

MINERALOGY, O^{18}/O^{16} , AND D/H RATIOS OF CLAY-RICH SEDIMENTS FROM DEEP SEA DRILLING PROJECT SITE 180, ALEUTIAN TRENCH¹

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Abstract—Mineralogy, O^{18}/O^{16} , and D/H ratios have been determined in five size fractions (<0.1 , 0.1 – 0.5 , 0.5 – 1.0 , 1.0 – 2.0 , and >2.0 μm) of seven samples taken from 500 m of Pleistocene deep-sea sediments cored at Deep Sea Drilling Project Site 180 in the Aleutian Trench. The depositional age of the samples spans the last 300,000 years; the samples have been interpreted by others to be continental detritus weathered from a mixed igneous, metamorphic, and sedimentary source area and then deposited by ice-rafting and turbidity currents. The minerals present are quartz, feldspar, illite, chlorite and/or non-expandable vermiculite, and expandable vermiculite and/or mixed-layer illite/expandable vermiculite. The relative amounts of quartz, feldspar, and total clay vary with particle size, but are nearly constant from sample to sample for a given particle size. δO^{18} values of the four coarser size fractions range from $+9.7$ to $+12.0\%$ with variations attributable to changes in quartz/feldspar and clay/(quartz + feldspar) abundances. Values of δO^{18} for the expandable vermiculite-rich <0.1 - μm size fraction range from $+12.1$ to $+16.3\%$ which indicates some oxygen isotope exchange at surface temperatures between meteoric waters and the parent rock during vermiculite formation. Values of δD range from -46 to -74% with variations attributable to changes in amounts of different clay minerals present. There is no mineralogic or isotopic evidence of post-depositional reactions in the coarser size fractions, but a general change in δD of the vermiculite-rich, <0.1 - μm size fraction from about -50% to about -70% with increasing depth may be due either to post-depositional isotopic exchange or to climatic changes in the terrestrial weathering environment.

Key Words—Deep sea sediments, D/H, Isotopic ratios, O^{18}/O^{16} , Provenance.

INTRODUCTION

The potential use of O^{18}/O^{16} and D/H ratios of silicates as provenance indicators has been recognized since the first detailed study of oxygen and hydrogen isotopes in sediments was made by Savin and Epstein (1970a, 1970b, 1970c). R. N. Clayton and M. L. Jackson and their colleagues published a series of papers (e.g., Clayton *et al.*, 1972; Churchman *et al.*, 1976; Mokma *et al.*, 1972) detailing the variation of δO^{18} of aeolian quartz with particle size and geography. Lawrence (1979) used δO^{18} to decipher source areas of Recent sediments in the south Atlantic Ocean. The present study incorporates detailed mineralogy and δD data with δO^{18} data of several grain-size fractions of deep-sea sediments. The emphasis in this paper is on: (1) how grain size is critical to the interpretation of δO^{18} and δD in a multicomponent detrital sediment; (2) how diagenesis is relatively unimportant in controlling the δO^{18} and δD

of these sediments; and (3) how δD of the sediments might complement mineralogy and δO^{18} in defining the nature of the source area.

The samples for this study were taken from Deep Sea Drilling Project (DSDP) cores from site 180, Leg 18, in the Aleutian Trench (Table 1). The sediments have been interpreted to be continental detritus transported to their depositional sites by ice-rafting and turbidity currents (von Huene *et al.*, 1973). Hayes (1973) demonstrated that the clays in the Aleutian Abyssal Plain are dominated by a complex assemblage of detrital, well-crystallized, terrigenous chlorite, mica, and vermiculite plus some mixed-layered clays. A study (Slatt and Piper, 1974) of heavy and light minerals of sands and coarse silts in Site 180 and nearby DSDP sites indicated two source areas: (1) pyroxene-rich sands, with lithic fragments of volcanic and sedimentary rocks, were eroded from the Alaska Peninsula and south-central Alaska; and (2) an altered-grain assemblage, with lithic fragments that include metamorphic and plutonic components, was eroded from southeastern and south-central Alaska.

