

CHEMOD: An automated chemical and modal analysis technique

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ABSTRACT

A digital image-processing technique was modified to provide a more accurate modal analysis of sediments than the standard point-counting procedure. A digital backscattered-electron image covering an area that includes at least 300 grains is processed to form a binary image template for beam control. The automated process re-examines each feature or grain and collects an X-ray spectrum with an energy-dispersive spectrometer. The advantage of this procedure is that instead of analyzing a single spot on a grain, our system rasters the electron beam over the entire grain surface. The EDS spectra therefore average chemical variations over the entire grain. The stored analyses are background corrected, and the element intensities are utilized to calculate oxide weight percentages for mineral identification. All analyses were by a Tracor Northern image-processing system (IPA-5700) adapted to a JEOL 733 Superprobe.

To demonstrate the utility of the analytical technique, six grain mounts of sand- and silt-sized particles from the Cubits Gap crevasse-splay, Mississippi Delta, were analyzed. There were no size-dependent differences in the mineralogy of the samples. Quartz makes up 50–60 vol% of the samples analyzed. Feldspars and lithic fragments occur in approximately equal quantities, and each may constitute 20–30 vol% of the material. The carbonates and accessory minerals account for less than 5% of the rock volume. Other mineral and rock fragment categories may be selected by modifying the elements to be analyzed and adjusting the classification procedure accordingly.

The technique—referred to as CHEMOD—provides more reliable estimates of feldspar abundances than optical methods because it is based on chemical composition. This chemical approach provides estimates of compositional diversity within groups of minerals or rock types. The image-analysis technique also measures grain size and shape parameters. The outcome is an accurate and reproducible chemical, mineral, and textural analysis from a single polished specimen.

INTRODUCTION

In this paper, we describe an automated technique for the acquisition of chemical composition and modal mineral data. We refer to the analytical technique as CHEMOD for simplicity. It employs an electron microprobe and digital image-processing procedures to analyze the major-element variations in individual mineral grains, to establish their identity, and to determine the volume fraction of each detected mineral. Whole-rock data can be obtained by combining the individual grain results. The utility of CHEMOD is demonstrated for sand- and silt-sized grains from Mississippi River sediments, but the procedure can be applied to mineralogic investigations of other sedimentary, igneous, and metamorphic rocks.

Mineral content, chemical composition, and texture, including the shape and size of mineral constituents, are the chief rock properties measured by petrologists. Sedimentary petrologists, in particular, have used these characteristics to identify source areas and to assess changes occurring during transportation and subsequent burial. In diagenetic studies, it is important to have analyses of spe-

cific minerals or rock fragments in order to follow the chemical reactions that have taken place. The investigator rarely has these data available because of the tedium and uncertainties associated with the currently employed analytical methods.

The standard method for mineral analysis and classification of coarse-grained materials is based on point counts of petrographic thin sections. This optical-microscope method has been refined and standardized over the years so that a skilled operator may produce results for a single specimen in less than 30 min. However, mineral identifications, especially the recognition of untwinned feldspars, are uncertain, and no direct chemical information is attainable. There are often significant differences in mineral ratios of similar samples when determined by different authors. For example, Russell (1937) and Potter (1978) reported quartz/feldspar ratios for the fine-sand fraction of Mississippi River sediments of 3.3 and 5.1, respectively. CHEMOD provides a standardized technique that enables one to determine whether these differences are real sample variations or are simply due to the deviation in results by different petrographers.

Quantitative results may be obtained by X-ray powder-diffraction methods. These techniques are accompanied by improved mineral identifications, but they lack direct measurements of chemical variability. The XRD techniques also require a powdered sample, so spatial relationships among minerals cannot be discerned.

