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A METHOD FOR THE QUANTITATIVE ESTIMATION OF CLAY MINERALS IN NORTH PACIFIC DEEP-SEA SEDIMENTS

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Abstract—The addition of a 10% talc internal standard to North Pacific sediments allows the relative abundances of clay minerals to be determined both accurately and precisely by X-ray powder diffractometry. Linear programming can be used to generate factors for converting talc-normalized peak areas to weight percentages; hence, absolute clay-mineral abundances can be estimated. This procedure minimizes residuals (nondiffracting or poorly crystalline components), but its accuracy is untested. Even this procedure results in an average residual of almost 30% for North Pacific sediments; other peak-area to weight conversion schemes generate even larger values.

In general, there is no correlation between clay-mineral abundances estimated from talc-normalized peak areas and abundances derived from the assumption that the sum of smectite, illite, kaolinite, and chlorite is 100%. This accounts for the past difficulties in relating bulk-sediment chemistry to clay mineralogy.

Key Words—Clay Distribution, Clay Quantification, Deep-sea, Pacific, X-ray Methods.

INTRODUCTION

Over the past 15 years, a number of papers have described the distribution and abundance of clay minerals within major ocean basins (Biscaye, 1965; Gorbunova, 1963; Griffin and Goldberg, 1963; Kolla *et al.*, 1976; Venkatarathnam and Biscaye, 1973; Yeroshchev-Shak, 1962) and even the world ocean (Griffin *et al.*, 1968; Rateev *et al.*, 1969). In all cases, however, the abundance values were derived from the assumptions that peak-area ratios from X-ray powder diffractograms are related to the corresponding weight ratios of clay minerals by known simple factors (Biscaye, 1965; modified from Johns *et al.*, 1954), and that clay minerals constitute 100% of the fine fraction of deep-sea sediments.

These assumptions have allowed large numbers of samples to be analyzed and compared. The resulting global and oceanic maps tell us a great deal about the influence of source areas, dispersion, and bottom transport in establishing the nature of nonbiogenic pelagic sediments. At the same time, however, such clay-mineral values cannot be used for estimating the quantitative importance of various sediment sources, or for comparison with other geochemical data.

During a study of the provenance of North Pacific deep-sea sediments, it became obvious that efforts to relate the mineralogy of sediment samples to their elemental compositions were limited by the qualitative nature of estimates of mineral abundances. An attempt has been made to improve these estimates by using an internal standard in samples analyzed by X-ray powder diffraction, and by reassessing the factors used to convert ratios of peak areas to weight fractions of North Pacific clays.

METHODOLOGY

All samples were taken from the tops of cores in the Oregon State University and Scripps Institution of Oceanography collections (locations on Figure 2 and subsequent maps). These cores are stored in sealed tubes under refrigeration. Thus, contamination is minimal, and all the raw samples were still moist when processed. They were separated into three fractions: finer than 2 μm, 2–20 μm, and coarser than 20 μm, by repeated Stokes'-Law settling. This step also removed salts dissolved in interstitial waters. The present study deals only with the finest fraction, which is widely dispersed by bottom currents. A single bottom sample of such fine material is representative of a large surrounding area (Heath *et al.*, 1970). Amorphous ferromanganese oxides were removed by the dithionite-citrate-bicarbonate treatment of Mehra and Jackson (1960), and the residues were saturated with Mg before being freeze-dried for storage.

For X-ray powder diffraction analysis, 300 mg of clay was dispersed in 5 ml of deionized water. A similar suspension of talc, prepared by wet-grinding and Stokes'-Law settling, was made at the same time. A homogenized mixture of 1.8 ml of clay suspension and 0.2 ml of talc suspension was used to make two oriented clay aggregates by rapidly drawing the mixture onto a porous silver plug in an X-ray planchet attached to a vacuum flask. The aggregates were solvated with ethylene glycol and X-rayed using diffracted-beam monochromatized CuKα radiation. Diffractograms were recorded digitally at 0.02°2θ intervals, filtered using the Gaussian filter of Rex (1970), and digitally plotted with the

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maximum peak intensity at full scale. Peak areas were measured with a planimeter, after subtraction of a standardized background. The areas under the overlapping 9–10-Å illite-talc and 3.4-Å kaolinite-chlorite peaks were partitioned using a Dupont 310 Curve Resolver. A measure of the precision of the analyses is given by the differences between data from the paired planchets of each sample. For the clay minerals, the mean absolute differences (using Biscaye's (1965) conversion factors) were 2.0, 2.3, 0.8, and 1.6% for smectite, illite, kaolinite, and chlorite, respectively. These correspond to relative differences of 8.6, 6.0, and 8.5, and 7.1% of the amounts present. For the minor components quartz, amphibole, and plagioclase, the mean absolute differences between paired planchets were 1.2, 1.8, and 0.7%, respectively, corresponding to relative differences of 26, 57, and 52%.

DATA REDUCTION

To simplify processing, all peak areas were ratioed to the area of the 9.3 Å peak of the 10% talc internal standard; mineral symbols in the following discussion refer to these talc-normalized peak areas. The mineral designators and corresponding peaks are: S = smectite 17 Å (001), expanded by ethylene glycol; I = illite 10 Å (001); K = kaolinite 7 Å (001); C = chlorite 7 Å (002); A = amphibole 8.4 Å (110); Q = quartz 3.3 Å (101); and P = plagioclase 3.2 Å complex. The 7 Å peak is divided between kaolinite and chlorite in proportion to the relative intensities of their 3.58 Å (002) and 3.54 Å (004) peaks, respectively (Biscaye, 1964).

Biscaye's method

For each sample, the percentages of the clay minerals are given by:

$$\text{Illite (for example)} = \frac{4\text{I} \times 100}{\text{S} + 4\text{I} + 2\text{K} + 2\text{C}} \qquad (1)$$

where the weighting factors to convert peak areas to relative weight fractions for smectite, illite, kaolinite, and chlorite are 1, 4, 2, and 2, respectively (Biscaye, 1965, after Johns *et al.*, 1954).