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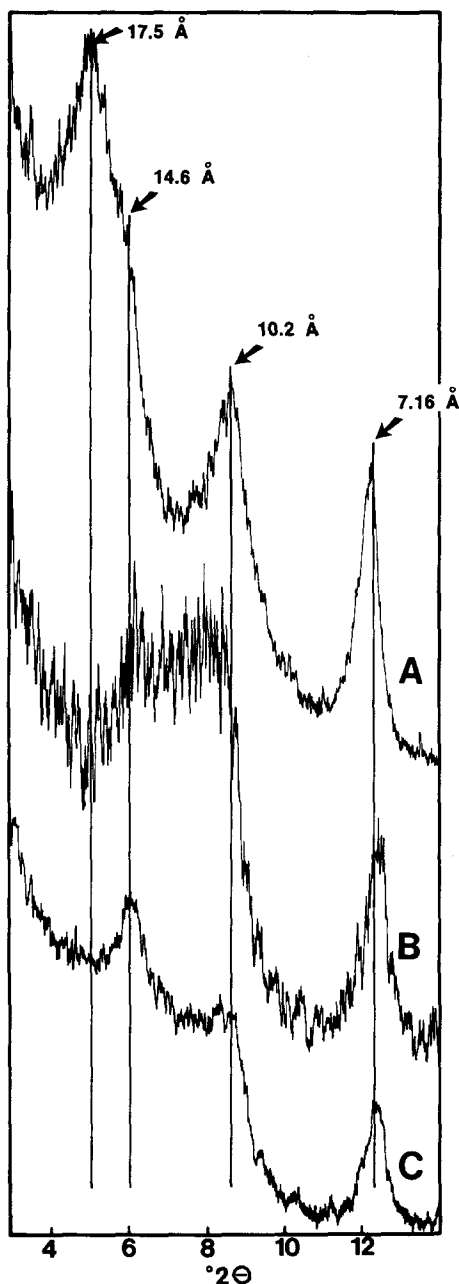


Figure 1. X-ray powder diffraction patterns of the $<0.1\text{-}\mu\text{m}$ size fraction of sample G. All patterns are from preferentially oriented slurry mounts. A = ethylene glycol; B = K-saturated; C = K-saturated followed by ethylene glycol.

ANALYTICAL TECHNIQUES

The samples were dispersed and treated to remove carbonates, noncrystalline and organic matter, and free iron oxides by the methods of Jackson (1974). The samples were then separated by centrifugation techniques into $<0.1\text{-}$, $0.1\text{-}0.5\text{-}$, $0.5\text{-}1.0\text{-}$, $1.0\text{-}2.0\text{-}$, and $>2.0\text{-}\mu\text{m}$

size fractions. Each size fraction of each of the seven samples was examined by X-ray powder diffraction (XRD) using a Norelco XRD-7 diffractometer equipped with a single crystal monochromator using $\text{CuK}\alpha$ radiation. Randomly oriented powder mounts were prepared and scanned from 33° to $18^\circ 2\theta$. Quantitative estimates of the amounts of quartz, feldspar, and total "clay" (phyllosilicates) were made by measuring peak heights and using the method of Schultz (1964). The precision of these measurements is estimated to be about $\pm 10\%$ of the amount present. Preferentially oriented sample mounts were prepared by dispersing a small amount of sample in distilled water and allowing the dispersion to evaporate on a glass slide. These slides were scanned from 33° to $2^\circ 2\theta$ both before and after saturation with ethylene glycol. Peak heights were measured for those samples that had been treated with ethylene glycol so that relative intensity ratios could be determined. Peaks used were expandable clays ($5.1^\circ 2\theta = 17 \text{ \AA}$), illite ($8.9^\circ 2\theta = 10 \text{ \AA}$), and chlorite ($12.2^\circ 2\theta = 14 \text{ \AA}$). The $<0.1\text{-}\mu\text{m}$ size fractions were K-saturated and X-rayed before and after solvation with ethylene glycol to check for the presence of vermiculite.

Oxygen was extracted from the samples by reacting the samples in vacuum with fluorine at 550°C (Taylor and Epstein, 1962) after first placing the samples in a drybox and then outgassing at 250°C to eliminate interlayer and adsorbed water (Savin and Epstein, 1970a). The extracted O_2 was converted quantitatively to CO_2 and the gas analyzed on a mass spectrometer. Hydroxyl hydrogen was expelled as H_2O by heating the sample up to 1300°C after removing interlayer and adsorbed water by heating under vacuum for about 24 hr at $250\text{-}300^\circ\text{C}$. Hydrogen was formed by reacting the H_2O vapor with uranium (Godfrey, 1962), and the D/H ratio was determined using a mass spectrometer (Friedman, 1953). The δO^{18} values are based on single analyses; from past experience, the standard deviation is probably better than 0.2‰ . Most of the samples were analyzed twice for D/H and the precision of these analyses is $\pm 2\text{‰}$. All of the analyses presented here are in the usual notation and are relative to Standard Mean Ocean Water (SMOW) (Craig, 1961).