Electron-microprobe analysis of small volumes of mineral grains was recognized for its potential in sedimentary petrology more than a decade ago (Blatt et al., 1972; Potter, 1978) and has become a routine analytical technique in many mineralogical laboratories (Jones, 1985). Uses of image processing or point analyses in petrology are discussed by Albee et al. (1977), Quick et al. (1981), Ehrlich et al. (1984), Jones (1985), Nicholls and Stout (1986), and Schäfer and Teyssen (1987). In these studies, data were collected at points on a grid by microprobes equipped with an automated stage or digital beam control, and long run times were required. Other methods for the mineralogical analysis of sandstones by image-processing techniques have been discussed by Dilks et al. (1984) and Dilks and Graham (1984, 1985). They have used gray-level segmentation procedures on digital backscattered-electron images to discriminate minerals by their backscattered-electron intensities.

CHEMOD differs from other image-processing systems in significant ways that provide important advantages. (1) Our microprobe system is equipped with digital beam control that allows the acquisition of a digital backscattered-electron image that is used as a template for subsequent automated analysis of discrete grains. (2) Instead of analyzing a single spot on each grain, our system rasters the electron beam over the entire grain surface. (3) The energy-dispersive spectrometer (EDS) system measures the X-ray intensities of 16 or more elements in less than 20 s (live time). (4) Classification of grains is computer controlled and is based on X-ray intensities rather than on properties that are more difficult to quantify, such as backscattered coefficients (Dilks and Graham, 1985) or backscattered-electron energies (Jones, 1985). (5) Our system measures grain size and textural parameters including grain area, diameter, perimeter, length, width, shape factor, grain orientation, and aspect ratio. The outcome is an accurate and reproducible chemical, mineral, and textural analysis from a single polished specimen.

MATERIALS AND METHODS

Six polished surface grain mounts of size-fractioned separates from Cubits Gap crevasse-splay sediments, Mississippi Delta, were analyzed by CHEMOD. The samples were collected from traction-transported and suspension-deposited lithofacies (DiMarco et al., 1986). The sand grains were coarser than 62 μm in diameter, and the particle size of the silts varied between 5 and 62 μm . Clay minerals and micaceous particles other than those in rock fragments were essentially absent in these coarser separates.

CHEMOD was developed for use with an energy-disper-

sive spectrometer on the JEOL 733 Superprobe and Tracor Northern IPA-5700 image-processing system at Louisiana State University. The procedure should be readily adaptable for use with other analytical systems. A complete analysis requires approximately 120 min, but the operator is only required for about 20 of those minutes. The major steps in the procedure are listed here and then discussed in some detail: (1) Obtain a backscattered-electron image containing approximately 300 mineral grains. (2) Produce a binary image template. (3) Scan each feature using the template and accumulate data for stereological analysis and individual-grain EDS spectra. (4) Convert background-corrected peak intensities to oxide weight percentages. (5) Use oxide weight percentages to classify minerals. (6) Output results.

Backscattered-electron image

The grain mounts are scanned at variable magnifications to select a representative area. A digital backscattered-electron image containing 512 \times 512 pixels is then obtained at a magnification sufficient to include approximately 300 grains in the field of view. This number of grains was selected because in point-count-based optical methods, it is the minimum number required to ensure that, at the 0.95 confidence level, a grain that makes up 1 vol% of the sample would be counted. Higher-magnification images of multiple areas with fewer grains could be employed when more detailed individual grain data are desired.

Binary template

A black-and-white binary-image template is prepared by manually selecting an intensity-threshold value from the gray-level histogram of the backscattered-electron image that separates mineral grains from the epoxy mounting material. This template guides the electron beam to individual mineral particles in subsequent procedures.

Grains in contact with one another produce composite grain images, and they must be separated in order to produce accurate results. For example, if a quartz and feldspar grain were in contact, they would be classified as a rock fragment with a composition dependent on the relative area percent of each mineral in the composite. This misclassification would underestimate the modal abundances of both quartz and feldspar. To avoid such an error, images are separated by automated erosion and dilation procedures. The erosion is performed at the edges of grains as identified by the binary-image template. This process works equally well for grain mounts or whole-rock thin sections. A small amount of manual editing may be required where automated pixel erosion is incomplete. One may exclude exceptionally large or small grains by specifying maximum and minimum grain area limits for each analysis.

