Data report: refined method for calculating percentages of kaolinite and chlorite from X-ray diffraction data, with application to the Nankai margin of southwest Japan¹

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Abstract

Calculating accurate percentages of kaolinite and chlorite in marine sediments using X-ray diffraction (XRD) data is challenging because the minerals have similar d-values. Most approaches use a peak-intensity ratio or fitted-peak-area ratio and assume a linear fit between the ratio and the concentration of each mineral. To test this assumption, we mixed various proportions of kaolinite and chlorite standards and calculated each mineral's individual peak area (PA) at ~3.5 Å along with the total area (TA) of the composite peak. A power function provides the best correlation coefficient for regression curves of the ratio of PA/TA versus the actual mineral concentration (weight percent). The average error using a doubled half-peak area approach is 2.6%. We tested the utility of the method by analyzing data from natural mudstone specimens from Ocean Drilling Program Sites 1177 and 1178 (central and western Nankai Trough). This documentation of temporal changes in concentrations of chlorite and kaolinite provides a basis for improved XRD analyses and interpretations of sediment provenance during the Integrated Ocean Drilling Program's Nankai Trough Seismogenic Zone Experiment.

Introduction

Semiquantitative analysis of clay minerals is commonplace in such fields as sedimentology, paleoceanography, and paleoclimate (e.g., Petschick et al., 1996; Svensson et al., 2000; Fagel et al., 2003; Liu et al., 2003). Most techniques are based on X-ray diffraction (XRD), but accurate quantitative analysis of clays and clay minerals remains a formidable challenge (Brindley, 1980; Reynolds, 1989; Snyder and Bish, 1989; McManus, 1991; Moore and Reynolds, 1997). It is difficult to reproduce with precision the intensities generated by broad reflections of poorly crystalline clay minerals, so researchers typically use values of peak area in combination with sets of weighing factors (Biscaye, 1965; Cook et al., 1975; Heath and Pisias, 1979; Fagel et al., 2003; Underwood et al., 2003). Accuracy improves if the analytical methods include calibration with internal standards, use of single-line reference intensity ratios, and some fairly elaborate sample preparation steps to ensure uniformity of random particle orientations (Środoń et al., 2001; Omotoso et al., 2006). However, those kinds of approaches are too laborious to be practical for large suites of samples, such as those stemming from the Nankai Trough Seismogenic Zone Experiment (NanTroSEIZE).

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In marine geology, the most widely used factors for calculating percentages of smectite, illite, and kaolinite + chlorite were established by Biscaye (1965). Unfortunately, the d-values of kaolinite (001) and chlorite (002) reflections are nearly identical at ~7 Å, as are the kaolinite (002) and chlorite (004) reflections at \sim 3.5 A. This overlap makes it difficult to separate peak intensities or peak areas before calculating their relative abundances. Heating to 550°C or boiling with hydrochloric acid helps to confirm the presence/absence of the two minerals (Nelson and Roy, 1953; Brindley, 1961; Martin Vivaldi and Gallego, 1961), but those steps are time consuming, and comparisons of peaks by subtraction of intensity or area values before and after treatment are flawed because two separate specimens are being analyzed, thereby propagating the error. Consequently, if a specimen contains both kaolinite and chlorite, completing XRD scans before and after the treatment cannot resolve the individual contents of either mineral very accurately.

Biscaye (1964) showed that the ratio of heights of the overlapping 7.16–7.08 Å peaks are approximately the same as the ratio of heights of the double peaks with d-values of 3.58–3.54 Å. Most experts agree that the peaks at ~3.5 Å are better for semiquantitative analyses because of their slightly wider separation (e.g., Petschick et al., 1996; Fagel et al., 2003). The peak-intensity and fitted-peak-area ratios represent two ways to calculate the proportion of each mineral, although the fit between either one of the ratios and the true mineral abundance is nonlinear (Elverhøi and Rønningsland, 1978). Per unit weight, the kaolinite intensity is twice the chlorite intensity, so smaller contents of chlorite are detectable only as a shoulder on the double peak. To create better statistical fits and improve the XRD technique's accuracy, we mixed and analyzed standards of chlorite and kaolinite in known proportions by weight, and we computed two regression curves for the relation between peak-area ratio and weight percent. This report documents those test results and provides a thorough analysis of the error. By reducing the error, we hope to improve the detail and reliability of XRD results during NanTroSEIZE.