RESULTS AND DISCUSSION

The interpretation of the clay mineralogy is similar to that made by Hayes (1973) and will not be discussed at length here. Figure 1 shows "oriented" XRD patterns of the $<0.1\text{-}\mu\text{m}$ fraction of sample G which is representative of the other samples. Briefly, the peak positions shown in Figure 1a (ethylene glycol), Figure 1b (K-saturated), and Figure 1c (K-saturation followed by ethylene glycol) argues for a mixture of illite, chlorite and/or vermiculite, and expandable vermiculite and/or mixed-layer illite/expandable vermiculite.

Figure 2 summarizes the variation of bulk and clay

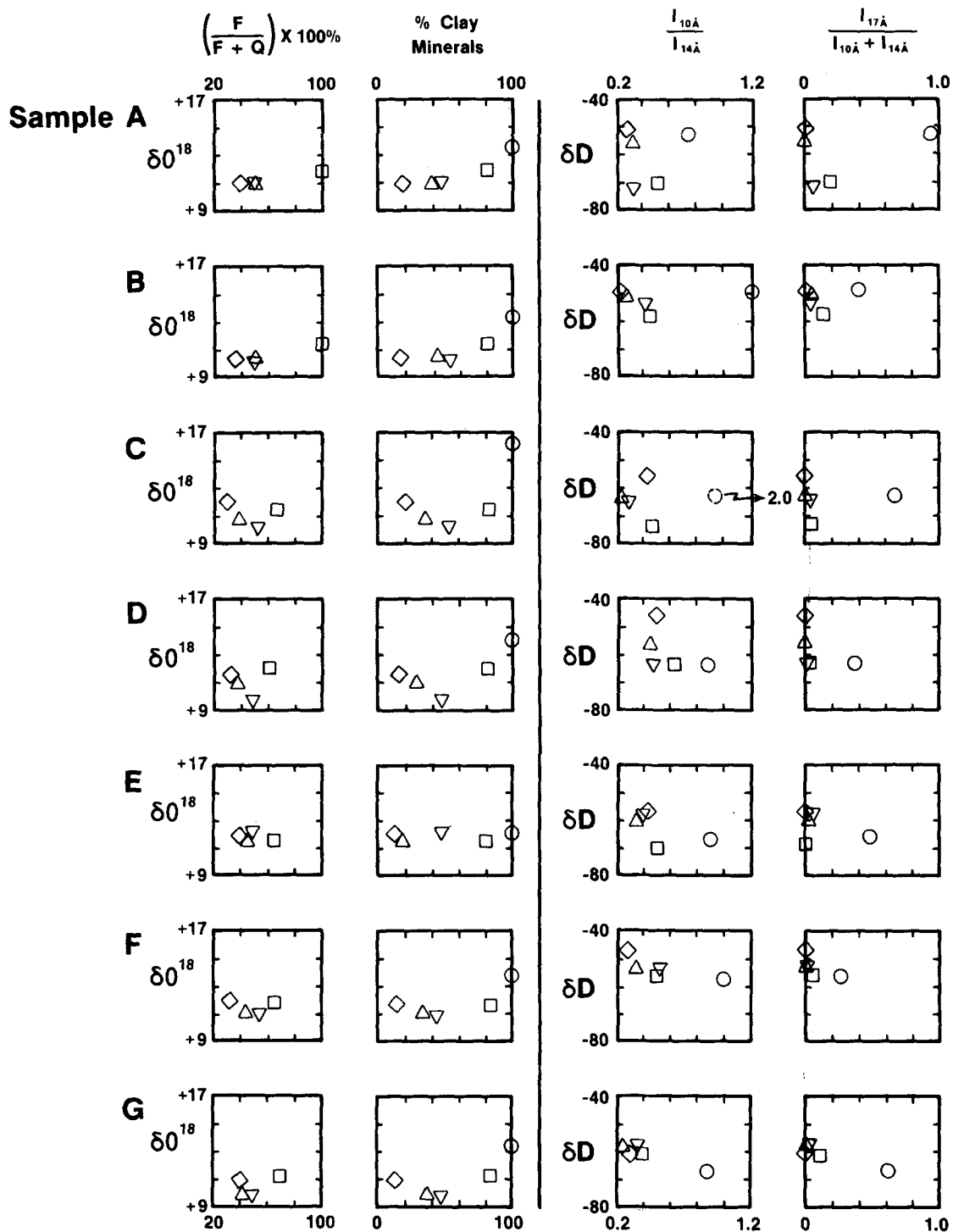


Figure 2. Variations of δO^{18} and δD with size fraction and mineralogy. Size fractions (μm) are: circles, <0.1 ; squares, $0.1-0.5$; inverted triangles, $0.5-1.0$; triangles, $1.0-2.0$, and diamonds, >2.0 . Column 1: δO^{18} vs. $[\text{feldspar}/(\text{feldspar} + \text{quartz})] \times 100\%$. Column 2: δO^{18} vs. % total clay. Column 3: δD vs. (I_{10A}/I_{14A}) . Column 4: δD vs. $[I_{17A}/(I_{10A} + I_{14A})]$.

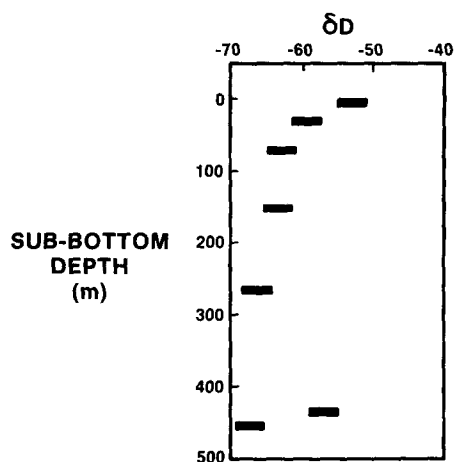


Figure 3. δD of 17-Å vermiculite-rich $<0.1\text{-}\mu\text{m}$ fraction vs. subbottom depth.

mineralogy, δO^{18} , and δD with size fraction. The δO^{18} values are compatible with a mixed assemblage of minerals derived from weathering of igneous (Taylor, 1968), metamorphic (Garlick and Epstein, 1967), and sedimentary (Savin and Epstein, 1970c; Churchman *et al.