The modified binary-image template is used to examine and to collect data on each grain. EDS spectra are

generated as the beam is rastered within the confines of each feature in the template. The EDS system provides reliable X-ray intensities from the entire field of examination because it is not subject to the defocusing errors associated with wavelength-dispersive spectrometers. Analyses were acquired at 15 kV and a probe current of approximately 0.01 μ A producing an input count rate of 2000 counts per second. X-ray counts are accumulated for a preset live time of 20 s or until 1024 counts are registered in an individual channel. This collection time produces a satisfactory quantitative analysis within a reasonable time, and the results are obtained with minimal sample damage due to prolonged exposure to the electron beam. Counting times less than 20 s could not be employed because light elements (such as Na) could not be detected at the 1–2 oxide weight percent level.

Each feature is numbered automatically by the IPA-5700 program while X-ray spectra and data for stereological calculations are stored. In initial experiments, background-corrected intensity values for 16 elements (Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, Cr, Mn, Fe, Ni, Cu, and Zn) were obtained. The trace elements could not be used with the same degree of confidence as the major elements; therefore, in our application, only data for Na, Mg, Al, Si, K, Ca, Ti, and Fe were used in the calculations for mineral classification and bulk chemical composition. Other applications might take advantage of many more elemental intensities. Computed stereological parameters include grain area, diameter, perimeter, length, width, shape factor, grain orientation, and aspect ratio. Only grain-area values are used in CHEMOD since it is the only stereological parameter required for a modal analysis.

Convert peak intensities to weight percentages

Thirty mineral standards (Table 1) form the basis for the correlation of the normalized EDS intensities (peak counts per total counts \times 100) of eight major elements and the weight percents of their oxides. The relationship is linear (Fig. 1) except where absorption or fluorescence is a problem as for Si and Fe (Bence and Albee, 1968; Pyman et al., 1978). The calibration curves are to some degree instrument dependent, but if the same standards and similar instrumental operating conditions are utilized, the results should be comparable. The calibration avoids the use of Bence-Albee or ZAF corrections that entail considerable computation time. For elements that show a well-defined correlation between weight percent and normalized intensity ($r \geq 0.9$), the equations of best-fit lines are employed to calculate the chemical composition of each grain. Although the y intercepts are small positive or negative values, the equations could still be used for the calculation because the intensities of the major elements were great enough to be unaffected by the intercept error. When intensities are low, this error and other uncertainties reduce the reliability of the calibration and preclude the use of intensity data for elements that make up less than 1 wt% of the analyzed grain.

TABLE 1. List of mineral standards analyzed by CHEMOD for correlation of normalized elemental intensities with values for weight percent oxide

1. PSU Orthoclase OR-1A
2. Microcline USNM #143966
3. Amelia Albite
4. UO Biotite M1
5. Kakanui Hornblende USNM #143965
6. Lake Co. Plagioclase USNM #115900
7. PSU Engles Amphibole
8. Amphibole QC 81-23
9. PSU Na-Amphibole 6-040
10. UO Actinolite AM-9
11. UO Tremolite AM-7
12. PSU Biotite R-2208
13. Johnstown Hypersthene USNM #746
14. Kakanui Pyrope USNM #143968
15. San Carlos Olivine USNM #111312/444
16. Rockport Fayalite USNM #85276
17. Kakanui Augite USNM #122142
18. Tiebaghi Chromite USNM #117075
19. Minas Geras Magnetite USNM #114887
20. Ilmen Mtns. Ilmenite USNM #96189
21. Durango Apatite USNM #104021
22. Scapolite USNM #R6600-1
23. Volcanic Glass VG-2 USNM #111240/52
24. Volcanic Glass VG-A99 USNM #113498/1
25. Rhyolite Glass VG-568 USNM #72854
26. Hot Springs Quartz USNM #R17701
27. UO Al-Ti Diopside Glass GL-34
28. UO Cr-Mn-Ni Diopside Glass GL-41
29. PSU Sphene
30. PSU Riebeckite R-2535

Note: PSU = Pennsylvania State University. UO = University of Oregon. USNM = Smithsonian Institution—U.S. National Museum.

