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Separation and Identification of the Silt-Sized Heavy-Mineral Fraction in Sediments

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By JUDITH A. COMMEAU, LAWRENCE J. POPPE, and
ROBERT F. COMMEAU

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Abstract

The separation of silt-sized minerals by specific gravity is made possible by using a nontoxic, heavy liquid medium of sodium polytungstate and water. Once separated, the silt-sized heavy-mineral fraction is prepared for analysis with a scanning electron microscope equipped with an automatic image analyzer and energy-dispersive spectrometer. Particles within each sample are sized and sorted according to their chemistry, and the data are tabulated in histograms and tables. Where possible, the user can define the chemical categories to simulate distinct mineral groups. Polymorphs and minerals that have overlapping compositions are combined into a group and differentiated by X-ray diffraction. Hundreds of particles can be rapidly sized and classified by chemistry. The technique can be employed on sediments from any environment.

INTRODUCTION

Analyses of the heavy-mineral fraction of sediments provide information on the provenance, depositional environment, and resource potential of the areas sampled. At present, heavy-mineral analyses are conducted on the sand fraction because of the difficulties encountered in suspending or wetting silt-sized material in organic heavy liquids and in identifying the mineralogy of this size fraction by petrographic microscope. However, most of the heavy minerals occur in the finer fractions because of their greater specific gravity (>2.85). In addition, sediments from most low-energy depositional environments contain little or no sand-sized heavy minerals from which to obtain statistically significant petrographic data.

The recent introduction of a new heavy liquid medium, an aqueous solution of sodium polytungstate [$3\text{Na}_2\text{WO}_4 \cdot 9\text{WO}_3 \cdot \text{H}_2\text{O}$] (SPT) (Callahan, 1987; Gregory and Johnston, 1987), and the use of a scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectrometer (EDS) and an automatic image analyzer (AIA) enable us to examine the grain size, chemistry, and

mineralogy of the silt-sized heavy-mineral fraction of sediments. The SPT solution is nontoxic, readily adjustable in density from 1.0 to 3.1 g/cm^3 , reusable, and, in the absence of calcium ions, chemically inert. It is also an ideal substitute for the carcinogenic halogenated lower alkanes such as tetrabromoethane and bromoform. The SEM/EDS/AIA equipment allows us to automatically determine the size and shape of hundreds of particles at a time and categorize them by chemistry.

The samples analyzed to demonstrate the procedures presented in this report were collected during the Atlantic Margin Program (Emery, 1966), an extensive study of the geological, biological, and hydrological characteristics of the Continental Shelf, slope, and rise of the Eastern United States. These surficial-sediment grab samples were selected because data on grain size, mineralogy, and chemistry of the sand fraction have been compiled (Ross, 1970; Hathaway, 1971). The analysis of the silt-sized fraction from these samples would complement these data and allow us to identify trends between the two size fractions.

METHODS

Sample Preparation

Size Fractionation and Heavy-Mineral Separation

The silt-sized particles must be isolated from the coarser and finer fractions before the heavy-mineral separation is performed (fig. 1). The sand fraction is separated by wet-sieving the samples through a 62- μm (ASTM number 230) sieve and is reserved for sand-fraction heavy-mineral analyses. The clays less than 4 μm in average diameter are removed from the fine fraction by decantation (Folk, 1974) and may be reserved for identification by X-ray powder diffraction. The decantation procedure involves settling the sample twice in a 0.5 percent sodium metaphosphate solution to de-flocculate the particulates and at least five additional times in distilled water. The samples are shaken and ultrasonified between each decantation to ensure that the sample is completely disaggregated. Decantation also serves to remove most of the free cations that

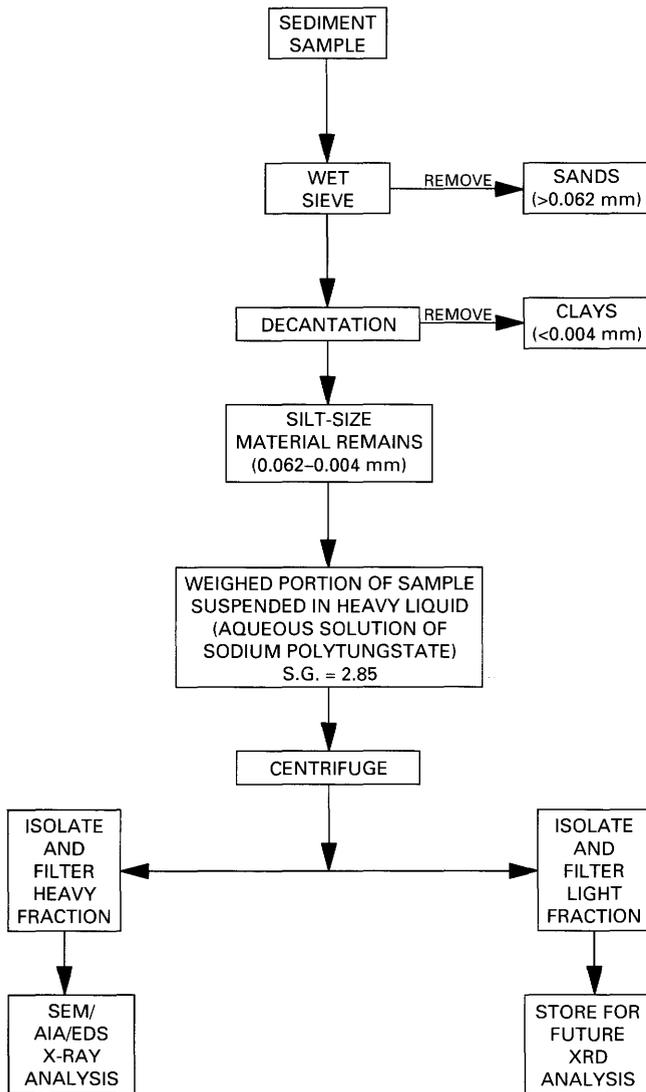


Figure 1. Flow diagram outlining the procedures used to separate the clay, silt, and sand fractions of a sample and to isolate the heavy-mineral fraction for SEM/AIA/EDS analysis.

may react with the SPT, such as calcium ions, which form an insoluble precipitate of calcium tungstate. After the decantation procedure, the silt fraction is dried in a convection oven at 60 °C.

The separation of the heavy minerals from the silt fraction is performed according to the following procedure.

