A K350	7A K350	7A MgEG	3.5A MgEG	3.3A MgEG
31121	0.67	0.41	0.36	0.22	0.90	1.38	0.87	0.84
31122	0.63	0.43	0.27	0.19	0.93	1.36	1.09	0.86
31123	0.73	0.42	0.20	0.19	0.91	1.39	0.85	0.84
31131	1.14	0.77	1.02	0.36	1.08	1.40	1.03	0.78
31132	0.99	0.47	0.84	0.23	1.11	1.79	0.82	0.80
31133	1.13	0.67	0.66	0.30	1.10	1.75	0.83	0.72
31211	1.14	0.46	0.40	0.39	1.54	1.44	0.84	0.80
31212	0.97	0.48	0.46	0.39	1.55	1.46	1.05	0.84
31213	1.10	0.48	0.60	0.39	1.55	1.42	0.85	0.84
31221	0.78	0.51	0.40	0.14	1.07	1.44	0.89	0.84
31222	0.64	0.50	0.34	0.16	1.26	1.52	0.91	0.84
31223	0.65	0.50	0.43	0.37	1.04	1.53	0.89	0.84
31231	1.27	0.50	0.50	0.48	1.56	1.44	0.84	0.78
31232	1.56	0.54	0.65	0.40	1.49	1.80	0.86	0.82
31233	1.59	0.48	0.70	0.43	1.50	1.44	0.83	0.80
31311	0.75	0.48	0.36	0.47	1.09	1.83	0.86	0.76
31312	0.84	0.48	0.49	0.36	1.11	1.83	0.89	0.84
31313	0.90	0.50	0.37	0.43	1.12	1.85	0.85	0.82
31321	0.71	0.45	0.24	0.15	0.83	1.46	0.88	0.82
31322	0.69	0.46	0.25	0.28	0.85	1.48	0.89	0.82
31323	0.60	0.49	0.55	0.16	0.87	1.85	0.93	0.84
31331	1.28	0.48	0.63	0.29	0.95	1.48	0.84	0.76
31332	1.38	0.50	0.66	0.25	0.95	1.80	0.85	0.76
31333	1.36	0.53	0.55	0.22	0.97	1.80	0.83	0.74
32111	0.82	0.50	0.38	0.25	1.33	1.73	0.79	0.76
32112	0.94	0.75	0.49	0.41	1.28	1.73	0.85	0.80
32113	0.82	0.63	0.35	0.27	1.35	1.77	1.00	0.80
32121	0.49	0.43	0.26	0.19	0.96	1.50	0.90	0.76
32122	0.46	0.43	0.28	0.17	0.84	1.50	0.93	0.76
32123	0.58	0.41	0.27	0.18	0.84	1.68	0.93	0.78
32131	1.10	0.69	0.52	0.27	1.35	1.70	0.91	0.70
32132	1.24	0.52	0.36	0.29	1.37	1.65	0.93	0.72
32133	1.24	0.63	0.67	0.41	1.38	1.70	0.91	0.74
32211	0.64	0.55	0.36	0.30	1.30	1.57	0.85	0.66
32212	0.70	0.63	0.27	0.25	1.33	1.50	0.69	0.64
32213	0.82	0.66	0.40	0.22	1.31	1.57	0.85	0.64
32221	0.56	0.40	0.24	0.16	0.85	1.43	0.71	0.58
32222	0.50	0.40	0.23	0.12	0.83	1.47	0.61	0.56
32223	0.42	0.53	0.19	0.16	0.88	1.45	0.59	0.58
32231	1.25	0.63	0.55	0.28	1.50	1.70	0.96	0.76
32232	1.18	0.52	0.40	0.28	1.50	1.67	0.78	0.78

Table 15 (continued)

Sample No.	17A Mg-EG	10A Mg-EG	14A Mg-EG	14A K350	7A K350	7A MgEG	3.5A MgEG	3.3A MgEG
32233	1.19	0.61	0.55	0.30	1.50	1.70	0.99	0.80
32311	0.80	0.66	0.37	0.39	1.38	1.67	0.99	0.80
32312	0.70	0.61	0.38	0.30	1.41	1.70	0.81	0.78
32313	0.85	0.63	0.39	0.47	1.40	1.75	1.05	0.82
32321	0.58	0.40	0.24	0.25	1.15	1.50	0.94	0.74
32322	0.51	0.41	0.24	0.16	1.18	1.55	0.75	0.74
32323	0.60	0.44	0.17	0.19	1.18	1.53	0.95	0.74
32331	1.22	0.65	0.46	0.30	1.47	2.13	1.01	0.78
32332	1.24	0.66	0.66	0.37	1.45	1.75	1.04	0.82
32333	1.20	0.63	0.69	0.30	1.47	1.75	1.05	0.84
33111	0.77	0.59	0.36	0.31	0.49	1.13	0.61	0.82
33112	0.85	0.63	0.28	0.42	0.52	1.15	0.74	0.86
33113	1.04	0.51	0.44	0.39	0.52	1.15	0.76	0.84
33121	0.53	0.49	0.23	0.17	0.43	1.17	0.80	0.92
33122	0.74	0.49	0.26	0.24	0.43	1.21	0.80	0.87
33123	0.64	0.40	0.20	0.21	0.42	1.50	0.83	0.96
33131	1.62	0.53	0.41	0.32	0.49	1.19	0.74	0.88
33132	1.57	0.46	0.33	0.28	0.49	1.21	0.76	0.79
33133	1.42	0.47	0.58	0.34	0.49	1.45	0.75	0.77
33211	0.79	0.63	0.45	0.35	0.71	1.20	0.75	0.82
33212	0.84	0.56	0.36	0.33	0.71	1.41	0.71	0.82
33213	0.77	0.55	0.45	0.43	0.67	1.21	0.79	0.82
33221	0.62	0.44	0.20	0.27	0.56	1.50	0.81	0.92
33222	0.62	0.55	0.30	0.26	0.55	1.25	0.85	0.90
33223	0.53	0.57	0.21	0.24	0.59	1.25	0.83	0.86
33231	1.29	0.59	0.38	0.72	0.70	1.40	0.67	0.96
33232	1.33	0.61	0.48	0.56	0.67	1.37	0.85	0.98
33233	1.15	0.64	0.55	0.54	0.70	1.40	0.83	0.96
33311	0.73	0.61	0.33	0.26	0.50	1.57	0.87	0.82
33312	0.75	0.63	0.40	0.21	0.54	1.55	0.70	0.80
33313	0.72	0.53	0.38	0.22	0.55	1.55	0.89	0.86
33321	0.56	0.49	0.24	0.11	0.34	1.74	0.81	0.76
33322	0.59	0.49	0.25	0.09	0.32	1.45	0.81	0.76
33323	0.59	0.45	0.24	0.17	0.29	1.40	0.83	0.74
33331	1.49	0.68	0.58	0.34	0.60	1.60	0.94	0.88
33332	1.29	0.65	0.42	0.35	0.59	1.89	0.95	0.86
33333	1.17	0.65	0.63	0.31	0.56	1.55	0.97	0.88

Table 16. Percent Clay Mineral Composition for the Preliminary Study.