Classification of minerals

Figure 2 illustrates the flow chart of the classification program. Individual grain composition data are used to assign the particle to one of eight categories. For the Mississippi Delta sediments, the major grain types are quartz, impure quartz, sodium feldspar, calcium feldspar, potassium feldspar, calcite, dolomite, and lithic fragments. A range of oxide weight percent values is permitted within each category because the detrital grains are commonly impure. This provision for small variations is similar to that employed in optical petrography in that an albite or quartz grain may contain inclusions of other minerals, but our ranges are based on empirical measurements of chemical composition. For example, the sodium feldspar category accounts for the possible incorporation of up to 5 wt% quartz in a grain in addition to potential solid solution or intergrowths with calcium and potassium feldspars. For this classification, the sodium feldspar is a feldspar grain with Na dominant over Ca and K. Individual sample results must be consulted to determine the precise composition of a grain counted in this category. Similar arguments apply to the other categories, especially the one termed "impure quartz," which is probably a mixture of quartz and $<2\text{-}\mu\text{m}$ mica crystals typical of some recycled sedimentary rock fragments. Lithic particles are essentially the igneous, sedimentary, and metamorphic rock fragments that would not fit into one of the specific mineral dominant categories.

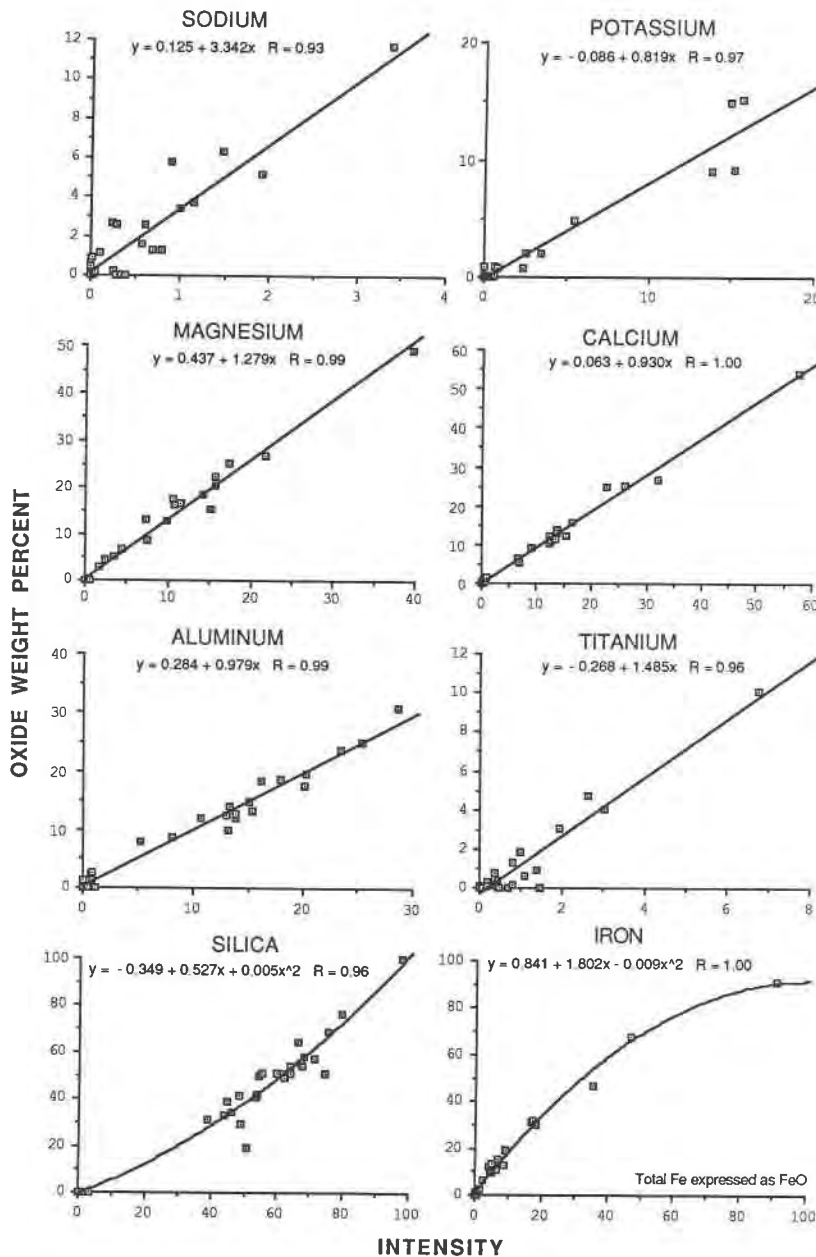


Fig. 1. Graphs showing the correlation between EDS-normalized intensities and oxide weight percent of the major elements used in CHEMOD mineral classification. Instrumental operating conditions used were 15 kV, 10-nA probe current, focused beam, 40° take-off angle.