Methods

Two groups of standard minerals (C-group and K-group) were mixed: a poorly crystalline kaolinite standard, Clay Minerals Society (CMS) KGa-1, and a variety of chlorite (ripidolite), CMS standard CCa-2. We selected those source clays standards because they are easily accessible to the research community and they have been thoroughly analyzed by other research (e.g., Chipera and Bish, 2001; Vogt et al., 2002). Proportions by weight are similar for the two

groups of mixtures but not identical (Table T1). Prior to blending, each standard was suspended in ~500 mL of distilled water with sodium hexametaphosphate dispersant and disaggregated using an ultrasonic cell disrupter. Particles <2 µm equivalent settling diameter were separated by centrifugation (1000 rpm for 2.4 min; \sim 320× g). The average concentration of each suspension was determined by extracting three aliquots and drying at 75°C to obtain dry weight of clay per unit volume of suspension, corrected for weight of dispersant. The two components were measured by pipette and their volumetric proportions converted to dry weights and weight percentages (Table T1). Oriented aggregates on glass slides were prepared using the filter-peel method and 0.45 µm membranes (Moore and Reynolds, 1997).

Working with an older analog diffractometer, Biscaye (1964) advocated the use of slow scanning rates (0.2°2θ/m; 0.01°2θ step) to create higher intensity and reproducibility of the double peaks at ~3.5 Å. We achieved optimal results with a faster scan (1°2θ/m; 0.01°2θ step) from 23° to 28°2θ. The instrument is a Scintag Pad V X-ray diffractometer with CuKα radiation (1.54 Å) and a Ni filter, set to 40 kV and 30 mA. Slits were 0.5 mm (divergence) and 0.2 mm (receiving). We processed the digital data using MacDiff software (version 4.2.5) to establish a baseline of intensity, provide smooth counts, and correct for offset of peak positions caused by misalignment of the instrument's detector and small differences in the orientation of slides within the sample changer.

The uncorrected position of the kaolinite peak of interest ranges from 25.09° to 25.19°20, and the uncorrected chlorite peak ranges from 25.33° to 25.48°20. The peak positions were corrected to either kaolinite (002) at ~24.8°20 (d-value = 3.58 Å) or chlorite (004)at $\sim 25.1^{\circ}2\theta$ (d-value = 3.54 Å) (Fig. F1). The corrected peak profiles were used to locate the centers of peaks, from which the maximum intensities were determined. When both crests are resolved, angular separation between the two overlapping peaks averages $0.27^{\circ}2\theta$ (Fig. F1), which is consistent with synthetic diffractograms (e.g., Moore and Reynolds, 1997). Therefore, we always assumed that the crest of the subsidiary peak is separated from the crest of the dominant peak by 0.27°20 when picking its point of maximum intensity on the shoulder of the composite (Fig. F1). In addition, we modeled the peaks using the Pearson II peak-fitting function of MacDiff on the corrected diffractogram to obtain the fitted area of those two peaks (Figs. F1, F2). The least residuum reduction was set to 0.001, and the largest loop number of refinement was set at 100.

On all the diffractograms generated for the standard mixtures (Fig. F3), we found that one side of the dominant peak is not distorted by interference from



the minor peak. Using only the undistorted side makes it easy to compute the dominant peak's total area by multiplying its half-peak area by $2 \times$ (Fig. F2). One important assumption here is peak symmetry, which does not necessarily hold true for natural mixtures of clay minerals (Moore and Reynolds, 1997). Nevertheless, the ratio of the dominant peak area (PA) to the total area (TA) of the composite peak should correlate with each mineral's abundance. For mixtures in which both peak crests are distinct, each mineral's undistorted half-peak area was measured and doubled to compute both of the respective PA values.