Sample No.	SMECT	ILL	VERM	CHLOR	KAOL	QUARTZ
11111	62	10	00	12	11	04
11112	71	09	00	10	09	02
11113	64	09	00	11	12	04
11121	55	10	00	08	19	09
11122	51	17	01	07	17	07
11123	56	14	00	09	14	07
11131	63	09	00	11	11	06
11132	62	10	00	12	11	05
11133	63	09	00	10	11	08
11211	61	11	00	09	14	05
11212	60	12	00	11	13	03
11213	63	09	00	10	14	04
11221	58	15	01	07	14	06
11222	63	10	01	06	13	07
11223	63	13	01	05	12	07
11231	61	11	00	14	10	05
11232	64	07	00	15	09	05
11233	60	08	00	16	11	05
11311	59	11	00	11	14	05
11312	57	11	00	12	15	05
11313	60	10	00	10	15	05
11321	58	12	00	06	17	07
11322	59	12	00	08	15	06
11323	58	12	00	07	15	07
11331	61	09	00	15	12	04
11332	64	10	00	12	11	04
11333	60	10	00	14	12	05
12111	60	09	00	12	13	05
12112	56	10	00	16	14	05
12113	60	09	00	10	15	06
12121	60	09	00	09	15	08
12122	63	09	00	07	15	07
12123	54	10	00	13	16	07
12131	45	14	00	22	14	04
12132	48	12	00	20	15	05
12133	45	14	00	20	17	04
12211	61	10	01	08	16	06
12212	58	12	00	11	14	04
12213	63	10	01	07	14	05

Table 16 (continued)

Sample No.	SMECT	ILL	VERM	CHLOR	KAOL	QUARTZ
12221	64	09	00	06	14	06
12222	59	10	00	06	17	08
12223	60	10	02	04	16	07
12231	66	09	00	11	11	04
12232	67	10	00	09	10	03
12233	66	08	00	12	10	04
12311	61	11	00	09	13	05
12312	58	10	00	13	13	05
12313	60	10	00	10	14	06
12321	61	11	04	04	14	06
12322	61	11	01	04	16	07
12323	60	09	00	08	15	07
12331	69	06	00	12	09	04
12332	66	07	00	12	10	05
12333	68	08	00	10	10	05
13111	67	09	00	10	12	02
13112	67	08	00	11	11	02
13113	64	11	00	12	10	03
13121	62	07	00	06	17	08
13122	59	09	00	09	17	06
13123	57	08	00	09	17	08
13131	64	08	00	20	07	01
13132	69	07	00	14	07	02
13133	72	07	00	14	07	01
13211	59	07	00	14	14	06
13212	62	06	00	12	14	06
13213	62	07	00	11	14	06
13221	64	08	01	05	14	05
13222	64	08	00	07	16	06
13223	62	08	00	09	16	06
13231	69	05	00	16	08	03
13232	71	05	00	13	08	03
13233	68	06	00	16	07	02
13311	58	07	00	17	13	05
13312	60	07	00	13	13	07
13313	57	09	00	17	12	05
13321	62	07	00	08	17	06
13322	62	08	00	06	17	07
13323	61	09	01	05	17	07

Table 16 (continued)

Sample No.	SMECT	ILL	VERM	CHLOR	KAOL	QUARTZ
13331	67	08	00	13	10	03
13332	70	06	00	11	10	03
13333	72	05	00	10	10	03
21111	00	41	00	26	29	04
21112	00	39	00	24	30	07
21113	00	37	00	23	33	06
21121	00	33	00	14	37	15
21122	00	35	00	14	37	15
21123	00	36	00	14	37	13
21131	00	37	00	25	30	08
21132	00	35	00	28	29	08
21133	00	41	00	28	26	05
21211	00	35	00	21	32	11
21212	00	39	00	21	33	08
21213	00	39	00	21	31	09
21221	00	43	00	10	36	11
21222	00	41	00	11	37	11
21223	00	41	00	10	36	13
21231	00	37	00	21	33	10
21232	00	43	00	21	29	07
21233	00	39	00	21	33	07
21311	00	30	00	19	38	12
21312	00	35	00	16	37	13
21313	00	37	00	15	36	12
21321	00	38	00	11	39	11
21322	00	37	00	13	37	13
21323	00	38	00	11	38	13
21331	00	48	00	18	29	05
21332	00	48	00	16	31	05
21333	00	43	00	14	34	08
22111	00	57	00	14	28	01
22112	00	41	00	18	33	08
22113	00	42	00	14	35	09
22121	00	41	00	08	42	09
22122	00	40	00	10	40	11
22123	00	40	00	08	41	11
22131	00	42	00	19	34	05
22132	00	40	00	20	32	07
22133	00	42	00	19	31	08

Table 16 (continued)

Sample No.	SMECT	ILL	VERM	CHLOR	KAOL	QUARTZ
22211	00	42	00	21	30	07
22212	00	36	00	18	35	11
22213	00	40	00	22	30	09
22221	00	40	00	09	38	13
22222	00	40	00	09	38	13
22223	00	39	00	10	38	13
22231	00	44	00	16	33	06
22232	00	47	00	17	32	04
22233	00	41	00	17	35	07
22311	00	39	00	17	32	11
22312	00	35	00	17	36	12
22313	00	35	00	19	33	14
22321	00	39	00	11	37	14
22322	00	45	00	09	34	12
22323	00	39	00	10	38	13
22331	00	39	00	16	37	08
22332	00	39	00	18	34	09
22333	00	40	00	17	33	10
23111	00	42	00	17	30	11
23112	00	43	00	15	32	10
23113	00	41	00	18	30	11
23121	00	39	00	12	33	15
23122	00	41	00	10	34	15
23123	00	41	00	12	34	13
23131	00	44	00	19	29	08
23132	00	42	00	20	29	09
23133	00	42	00	23	29	07
23211	00	43	00	19	32	07
23212	00	45	00	18	30	08
23213	00	48	00	16	30	06
23221	00	42	00	09	34	14
23222	00	45	00	08	33	14
23223	00	43	00	12	33	12
23231	00	35	00	18	29	17
23232	00	34	00	25	27	14
23233	00	35	00	24	27	15
23311	00	38	00	15	30	16
23312	00	39	00	14	28	18
23313	00	39	00	15	29	17