Output results

A variety of choices is available for presenting the results of CHEMOD. Individual, or combined, chemical and mineral percentage data for single and multiple samples may be printed in tabular form. Volume percentage estimates of minerals and rock fragments are calculated by summing the area measurements for the grains in a particular category and then dividing by the total of all measured grain areas. Graphic subroutines supplied with the IPA-5700 also permit several choices for histograms or

scatter plots of variables. Serial port interfaces permit the data files to be transferred to larger computer systems for other statistical analyses.

The average chemical composition of the whole sample can be determined from the individual grain data by summing the fractional contributions. For example, the weight percent SiO_2 in the whole rock (W_{SiO_2}) can be determined by

$$W_{\text{SiO}_2} = \sum_{g=1}^n \frac{a_g W_{\text{SiO}_2,g}}{A_n}$$

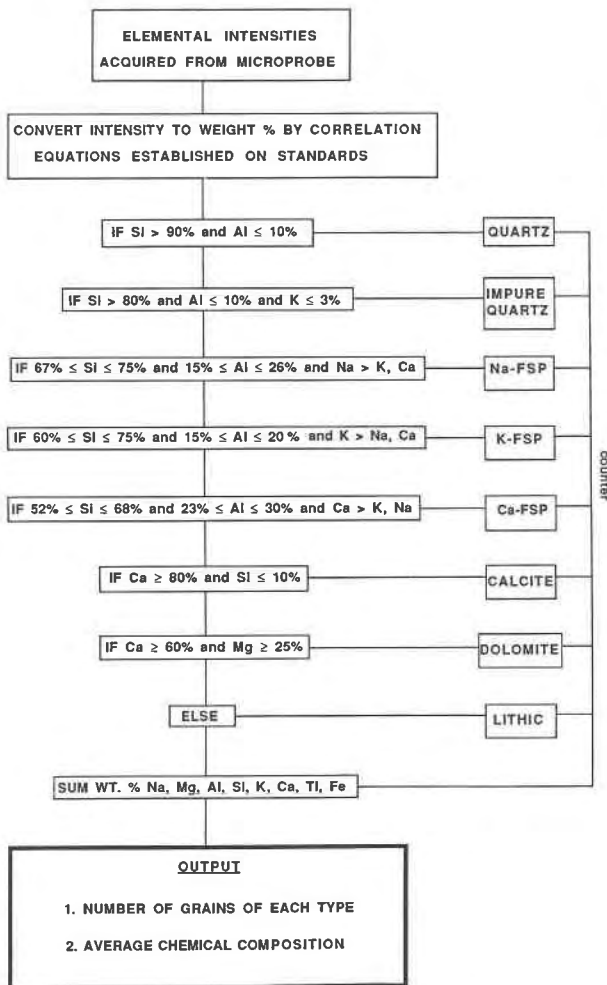


Fig. 2. Flow chart outlining the system for assigning grains to a mineral or lithic class based on their chemical compositions.

where a_g and $W_{SiO_2,g}$ are the area and the weight percent SiO_2 of an individual grain (g) and A_n is the total area summed over the total number (n) of all grains analyzed. A large variety of similar computations can be tailor-made for special applications from the basic data files generated by CHEMOD.