*, 1976) rocks. Columns one and two show how δO^{18} varies with the amount of feldspar in the feldspar + quartz component and total clay component, respectively. The highest δO^{18} values are from the clay-rich, $<0.1\text{-}\mu\text{m}$ fraction, except for sample E, and the lowest δO^{18} values commonly occur in the feldspar-rich, but clay-poor, $0.5\text{--}1.0\text{-}\mu\text{m}$ size fraction (column one, samples C, D, F, and G). That the δO^{18} values of the coarser size fractions of samples A, B, and E do not vary significantly with size fraction may be due largely to a fairly constant feldspar/quartz ratio in each size fraction, but it may also reflect a greater quantity of sediment derived from an igneous and/or high-grade metamorphic terrain rather than from a sedimentary and/or low-grade metamorphic terrain. The δO^{18} values of coexisting quartz, feldspar, and phyllosilicates weathered from high temperature rocks would form a smaller cluster than δO^{18} values of those weathered from low temperature rocks (Taylor, 1968).

There is no consistent trend of δO^{18} with depth in even the finest size fractions in these samples. Thus, diagenesis is considered to be unimportant in its effect on the δO^{18} of these samples. Yeh and Savin (1976) reported a small, but measurable amount of oxygen isotope exchange between clay minerals and sea water. However, their samples were rich in clay minerals, and the sample that experienced the most exchange (minimum of 27% exchange for the $<0.1\text{-}\mu\text{m}$ size fraction of the 2,725,000 year old SCAN 29P sample) was very smectite-rich. Apparently, smectite is much more susceptible to oxygen isotopic exchange than illite and

Table 1. Core, interval, depth, inferred age, and glacial status of samples studied.