1. An 82.75-mass percent solution of SPT in water (827.5 g SPT in 172.5 g water equals 1,000 g solution) is needed to create a solution with the desired density of 2.85 g/cm³. Other densities (1.0–3.1) can easily be made by varying the mixture (fig. 2). We recommend adding slightly more distilled water than necessary to facilitate the dissolution of SPT and then evaporating the suspension down to the desired density in a convection oven set to a temperature that is less than 60 °C. Using a higher temperature to accelerate the

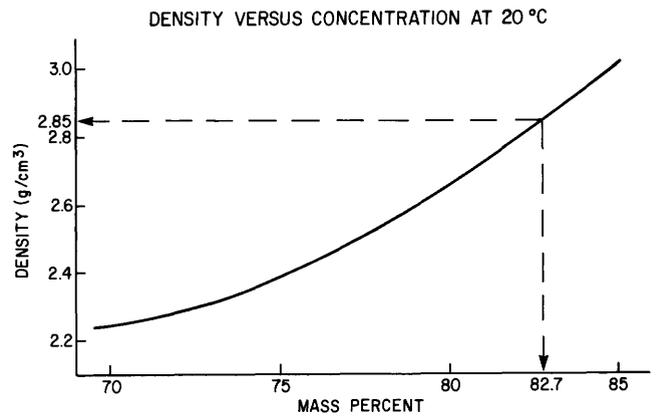


Figure 2. Plot of density versus the concentration of sodium polytungstate in distilled water at 20 °C. A solution with a density of 2.85 g/cm³ at 20 °C is obtained by preparing an 82.7 mass percent solution (e.g., 827 g solid and 173 g water).

evaporation process will result in the irreversible decomposition of SPT into sodium tungstate, Na₂WO₄, which is insoluble in water (unpublished data, J.M. Robbins, Oregon State University).

The pH of the SPT–distilled water solution is about 3.2 to 4.3. If calcium carbonate is present in the sample to be processed, we recommend adding ammonium hydroxide (30 percent) until the pH is about 7.0. This level limits the dissolution of the carbonates and the production of calcium ions that result in the precipitation of calcium tungstate.

2. Before the silt samples are mixed with the SPT solution, it is necessary to prewet the samples to overcome surface-tension effects. Samples of known weight (3–4 g) are placed in preweighed, wide-mouthed, glass or Teflon jars and wetted with a small amount of distilled water to form a slurry. Teflon containers are preferred because SPT sticks to glass upon drying and, if the suspension is accidentally brought to dryness, the pressure of crystallization may shatter glass (Savage, 1988). The jar should be evacuated to remove any air bubbles still adhering to the silt grains. The evacuation procedure is easily accomplished by using plastic tubing to attach a portable, rotary-vane vacuum pump to a rubber stopper large enough to replace the lid on the jar.
3. A known weight (60 to 100 g) of SPT solution adjusted to a density of 2.85 g/cm³ is added to the silt/water slurry, and the silt sample is homogenized and disaggregated using an ultrasonic probe. A convection oven set to less than 60 °C can be used to evaporate the entire suspension to the desired density. Because the weight of the entire suspension and the individual weights of the sample, the container, and the added SPT solution are known, the weight of the water to be evaporated (that is, the water added to the sample to form the slurry) can be calculated.

4. The suspension is washed into plastic centrifuge tubes using a squeeze bottle containing SPT solution (density, 2.85 g/cm^3). The number of centrifuge tubes used depends on the amount of sample to be processed. We recommend no more than 1.0 g per centrifuge tube because too much sample will cause the grains to interfere with each other and result in an incomplete separation.
5. The weight of each tube is noted and adjusted using the stock SPT solution to ensure rotor balance in the centrifuge. We used an ultra-high-speed centrifuge for a minimum time of 10 minutes at 15,000 rpm to complete the separation of the heavy and light mineral fractions of a 4- to 62- μm silt sample.
6. After centrifugation, the separated light-mineral fraction floating on the surface of the heavy liquid may be removed from the centrifuge tubes by either of two techniques. First, the light-mineral fraction can be isolated by using a scoop, spatula, and (or) pipet and rinsed into a beaker using distilled water. Alternatively, the bottom 1 to 2 cm of the centrifuge tube may be submerged in liquid nitrogen. This submersion freezes the heavy fraction into a plug and allows the unfrozen light fraction to be decanted into a beaker.
7. The light-mineral fraction is washed out of the beaker with distilled water into a Millipore filtration device. The beaker should be thoroughly rinsed with distilled water to ensure that no sample remains. The material is suction-mounted onto preweighed 0.45- μm Millipore filters and rinsed with liberal amounts of distilled water. Failure to wash the sample with enough distilled water will allow any remaining SPT to recrystallize in the sample and adversely affect the sample weight determinations. To prevent filter clogging and to speed the filtration process, no more than 1.0 to 1.5 g of sediment should be mounted on any one filter.
8. The heavy-mineral fraction contained in the centrifuge tubes is washed with distilled water into a beaker. Next, the beaker is filled with distilled water and ultrasonified to disaggregate any small amount of calcium tungstate that may have precipitated during the separatory process. If present, calcium tungstate is usually very fine-grained and may be removed by decantation. The heavy minerals are rinsed with distilled water into the filtration device and suction-mounted onto a preweighed 0.45- μm Millipore filter. The filter is then rinsed with liberal amounts of distilled water to remove any remaining SPT.
9. The filters containing the light and heavy fractions are air-dried and then weighed. The weights of the light- and heavy-mineral fractions are determined by subtracting the weights of the clean filters from the filter-plus-sediment weights. The weight percentage of the heavy and light minerals in the original sample (step 2) is determined from the weight of the light-

and heavy-mineral fraction and the weight of the original sediment.

10. The filtered SPT solution diluted during the washings can be recovered. The solution is evaporated in a plastic crystallizing dish placed in an oven set to less than $60 \text{ }^\circ\text{C}$ until the density of the solution is 2.85 g/cm^3 . The recovered solution can be stored indefinitely in any tightly sealed plastic container.

Preparation of Samples for Analysis

Ultrasmooth carbon SEM mounts are used as the substrate for mounting the samples. The ultrasmooth surface prevents fine particles from being lost in voids or irregularities and ensures that the backscattered-electron signal generated from this surface is a constant, uniform level. A difference in backscattered-electron signal emitted from the sample and the carbon substrate allows the image analyzer to identify and measure particles. X-rays from the carbon matrix are not detectable by the EDS system, so there is no interference with characteristic X-rays from elements being analyzed.

The surface of the carbon mount is covered with a thin coating of adhesive solution. The solution is prepared by dissolving the adhesive cement from a piece of cellophane tape with amyl acetate (Murphy, 1982). The glue should be allowed to become tacky before the sample is applied to avoid the wicking of adhesive around and over the particles by capillary action.

Next, a representative split is taken from the dry, silt-sized, heavy-mineral separation by removing approximately one-third of the sample from a filter. The sample is mixed well and lightly dispersed over the surface of the SEM mount, being careful to minimize particle overlap and aggregation. This step can be performed as a dry dispersion by dusting the surface with the sample grains or as a wet dispersion by suspending the sample in distilled water and placing a bead of suspended sample on the mount and allowing it to dry.

Once the sample mount is thoroughly dried, the surface is carbon-coated in a vacuum evaporator. The carbon coating provides an electrical path to ground when the electron beam of the SEM is scanned over the surface of the sample grains.