DISCUSSION OF RESULTS

The backscattered-electron image of a section of a standard field of view is presented in Figure 3. This enlarged view illustrates the subrounded to angular and embayed margins of the grains and the gray-level contrasts among the mineral grains and the epoxy mounting material (black). At the lower magnification required to obtain 300 grains in a field of view, grains in close contact (E) appear as a single feature and must be separated in order to form a proper binary template. Based on EDS spectra and the derived oxide weight percentages, the grains in this field of view are identified as quartz (Q), potassium feldspar (K), sodium feldspar (N), calcium feldspar (C), and lithic

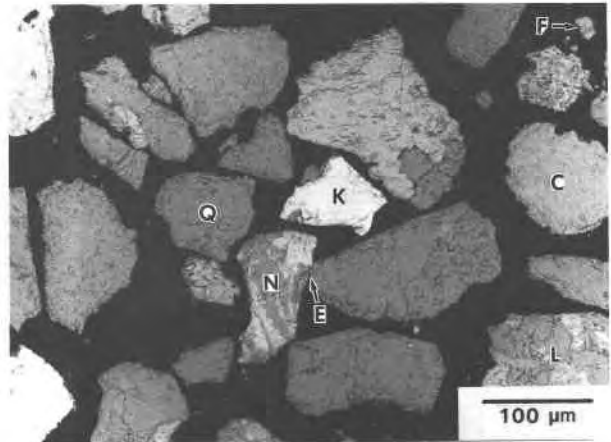


Fig. 3. Backscattered-electron image showing a portion of the standard field of view analyzed by CHEMOD. Labels show a grain contact zone (E); quartz (Q); potassium feldspar (K); sodium feldspar (N); calcite (C); lithics (L); and fragments (F).

fragments (L). The backscattered-electron image clearly demonstrates the mixed nature of the two feldspar grains near the center of the figure, and the individual grain chemical analyses confirm that they are mixtures of sodium and potassium feldspars. Tiny fragments of grains (F) were excluded from this CHEMOD analysis by setting the lower area limit at a value equal to approximately $400 \mu m^2$.

The sand fraction of the Cubits Gap samples have a weighted average quartz : feldspar : lithic fragment (Q:F:L) ratio of 56:21:24, which identifies them as a sublithic arenite. The silt fraction of the same samples yields roughly similar results (Q:F:L = 58:19:23) to those reported for the sands, although the arenite terminology is not applicable. The sands contain more lithic fragments than the average for Mississippi River sediments reported by Russell (1937) (Q:F:L = 69:21:10) and more feldspar than the amount reported by Potter (1978) (Q:F:L = 61:12:27). These differences are expected because CHEMOD utilizes information from the whole grain in its classification program. The results are more reliable than petrographic analyses because they are based on more objective measurements.

The complete results of the mineral determinations are listed in Table 2. Quartz is the major mineral, constituting 50–60 vol% of the sand and silt fractions. Feldspars and lithic fragments occur in approximately equal quantities and each may account for 20–30 vol% of the material. The carbonates and other mineral (or rock) categories make up less than 5% of the rock volume. The values reported for sands compare very well with those obtained by manual identification procedures with the SEM.

Comparison of the SEM and electron-microprobe results provides a reasonable estimate of the reproducibility and accuracy of CHEMOD. Because of the manual supervision of the SEM-acquired EDS spectra, SEM values are