Results

Tabulation of the results for standard mineral mixtures appears in Table T1. Regression analysis shows that a power function yields the best correlation coefficient (r) for the ratio of PA to TA versus the known abundance of the mineral (Fig. F4). Linear regression, in comparison, shifts the intercept away from the origin, which is nonsensical. Two equations were constructed. For kaolinite-dominant specimens (kaolinite wt% = $100 \times (PA/TA)^{1.613}$, where r = 0.9870) and for chlorite-dominant specimens (chlorite wt% = $100 \times (PA/TA)^{1.433}$, where r = 0.9974). This pair of equations allows researchers a choice depending on which of the two minerals is dominant in the natural mix.

Table T1 also compares the calculated mineral abundances using three approaches (Fig. F5). The doubled half-peak area method yielded the best agreement between the calculated abundance and the actual abundance (Fig. F5). The maximum error is 7.7%, and the average error is only 2.6%. Using the intensity-ratio method, the average error increases to 7.4%, but those results become more accurate with well-balanced proportions (chlorite to kaolinite ratio close to 50:50). Using the fitted-area-ratio method, the average error is 5.1%, and those results are more reliable when kaolinite contents are between 20% and 80%. The maximum errors increase to 17.6% and 12.3%, respectively, using intensity-ratio and fittedarea-ratio (Table T1). Thus, for use with a broad range of potential mixtures in natural samples, the doubled half-peak area method is judged to be the most accurate.

Quantitative accuracy of XRD results is known to be affected by a long list of natural variables and laboratory artifacts, including the type of diffractometer, sample disaggregation technique, chemical pretreatment, particle size separation, size and shape distribution within a selected size fraction, chemical composition of clay minerals (e.g., content of iron in chlorite), structural ordering and crystallinity, the degree of

preferred orientation of crystallites on the scanned surface, and peak-fitting algorithms (Reynolds, 1989; Moore and Reynolds, 1997; Ottner et al., 2000; Środoń, 2002). Besides those variables, the thickness of aggregates on a glass slide will have some effect on the peak intensities and peak areas. This inconsistency in thickness helps explain the scatter of our results across similar ranges weight percentages for a given mineral (Fig. F3). Finally, we assumed ideal Pearson II peak shapes when computing the halfpeak areas, but the instrumental peak shape of a Bragg-Brentano diffractometer is usually somewhat asymmetric.

Unquestionably, the two CMS source clays KGa-1 (kaolinite) and CCa-2 (chlorite) are not representative of all possible clay mineral assemblages in natural marine sediments. The refinement probably could be improved even more by analyzing additional mixtures with a broader range of chemical compositions and crystallinities, particularly for chlorite, but then extra attention would be required to match by iteration each natural assemblage with its best-fit standard mixture. Errors can also increase in a more unpredictable way if the natural sediment contains minerals in addition to kaolinite and chlorite whose reflections interfere with the 3.5 Å double peaks (Biscaye, 1964). Although imperfect, we have demonstrated that the doubled half-peak area approach is consistently more accurate than estimates from peak-intensity ratio and fitted-peak-area ratio.

Application of the method

The goal of refining the XRD methods for chlorite and kaolinite was to improve the detail of large data sets generated for NanTroSEIZE, without adding excessive amounts of time and complexity to sample preparation and data reduction. To test the utility of the doubled half-peak area approach, we took advantage of previously unpublished XRD data from ODP Sites 1177 and 1178 for which the reflections at ~3.55 Å had been recorded (Table T2). Kaolinite and chlorite contents for those specimens (in central and western Nankai Trough) were initially reported as one undifferentiated value, ranging from 0% to 32% of the clay-size fraction (Steurer and Underwood, 2003; Underwood and Steurer, 2003). Those authors, as well as Underwood et al. (1993), using samples from ODP Site 808, treated a small number of representative samples with acid to resolve how much kaolinite contributes to the composite 7 Å peak area. Results from Sites 808 and 1173 show that kaolinite makes up <20%, and averages ~14%, of the chlorite-kaolinite composite (based on acid-treatment calculations). Three values from Site 1177 range from 10% to 60% kaolinite in the chlorite-kaolinite composite. Only



one sample (190-1177A-46R-1, 120 cm) was analyzed by both methods and yielded a value of 60% for the ratio of treated to untreated peak area, but it is worth noting that the total chlorite + kaolinite in this claysize fraction is only 4% (Steurer and Underwood, 2003). For the same sample, we obtained a 50:50 ratio of kaolinite to chlorite using the doubled halfpeak area method (Table T2).