Sample Analysis—Particle Size, Shape, and Chemistry

The system used for the analysis of silt-sized heavy minerals consists of an Etec Autoscan, Model U-1, SEM; a KeveX 7000 EDS system equipped with a conventional beryllium window detector; and a LeMont Scientific AIA unit (fig. 3). The sample is placed in the SEM and is analyzed using a beam acceleration potential of 20 keV and approximately 400 picoamperes of beam current. The

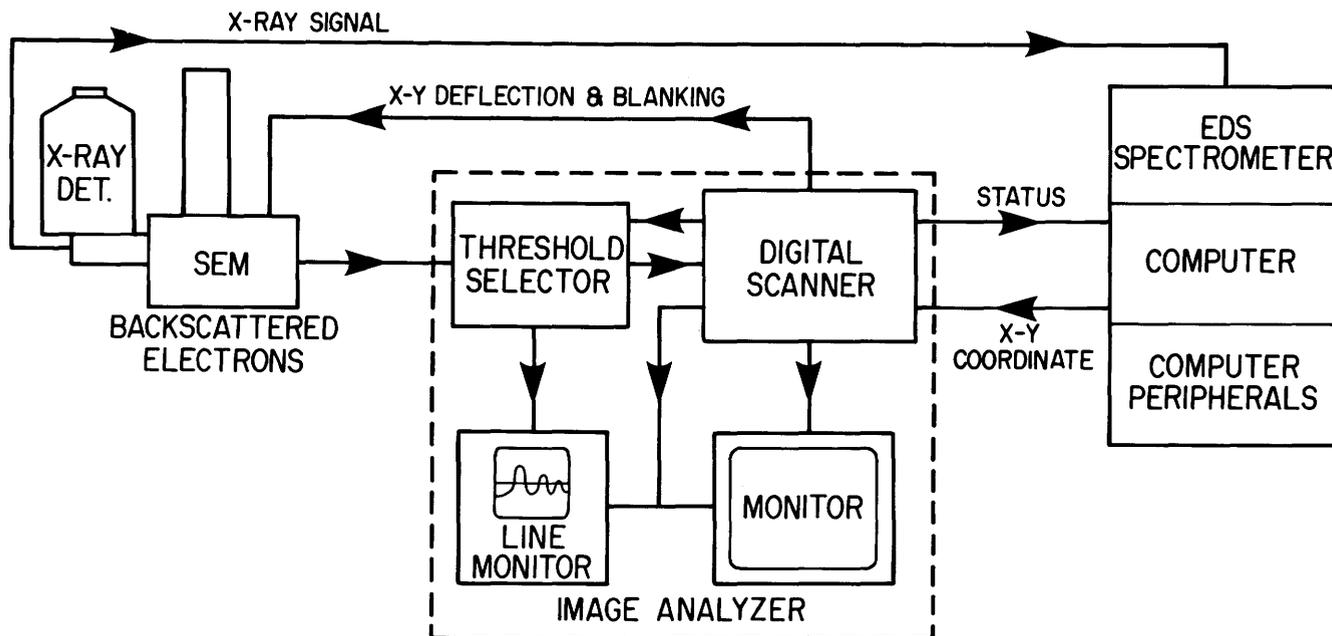


Figure 3. Schematic of the SEM/AIA/EDS system.

specimen tilt angle is set to 0 degrees and the working distance is approximately 23 mm. X-ray spectra are acquired for 4 seconds (live time) at a takeoff angle (the angle between the surface of the specimen and a line projected to the center of the X-ray detector) of 18 degrees. The backscattered-electron signal is selected as the input to the AIA system because it gives the best ratio of signal (on-particle) to noise (off-particle) for samples containing particles that vary widely in average atomic number.

In the SEM, the computer-driven electron beam automatically scans a field of particles, and the AIA system measures the length of a user-specified number of lines projected at regular angular intervals through the center of each particle (fig. 4). The AIA system then instructs the EDS unit to collect an X-ray spectrum from the center of the particle, identifies the peaks of elements present in the spectrum, and calculates the interelement peak ratios. The fundamental measurements for each particle (the projected-line data) are stored on computer disk along with the element data. From the fundamental particle data, the AIA system automatically calculates user-selected parameters, or derived physical measurements, such as area, average diameter, length, width, and (or) length-to-width ratio, for each of the particles. The derived physical measurements calculated for each particle can be sorted by chemistry into categories by the system's chemistry definition file and output as histograms and statistical tables.

A chemistry definition file consists of user-defined chemical categories called *macro classes* that allow measured particles to be sorted according to composition by their relative elemental X-ray intensities. Each macro class represents a specific mineral or a series of minerals of similar composition. Table 1 is a partial listing of actual

computer output showing macro classes created to sort X-ray spectral data collected from particulate samples from the test study area (see below). For example, macro class 1 defines the relative X-ray intensity criteria for titanium oxide minerals (rutile, leucosene, anatase, brookite, etc.). For a particle to fit this macro class, the X-ray spectrum must meet three criteria. First, the X-ray spectrum of the particle must contain a titanium peak. Second, the ratio of the titanium X-ray peak intensity to the sum of all the element intensities (including titanium) measured in the spectrum must be equal to or greater than 0.6 and equal to or less than 1.0. This criterion sets both the *lower and upper limits* because the titanium peak must contain at least 60 percent of all X-ray peak counts and can contain up to 100 percent of all counts. Third, the ratio of X-ray peak intensities from any one of the other elements to all the elements (including titanium) in the spectrum must be equal to or less than 0.05. Thus, the *upper limit* for the individual peak intensities for each of the other elements cannot exceed 5 percent of the total counts.

As each macro class is created, the user may enter a specific gravity for that class. Because each particle is sorted according to its chemistry and is stored in a class along with its derived physical measurements, weight percentages of minerals (macro classes) can be calculated. For example, the weight percentage of particles within a specific size range of a chemical category can be calculated by summing the masses of all the measured particles sorted into that size range, dividing that sum by the total sample mass (the total mass of all particles measured), and multiplying by 100. The mass of each particle is equal to its volume times the value of the specific gravity entered by the user for that chemical category. The algorithm used for the