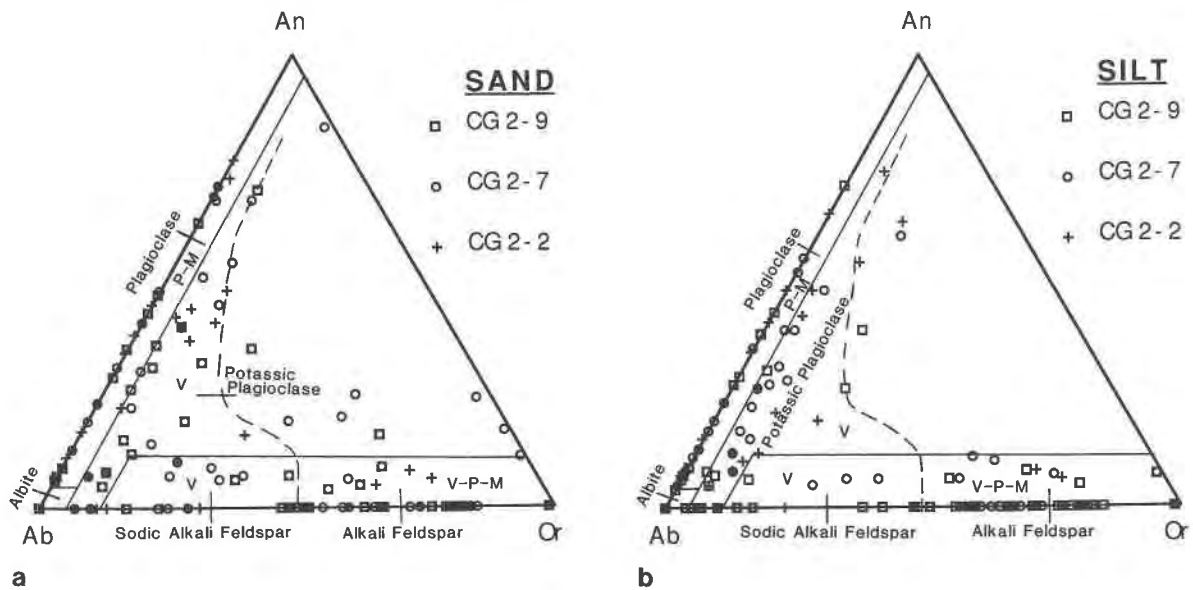


Fig. 4. Composition of feldspars in the (a) sand and (b) silt fraction of Cubits Gap sediments. The classification of feldspar compositions is from Maynard (1984). The letter symbols indicate source rock types for each compositional zone according to Trevena and Nash (1981): volcanic (V); plutonic (P); metamorphic (M).

considered the standard against which to compare the automated-microprobe results. Of course some small error is expected by this comparison because different numbers of grains and slightly different fields of view were examined. At the 50–60 vol% level, the average relative error in the quartz determination is 4.7% of the amount reported. Errors in the total feldspar and lithic fragment estimates average 11% of the amount reported. Larger relative errors occur when the grains make up less than 10 vol% of the sample.

The errors are due to two main factors. A true standard is not available for compilations of grain-by-grain measurements, so we cannot really derive a quantitative estimate of accuracy. Reproducibility errors are primarily due to the statistical counting error associated with the 20-s accumulation time of the EDS spectrum. Net peak

intensities for some elements are as low as 100 counts, so variations of $\pm 10\%$ are expected in the estimates of oxide weight percentages. The total standard deviations of the Na_2O , CaO , and SiO_2 estimates obtained from the calibration curves are ± 0.7 , ± 0.7 , and ± 3.3 wt%, respectively. Similar values are obtained for the other oxides.

The total error arising from these two causes are of approximately the same magnitude as those determined by comparing the SEM and electron-microprobe results. Typical expected errors for major (greater than 50 vol%) and minor (roughly 20 vol%) constituents are approximately 5% and 10%, respectively. For less abundant grains, the errors are higher.

Individual feldspar chemical analyses are plotted in Figure 4 for the sand and silt fractions. There are points

TABLE 2. Mineral-classification results of CHEMOD for six Cubits Gap samples

Mineral (%)	CG 2-2 sand		CG 2-7 sand		CG 2-9 sand		CG 2-2 silt	CG 2-7 silt	CG 2-9 silt
	Probe	SEM	Probe	SEM	Probe	SEM	Probe	Probe	Probe
Quartz	58	59	50	54	50	48	60	50	55
Impure Qtz	2	—	2	—	3	—	3	2	3
Feldspar total	20	16	24	24	19	18	17	22	20
Ab	7	3	9	6	5	2	8	8	8
Or	8	10	11	12	10	10	5	10	10
An	5	3	4	6	4	6	4	4	2
Calcite	0	1	<1	1	1	4	0	<1	1
Dolomite	1	—	1	—	1	—	<1	0	2
Lithics	19	24	23	20	26	28	20	26	19
Total grains	344	239	385	472	363	413	302	338	346

Note: Manual SEM analyses of the same samples in the sand size fraction are listed for comparison.