Table 16 (continued)

Sample No.	SMECT	ILL	VERM	CHLOR	KAOL	QUARTZ
23321	00	35	00	14	34	16
23322	00	36	00	12	35	17
23323	00	39	00	12	31	18
23331	00	49	00	13	29	09
23332	00	48	00	18	29	05
23333	00	49	00	14	31	06
31111	15	30	04	12	30	09
31112	16	32	01	13	31	07
31113	18	31	05	13	25	08
31121	14	33	06	09	28	11
31122	13	36	03	08	28	11
31123	15	36	00	08	29	11
31131	15	39	17	09	18	03
31132	15	28	18	07	26	07
31133	16	38	10	09	25	03
31211	20	32	00	14	25	08
31212	17	33	02	14	25	08
31213	18	31	07	13	23	08
31221	14	37	09	05	26	08
31222	12	38	07	06	29	09
31223	12	36	02	13	28	08
31231	21	33	01	16	23	07
31232	22	30	07	11	25	06
31233	24	27	08	13	21	07
31311	13	33	00	16	31	07
31312	14	32	04	12	30	08
31313	15	33	00	14	31	07
31321	14	37	04	06	30	10
31322	14	37	00	11	29	09
31323	10	33	13	05	31	08
31331	20	30	11	09	23	06
31332	20	29	12	07	26	06
31333	20	31	10	07	27	05
32111	14	35	05	09	30	07
32112	14	44	02	12	25	03
32113	13	41	03	09	29	05
32121	11	37	03	08	32	09
32122	10	37	05	07	32	09
32123	12	33	04	07	34	10

Table 16 (continued)

Sample No.	SMECT	ILL	VERM	CHLOR	KAOL	QUARTZ
32131	16	41	07	08	25	03
32132	21	35	02	10	27	05
32133	17	36	07	12	24	04
32211	12	41	02	11	29	05
32212	13	46	01	09	28	03
32213	14	44	06	07	26	02
32221	13	37	04	07	33	06
32222	12	37	05	06	34	06
32223	09	47	01	07	32	04
32231	18	37	08	08	25	04
32232	19	34	04	09	27	06
32233	18	36	07	09	25	05
32311	13	43	00	13	27	05
32312	12	41	03	10	29	05
32313	13	39	00	15	27	05
32321	13	35	00	11	33	09
32322	11	36	03	07	34	09
32323	13	38	00	08	33	09
32331	17	36	05	08	30	04
32332	17	36	08	10	24	05
32333	17	35	11	08	24	05
33111	14	44	02	12	21	07
33112	15	44	00	15	20	07
33113	19	37	02	14	21	08
33121	11	42	03	07	25	12
33122	05	40	01	10	25	10
33123	13	33	00	09	31	14
33131	26	34	03	10	19	08
33132	27	32	02	10	21	08
33133	22	30	08	11	23	07
33211	14	44	03	12	21	06
33212	15	40	01	12	25	07
33213	14	40	01	16	22	07
33221	12	35	00	11	30	12
33222	12	43	02	10	24	09
33223	11	46	00	10	25	09
33231	18	34	00	21	20	07
33232	20	36	00	17	20	08
33233	17	38	00	16	21	07

Table 16 (continued)

Sample No.	SMECT	ILL	VERM	CHLOR	KAOL	QUARTZ
33311	13	42	02	09	27	06
33312	13	42	06	07	26	05
33313	13	38	06	08	28	08
33321	11	38	05	04	34	08
33322	12	40	07	04	30	08
33323	13	39	03	07	38	09
33331	20	37	07	07	22	09
33332	18	37	02	09	27	05
33333	17	37	09	10	22	06

Table 17. ANOVA for Main Effects on the 17A Peak (MgEG) for All Three Soils Studied–Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Squares	Variance Component	F-Value
Total	242	1108.160	4.579	6.635	
Soils	2	972.767	486.383	5.997	780.43*
Clay Sep.	6	3.739	0.623	-0.028	0.45
Slide	18	24.946	1.386	-0.045	0.77
Field/Slide	54	96.770	1.792	0.577	29.21*
Error	162	9.937	0.061	0.061	

* - significant at $\alpha = 0.05$

Table 18. ANOVA for Main Effects on the 10A Peak (MgEG) for All Three Soils Studied–Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Squares	Variance Component	F-Value
Total	242	8.243	0.034	0.047	
Soils	2	6.294	3.147	0.038	81.14*
Clay Sep.	6	0.233	0.039	0.001	2.08
Slide	18	0.336	0.019	0.000	0.97
Field/Slide	54	1.028	0.019	0.006	8.77*
Error	162	0.353	0.002	0.002	

* - significant at $\alpha = 0.05$

Table 19. ANOVA for Main Effects on the 14A Peak (MgEG) for All Three Soils Studied–Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Squares	Variance Component	F-Value
Total	242	4.991	0.021	0.028	
Soils	2	2.043	1.021	0.012	16.34*
Clay Sep.	6	0.375	0.063	0.002	10.64*
Slide	18	0.106	0.006	-0.003	0.17
Field/Slide	54	1.854	0.034	0.010	9.07*
Error	162	0.613	0.004	0.004	

* - significant at $\alpha = 0.05$

Table 20. ANOVA for Main Effects on the 14A Peak (K350) for All Three Soils Studied–Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Squares	Variance Component	F-Value
Total	242	5.170	0.021	0.027	
Soils	2	0.550	0.275	0.003	18.45*
Clay Sep.	6	0.089	0.015	0.000	0.59
Slide	18	0.451	0.025	-0.004	0.38
Field/Slide	54	3.534	0.065	0.021	19.45*
Error	162	0.545	0.003	0.003	

* - significant at $\alpha = 0.05$

Table 21. ANOVA for Main Effects on the 7A Peak (MgEG) for All Three Soils Studied–Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Squares	Variance Component	F-Value
Total	242	134.659	0.143	0.185	
Soils	2	20.615	10.307	0.121	21.82*
Clay Sep.	6	2.835	0.472	0.005	1.44
Slide	18	5.903	0.328	0.028	4.16*
Field/Slide	54	4.259	0.079	0.024	12.20*
Error	162	1.047	0.006	0.006	

* - significant at $\alpha = 0.05$

Table 22. ANOVA for Main Effects on the 7A Peak (K350) for All Three Soils Studied–Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Squares	Variance Component	F-Value
Total	242	44.365	0.183	0.224	
Soils	2	20.073	10.036	0.102	5.76*
Clay Sep.	6	10.450	1.742	0.056	7.67*
Slide	18	4.087	0.227	0.005	1.28
Field/Slide	54	9.594	0.178	0.059	177.52*
Error	162	0.162	0.001	0.001	