Biscaye's method (corrected)

Because all aggregates contained an internal standard, the normalized peak areas can be compared from sample to sample. Thus Biscaye's (1965) assumption that the sum of the clay minerals is 100% in each sample can be modified to let the sum total 100% only for the sample with the maximum value of (S + 4I + 2K + 2C). Eq. (1) now becomes:

$$\text{Illite (corrected)} = \frac{4\text{I} \times 100}{(\text{S} + 4\text{I} + 2\text{K} + 2\text{C})_{\max}} \qquad (2)$$

This correction yields ratios of the abundances of each mineral between samples that are correct in an absolute sense, regardless of possible errors in the factors used to convert peak areas to concentrations. This procedure generates a residual for most samples, where the residual (R) is given by:

$$\text{R} = 100\left[1 - \frac{(\text{S} + 4\text{I} + 2\text{K} + 2\text{C})_{\text{sample}}}{(\text{S} + 4\text{I} + 2\text{K} + 2\text{C})_{\max}}\right] \qquad (3)$$

R is a measure of poorly crystalline minerals (which will be underestimated by the Biscaye factors) and of phases that are X-ray amorphous.

Because the denominator of Eq. (2) and (3) is constant for all the samples in the set, Eq. (2) can be modified to:

$$\text{Illite (corrected)} = 40\text{I} \qquad (4)$$

where the factors for converting the areas of talc-normalized smectite, illite, kaolinite, and chlorite peaks to weight percentages are 10, 40, 20, and 20, respectively.

Internally generated factors

The general form of the equation describing the preceding method is:

$$\begin{aligned}
&\text{F}_1\text{A}_{11} + \text{F}_2\text{A}_{12} + \cdots \text{F}_n\text{A}_{1n} + \text{R}_1 = 100\\
&\text{F}_1\text{A}_{21} + \text{F}_2\text{A}_{22} + \cdots \text{F}_n\text{A}_{2n} + \text{R}_2 = 100\\
&\quad\vdots \qquad\quad \vdots \qquad\quad\quad \vdots \qquad\quad \vdots\\
&\text{F}_1\text{A}_{m1} + \text{F}_2\text{A}_{m2} + \cdots \text{F}_n\text{A}_{mn} + \text{R}_m = 100
\end{aligned} \qquad (5)$$

where $\text{A}_{m1\cdots mn}$ are the talc-normalized peak areas from X-ray powder diffractograms of n minerals in m samples, and $\text{F}_{1\cdots n}$ are the factors to convert the areas to weight percentages. If the F and R values are treated as unknowns, then for n minerals and m samples, there are m equations in m + n unknowns—clearly an underdefined system. One method of handling such a system is to minimize the R values. A simple least-squares minimization procedure is not satisfactory, however, because many of the F values will be negative; not a very useful attribute for quantitative mineralogy!

Instead, a minimization technique of linear programming (Heady and Candler, 1958; Geary and McCarthy, 1964) has been employed. The form used has the following characteristics:

(1) $\text{A}_{ij} \geq 0$ for all i and j (6)

(2) $\text{F}_j > 0$ for all j (7)

(3) $\sum_{j=1}^{n} \text{F}_j\text{A}_{ij} \leq 100$ for all i (8)

(4) $\text{C}_1\text{F}_1 + \text{C}_2\text{F}_2 + \cdots \text{C}_n\text{F}_n = \text{U}$, where U is maximized (9)

The coefficients $\text{C}_{1\cdots n}$ are defined by the user.

Eq. (6) prohibits negative peak areas; Eq. (7) prohibits negative or zero conversion factors (the simplest solution for the North Pacific data, for example, set all but the illite factors to zero and allowed the R values

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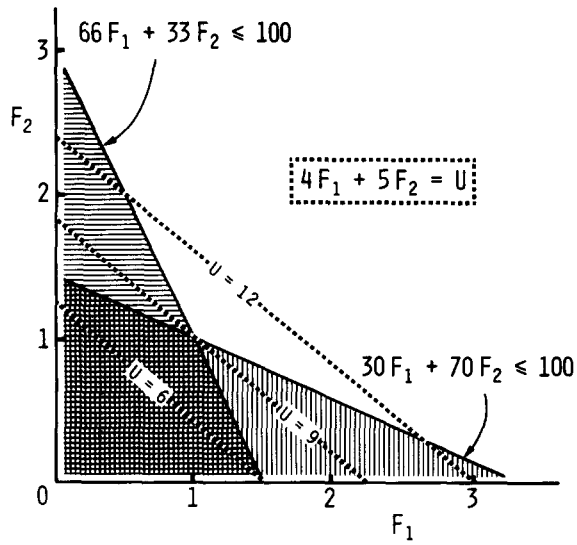


Figure 1. Graphic illustration of the use of linear programming to derive coefficients for a 2-sample, 2-mineral system. See text for details.

to take up the slack); and Eq. (8) is a restatement of (5). Eq. (9) is discussed below.

The use of linear programming can be illustrated by the simple example shown in Figure 1. In this case, only two samples, each containing the same two minerals are considered. In one case the peak areas for the minerals are 66 and 33 units, respectively. The values of the F factors satisfying Eq. (8) for this sample must fall in the horizontally shaded region of Figure 1. Similarly, the second sample has peak areas of 30 and 70 units, so that F values must fall in the vertically shaded region. Clearly, the cross-hatched region contains all values of F_1 and F_2 that satisfy both requirements. In addition, C_1 and C_2 of Eq. (9) have been set equal to 4 and 5, respectively. The locus of Eq. (9) for three values of U (6, 9, and 12) is shown on Figure 1. The maximum value of U that falls in the cross-hatched region is 9, corresponding to an F_1 value of 1 and an F_2 value of 1. In this example, there are no residuals.