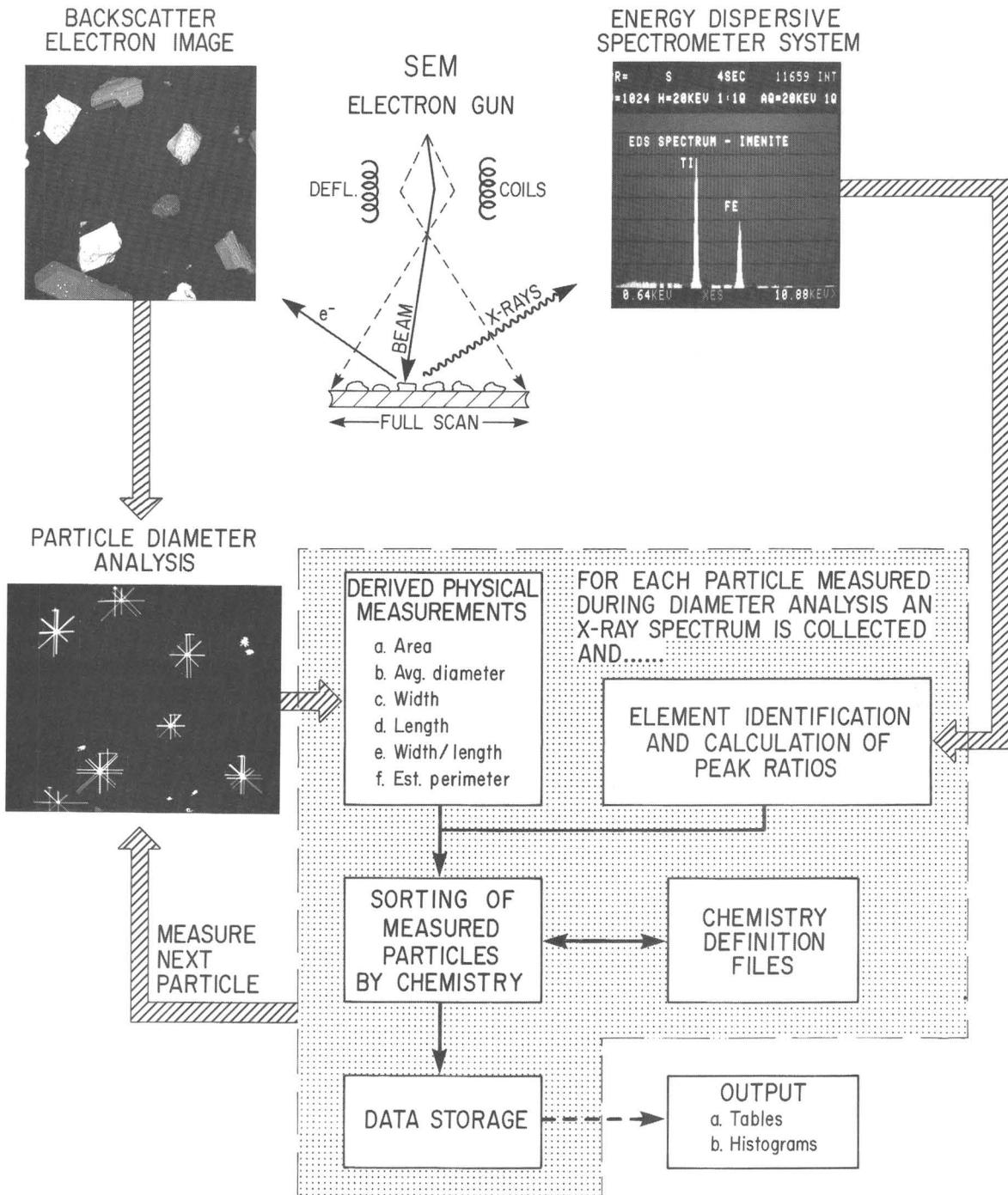


Figure 4. Flow diagram of procedures performed during SEM/AIA/EDS analysis.

calculation of the particle volume is based on the assumption that the two-dimensional particle is an ellipse with its length the major axis, *A*, and its width the minor axis, *B*. Rotation of the ellipse about its major axis creates a prolate spheroid with a volume equal to

$$\frac{4}{3}\pi \cdot 5A \cdot 5B \cdot 5B$$

Based on the shapes of mineral grains from the samples observed in the SEM, this mathematical model is an acceptable routine to estimate particle volume.

Table 1. Partial list of user-defined chemical categories

[The chemical categories are programmed into the chemical definition file and represent a specific mineral or a series of minerals]

Specification type	Lower limit	Upper limit	(Weighting)
Ti oxides, MACRO CLASS 1			
Ti fraction of X-ray count	0.600	1.000	1000
Other element fractions:	0.000	0.050	1000
i.e., NAMGALSI P SCL KCACRMNFENICUZNPB			
Parameter statistics provided along with histograms and tables			
Specific gravity		=4.200	
Fe Ti oxide, MACRO CLASS 2			
Fe fraction of X-ray count	0.100	1.000	1000
Ti fraction of X-ray count	0.100	1.000	1000
Other element fractions:	0.000	0.050	1000
i.e., NAMGALSI P SCL KCACRMNNICUZNPB			
Parameter statistics provided along with histograms and tables			
Specific gravity		=4.700	
Ca Ti Silicate, MACRO CLASS 3			
Ca fraction of X-ray count	0.100	1.000	1000
Ti fraction of X-ray count	0.100	1.000	1000
Si fraction of X-ray count	0.050	1.000	1000
Other element fractions:	0.000	0.020	1000
i.e., NAMGAL P SCL KCRMNFENICUZNPB			
Parameter statistics provided along with histograms and tables			
Specific gravity		=3.500	

Table 2 lists the chemical categories (macro classes) as they appear in the chemistry definition file prepared for the test study area on the Outer Continental Shelf near Georges Bank. Minerals corresponding to each macro class are also tabulated. The parameters for each macro class are based on chemical criteria that may fit a number of minerals that have overlapping compositions. If necessary, further refinement of the mineralogy within each macro class is accomplished by X-ray diffraction analysis (XRD) and examination of particle morphology in the SEM. Two macro classes, Si Oxide and Fe Stain Si Ox., represent quartz and iron-stained quartz, respectively. A small but significant number of quartz grains adhered to the wall of the test tube or to the heavy-mineral grains during the heavy-liquid separation procedure and were not separated with the other light-mineral grains.

A 10-Å phyllosilicate group is included in the chemistry definition file for the purpose of particle identification, but the mathematical model listed above is not appropriate for the calculation of weight percentages of micaceous minerals. Furthermore, the separation of phyllosilicates using heavy liquids is problematic because the specific gravities of the micas often straddle that of the heavy liquid and not all of the grains are removed in the heavy-mineral residue. As a result, the K Al Silicate macro class data have been removed from the weight percentages shown in table 5.

To evaluate the ability of the image analysis system to sort particles by chemistry into predefined chemical categories, we prepared a sample mount containing a known

number of particles from five different minerals. The results in table 3 show that the image analysis system properly classified 90 of the 93 mineral grains (over 96 percent of the grains) mounted for the test. The system was not able to classify three of the grains because of their rough surface

Table 2. List of chemical categories or macro classes

[These macro classes were prepared for the test study area off southeastern New England. Minerals corresponding to each macro class are also tabulated]

Macro Class	Mineral(s)
1. Ti Oxide	Rutile, leucoxene, anatase, brookite
2. Fe Ti Oxide	Ilmenite
3. Ca Ti Silicate	Sphene
4. Mg Carb.	Magnesite
5. Al Oxide	Corundum
6. Fe Oxide/Carb.	Magnetite, hematite, goethite, siderite
7. Si Oxide	Quartz
8. Zr Si Oxide	Zircon
9. Ca Phosphate	Apatite, monazite, vivianite
10. Fe Zn Cu Sulph.	Pyrite, sphalerite, chalcocopyrite
11. Ca Al Silicate	Epidote
12. Ca Fe Silicate	Andradite (garnet)
13. Fe Al Silicate	Staurolite, almandine (garnet)
14. Al Silicates	Sillimanite, topaz, beryl, andalusite, spodumene, kyanite
15. Fe Stain Si Ox.	Iron-stained quartz
16. Na Ca Mg Sil.	Amphibole, pyroxene, tourmaline
17. K Al Silicate	10-Å phyllosilicate group
18. Mg Al Oxide	Spinel
19. Fe Mn Oxide	Ferromanganese
20. Misc. Macro	Other minerals