* - significant at $\alpha = 0.05$

Table 23. ANOVA for Main Effects on the 3.5A Peak (MgEG) for All Three Soils Studied—Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Squares	Variance Component	F-Value
Total	242	12.311	0.051	0.068	
Soils	2	7.397	3.698	0.045	63.14*
Clay Sep.	6	0.351	0.059	-0.002	0.49
Slide	18	2.137	0.119	0.100	3.75*
Field/Slide	54	1.711	0.032	0.009	7.19*
Error	162	0.714	0.004	0.004	

* - significant at $\alpha = 0.05$

Table 24. ANOVA for Main Effects on the 3.3A Peak (MgEG) for All Three Soils Studied—Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Squares	Variance Component	F-Value
Total	242	10.449	0.043	0.055	
Soils	2	5.913	2.956	0.034	16.96*
Clay Sep.	6	1.046	0.174	0.003	2.18
Slide	18	1.441	0.080	0.005	2.49*
Field/Slide	54	1.734	0.032	0.010	16.49*
Error	162	0.315	0.002	0.002	

* - significant at $\alpha = 0.05$

Table 25. ANOVA of Percent Smectite Composition for All Three Soils Studied–Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Squares	Variance Component	F-Value
Total	242	16.925	0.070	0.104	
Soil	2	16.596	8.298	0.102	1463.49*
Clay Sep.	6	0.034	0.006	0.000	1.85
Slide	18	0.055	0.003	0.000	0.82
Field/Slide	54	0.201	0.004	0.001	15.28*
Error	162	0.039	0.000	0.000	

* - significant at $\alpha = 0.05$

Table 26. ANOVA of Percent Illite Composition for All Three Soils Studied–Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Squares	Variance Component	F-Value
Total	242	5.003	0.021	0.030	
Soil	2	4.656	2.328	0.029	180.88*
Clay Sep.	6	0.077	0.013	0.000	11.92*
Slide	18	0.019	0.001	0.000	0.37
Field/Slide	54	0.158	0.003	0.001	5.09*
Error	162	0.093	0.001	0.001	

* - significant at $\alpha = 0.05$

Table 27. ANOVA of Percent Vermiculite Composition for All Three Soils Studied–Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Square	Variance Component	F-Value
Total	242	0.219	0.001	0.001	
Soil	2	0.090	0.045	0.001	18.58*
Clay Sep.	6	0.015	0.002	0.000	3.55*
Slide	18	0.012	0.001	0.000	0.63
Field/Slide	54	0.059	0.001	0.000	4.06*
Error	162	0.043	0.000	0.000	

* - significant at $\alpha = 0.05$

Table 28. ANOVA of Percent Chlorite Composition for All Three Soils Studied–Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Squares	Variance Component	F-Value
Total	242	0.579	0.002	0.003	
Soil	2	0.185	0.093	0.001	29.77*
Clay Sep.	6	0.019	0.003	0.000	0.83
Slide	18	0.067	0.004	0.000	0.77
Field/Slide	54	0.261	0.005	0.001	16.66*
Error	162	0.047	0.000	0.000	

* - significant at $\alpha = 0.05$

Table 29. ANOVA of Percent Kaolinite Composition for All Three Soils Studied–Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Square	Variance Component	F-Value
Total	242	1.987	0.008	0.012	
Soil	2	1.693	0.847	0.010	99.01*
Clay Sep.	6	0.051	0.009	0.000	5.65*
Slide	18	0.027	0.001	0.000	0.47
Field/Slide	54	0.172	0.003	0.001	11.68*
Error	162	0.044	0.000	0.000	

* - significant at $\alpha = 0.05$

Table 30. ANOVA of Percent Quartz Composition for All Soils Studied–Preliminary Study

Source	Degrees of Freedom	Sum of Squares	Mean Squares	Variance Component	F-Value
Total	242	0.291	0.001	0.001	
Soil	2	0.114	0.057	0.001	16.06*
Clay Sep.	6	0.021	0.003	0.000	4.06*
Slide	18	0.016	0.001	0.000	0.40
Field/Slide	54	0.119	0.002	0.001	17.03*
Error	162	0.021	0.000	0.000	

* - significant at $\alpha = 0.05$

Table 31. Complete Peak Area Measurements (in.^{2}) for the Main Study

Sample No.	17A MgEG	10A MgEG	14A MgEG	14A K350	7A K350	7A K500	7A MgEG	3.5A MgEG	3.3A MgEG
11	4.45	0.35	0.52	0.59	0.18	0.00	0.73	0.47	0.47
12	5.65	0.38	0.60	0.62	0.26	0.00	0.65	0.41	0.44
21	2.40	0.39	0.59	0.41	0.27	0.00	0.63	0.43	0.67
22	2.50	0.33	0.34	0.49	0.48	0.00	0.36	0.37	0.58
31	1.39	0.23	0.19	0.42	0.25	0.00	0.36	0.38	0.61
32	2.09	0.28	0.19	0.54	0.27	0.00	0.48		0.54
41	2.07	0.14	0.21	0.44	0.39	0.00	0.43	0.23	0.36
42	1.85	0.12	0.18	0.52	0.18	0.00	0.59		0.50
51	5.17	0.09	0.25	0.33	0.73	0.00	0.61	0.33	0.33
52	2.89	0.06	0.17	0.48	0.21	0.00	0.60		0.42
61	8.56	0.22	0.72	0.66	0.39	0.00	1.38	0.59	0.56
62	4.27	0.14	0.30	0.62	0.47	0.00	0.76		0.55
71	15.53	0.28	0.00	0.75	0.22	0.00	1.01	0.52	0.73
72	7.20	0.16	0.35	0.59	0.13	0.00	0.66	0.37	0.65
81	5.04	0.81	1.29	0.90	0.29	0.00	2.00	1.20	1.10
82	3.19	0.46	0.63	0.63	0.28	0.00	1.32	0.71	0.72
91	0.93	0.79	0.42	0.80	0.45	0.00	1.98	0.91	0.62
92	0.91	0.68	0.60	0.88	0.53	0.00	1.85	1.31	0.78
101	0.35	0.53	0.20	0.69	0.46	0.00	1.32	1.05	1.08
102	1.21	0.84	0.69	0.61	0.44	0.00	1.50	1.01	0.91
111	0.22	0.99	0.31	0.89	0.57	0.00	2.51	1.35	0.87
112	0.51	0.89	0.49	0.60	2.11	0.00	2.73	1.40	0.85
121	0.13	1.21	0.31	0.59	1.83	0.00	3.20	1.76	1.04
122	0.31	0.72	0.49	0.69	1.54	0.00	1.80	1.20	0.74
131	0.00	1.01	0.35	0.72	1.64	0.00	2.81	2.24	1.32
132	0.00	0.71	0.35	0.58	1.65	0.14	2.55	1.15	0.75
141	4.61	0.40	0.35	0.42	0.36	0.00	0.72	0.50	0.53
142	4.10	0.38	0.22	0.47	0.44	0.00	0.55	0.36	0.38
151	5.52	0.14	0.35	0.55	0.14	0.00	0.44	0.27	0.44