For the North Pacific sediments, the calculation has been extended to 7 minerals and 143 samples. Because of the trace concentrations of, and consequent poor relative analytical precisions for quartz, plagioclase, and amphibole, stable factors for these minerals could not be generated. Based on the work of Borg and Smith (1969) and Rex (1970), and on our own experiments, factors of 9.2 and 39, respectively, were estimated, to convert talc-normalized quartz and amphibole peak-areas to weight percentages (quartz areas were first reduced by 12.5% of the measured illite areas to correct

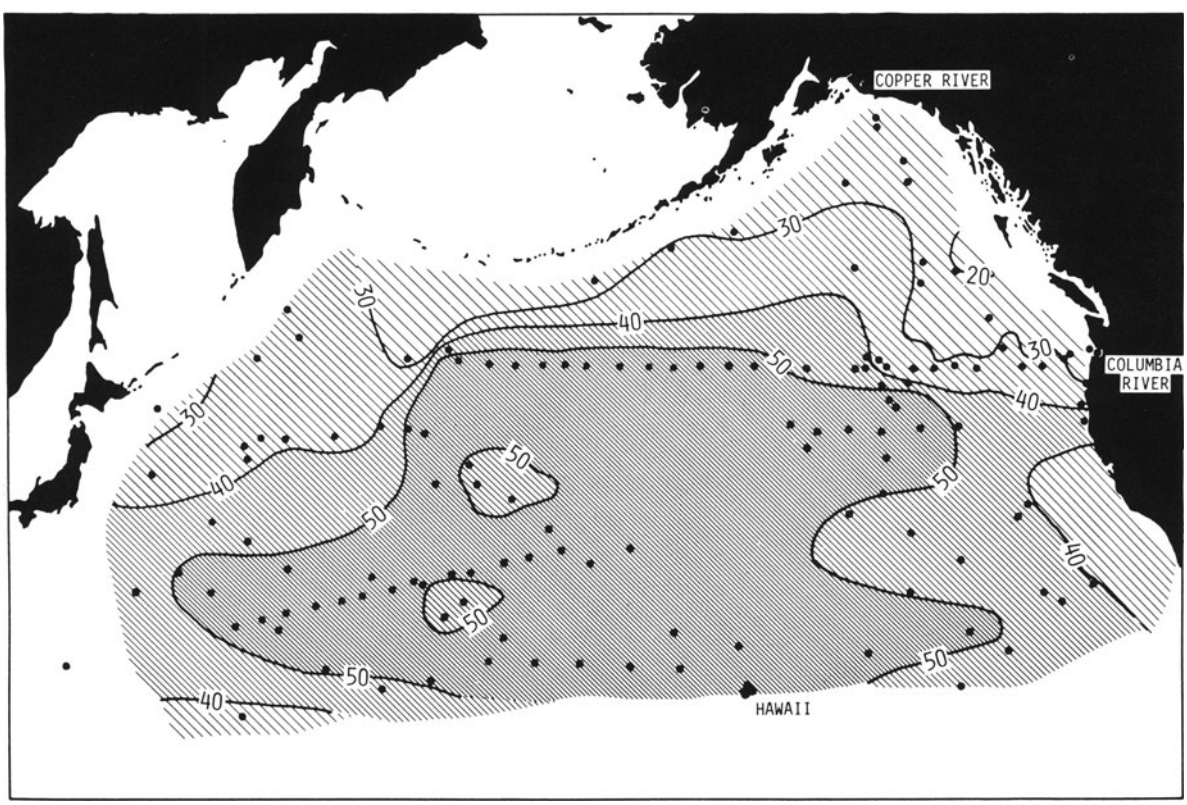


Figure 2. Illite abundances (weight percent) in the fine fraction of North Pacific sediments, as estimated by Biscaye's (1965) technique.

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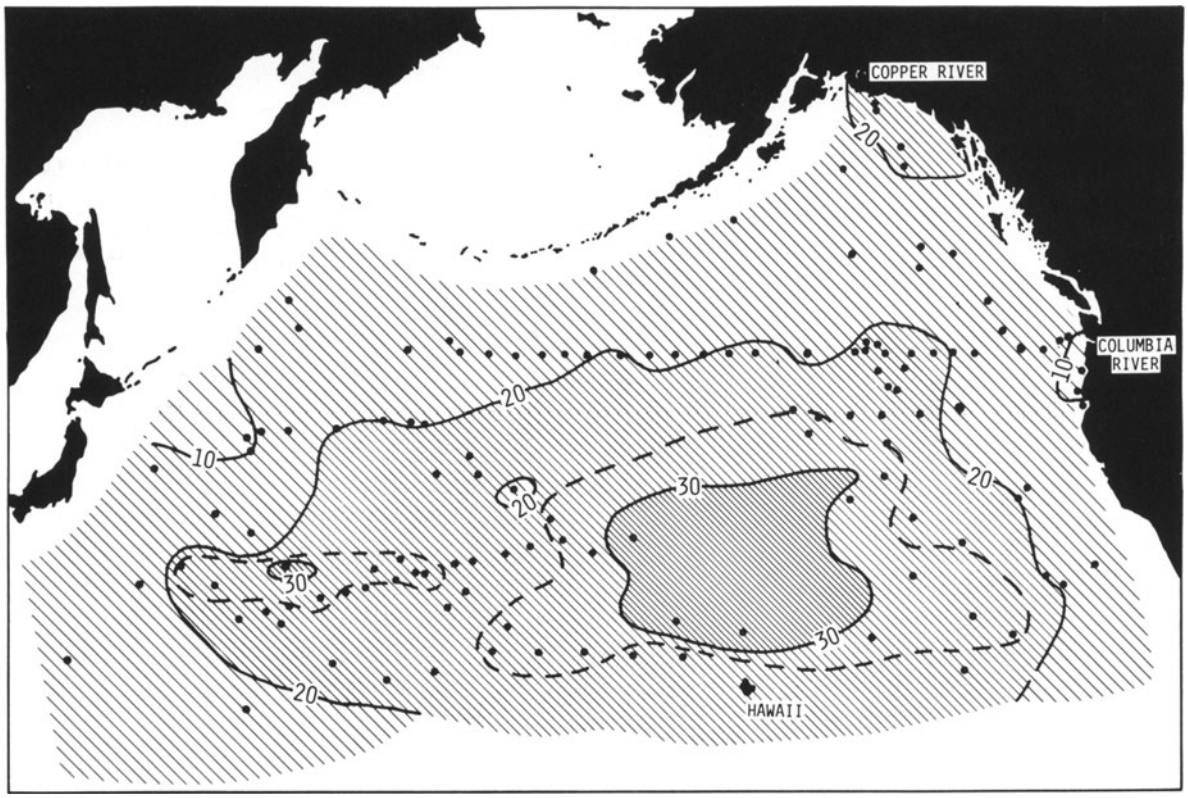