Sample	Core	Section	Interval (cm)	Approximate sub-bottom depth (m)	Age (m.y.) ¹	Glacial status ¹
A	1	1	85–98	1	0.01	Interglacial
B	4	1	120–129	29	0.025	Glacial
C	8	3	50–59	69	0.05	Glacial
D	12	2	100–107	149	0.14	Glacial
E	17	3	85–92	264	0.18	Interglacial
F	22	4	20–40	436	~0.3	Glacial
G	24	3	91–100	454	~0.3	?

¹ Estimated ages and interpretation of glacial events from von Huene and Kulm (1973).

chlorite, which are the dominant clays in the Site 180 samples.

Columns three and four of Figure 2 summarize the variations of clay mineralogy and δD with size fraction and sample depth. The data indicate that 17-Å material (expandable vermiculite and/or mixed-layer illite/expandable vermiculite) increases with decreasing size fraction (column 4). Also, 14-Å material (chlorite and/or non-expandable vermiculite) generally increases with increasing size fraction at the expense of 10-Å material (illite) in samples A, B, F, and G, but remains about the same or changes erratically in samples C, D, and E (column 3). The δD values vary both with size fraction and, for the $<0.1\text{-}\mu\text{m}$ size fraction, with depth. Columns 3 and 4 indicate that, at all depths, δD decreases (becomes more negative) with increasing abundance of 10-Å material, which is usually most abundant in the $0.1\text{--}0.5\text{-}\mu\text{m}$ size fraction and, in the samples from the deeper parts of the hole, δD decreases with increasing amounts of 17-Å material, which is most abundant in the $<0.1\text{-}\mu\text{m}$ size fraction. Thus, the δD of a given size fraction can be thought of as a mixture of 3 components: (1) low δD ($<-60\text{‰}$) 10-Å material; (2) high δD ($>-60\text{‰}$) 14-Å material; and (3) 17-Å material which generally increases from $<-65\text{‰}$ near the bottom of the hole to $>-55\text{‰}$ near the top of the hole. There is no unique interpretation to this data. The δD of a phyllosilicate has been shown to increase with increasing crystallization temperature, increasing δD of water present during crystallization, and decreasing Fe/(Al + Mg) in the octahedral lattice sites (Suzuoki and Epstein, 1976). Thus, the differences in δD values among the coarser size fractions could be attributed to 10-Å material that is more Fe-rich and/or that formed at a lower temperature than the 14-Å material. For samples such as A and D, for which the δD changes significantly with size fraction in the coarser size fractions, but for which the 10 Å/14 Å ratio does not change, the difference in δD values might be due to a high concentration of high-temperature 2M illite polytype in the

coarser size fractions versus a high concentration of low temperature 1Md illite polytype in the finer size fractions. Such variations with grain size in the ratio 1Md/2M of illite polytypes have been documented in shales (Velde and Hower, 1963).

Figure 3 shows that, except for sample E, there is a general decrease with depth of the δD of the $<0.1\text{-}\mu\text{m}$ size fraction. Small variations of δD with depth in other deep-sea samples have been interpreted to be due to isotopic exchange between clay minerals and sea water (Yeh and Epstein, 1978). However, those measurements were made on $<0.1\text{-}\mu\text{m}$, smectite-rich, 2–3 m.y. old sediments, and the δD values showed an increasing trend with depth (Yeh and Epstein, 1978), rather than a decreasing trend with depth as at Site 180. This, plus the facts that sample F does not fit the trend of decreasing δD with increasing depth and that there is no evidence for post-depositional oxygen isotopic exchange in the $<0.1\text{-}\mu\text{m}$ size fraction, suggests that some factor other than post-depositional isotopic exchange may be responsible for the variation of δD with depth in the $<0.1\text{-}\mu\text{m}$ size fraction. The alternative is that the δD variations are inherited from the source area. The average δO^{18} value of +14‰ for the expandable vermiculite-rich, $<0.1\text{-}\mu\text{m}$ fraction dictates via mass balance that the δO^{18} value of the pure expandable vermiculite-rich component would be more positive than +14‰. Such a value is incompatible with an O¹⁸/O¹⁶ ratio inherited entirely from an igneous (Taylor, 1968) or metamorphic (Garlick and Epstein, 1967) ferromagnesian parent mineral and thus suggests an approach toward oxygen isotopic equilibrium at surface temperatures between meteoric waters and the newly-formed vermiculite weathering product. If the oxygen isotopes approached equilibrium in the weathering environment, it is not unlikely that the hydrogen isotopes also would have done so. The degree to which isotopic equilibrium between the vermiculite and meteoric water may have been approached is impossible to evaluate because the δD of the meteoric water is unknown and the hydrogen isotope fractionation between vermiculite (or even smectite, cf. Yeh and Epstein, 1978) and water is poorly known. However, it is possible that the general increase in δD of the $<0.1\text{-}\mu\text{m}$ fraction from about –70‰ to about –50‰ may reflect a climate-induced increase through time in the average δD of precipitation in the source rock areas. A test of this hypothesis would necessitate: (1) acquiring fairly pure 17-Å vermiculitic material, possibly by making size-cuts smaller than $<0.1\text{-}\mu\text{m}$; (2) determining Fe/(Al + Mg) of the material to estimate the extent to which chemical changes affect the δD trends; and (3) determining δD of closely-spaced samples to determine to what extent δD fluctuations correlate with established Pleistocene temperature curves, making the assumption that deposition in the Aleutian Trench occurred not long after weathering and erosion from the source areas.