Table 31 (continued)

Sample No.	17A MgEG	10A MgEG	14A MgEG	14A K350	7A K350	7A K500	7A MgEG	3.5A MgEG	3.3A MgEG
152	6.80	0.17	0.00	0.41	0.12	0.00	0.41	0.27	0.37
161	4.51	0.11	0.00	1.17	0.19	0.00	0.29	0.30	0.70
162	14.61	0.19	0.00	0.52	0.24	0.00	0.42	0.21	0.54
171	6.30	0.11	0.00	0.65	0.21	0.00	0.22	0.21	0.55
172	15.93	0.28	0.00	0.79	0.22	0.00	0.55	0.35	1.06
181	2.68	0.16	0.00	0.78	0.16	0.00	0.15	0.27	0.46
182	13.30	0.35	0.00	0.70	0.11	0.00	0.48	0.34	1.03
191	2.57	0.19	0.00	0.68	0.13	0.00	0.23		0.68
192	16.40	0.38	0.00	0.84	0.13	0.00	0.51	0.35	0.88
201	8.30	0.57	0.00	1.15	0.00	0.00	0.87	0.61	1.03
202	7.57	0.63	0.00	1.31	0.00	0.00	0.93	0.43	0.65
211	6.25	0.60	0.00	0.91	0.12	0.00	0.97	0.73	1.17
212	9.71	0.95	0.00	1.24	0.00	0.00	1.01	0.65	0.87
221	2.59	0.28	0.00	1.08	0.60	0.00	0.49	0.45	0.72
222	11.00	0.53	0.00	0.68	0.14	0.00	1.01	0.67	0.99
231	8.50	0.22	0.00	1.13	0.53	0.00	0.52		0.75
232	4.59	0.16	0.00	0.68	0.16	0.00	0.38		0.63
241	11.00	0.12	0.00	0.79	0.34	0.00	0.39		0.77
242	3.22	0.11	0.00	0.73	0.16	0.00	0.29		0.74
251	8.50	0.34	0.00	0.99	0.19	0.00	0.79		0.62
252	6.70	0.30	0.00	1.01	0.08	0.00	0.95	0.47	0.62
261	8.90	0.50	0.00	1.20	0.13	0.00	1.02	0.46	0.44
262	8.28	0.52	0.00	1.07	0.15	0.00	1.33	0.71	0.71
271	8.87	0.87	0.00	0.80	3.60	0.00	4.13	2.03	1.21
272	8.16	0.88	0.00	0.74	2.84	0.00	4.16	2.11	0.91
281	1.22	1.51	1.18	0.92	0.82	0.00	3.30	2.36	2.10
282	1.57	1.33	0.63	0.69	1.05	0.00	3.24	2.62	1.90
291	0.00	1.59	0.41	0.13	4.72	0.00	5.59	3.78	1.47
292	0.00	1.50	0.23	0.21	5.63	0.00	5.06	3.13	1.15

Table 31 (continued)

Sample No.	17A MgEG	10A MgEG	14A MgEG	14A K350	7A K350	7A K500	7A MgEG	3.5A MgEG	3.3A MgEG
301	0.00	1.10	0.00	0.00	4.63	0.00	6.06	3.19	0.73
302	0.00	0.57	0.00	0.16	2.46	0.00	2.44	1.38	0.35
311	0.00	1.37	0.00	0.23	0.94	0.00	5.03	2.59	1.20
312	0.00	1.61	0.00	0.21	1.79	0.00	4.59	2.64	1.09
321	0.00	2.03	0.44	0.49	2.10	0.00	3.03	2.15	1.91
322	0.00	2.05	0.62	0.61	2.97	0.00	3.56	2.05	1.61
331	0.00	1.65	0.39	0.58	2.03	0.00	3.63	2.31	1.60
332	0.00	1.91	0.49	0.71	2.53	0.00	3.75	2.07	1.54
341	0.00	1.97	0.56	0.56	3.25	0.00	3.47	1.91	1.53
342	0.00	1.92	0.54	0.66	1.24	0.00	3.22	2.11	1.33
351	0.00	1.15	0.00	0.00	1.67	0.00	2.23	1.42	0.89
352	0.00	0.76	0.00	0.00	0.68	0.00	1.93	1.03	0.71
361	0.00	1.69	0.00	0.00	3.97	0.00	5.22	2.75	1.00
362	0.00	1.40	0.44	0.28	1.12	0.00	4.69	2.69	1.11
371	0.00	1.20	0.00	0.00	1.04	0.00	4.78	2.49	0.89
372	0.00	1.29	0.00	0.22	0.81	0.00	4.95	2.41	0.90
381	8.53	0.62	0.00	0.55	0.79	0.00	1.44	1.03	1.40
382	8.45	0.58	0.00	0.49	0.61	0.00	1.33	0.94	1.13
391	6.60	0.88	0.95	0.54	0.42	0.00	1.65	1.49	1.51
392	5.00	0.69	1.05	0.81	0.49	0.00	1.60	1.31	1.67
401	5.85	0.44	0.00	0.45	0.20	0.00	0.82	0.55	0.47
402	5.12	0.53	0.00	0.31	0.23	0.00	0.92	0.48	0.56
411	5.82	0.53	0.80	0.34	0.11	0.00	0.89	0.49	0.58
412	6.24	0.63	0.96	0.30	0.32	0.00	0.97	0.73	0.69
421	0.95	1.26	0.45	0.49	1.15	0.00	2.40	1.43	1.19
422	1.27	1.11	0.45	0.34	1.69	0.00	2.43	1.33	1.10
431	0.00	1.30	0.48	0.40	3.36	0.00	2.66	1.61	1.24
432	0.00	1.58	0.41	0.41	0.96	0.00	2.60	1.39	1.00
441	0.00	1.34	0.43	0.57	3.13	0.00	3.97	2.15	1.07

Table 31 (continued)