TABLE 3. Average chemical results of CHEMOD for six Cubits Gap samples, compared with Potter's (1978) average chemical composition of Mississippi River sands for the same oxides

	CG 2-2 sand	CG 2-7 sand	CG 2-9 sand	CG 2-2 silt	CG 2-7 silt	CG 2-9 silt	Average	Potter's average
SiO ₂	81.10	78.21	78.06	81.14	76.48	77.50	78.75	80.15
Al ₂ O ₃	8.02	9.85	8.68	7.03	10.13	7.49	8.52	6.43
FeO + Fe ₂ O ₃	0.72	0.87	0.97	1.81	1.25	0.61	1.04	2.47
TiO ₂	1.36	0.99	1.15	1.77	1.11	0.93	1.22	0.42
CaO	2.02	2.10	2.47	1.69	1.55	3.62	2.24	3.32
MgO	1.71	1.70	1.68	1.60	1.35	2.43	1.75	0.85
Na ₂ O	3.35	3.16	3.97	3.76	5.81	4.88	4.16	1.19
K ₂ O	1.81	2.14	2.09	1.33	1.92	1.98	1.88	1.20

that plot within the regions of volcanic, plutonic, and metamorphic feldspar identified by Trevena and Nash (1981) and modified by Maynard (1984). Most of the plagioclases are between 50% and 95% albite, and the alkali feldspars are 45% to 90% orthoclase. The complex nature of the sediments is typical of what is expected in a river such as the Mississippi that drains a large region of complex geology. Some of the grains are apparently complex aggregates that could not be differentiated by CHEMOD, because they have compositions that plot within an area where no naturally occurring feldspars have been recognized. These complex grains are less important volumetrically in the silt fractions because the smaller grains are more likely to be single minerals. In the future we hope to develop a procedure for the distinction of single grains of an isomorphous substitution series and intergrowths of different grains.

The chemical composition (Table 3) of the Cubits Gap samples is variable, but the average is similar to the average reported for Mississippi River sediments by Potter (1978). CHEMOD analytical determinations produce higher estimates of Na₂O, K₂O, and MgO than Potter reported. These and other differences are due in large part to the different procedures for the collection and analysis of the sediments. The CHEMOD values are particularly useful because they can be related directly to grains in the sample, and the calculations avoid any assumption of stoichiometry. Individual grain results such as these should be especially valuable in studies of diagenesis and provenance.

CONCLUSIONS

CHEMOD is an improved procedure for the automated determination of chemical composition and modal analysis of a wide variety of particulate materials. In less than 2 h per sample, one can obtain analytical data on approximately 300 grains. A chief advantage of the technique is its ability to analyze the entire surface of a polished grain rather than a single point.

As applied to sediments from the Mississippi River, CHEMOD produces a mineral analysis that agrees reasonably well with petrographic results obtained by others. However, this method is automated and eliminates some of the operator-induced errors of standard petrographic procedures, especially those produced by the misidenti-

fication of albite. Volume percentages of minerals obtained with CHEMOD are comparable to those obtained by optical methods, but this technique compiles chemical results and grain-size determinations at the same time. In the demonstrated application, Na, Mg, Al, Si, K, and Ca oxide weight percentages were sufficient to recognize eight mineral and grain types in the Cubits Gap sediments, but a modified program could readily employ X-ray intensity data on more elements. Adjustments to the mineral-classification program could facilitate the analysis of mafic and accessory minerals that may be of more importance to the igneous or metamorphic petrologist.

The most obvious limitation of CHEMOD is the inability to distinguish grains with identical chemical compositions, such as calcite and aragonite. Lithic rock fragments of sedimentary, igneous, or metamorphic origin are also impossible to separate if they have similar compositions. Future work will focus on expanding the classification procedures and employing pattern recognition techniques to distinguish grains with different textures. More elaborate modifications of CHEMOD should expand its utility as a complete mineral, chemical, and textural data-collection procedure for igneous and metamorphic petrologists as well as for sedimentary petrologists.

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MANUSCRIPT RECEIVED FEBRUARY 12, 1988

MANUSCRIPT ACCEPTED AUGUST 1, 1988