In summary, δO^{18} and δD values of these sediments are not affected by diagenesis, but they are strongly dependent on the mineralogy, and thus the size fraction, that is analyzed. The δO^{18} and δD values are compatible with a mixed igneous-metamorphic-sedimentary source area, and though this study did not attempt to define the locality of the source area as Slatt and Piper (1974) did using heavy mineral data, the δD data show sufficient variation with size fraction, and thus with mineralogy or environment of formation, to warrant the consideration of δD values as a provenance tool, perhaps as useful as δO^{18} values.

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Резюме—Минералогия, соотношения O^{18}/O^{16} и D/H были определены для пяти фракций ($<0,1$, $0,1-0,5$, $0,5-1,0$, $1,0-2,0$, и $>2,0$ μm) семи образцов, выбранных из 500 м плейстоценовых глубинно-морских осадков, взятых из места реализации Проекта Глубоко-Морского Бурения 180 в Алеутовом Рове. Осадочный период образцов охватывает последние 300 000 лет. Образцы были описаны другими авторами, как континентальный детритус, выветренный из смешанных приогенных, метаморфических и осадочных мест и потом осаждённый в результате ледникового движения и турбидительных течений. Присутствующие минералы это кварц, фельдшпат, иллит, хлорит и/или не расширяющийся вермикулит, а также расширяющийся вермикулит и/или смешанно-слоистый иллит/расширяющийся вермикулит. Относительные количества кварца, фельдшпата и общей глины изменяются с размером частиц, но почти постоянны для разных образцов данного размера частиц. Величины δO^{18} для четырёх грубозернистых фракций находятся в пределе $+9,7-+12\%$ с вариациями, связанными с изменениями в количествах кварц/фельдшпат и глина/(кварц + фельдшпат). Величины δO^{18} для фракции размером $<0,1$ μm , богатой в расширяющийся вермикулит находятся в пределе $+12,1-+16,3\%$. Это указывает на изотопный обмен некоторого количества кислорода между атмосферными водами и материнской породой при температурах поверхности во время образования вермикулита. Величины δO находятся в пределе -46 до -74% с вариациями, связанными с изменениями количества различных присутствующих глинистых минералов. Нет никаких минералогических или изотопных доказательств послеосадительных реакций в грубозернистых фракциях, но основное изменение величины фракции размером $<0,1$ μm , богатой в вермикулит, от около -5% до около -70% с увеличивающейся глубиной может быть вызвано или послеосадительным изотопным обменом, или климатическими изменениями в сухопутной среде выветривания. [E.C.]