Sample No.	17A MgEG	10A MgEG	14A MgEG	14A K350	7A K350	7A K500	7A MgEG	3.5A MgEG	3.3A MgEG
442	0.00	1.40	0.48	0.54	2.63	0.00	3.81	2.06	1.08
451	0.00	0.64	0.00	0.00	0.29	0.00	1.90	1.24	0.95
452	0.00	0.68	0.00	0.18	0.24	0.00	1.90	1.21	1.01
461	0.00	0.84	0.57	0.81	1.13	0.00	7.40	4.63	0.86
462	0.00	0.77	0.55	0.71	1.56	0.00	7.60	4.03	0.80
471	0.00	1.21	0.62	0.49	4.25	0.00	7.50	5.37	1.45
472	0.00	1.00	0.58	0.54	4.06	0.00	7.25	3.80	1.10
481	0.00	1.30	0.63	0.56	4.06	0.00	6.55	4.59	1.39
482	0.00	1.28	0.54	0.41	2.33	0.00	6.35	3.75	1.20
491	0.00	1.35	0.54	0.45	1.14	0.00	3.59	2.81	1.37
492	0.00	1.39	0.54	0.59	0.87	0.00	4.25	2.81	1.52
501	1.53	0.70	0.00	0.39	0.10	0.00	1.75	1.65	1.69
502	2.27	0.90	0.00	0.27	0.14	0.00	2.21	1.57	1.76

Table 32. Complete Data Table for Modification IV

	PSM	PIL	PVM	PCA	PKA	PQU	ECEC	LCEC	TKIL8
1	54.3	15.7	0.0	13.1	15.0	2.0	62.70	56.25	15
2	39.7	22.6	0.0	14.1	17.9	5.6	50.43	47.50	15
3	35.2	21.2	0.0	18.3	17.1	8.2	46.62	42.50	14
4	43.3	10.7	0.0	17.1	21.7	7.1	52.18	42.50	13
5	58.9	4.7	0.0	12.7	18.7	5.0	64.88	51.25	8
6	57.3	6.7	0.0	12.4	19.3	4.1	63.71	46.25	11
7	70.1	5.7	0.0	9.2	11.1	3.7	74.85	62.50	8
8	34.4	21.0	0.0	13.2	27.8	3.7	45.18	32.50	20
9	9.6	30.9	0.0	17.7	40.2	1.6	25.03	21.25	27
10	9.0	33.3	0.0	16.2	35.0	6.3	24.36	20.00	30
11	3.2	34.3	0.0	13.4	47.6	1.4	19.02	20.00	27
12	1.6	36.7	0.0	12.7	47.3	1.7	17.76	17.50	27
13	0.0	33.0	0.0	12.6	50.7	3.6	15.52	17.50	29
14	51.3	19.6	0.0	11.3	15.8	1.9	60.36	40.00	19
15	67.4	7.6	0.0	11.6	10.2	3.5	73.02	58.75	14
16	75.6	4.3	0.0	13.1	4.7	2.2	80.37	72.50	11
17	72.3	5.5	0.0	11.8	5.4	4.9	77.17	62.50	11
18	72.8	8.1	0.0	9.1	5.6	4.5	77.64	63.75	17
19	37.7	15.2	0.0	27.2	9.2	10.7	49.25	42.50	19
20	48.7	17.5	0.0	17.9	13.1	2.8	58.65	48.75	20
21	46.8	21.0	0.0	14.8	13.9	3.4	56.98	43.75	23
22	59.0	12.6	0.0	12.8	12.0	3.5	66.38	48.75	22
23	57.6	8.1	0.0	18.8	9.6	5.9	65.20	46.25	17
24	47.6	6.6	0.0	24.3	10.7	10.8	56.38	53.75	15
25	54.5	11.1	0.0	16.7	14.6	3.1	62.69	47.50	18
26	52.0	14.2	0.0	15.8	16.4	1.5	60.85	37.50	26
27	36.0	16.3	0.0	7.2	38.6	1.8	45.05	28.75	22
28	12.2	35.0	0.0	10.1	37.3	5.4	26.51	22.50	29
29	0.0	35.1	1.9	1.7	60.4	0.8	17.30	17.50	24
30	0.0	28.6	0.0	2.2	69.2	0.0	13.24	12.50	21
31	0.0	37.0	0.0	2.7	59.7	0.5	14.71	17.50	23
32	0.0	50.2	0.0	7.4	40.5	1.9	17.68	12.50	37
33	0.0	44.3	0.0	8.0	46.1	1.5	16.81	12.50	38
34	0.0	49.5	0.0	7.7	42.5	0.2	17.72	10.00	33
35	0.0	46.9	0.0	0.0	51.9	1.1	15.90	23.75	27
36	0.0	36.0	3.5	2.4	57.9	0.2	20.46	17.50	24
37	0.0	33.0	0.0	1.4	65.2	0.0	13.91	17.50	24
38	53.2	16.0	0.0	6.9	18.4	5.4	60.52	47.40	19
39	33.7	20.5	0.0	7.6	31.5	6.5	43.43	42.50	22
40	52.6	20.1	0.0	7.8	18.0	1.5	61.08	47.50	20

Table 32 (continued)

	PSM	PIL	PVM	PCA	PKA	PQU	ECEC	LCEC	TKIL8
41	49.6	19.0	9.1	5.3	15.3	1.6	72.95	38.75	21
42	9.1	40.3	0.9	6.3	41.7	1.6	25.71	17.50	30
43	0.0	47.6	0.0	7.2	44.1	1.1	17.24	12.50	33
44	0.0	38.0	0.0	7.7	53.9	0.3	15.76	12.50	33
45	0.0	35.9	0.0	6.0	51.6	6.5	14.72	31.25	23
46	0.0	16.1	0.0	7.6	75.1	1.1	11.97	20.00	19
47	0.0	20.6	0.6	5.2	71.1	2.5	13.24	17.50	20
48	0.0	26.4	1.0	4.9	66.0	1.7	14.99	17.50	20
49	0.0	37.2	0.0	6.9	53.0	2.8	15.33	12.50	34
50	14.3	30.2	0.0	7.0	37.6	10.9	26.85	30.00	24