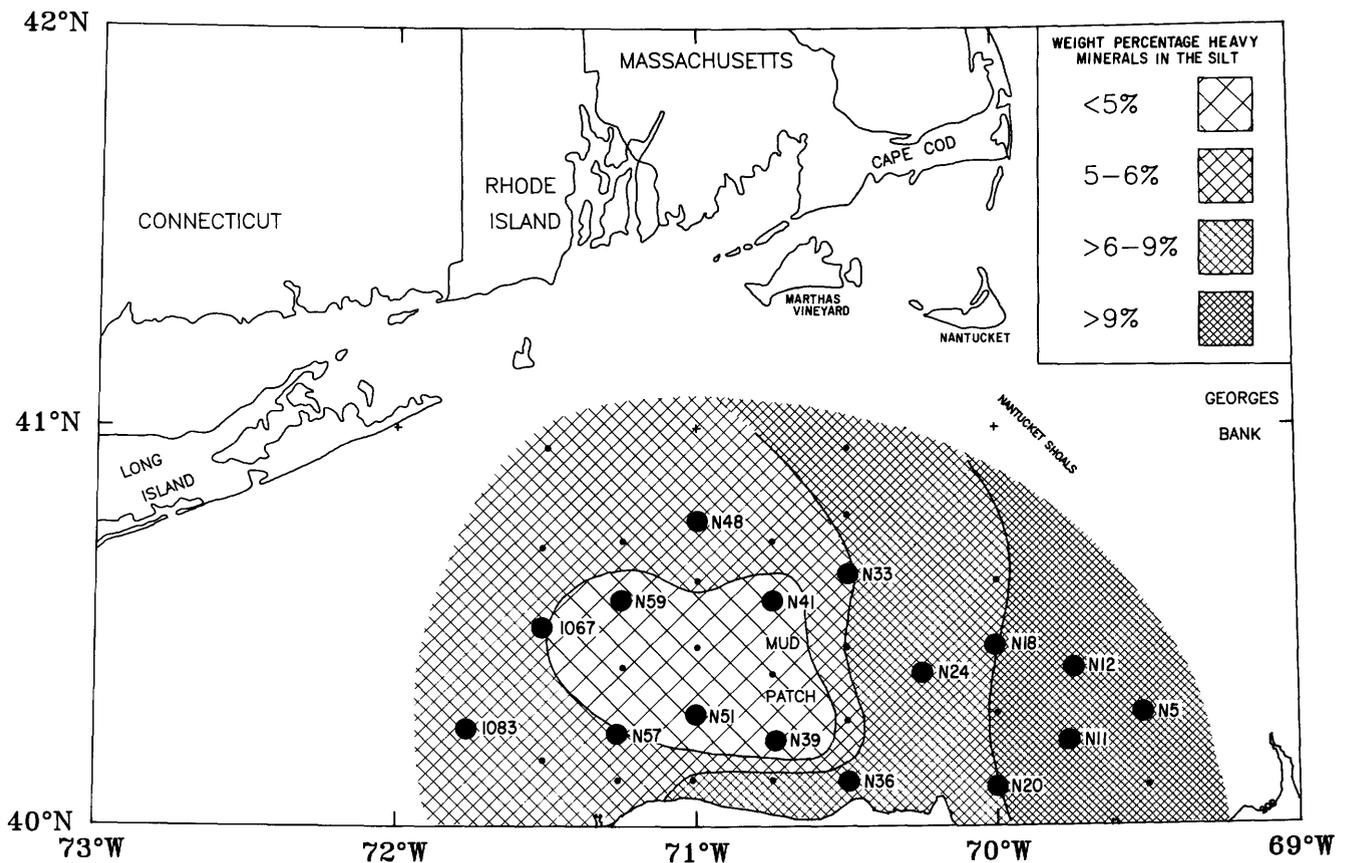


Figure 5. Sample locations of surficial sediments collected in the test study area. Patterned areas show the weight percentage of heavy minerals in the silt fraction. Samples from locations marked by large dots with identification numbers were analyzed by SEM/AIA/EDS analysis.

topography and the low, 18-degree, X-ray takeoff angle used in our SEM/EDS unit. After the system locates and measures a grain, it centers the beam on the grain to collect a spectrum. Occasionally, this location is in a recess on the surface, and the X-ray path to the EDS detector is obstructed. However, this obstruction is less of a problem with most newer SEM/EDS systems because they are equipped with inclined EDS detectors that allow the X-rays to be collected at takeoff angles of 35 to 40 degrees.

TEST STUDY AREA—RESULTS AND DISCUSSION

Mineralogical studies were made on the silt fraction of the surficial sediment from the Outer Continental Shelf area near Georges Bank (fig. 5). The area map shows sample locations (large and small dots) of surficial sediments collected in the study area and the weight percentage of heavy minerals in the silt fraction. The percentages were calculated from samples collected from all locations. Table 4 lists the weight percentages of silt-sized heavy minerals and compares them with data on the sand fraction listed in Hathaway (1971) and Poppe and others (1991). Samples from the map locations marked with identification numbers

were analyzed by SEM/AIA/EDS analysis, and selected examples of actual data output for sample station N5 are shown in figures 6–10. Figures 6–8 illustrate how the particle count data can be sorted either in terms of numbers of particles or in percentages of the total population into size ranges for each chemical category. Weight percentage data are presented in figures 9 and 10. The relative abundances of the nonmicaceous, heavy minerals contained in the silt fraction for all of the sample locations numbered on the map are presented in table 5. The mineralogy and the

Table 3. Results from a test sample, particles mounted versus identified

[Test results from a specially prepared SEM mount showing the number of particles identified and sorted by the automatic image analyzer system into macro classes or mineral groups versus the actual number mounted for each mineral]

Mineral	Macro class	Number mounted	Number counted
Rutile	Ti Oxide	14	14
Apatite	Ca Phosphate	19	19
Pyrite	Fe Zn Cu Sulph.	19	19
Epidote	Ca Al Silicate	20	19
Topaz	Al Silicates	21	19
Total mineral grains		93	90

Table 4. Weight percentages of silt-sized minerals from test area

[Weight percentages of silt-sized light and heavy minerals determined by the heavy-liquid separation technique for samples collected from the Continental Shelf off southeastern New England. Sand-sized, heavy-mineral data are presented for comparison. Dashes (-) in data columns denote no data]