Figure 3. Illite abundances (weight percent) in the fine fraction of North Pacific sediments as estimated by Biscaye's (1965) technique corrected by text equation (2).

for overlap of the illite (003) and quartz (101) peaks). A factor of 49, based on earlier work by Heath (1968), was used to convert talc-normalized plagioclase peak-areas to weight percentages.

The selection of the C coefficients in Eq. (9) requires some explanation. Obviously, there is no *a priori* reason for assigning a greater "value" to one mineral than to another. Fortunately, the final solution is quite insensitive to the choice of these coefficients. This is equivalent to changing the slope of the "U" lines in Figure 1 without changing their maximum-value intersection with the apex of the cross-hatched field. Apparently, the multidimensional apex for the North Pacific samples is sufficiently peaked that changing the C values from unity to the Biscaye ratios, for example, does not alter the F array. The F values generated by the linear programming analysis that were used to convert talc-normalized smectite, illite, chlorite, and kaolinite peak-areas to weight percentages are 8.0, 75.2, 8.5, and 57.1, respectively.

RESULTS

Only illustrative results are described here. Data lists, maps of mineral distributions, and more complete geologic and oceanographic interpretations of the patterns will be presented elsewhere.

Figure 2 shows the distribution of illite in North Pacific sediments finer than 2 μm, calculated according to Biscaye's (1965) method. The pattern is consistent with earlier maps by Griffin *et al.* (1968) and Rateev *et al.* (1969), although it shows more detail because of the larger number of samples. The agreement between our numerical values and those of Griffin *et al.* (1968) is an indication of the degree to which standardization of sample preparation and analytical techniques have allowed clay-mineral data from different laboratories to be merged.

If, however, the illite values are adjusted by Eq. (2) to produce correct absolute ratios from sample to sample, the picture changes dramatically (Figure 3). The highest values are reduced by almost a factor of two, with some tendency for the east–west contours of Figure 2 to parallel the ocean margins more closely. Inasmuch as the large illite values of Figure 2 have been attributed to the deposition of eolian material derived from Asia and North America (Griffin *et al.*, 1968), Figure 3 implies that such material is substantially diluted adjacent to land areas, presumably by illite-poor fluvial debris.

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The correlation between the raw and adjusted Biscaye (1965) values is poor (Figure 4), with the adjusted values ranging up to an order of magnitude lower than the raw values. The other clay minerals display similar patterns (Figure 4). Only chlorite shows some correlation between adjusted and raw values—a reflection of a negative correlation between chlorite and residual values. The patterns of Figure 4 clearly explain why geochemists have such difficulty in correlating clay mineralogy with other compositional properties of deep-sea sediments.

The residuals generated by Eq. (2), which average 47%, form geographically coherent patterns (Figure 5). Low values cluster along the northern edge of the basin, where the sediments are dominated by mechanically weathered debris of glacial origin. In contrast, the high values off the Columbia River and east Asia mark more intensely chemically weathered debris derived primarily from igneous rocks of intermediate composition. The high values in the center of the region are found in samples rich in biogenic opal and authigenic components.

The effect of linear programming (l.p.) in reducing residuals is clearly illustrated by comparing Figures 5 and 6. The basic pattern remains the same, confirming that the residual values are geologically meaningful, rather than mathematical artifacts. However, the l.p. residuals (which average 29%) are 10 to 30% lower than the adjusted Biscaye residuals. The pattern of reduction is not random (Figure 7), indicating that the abundances of the four major minerals (which are proportional to the normalized peak areas in both cases) do not vary randomly in our sample suite.

The illite abundances estimated using the l.p. factor are closer to the raw Biscaye estimates than to the adjusted values. The overall correlation in the former case is poor, however (Figure 8), again indicating why conventional clay mineral estimates are so difficult to interpret geochemically.

The distribution of illite values estimated using the l.p. factor (Figure 9) shows a well-defined band of maxima at about 30°N, in good agreement with the band of eolian quartz described by Rex and Goldberg (1958) and Heath *et al.* (1973). The pattern differs from Figure 2 principally in the low concentrations off east Asia, due to large residual values, and in the better defined southern limit of the illite-rich deposits.