Table 33. Complete Data Table for Modification V

	PSM	PIL	PVM	PCA	PKA	PQU	ECEC	LCEC	TKIL8
1	47.2	13.7	0.0	11.3	26.0	1.7	55.58	56.25	15
2	33.7	19.2	0.0	12.0	30.3	4.7	44.07	47.50	15
3	30.0	18.2	0.0	15.6	29.2	7.0	40.93	42.50	14
4	35.6	8.8	0.0	14.1	35.6	5.8	44.33	42.50	13
5	49.8	3.9	0.0	10.6	31.3	4.2	56.04	51.25	8
6	48.0	5.6	0.0	10.4	32.4	3.5	54.68	46.25	11
7	63.2	5.2	0.0	8.2	20.1	3.3	68.23	62.50	8
8	26.9	16.4	0.0	10.3	43.5	2.8	37.12	32.50	20
9	6.9	22.0	0.0	12.6	57.4	1.1	20.17	21.25	27
10	6.7	24.8	0.0	11.9	51.9	4.7	20.17	20.00	30
11	2.2	23.2	0.0	9.1	64.5	1.0	15.47	20.00	27
12	1.1	24.9	0.0	8.6	64.2	1.2	14.63	17.50	27
13	0.0	22.0	0.0	8.3	67.3	2.4	13.01	17.50	29
14	44.3	16.9	0.0	9.7	27.2	1.7	53.23	40.00	19
15	61.2	6.7	0.0	10.5	18.5	3.1	67.04	58.75	14
16	72.2	4.1	0.0	12.6	9.0	2.1	77.14	72.50	11
17	68.5	5.2	0.0	11.2	10.3	4.7	73.58	62.50	11
18	68.9	7.7	0.0	8.6	10.6	4.2	73.91	63.75	17
19	34.6	13.9	0.0	24.9	16.8	9.8	45.84	42.50	19
20	43.0	15.5	0.0	15.8	23.1	2.5	52.76	48.75	20
21	41.2	18.5	0.0	13.0	24.3	3.0	51.08	43.75	23
22	52.7	11.3	0.0	11.5	21.5	3.1	60.18	48.75	22
23	52.5	7.4	0.0	17.1	17.4	5.4	60.19	46.25	17
24	42.9	6.0	0.0	21.9	19.3	9.8	51.67	53.75	15
25	47.6	9.8	0.0	14.6	25.4	2.6	55.81	47.50	18
26	44.7	12.3	0.0	13.6	28.1	1.3	53.49	37.50	26
27	26.0	11.8	0.0	5.1	55.7	1.3	34.71	28.75	22
28	8.9	25.4	0.0	7.3	54.3	3.9	21.52	22.50	29
29	0.0	21.9	1.2	1.0	75.3	0.5	13.79	17.50	24
30	0.0	16.9	0.0	1.3	81.8	0.0	11.11	12.50	21
31	0.0	23.2	0.0	1.7	74.7	0.3	12.22	17.50	23
32	0.0	35.7	0.0	5.2	57.6	1.3	14.89	12.50	37
33	0.0	30.3	0.0	5.5	63.1	1.0	14.03	12.50	38
34	0.0	34.7	0.0	5.4	59.7	0.1	14.83	10.00	33
35	0.0	30.9	0.0	0.0	68.3	0.7	13.22	23.75	27
36	0.0	22.8	2.3	1.5	73.4	0.1	15.88	17.50	24
37	0.0	20.2	0.0	0.8	78.9	0.0	11.58	17.50	24
38	44.9	13.5	0.0	5.8	31.2	4.5	52.32	47.40	19
39	25.7	15.6	0.0	5.7	47.9	5.0	34.97	42.50	22
40	44.6	16.9	0.0	6.6	30.4	1.3	52.95	47.50	20

Table 33 (continued)

	PSM	PIL	PVM	PCA	PKA	PQU	ECEC	LCEC	TKIL8
41	43.1	16.5	7.9	4.6	26.6	1.4	64.27	38.75	21
42	6.4	28.5	0.7	4.5	58.9	1.1	20.60	17.50	30
43	0.0	33.1	0.0	4.9	61.2	0.8	14.41	12.50	33
44	0.0	24.7	0.0	5.0	70.1	0.2	13.04	12.50	33
45	0.0	23.6	0.0	3.9	68.0	4.3	12.42	31.25	23
46	0.0	9.2	0.0	4.3	85.8	0.6	10.28	20.00	19
47	0.0	12.0	0.3	3.0	83.1	1.4	11.07	17.50	20
48	0.0	15.9	0.6	3.0	79.5	1.0	12.16	17.50	20
49	0.0	24.3	0.0	4.5	69.3	1.9	12.81	12.50	34
50	10.4	22.0	0.0	5.1	54.5	7.9	21.71	30.00	24

Table 34. Complete Data Table for Modification IX.

	PSM	PIL	PVM	PCA	PKA	PQU	ECEC	LCEC	TKIL8
1	48.6	17.7	0.0	14.6	16.8	2.2	58.11	56.25	15
2	34.5	24.6	0.0	15.4	19.4	6.1	46.18	47.50	15
3	30.3	22.8	0.0	19.7	18.4	8.8	42.52	42.50	14
4	37.9	11.7	0.0	18.8	23.8	7.8	47.62	42.50	13
5	59.3	5.7	0.0	13.8	17.5	3.7	65.65	51.25	8
6	51.8	7.5	0.0	14.0	21.9	4.7	59.08	46.25	11
7	65.3	6.7	0.0	10.6	12.9	4.3	70.81	62.50	8
8	29.5	22.5	0.0	14.1	29.9	3.9	41.12	32.50	20
9	7.8	31.4	0.0	18.0	41.0	1.6	23.54	21.25	27
10	7.4	34.0	0.0	16.5	35.7	6.4	23.00	20.00	30
11	2.6	34.5	0.0	13.5	47.9	1.4	18.52	20.00	27
12	1.3	36.8	0.0	12.7	47.5	1.7	17.51	17.50	27
13	0.0	33.0	0.0	12.6	50.7	3.6	15.52	17.50	29
14	45.8	21.8	0.0	12.5	17.6	2.1	55.86	40.00	19
15	62.3	8.6	0.0	13.4	11.8	4.0	68.81	58.75	14
16	71.3	5.0	0.0	15.5	5.6	2.6	76.93	72.50	11
17	67.6	6.4	0.0	13.7	6.3	5.8	73.31	62.50	11
18	68.1	9.5	0.0	10.6	6.5	5.2	73.75	63.75	17
19	32.7	16.4	0.0	29.4	9.9	11.6	45.17	42.50	19
20	43.2	19.3	0.0	19.8	14.6	3.1	54.18	48.75	20
21	41.4	23.2	0.0	16.3	15.4	3.7	52.58	43.75	23
22	53.5	14.3	0.0	14.6	13.6	4.0	61.89	48.75	22
23	52.1	9.1	0.0	21.2	10.9	6.7	60.70	46.25	17
24	42.1	7.3	0.0	26.8	11.8	11.9	51.81	53.75	15
25	49.0	12.5	0.0	18.7	16.3	3.4	58.18	47.50	18
26	46.5	15.9	0.0	17.7	18.3	1.7	56.33	37.50	26
27	31.0	17.5	0.0	7.7	41.6	2.0	40.75	28.75	22
28	10.0	35.9	0.0	10.4	38.2	5.6	24.72	22.50	29
29	0.0	35.1	1.9	1.7	60.4	0.8	17.30	17.50	24
30	0.0	28.6	0.0	2.2	69.2	0.0	13.24	12.50	21
31	0.0	37.0	0.0	2.7	59.7	0.5	14.71	17.50	23
32	0.0	50.2	0.0	7.4	40.5	1.9	17.68	12.50	37
33	0.0	44.2	0.0	8.0	46.1	1.5	16.81	12.50	38
34	0.0	49.5	0.0	7.7	42.5	0.2	17.72	10.00	33
35	0.0	46.9	0.0	0.0	51.9	1.1	15.90	23.75	27
36	0.0	36.0	3.5	2.4	57.9	0.2	20.46	17.50	24
37	0.0	33.3	0.0	1.4	65.2	0.0	13.91	17.50	24
38	47.6	17.9	0.0	7.7	20.6	6.0	55.79	47.40	19
39	28.9	22.0	0.0	8.1	33.8	7.0	39.34	42.50	22
40	47.0	22.4	0.0	817	20.0	1.7	56.48	47.50	20