Sample number ¹	Silt fraction weight %			Sand fraction weight %		Whole sample weight (%)	
	Whole sample	Light minerals	Heavy minerals	Heavy minerals	Silt-sized heavy minerals	Silt + sand-sized heavy minerals	
N5	12.7	89.18	10.82	² 1.6	1.37	2.6	
N6	10.0	90.59	9.41	-	.94	-	
N11-1	23.6	88.55	11.45	-	2.70	-	
N11-2	23.6	88.75	11.25	-	2.66	-	
N11-3	23.6	88.96	11.04	-	2.61	-	
N12-1	9.2	88.25	11.75	³ 2.4	1.08	3.1	
N12-2	9.2	88.85	11.15	-	1.03	-	
N12-3	9.2	88.62	11.38	-	1.05	-	
N17	10.3	91.40	8.40	-	.87	-	
N18	25.3	91.00	9.00	-	2.28	-	
N19	31.4	90.89	9.11	-	2.86	-	
N20-1	44.8	91.06	8.94	³ 1.8	4.01	4.8	
N20-2	44.8	90.75	9.25	-	4.14	-	
N20-3	44.8	91.03	8.97	-	4.02	-	
N24-1	38.9	92.73	7.27	³ 1.8	2.83	3.6	
N24-2	38.9	92.42	7.58	-	2.95	-	
N24-3	38.9	92.91	7.09	-	2.76	-	
N31	14.1	92.94	7.06	-	1.00	-	
N32	24.1	91.89	8.11	-	1.95	-	
N33	25.3	94.10	5.90	-	1.49	-	
N34	49.0	93.97	6.03	-	2.95	-	
N35-1	72.3	94.56	5.44	³ 2.1	3.93	4.2	
N35-2	72.3	94.57	5.43	-	3.93	-	
N35-3	72.3	94.91	5.09	-	3.68	-	
N36	10.2	93.17	6.83	² 1.4	.70	1.9	
N38	31.8	93.77	6.23	-	1.98	-	
N39	66.4	96.62	3.38	² .4	2.24	2.3	
N40	61.6	96.25	3.75	-	2.31	-	
N41	62.6	95.67	4.33	² .5	2.71	2.8	
N42-1	29.7	94.53	5.47	-	1.62	-	
N42-2	29.7	94.21	5.79	-	1.72	-	
N48	8.5	94.55	5.45	² .2	.46	.6	
N49	61.0	94.90	5.10	-	3.11	-	
N50	69.3	95.53	4.47	-	3.10	-	
N51-1	49.0	95.74	4.26	-	2.09	-	
N51-2	49.0	95.55	4.45	-	2.18	-	
N52	17.5	93.95	6.05	² 1.5	1.06	2.1	
N56	8.4	94.39	5.61	-	.47	-	
N57	23.6	94.94	5.06	-	1.19	2.9	
N58	35.3	95.78	4.22	-	1.49	-	
N59	19.5	95.79	4.21	² .3	.82	1.0	
N60-1	16.1	94.76	5.24	-	.84	-	
N60-2	16.1	94.11	5.89	-	.94	-	
N60-3	16.1	94.16	5.84	-	.94	-	
N64	6.1	94.19	5.81	-	.35	-	
1066-1	15.9	94.29	5.71	-	.91	-	
1066-2	15.9	94.11	5.89	-	.94	-	
1067	51.4	94.92	5.08	² 2.6	2.61	3.5	
1069	17.4	94.55	5.45	-	.94	-	
1083-1	18.6	94.75	5.25	-	.98	-	
1083-2	18.6	94.96	5.04	-	.94	-	
1083-3	18.6	94.87	5.13	-	.95	-	

¹For the purpose of computerized sorting and searches, the data base reported in Hathaway (1971) uses station numbers of standardized length for the samples used in this study, e.g., N005 for N5 and N011 for N11. The shorter style is used here for conformity with the usage in Poppe and others (1991). Hyphenated sample numbers denote replicate analyses.

²Data from Hathaway (1971).

³Data from Poppe and others (1991).

Table 5. Abundances of nonmicaceous, silt-sized heavy minerals in samples from the test study area

[The samples analyzed were surficial sediment samples collected from the Continental Shelf off southeastern New England (fig. 5). The mineralogy is based on data from the chemical class information from SEM/AIA/EDS analysis, observation of particle grain morphology in the SEM, and XRD. The values are in weight percent and are normalized to sum to 100. Asterisks indicate heavy minerals determined by XRD]

Sample ID	N5	N11	N12	N18	N20	N24	N33	N36	N39	N41	N48	N51	N57	N59	1067	1083
Rutile-anatase-brookite	5.74	6.17	5.42	5.69	5.29	10.50	6.70	8.57	7.41	3.81	3.98	6.07	3.73	5.00	1.97	4.73
Ilmenite	43.42	33.56	33.98	42.39	26.69	29.69	28.05	28.39	31.90	20.78	23.04	20.59	20.14	21.39	20.14	19.80
Sphene	0.53	0.38	2.38	0.69	2.73	0.95	0.05	0.11	6.87	0.60	4.45	2.10	3.19	1.67	1.48	2.84
Corundum	0.00	0.17	0.23	0.00	0.30	0.02	0.00	2.15	0.00	0.00	0.00	0.56	0.00	0.00	0.25	0.00
Magnetite-hematite-goethite-siderite	0.78	7.20	7.78	2.57	2.09	7.10	6.15	3.94	2.82	2.33	8.21	8.01	6.30	1.15	1.53	8.08
Zircon	20.30	8.37	13.83	5.07	6.59	3.54	9.41	3.90	0.51	2.11	8.70	5.85	3.06	2.32	4.98	0.25
Apatite-monzonite-vivianite	0.00	0.62	0.46	0.71	1.19	2.67	0.76	6.26	0.28	1.01	2.58	0.79	2.62	2.50	2.48	2.25
Pyrite	0.32	0.08	0.40	1.62	0.00	0.21	1.81	0.06	0.78	0.04	0.05	0.11	2.34	1.67	1.28	3.74
Spinel	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Epidote	5.45	5.63	4.44	9.26	6.22	6.07	11.11	9.07	6.87	12.08	7.93	11.79	10.93	7.67	14.98	8.58
Garnet-staurolite	2.91	7.44	7.32	2.96	12.15	6.46	13.34	2.55	5.08	6.69	5.28	7.73	8.92	10.84	9.02	10.16
Sillimanite-beryl-andalusite-topaz-spodumene-kyanite	2.57	2.29	1.01	2.96	1.91	3.56	2.18	0.91	1.10	4.12	3.42	2.88	1.70	6.51	4.54	2.39
Amphiboles	5.36	10.80	6.96	7.27	18.88	9.92	6.70	14.79	12.93	15.51	9.64	16.28	13.61	20.23	12.55	17.47
Pyroxenes	0.77	6.10	5.56	3.64	3.44	3.30	1.67	1.68	4.84	8.61	4.17	5.66	4.78	6.13	3.72	4.11
Tourmaline	3.57	1.88	2.78	4.85	3.42	1.98	3.90	3.36	4.04	10.33	1.93	3.54	4.77	4.29	2.79	7.71
Other FeMn coated-rock fragments	8.28	9.31	7.45	12.09	9.10	14.03	8.17	14.26	14.57	11.97	6.62	8.04	13.91	8.63	18.24	7.90