One justification for expanding the data set for the previously analyzed ODP samples from Nankai Trough is to ascertain whether or not the contents of kaolinite and chlorite changed systematically with time or stratigraphic position within the Shikoku Basin and Nankai trench wedge. Any such basinwide changes in the central and western Nankai Trough might have some bearing on interpretations of clay provenance and sediment dispersal for coeval strata within the NanTroSEIZE transect. To begin this comparison, we picked paleomagnetic polarity and calcareous nannofossil data from Sites 1177 and 1178 (Shipboard Scientific Party, 2001a, 2001b) and constructed composite age-depth models. Figure F6 shows how relative abundances of kaolinite and chlorite in the clay-size assemblage change as a function of time, as constrained by the age-depth models.

Most values of kaolinite are <10% of the total claysize fraction, and chlorite content is typically <20%. Strata at Site 1177 cover an age range of 1.6 to 18.6 Ma, so those samples provide a better view of temporal trends. At Site 1177, the kaolinite and chlorite contents are significantly lower for strata that are older than ~11 Ma, averaging only ~2% of the claysize fraction. This depletion occurs is because the supply of smectite was much higher during the early to middle Miocene (Steurer and Underwood, 2003). The ratio of chlorite to kaolinite is also higher in these older strata. At Site 1178, chlorite values don't show any systematic stratigraphic trend, although this scatter of results over time is complicated by occurrences of several thrust faults and an unconformity. Over comparable ranges of time at the two sites, the relative abundance of kaolinite gradually decreases by roughly 8 wt% with age.

Conclusions

Tests of standard mineral mixtures show that best-fit regressions are nonlinear for plots of both peak-intensity ratio and fitted-area ratio versus the concentration ratio of kaolinite and chlorite. Using the peak-intensity ratio method, accuracy improves if the chlorite to kaolinite ratio is close to 50:50. Using the fitted-area-ratio method, the results are more reliable when kaolinite contents are between 20% and 80%. Our refined approach uses a doubled peak-area ratio.

This reduces the error to a maximum of 7.7% and an average of 2.6% because it avoids measuring minor peaks obscured by the dominant peak's shoulder. Either of two equations can be used, depending on which mineral is dominant. For kaolinite-dominant specimens, kaolinite wt% = $100 \times (PA/TA)^{1.613}$. For chlorite-dominant specimens, chlorite wt% = $100 \times (PA/TA)^{1.433}$.

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Figure F1. Example of correction of X-ray diffractogram for a typical kaolinite and chlorite standard mixture. Kaolinite (002) peak is gray. The first step moves the original chlorite (004) peak crest (green) to $25.1^{\circ}2\theta$ (red). Separation between kaolinite (002) and chlorite (004) is fixed at $0.27^{\circ}2\theta$, after which the obscured kaolinite (002) peak intensity can be picked. Both peaks are then modeled using the Pearson II peak fitting function.

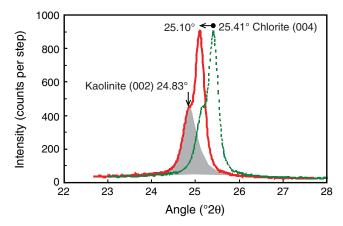
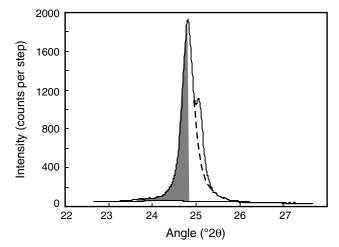




Figure F2. X-ray diffractogram of a typical kaolinite and chlorite standard mixture. The regions highlighted in gray show how to measure the half peak area and the total peak area. The dashed line helps outline the dominant peak.