Table 34 (continued)

	PSM	PIL	PVM	PCA	PKA	PQU	ECEC	LCEC	TKIL8
41	44.0	21.2	10.1	5.9	16.9	1.9	69.95	38.75	21
42	7.9	41.0	1.0	6.5	42.5	1.6	24.47	17.50	30
43	0.0	47.6	0.0	7.2	44.1	1.1	17.24	12.50	33
44	0.0	38.0	0.0	7.7	53.9	0.3	15.76	12.50	33
45	0.0	35.9	0.0	6.0	51.6	6.5	14.72	31.25	23
46	0.0	16.1	0.0	7.6	75.1	1.1	11.97	20.00	19
47	0.0	20.6	0.6	5.2	71.1	2.5	13.24	17.50	20
48	0.0	26.4	1.0	4.9	66.0	1.7	14.99	17.50	20
49	0.0	37.2	0.0	6.9	53.0	2.8	15.33	12.50	34
50	11.8	31.2	0.0	7.2	38.6	11.1	24.72	30.00	24

Table 35. Complete Data Table for Modification X

	PSM	PIL	PVM	PCA	PKA	PQU	ECEC	LCEC	TKIL8
1	41.7	15.1	0.0	12.5	28.7	1.9	50.95	56.25	15
2	28.9	20.6	0.0	12.9	32.5	5.0	40.01	47.50	15
3	25.6	19.3	0.0	16.6	31.0	7.4	37.17	42.50	14
4	30.8	9.5	0.0	15.2	38.3	6.3	40.11	42.50	13
5	50.5	4.9	0.0	11.7	29.8	3.1	57.10	51.25	8
6	42.5	6.1	0.0	11.5	35.9	3.8	49.92	46.25	11
7	57.9	5.9	0.0	9.5	22.9	3.8	63.66	62.50	8
8	22.7	17.3	0.0	10.9	46.0	3.0	33.49	32.50	20
9	5.6	22.3	0.0	12.8	58.2	1.1	19.05	21.25	27
10	5.5	25.0	0.0	12.1	52.6	4.7	19.09	20.00	30
11	1.7	23.3	0.0	9.1	64.8	1.0	15.07	20.00	27
12	0.8	25.0	0.0	8.6	64.2	1.2	14.43	17.50	27
13	0.0	22.0	0.0	8.3	67.3	2.4	13.01	17.50	29
14	38.9	18.5	0.0	10.7	29.9	1.8	48.65	40.00	19
15	55.8	7.6	0.0	11.9	21.1	3.6	62.44	58.75	14
16	67.5	4.8	0.0	14.7	10.5	2.5	73.27	72.50	11
17	63.6	6.0	0.0	13.0	11.9	5.4	69.43	62.50	11
18	63.9	8.9	0.0	10.0	12.3	4.9	69.71	63.75	17
19	29.7	14.9	0.0	26.7	18.1	10.6	41.76	42.50	19
20	37.7	16.9	0.0	17.4	25.3	2.7	48.39	48.75	20
21	35.9	20.1	0.0	14.1	26.5	3.2	46.71	43.75	23
22	47.1	12.6	0.0	12.8	24.0	3.5	55.44	48.75	22
23	47.0	8.2	0.0	19.2	19.5	6.0	55.57	46.25	17
24	37.7	6.5	0.0	23.9	21.1	10.7	47.18	53.75	15
25	42.2	10.8	0.0	16.0	28.0	3.0	51.20	47.50	18
26	39.3	13.4	0.0	15.0	30.8	1.4	48.91	37.50	26
27	22.0	12.4	0.0	5.4	58.7	1.4	31.15	28.75	22
28	7.2	25.9	0.0	7.5	55.3	4.0	20.05	22.50	29
29	0.0	21.9	1.2	1.0	75.3	0.5	13.79	17.50	24
30	0.0	16.9	0.0	1.3	81.8	0.0	11.11	12.50	21
31	0.0	23.2	0.0	1.7	74.7	0.3	12.22	17.50	23
32	0.0	35.7	0.0	5.2	57.6	1.3	14.89	12.50	37
33	0.0	30.3	0.0	5.5	63.1	1.0	14.03	12.50	38
34	0.0	34.7	0.0	5.4	59.7	0.1	14.83	10.00	33
35	0.0	30.9	0.0	0.0	68.3	0.7	13.22	23.75	27
36	0.0	22.8	2.3	1.5	73.4	0.1	15.88	17.50	24
37	0.0	20.2	0.0	0.8	78.9	0.0	11.58	17.50	24
38	39.5	14.8	0.0	6.4	34.2	5.0	47.60	47.40	19
39	21.7	16.4	0.0	6.1	50.5	5.3	31.48	42.50	22
40	39.2	18.6	0.0	7.3	33.4	1.4	48.38	47.50	20

Table 35 (continued)

	PSM	PIL	PVM	PCA	PKA	PQU	ECEC	LCEC	TKIL8
41	37.7	18.0	8.7	5.0	29.0	1.5	60.99	38.75	21
42	5.2	28.8	0.7	4.5	59.6	1.1	19.55	17.50	30
43	0.0	33.1	0.0	4.9	61.2	0.8	14.41	12.50	33
44	0.0	24.7	0.0	5.0	70.1	0.2	13.04	12.50	33
45	0.0	23.6	0.0	3.9	68.0	4.3	12.42	31.25	23
46	0.0	9.2	0.0	4.3	85.8	0.6	10.28	20.00	19
47	0.0	12.0	0.3	3.0	83.1	1.4	11.07	17.50	20
48	0.0	15.9	0.6	3.0	79.5	1.0	12.16	17.50	20
49	0.0	24.3	0.0	4.5	69.3	1.9	12.81	12.50	34
50	8.5	22.5	0.0	5.2	55.7	8.1	20.05	30.00	24