SAMPLE ID: SILT-SIZE HEAVY MINERALS N5											13-SEP-91		
POPULATION PERCENT VS. CHEMICAL CLASS													
TYP	CLASS NAME	%	0	1	2	3	4	5	6	7	8	9	10
MACR	TI OXIDES	4.55E**	0	0	0	0	0	0	0	0	0	0	0
MACR	FE TI OXIDE	29.55E*****	0	0	0	0	0	0	0	0	0	0	0
MACR	CA TI SILICATE	0.65E	0	0	0	0	0	0	0	0	0	0	0
MACR	MG CARBONATE	0.00E	0	0	0	0	0	0	0	0	0	0	0
MACR	AL OXIDE	0.00E	0	0	0	0	0	0	0	0	0	0	0
MACR	FE OXIDE/CARB.	1.95E*	0	0	0	0	0	0	0	0	0	0	0
MACR	SI OXIDE	3.25E**	0	0	0	0	0	0	0	0	0	0	0
MACR	ZR SI OXIDE	12.01E*****	0	0	0	0	0	0	0	0	0	0	0
MACR	CA PHOSPHATES	0.00E	0	0	0	0	0	0	0	0	0	0	0
MACR	FE ZN CU SULPH	1.30E*	0	0	0	0	0	0	0	0	0	0	0
*SUB	CU-S	0.00E	0	0	0	0	0	0	0	0	0	0	0
*SUB	ZN-S	0.00E	0	0	0	0	0	0	0	0	0	0	0
*SUB	FE-S	0.97E	0	0	0	0	0	0	0	0	0	0	0
MACR	CA AL SILICATE	5.84E***	0	0	0	0	0	0	0	0	0	0	0
MACR	CA FE SILICATE	1.95E*	0	0	0	0	0	0	0	0	0	0	0
MACR	FE AL SILICATE	1.95E*	0	0	0	0	0	0	0	0	0	0	0
MACR	AL SILICATES	2.27E*	0	0	0	0	0	0	0	0	0	0	0
MACR	FE STAIN SI OX	0.97E	0	0	0	0	0	0	0	0	0	0	0
MACR	NA CA MG SIL.	13.96E*****	0	0	0	0	0	0	0	0	0	0	0
MACR	K AL SILICATE	0.00E	0	0	0	0	0	0	0	0	0	0	0
MACR	MG AL OXIDE	0.00E	0	0	0	0	0	0	0	0	0	0	0
MACR	FE MN OXIDE	0.97E	0	0	0	0	0	0	0	0	0	0	0
MACR	MISC. MACRO	18.83E*****	0	0	0	0	0	0	0	0	0	0	0
MACR	SUM UNKNOWN	0.00E	0	0	0	0	0	0	0	0	0	0	0

Figure 6. Histogram of particle population percent versus chemical class (macro class). [Actual data output from the image analysis program for a sample collected from station N5 in the test study area (fig. 5).]

SAMPLE ID: SILT-SIZE HEAVY MINERALS N5											13-SEP-91
NUMBER OF PARTICLES BY CHEMISTRY AND AVERAGE DIAMETER (UM.) RANGE											
CHEMICAL CATEGORY \	< 4.00		< 8.00		<16.00		<31.00		<62.00		/ TOTAL
	E	0	E	0	E	0	E	0	E	0	
TI OXIDES	0	0	0	0	0	0	8	6			14
FE TI OXIDE	0	1	3	2	38	47					91
CA TI SILICATE	0	0	0	0	2	0					2
MG CARBONATE	0	0	0	0	0	0					0
AL OXIDE	0	0	0	0	0	0					0
FE OXIDE/CARB.	0	1	0	1	3	1					6
SI OXIDE	0	4	0	1	2	3					10
ZR SI OXIDE	0	0	0	0	15	22					37
CA PHOSPHATES	0	0	0	0	0	0					0
FE ZN CU SULPH	0	2	1	0	0	1					4
CA AL SILICATE	1	1	0	0	10	6					18
CA FE SILICATE	0	0	1	0	3	2					6
FE AL SILICATE	0	1	1	0	4	0					6
AL SILICATES	0	2	0	1	0	4					7
FE STAIN SI OX	0	0	0	0	0	3					3
NA CA MG SIL.	0	2	3	3	20	15					43
K AL SILICATE	0	0	0	0	0	0					0
MG AL OXIDE	0	0	0	0	0	0					0
FE MN OXIDE	0	0	0	0	2	1					3
MISC. MACRO	5	6	3	4	23	17					58
SUM UNKNOWN											0
TOTAL	6	20	12	12	130	128					308

Figure 7. The number of particles in user-specified size ranges within each chemical category. Symbols "E 0" in the column headings are exponential notation for ten raised to the zero power, or $10^0=1$. The numerical values in the headings are equal to the number indicated times ten raised to the power shown. [Actual data output from the image analysis program for a sample collected from station N5 in the test study area (fig. 5). UM.=Micrometers.]

SAMPLE ID: SILT-SIZE HEAVY MINERALS N5							13-SEP-91
PERCENT OF TOTAL PARTICLES BY CHEMISTRY AND AVERAGE DIAMETER (UM.) RANGE							
CHEMICAL CATEGORY \	< 4.00	< 8.00	<16.00	<31.00	<62.00	>62.00	/ TOTAL
	E 0	E 0	E 0	E 0	E 0	E 0	
TI OXIDES	0.	0.	0.	0.	2.60	1.95	4.55
FE TI OXIDE	0.	0.32	0.97	0.65	12.34	15.26	29.55
CA TI SILICATE	0.	0.	0.	0.	0.65	0.	0.65
MG CARBONATE	0.	0.	0.	0.	0.	0.	0.
AL OXIDE	0.	0.	0.	0.	0.	0.	0.
FE OXIDE/CARB.	0.	0.32	0.	0.32	0.97	0.32	1.95
SI OXIDE	0.	1.30	0.	0.32	0.65	0.97	3.25
ZR SI OXIDE	0.	0.	0.	0.	4.87	7.14	12.01
CA PHOSPHATES	0.	0.	0.	0.	0.	0.	0.
FE ZN CU SULPH	0.	0.65	0.32	0.	0.	0.32	1.30
CA AL SILICATE	0.32	0.32	0.	0.	3.25	1.95	5.84
CA FE SILICATE	0.	0.	0.32	0.	0.97	0.65	1.95
FE AL SILICATE	0.	0.32	0.32	0.	1.30	0.	1.95
AL SILICATES	0.	0.65	0.	0.32	0.	1.30	2.27
FE STAIN SI OX	0.	0.	0.	0.	0.	0.97	0.97
NA CA MG SIL.	0.	0.65	0.97	0.97	6.49	4.87	13.96
K AL SILICATE	0.	0.	0.	0.	0.	0.	0.
MG AL OXIDE	0.	0.	0.	0.	0.	0.	0.
FE MN OXIDE	0.	0.	0.	0.	0.65	0.32	0.97
MISC. MACRO	1.62	1.95	0.97	1.30	7.47	5.52	18.83
SUM UNKNOWN							0.00
TOTAL	1.95	6.49	3.90	3.90	42.21	41.56	100.00

Figure 8. The percentage of total particles in size ranges for each chemical category. Symbols "E O" in the column headings are exponential notation for ten raised to the zero power, or 10⁰=1. The numerical values in the headings are equal to the number indicated times ten raised to the power shown. [Actual data output from the image analysis program for a sample collected from station N5 in the test study area (fig. 5). UM.=Micrometers.]