DISCUSSION

A major unknown in linear programming (or any minimization procedure, for that matter) is the stability of the resulting coefficients. To gain insight to this problem, the calculation was carried out nine times, using the full data set and eight subsets created by randomly removing three to twenty samples. The results suggest uncertainties (1 standard deviation) in our factors for

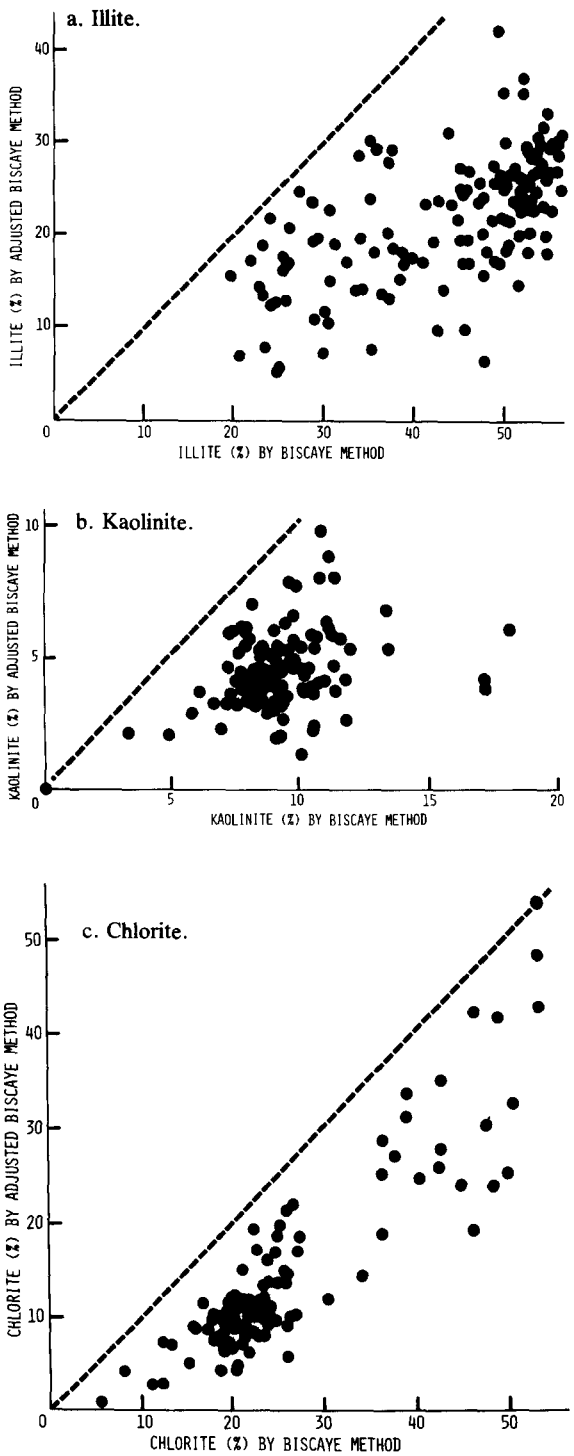


Figure 4. Clay mineral abundances (weight percent) estimated by Biscaye's (1965) technique compared with abundances estimated by the modification to the same technique given in text Eq. (2). a. Illite. b. Kaolinite. c. Chlorite.

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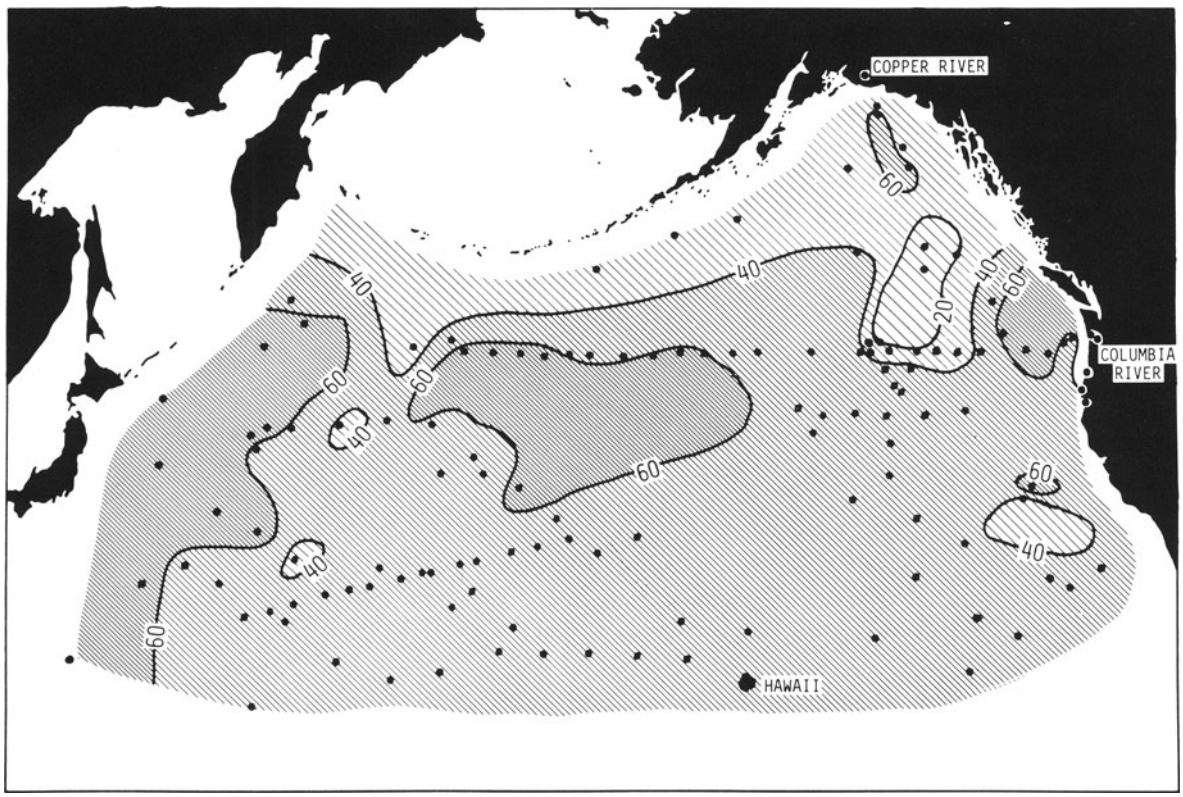


Figure 5. Percentage of the fine fraction of North Pacific sediments unaccounted for by clay minerals (residuals), as estimated by Biscaye's (1965) technique corrected by text Eq. (2).

smectite, illite, kaolinite, and chlorite of 11, 4, 15, and 7%, respectively.