Resümee—Es wurden die Mineralogie sowie die O^{18}/O^{16} - und D/H-Verhältnisse in 5 Kornfraktionen ($<0,1$; $0,1-0,5$; $0,5-1,0$; $1,0-2,0$; und $>2,0$ μm) von sieben Proben untersucht, die aus einem 500 m Bohrkern von pleistozänen Tiefseesedimenten genommen wurden, der im Rahmen des Tiefseebohrungsprojektes Site 180 im Aleutenbogen gezogen wurde. Das Ablagerungsalter der Proben umfaßt die letzten 300 000 Jahre; die Proben wurden von anderen Autoren als kontinentaler Verwitterungsschutt gedeutet, der von magmatischen, metamorphen und sedimentären Liefergebieten stammt und dann durch Eisverfrachtung und Suspensionsströme abgelagert wurde. Die vorhandenen Minerale sind Quarz, Feldspat, Illit, Chlorit und/oder nichtquellbarer Vermiculit, sowie quellbarer Vermiculit und/oder Illit/quellbarer Vermiculit-Wechselagerung. Die relativen Mengen an Quarz, Feldspat, und Gesamtton schwanken mit der Kornfraktion, sind aber bei gegebener Korngröße bei den verschiedenen Proben nahezu konstant. Die δO^{18} -Werte von 4 größeren Kornfraktionen reichen von $+9,7$ bis $+12,0\%$ mit Schwankungen, die auf die Veränderungen in den Quarz/Feldspat- und Ton/(Quarz + Feldspat)-Gehalten zurückzuführen sind. Die δO^{18} -Werte der Fraktion $<0,1$ μm , die reich an quellfähigem Vermiculit ist, reichen von $+12,1$ bis $+16,3\%$. Dies deutet darauf hin, daß ein Sauerstoffisotopen-Austausch bei Oberflächentemperaturen zwischen Niederschlagswasser und Ausgangsgestein während der Vermiculit-Bildung stattgefunden hat. Die δD -Werte reichen von -46 bis -74% mit Schwankungen, die auf Veränderungen im Gehalt der verschiedenen Tonminerale zurückzuführen sind. Weder bei der Mineralogie noch bei den Isotopen gibt es in den größeren Fraktionen einen Hinweis auf Reaktionen, die nach der Ablagerung stattgefunden haben. Eine generelle Veränderung des δD -Wertes der Vermiculit-reichen Fraktion $<0,1$ μm von etwa -50% bis etwa -70% mit zunehmender Tiefe kann entweder auf einen Isotopenaustausch nach der Ablagerung oder auf klimatische Schwankungen bei der terrestrischen Verwitterung zurückgeführt werden. [U.W.]

Résumé—La minéralogie, O^{18}/O^{16} et les proportions D/H ont été déterminées dans cinq groupes de taille différente ($<0,1$, $0,1-0,5$, $0,5-1,0$, $1,0-2,0$, et $>2,0 \mu\text{m}$) de sept échantillons pris dans 500 m de profonds sédiments marins pleistocènes tirés du Site 180 du Deep Sea Drilling Project dans la tranchée aleutienne. L'âge de déposition des échantillons couvre les dernières 300 000 années, ces échantillons ont été interprétés par d'autres comme étant des détritus continentaux altérés provenant d'une région-source à caractères mélangés ignée, métamorphique, et sédimentaire et ensuite déposés par transport par glace et par des courants de turbidité. Les minéraux présents sont le quartz, le feldspar, l'illite, la chlorite et/ou la vermiculite non-expansible, et la vermiculite expansible et/ou l'illite à couches mélangées/vermiculite expansible. Les quantités relatives de quartz, feldspar, et argile totale varient avec la taille de la particule, mais sont quasi constantes d'échantillon en échantillon pour une taille de particule donnée. Les valeurs δO^{18} pour les quatre groupes de tailles les plus grosses s'étagent de $+9,7$ à $+12,0\text{‰}$ avec des variations qui peuvent être attribuées à des changements dans les abondances de quartz/feldspar et d'argile/ (quartz + feldspar). Les valeurs δO^{18} du groupe de taille $<0,1-\mu\text{m}$ riche en vermiculite expansible s'étagent de $+12,1$ à $+16,3\text{‰}$, ce qui indique qu'aux températures de surface il y a un échange de l'isotope oxygène entre les eaux météoriques et la roche-mère pendant la formation de la vermiculite. Les valeurs δD s'étagent de -46 à -74‰ , avec des variations qui peuvent être attribuées à des changements dans les quantités des différents minéraux argileux présents. Il n'y a pas d'évidence minéralogique ou isotopique de réactions se passant après la déposition dans les groupes de grosses tailles, mais un changement général de δD dans le groupe de taille $<0,1-\mu\text{m}$ riche en vermiculite de -50‰ à à peu près -70‰ proportionnellement à la profondeur peut être dû soit à un échange isotopique après la déposition, soit à des changements climatiques dans l'environnement d'altération terrestre. [D.J.]