SAMPLE ID: SILT-SIZE HEAVY MINERALS N5											13-SEP-91		
WEIGHT PERCENT VS. CHEMICAL CLASS													
TYP	CLASS NAME	%	0	1	2	3	4	5	6	7	8	9	10
			0	0	0	0	0	0	0	0	0	0	0
MACR	TI OXIDES	5.13E***											
MACR	FE TI OXIDE	39.85E*****											
MACR	CA TI SILICATE	0.48E											
MACR	MG CARBONATE	0.00E											
MACR	AL OXIDE	0.00E											
MACR	FE OXIDE/CARB.	1.82E*											
MACR	SI OXIDE	1.38E*											
MACR	ZR SI OXIDE	17.99E*****											
MACR	CA PHOSPHATES	0.00E											
MACR	FE ZN CU SULPH	0.34E											
*SUB	CU-S	0.00E											
*SUB	ZN-S	0.00E											
*SUB	FE-S	0.03E											
MACR	CA AL SILICATE	4.88E**											
MACR	CA FE SILICATE	1.68E*											
MACR	FE AL SILICATE	1.16E*											
MACR	AL SILICATES	2.27E*											
MACR	FE STAIN SI OX	1.07E*											
MACR	NA CA MG SIL.	9.96E*****											
MACR	K AL SILICATE	0.00E											
MACR	MG AL OXIDE	0.00E											
MACR	FE MN OXIDE	1.29E*											
MACR	MISC. MACRO	10.71E*****											
MACR	SUM UNKNOWN	0.00E											

Figure 9. A histogram of weight percentages versus chemical category. [Actual data output from the image analysis program for a sample collected from station N5 in the test study area (fig. 5).]

SAMPLE ID: SILT-SIZE HEAVY MINERALS N5							13-SEP-91
WEIGHT PERCENT OF TOTAL SAMPLE BY CHEMISTRY AND AVERAGE DIAMETER (UM.) RANGE							
CHEMICAL CATEGORY \	< 4.00 E 0	< 8.00 E 0	<16.00 E 0	<31.00 E 0	<62.00 E 0	>62.00 E 0	/ TOTAL
TI OXIDES	0.	0.	0.	0.	2.10	3.03	5.13
FE TI OXIDE	0.	0.01	0.04	0.18	11.29	28.34	39.85
CA TI SILICATE	0.	0.	0.	0.	0.48	0.	0.48
MG CARBONATE	0.	0.	0.	0.	0.	0.	0.
AL OXIDE	0.	0.	0.	0.	0.	0.	0.
FE OXIDE/CARB.	0.	0.00	0.	0.03	1.16	0.63	1.82
SI OXIDE	0.	0.01	0.	0.02	0.40	0.95	1.38
ZR SI OXIDE	0.	0.	0.	0.	5.16	12.82	17.99
CA PHOSPHATES	0.	0.	0.	0.	0.	0.	0.
FE ZN CU SULPH	0.	0.01	0.01	0.	0.	0.32	0.34
CA AL SILICATE	0.00	0.00	0.	0.	2.10	2.77	4.88
CA FE SILICATE	0.	0.	0.02	0.	0.71	0.95	1.68
FE AL SILICATE	0.	0.01	0.01	0.	1.15	0.	1.16
AL SILICATES	0.	0.01	0.	0.03	0.	2.24	2.27
FE STAIN SI OX	0.	0.	0.	0.	0.	1.07	1.07
NA CA MG SIL.	0.	0.00	0.03	0.12	3.29	6.52	9.96
K AL SILICATE	0.	0.	0.	0.	0.	0.	0.
MG AL OXIDE	0.	0.	0.	0.	0.	0.	0.
FE MN OXIDE	0.	0.	0.	0.	0.64	0.65	1.29
MISC. MACRO	0.00	0.01	0.03	0.12	4.58	5.97	10.71
SUM UNKNOWN							0.00
TOTAL	0.00	0.06	0.14	0.50	33.05	66.25	100.00

Figure 10. Weight percentages for size ranges within each chemical category. Symbols "E O" in the column headings are exponential notation for ten raised to the zero power, or $10^0=1$. The numerical values in the headings are equal to the number indicated times ten raised to the power shown. [Actual data output from the image analysis program for a sample collected from station N5 in the test study area (fig. 5). UM.=Micrometers.]

abundances are derived from the chemical category (macro class) information from SEM/AIA/EDS analysis, from the observation of particle grain morphology in the SEM, and from XRD.

Poppe and others (1991) discuss the scientific relevance of the data listed in this report. The scientific study has revealed several important relationships with regard to the composition, abundance, and areal distribution of silt- and sand-sized heavy minerals in a modern depositional environment on the New England Continental Shelf. Some of the relationships are listed below.

1. Regardless of sediment texture, heavy minerals constitute a substantially greater weight percentage of the silt fraction than that of the sand fraction (table 4).
2. The silt-sized heavy mineral assemblage is diverse, is predominately detrital, and reflects the metamorphic and igneous character of the bedrock of New England and eastern Canada (table 5). The presence of extensively weathered ilmenite grains suggests that the provenance may also contain a sedimentary component.
3. There are pronounced compositional differences between the mineralogies of the silt and sand fractions. For example, corundum and the TiO_2 , epidote, and phosphate groups are enriched in the silt fraction (table 5); spinel and the amphibole, garnet + staurolite, and aluminum silicate groups are relatively

concentrated in the sand fraction (Ross, 1970; Hathaway, 1971).

4. TiO_2 is enriched in the silty sands southwest of Nantucket Shoals (fig. 5 and table 5). These sediments should be evaluated further for their economic potential.

SUMMARY

We have employed a new, nontoxic, heavy-liquid medium, an aqueous solution of SPT, to simplify the procedures necessary to separate silt-sized minerals by specific gravity. Samples of the silt-sized, heavy-mineral fraction were prepared for examination in a SEM equipped with an AIA and EDS system. Hundreds of particles within each sample were sized and sorted according to chemistry, and statistical data were output in histograms and tables. Chemical categories can be carefully designed by the user to simulate distinct minerals or mineral groups. Because of our scientific interests, the study area chosen was in a marine environment, but the same techniques can be employed on sediments from other environments. Particle feature analysis makes it possible to sort large numbers of particles by size and chemistry. Most manufacturers of current energy-dispersive X-ray equipment offer optional particle feature hardware and software as part of the digital imaging package.

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