A more difficult question concerns the validity of the philosophy underlying the method. Is the assumption that the amount of nondiffracting components must be as small as possible a reasonable one? From a purely thermodynamic viewpoint, it is. Amorphous phases will always be less stable than crystalline ones, so the latter should be created at the expense of the former in nature. There is ample observational evidence for such a trend, but even on a geologic time scale, such reactions proceed extremely slowly, particularly at 1 or 2°C on the deep-sea floor. Thus, the technique of minimizing residuals is certainly reasonable, but we have no independent evidence of its validity.

A closely related question concerns the validity of using the same conversion factors for all concentration ranges and over a geographic region as large as the North Pacific. Samples were diluted with ground glass (<2 μm) but no deviations in the talc-normalized peak areas from the expected linear response in the concentration range of our samples were found. This implies, for example, that realistic baselines for measuring peak areas have been selected.

The validity of the factors for all samples in our suite is much harder to assess. In the case of illite and associated minerals which seem to be derived from a well-mixed atmospheric reservoir, the method should be valid. Similarly, minerals such as quartz which show little variation in crystallinity in marine sediments should yield reliable data by this method. The case is less clear for minerals such as amphibole or smectite, which can have highly variable compositions, and which are supplied to North Pacific sediments from more than one source (both detrital and authigenic, in the case of smectite).

The question may be resolved by separating pure minerals from a spectrum of samples and determining the relation of talc-normalized peak areas to concentrations. Gibbs (1967) used this technique in a study of Amazon sediments. This difficult and time-consuming procedure has not been carried out in the present study.

Thus, the above technique represents an intermediate stage in the determination of absolute concentrations of clay minerals in fine sediments. For suites of samples containing varying proportions of the same minerals, the procedure used for this study will yield relative concentrations of a given mineral from sample to sample that have no systematic errors. Such data can solve many geochemical budgetary problems (e.g.,

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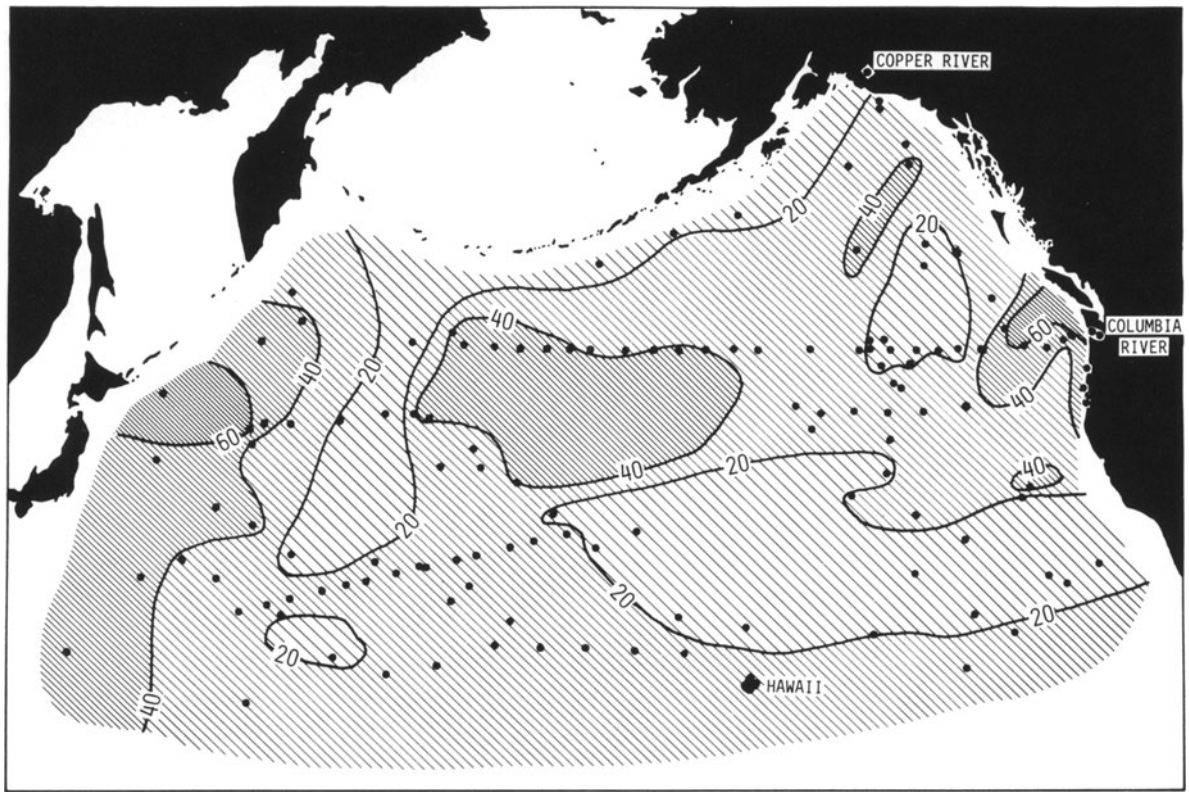


Figure 6. Percentage of the fine fraction of North Pacific sediments unaccounted for by clay minerals, quartz, feldspar, and amphibole (residuals) estimated by linear programming. See text for details.

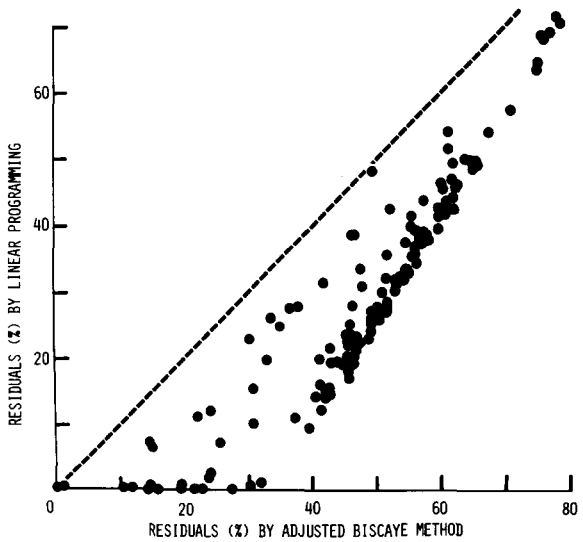


Figure 7. Fine-fraction residuals estimated by Biscaye's (1965) technique adjusted by text Eq. (2), compared to residuals estimated by linear programming.

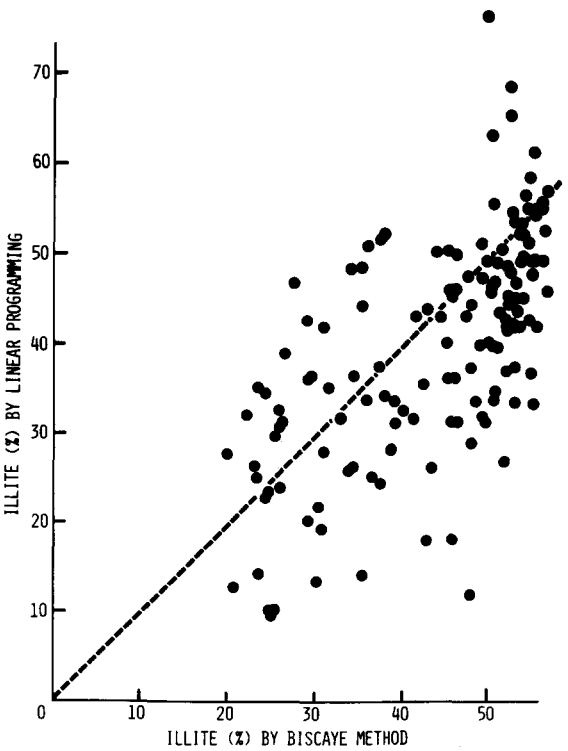


Figure 8. Poor correlation of illite abundances (weight percent) estimated by Biscaye's (1965) technique with abundances estimated by linear programming.

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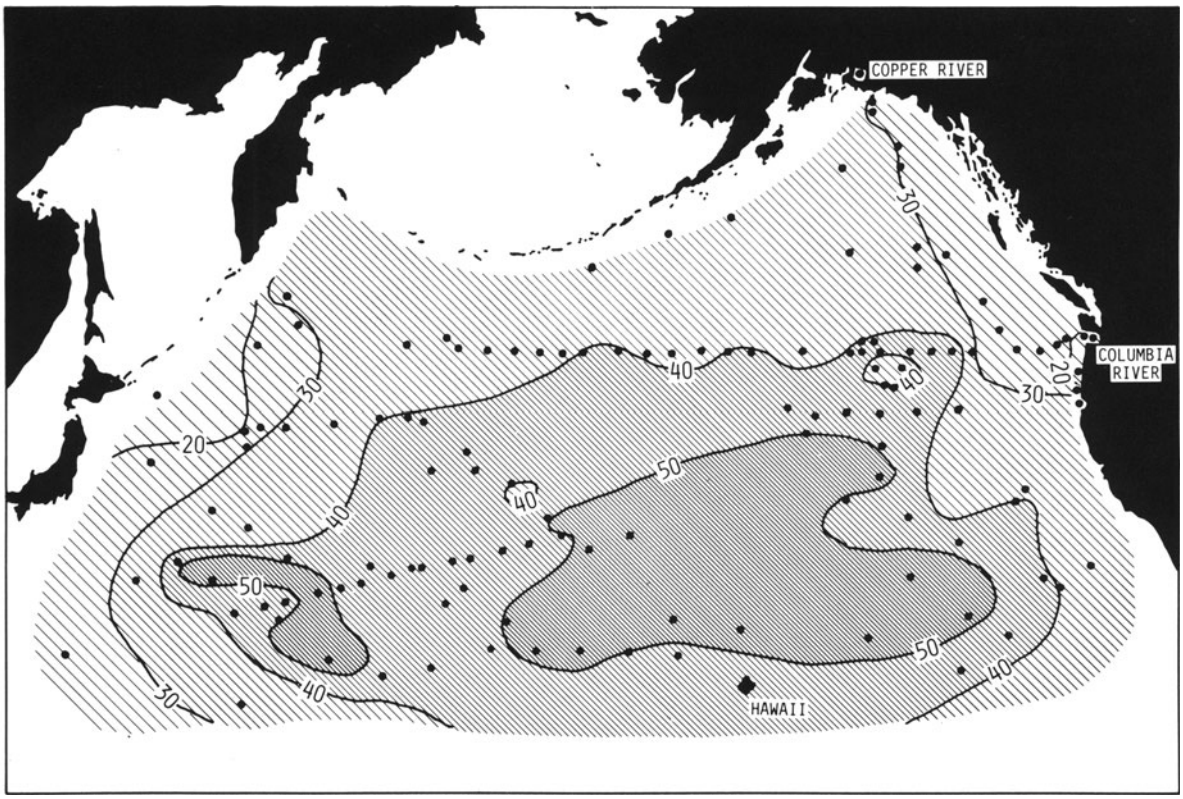


Figure 9. Illite abundances (weight percent) in the fine fraction of North Pacific sediments, as estimated by linear programming.

"what proportion of marine illite is deposited above and below 1000 m?"). Similarly, such data are adequate for mapping patterns of sediment dispersal, where each mineral is given equal weight in some form of multivariate analysis (e.g., Q-mode factor analysis). Because the concentrations selected may have systematic absolute errors, normative and modal compositions may still be difficult to relate, although the correspondence should be much better than if Biscaye (1965) concentrations are used.

The choice of factors to convert talc-normalized peak areas to weight percentages is debatable. The corrected Biscaye values retain the conventional within-sample mineral ratios, facilitating comparison with a large body of existing data. The linear programming values, however, generate concentrations that are closer to the true values, and therefore are more useful from a geochemical viewpoint, at least for North Pacific sediments. The factors to convert corrected Biscaye concentrations of smectite, illite, kaolinite, and chlorite to l.p. values are 0.8, 1.9, 2.9, and 0.43, respectively. In the absence of an internal standard, the l.p. relative weighting factors for the peak areas of smectite, illite, kaolinite, and chlorite are 1, 9.4, 7.1, and 1.1, respectively, in contrast to Biscaye's (1965) values of 1, 4, 2, and 2.

CONCLUSIONS

Precise clay mineral concentrations that accurately reflect sample to sample variations can be determined by adding a talc internal standard to deep-sea sediments. For North Pacific deep-sea deposits, as much as 80% of the sample is not accounted for by clay minerals if Biscaye's (1965) peak-area to weight-conversion factors are used. Even if factors calculated by linear programming to minimize residuals are used, as much as 70% of some samples is not accounted for.

Clay-mineral concentrations based on internal standards show no significant correlation with conventional estimates which assume that the clay minerals sum to 100%. It is not surprising that conventional values do not correlate well with other geochemical properties of deep-sea sediments.

The factors generated by linear programming suggest that relative to smectite, Biscaye's (1965) factors underestimate illite by a factor of 2 and kaolinite by a factor of 3.5, but overestimate chlorite by a factor of 2.

Application of the internal standard technique to a suite of 143 North Pacific sediments, using factors to convert talc-normalized peak areas to weight percentages estimated by linear programming, shows that the well-defined latitudinal band of eolian illite at 25–40°N

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is narrower than shown by Griffin *et al.* (1968) and is sharply bounded to both east and west by relatively illite-poor deposits. Most North Pacific sediments contain 20 to 50% nondiffracting material, with values rising to more than 60% off the Columbia River and northern Japan.

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Резюме—Добавление 10% талькового внутреннего эталона к Северо-Тихоокеанским осадкам позволяет определить с большой точностью и воспроизводимостью результатов относительные содержания глинистых минералов с помощью порошкового метода рентгеноструктурного анализа. С помощью линейного программирования могут быть получены множители для превращения площадей талько-нормализованных пиков в весовые проценты; следовательно могут быть оценены абсолютные содержания глинистых минералов. Эта процедура минимизирует остатки (не дифрагирующие или слабо кристаллизованные компоненты) но точность ее результатов не исследовалась. Даже эта процедура в среднем дает почти 30% остатков для Северо-Тихоокеанских осадков. Другие схемы преобразования пиковых площадей в вес дают даже большие величины.

В целом, не наблюдается коррелятции между содержаниями глинистых минералов, оцененных по талько-нормализованным пиковым площадям, и содержаниями, полученными в предположении, что сумма смектита, иллита, каолинита, и хлорита равна 100%. Это обуславливало в прошлом трудности в увязывании химического состава осадков и минералогии глин.

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Resümee—Die Zugabe eines inneren Standarts aus 10% Talk an Sedimente vom Nord Pazifik erlaubt die genaue und präzise Bestimmung der relativen Vorkommen von Tonmineralien durch Röntgenpulverdiagramme. Lineares programmieren kann benutzt werden, um Faktoren für die Umwandlung der auf Talk normalisierten Signalflächen in Gewichtsprozente zu erhalten; daher können absolute Vorkommen der Tonmieralien geschätzt werden. Diese Methode hält Rückstände (nicht diffraktierende oder schlecht kristallisierte Bestandteile) auf ein Minimum, aber ihre Genauigkeit ist unbestimmt. Selbst diese Methode ergibt einen durchschnittlichen Rückstand von 30% für Nord Pazifik Sedimente; andere Umrechnungen von Signalflächen in Gewicht bringen sogar noch größere Werte hervor.

Im Allgemeinen besteht kein Vergleich zwischen den Vorkommen von Tonmineralien, welche von auf Talk normalisierten Signalflächen erhalten wurden und denen, die von der Annahme, daß die Summe von Smektit, Illit, Kaolinit, und Chlorit 100% ist, abgeleitet wurden. Diese Tatsache erklärt die Schwierigkeiten, die man früher hatte, Mengen-Chemie mit Tonmineralogie zu verbinden.

Résumé—L'addition de 10% d'un standard de talc à des sédiments de l'océan pacifique nord permet la détermination exacte et précise par diffractométrie aux rayons-X des quantités relatives des minéraux argileux. La programmation linéaire peut être utilisée pour produire des facteurs pour la conversion de régions de maxima de normalisation au talc à des pourcentages de poids; ainsi, des abondances absolues de minéraux argileux peuvent être estimées. Ce procédé amoindri les résiduels (composants ne diffractant pas ou peu cristallins), mais son exactitude n'est pas testée. Même ce procédé résulte en un résiduel moyen de près de 30% pour les sédiments du Pacifique Nord; d'autres moyens de conversion de régions de maxima au poids produisent des valeurs plus grandes encore.

En général, il n'y a pas de corrélation entre les abondances de minéraux argileux estimées à partir des régions de maxima de normalisation au talc et les abondances dérivées de la présomption que la somme des smectites, illites, kaolinites, et chlorites est 100%. Ceci explique les difficultés rencontrées dans le passé pour établir une relation entre la chimie totale et la minéralogie des argiles.

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