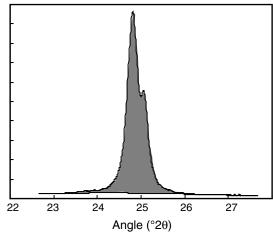


Figure F3. X-ray diffractograms of all kaolinite and chlorite standard mixtures used in this study. Numbers refer to known mineral percentages by weight. (Continued on next page.)

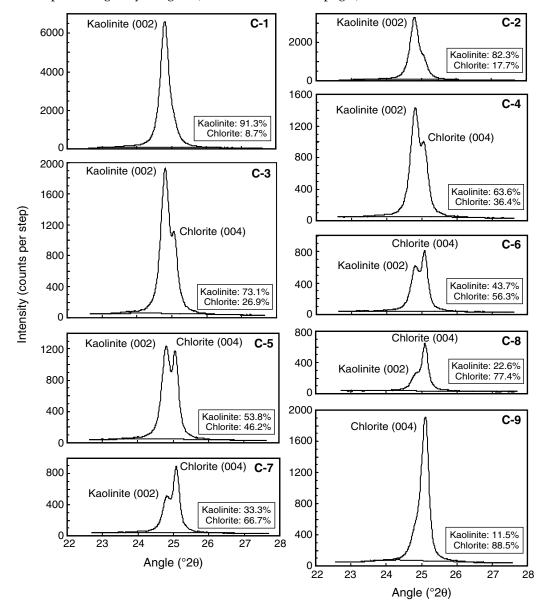


Figure F3 (continued).

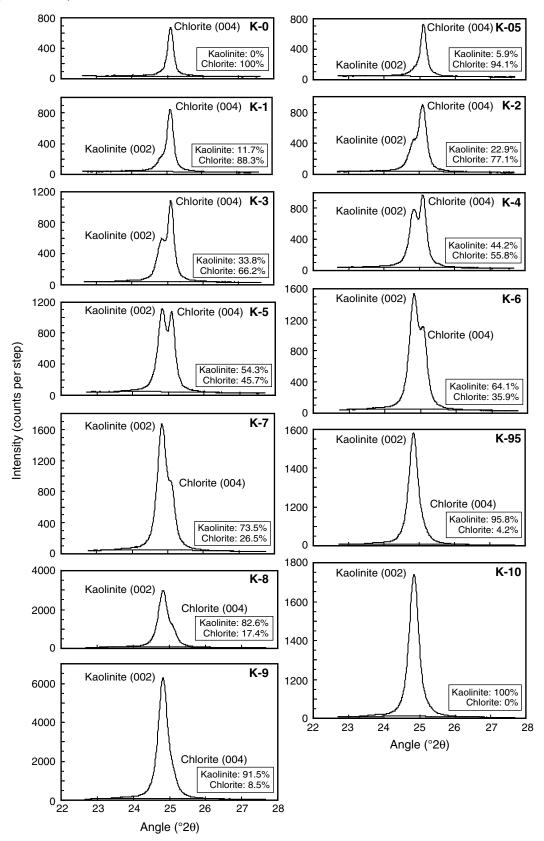




Figure F4. Plots of kaolinite and chlorite abundances versus peak-area ratios. The solid curves represent best fit power-law regression. Dashed lines represent linear regressions. r =correlation coefficient.

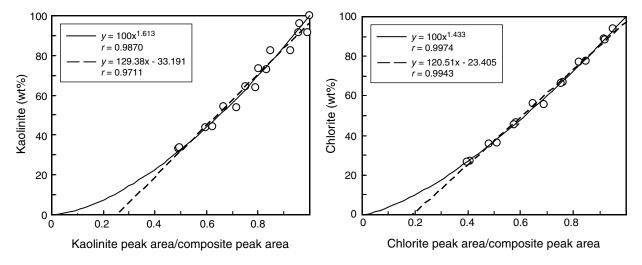




Figure F5. Plots of comparison of errors (known weight percent vs. calculated weight percent) from three different methods. Errors are consistently smaller using half-peak areas.

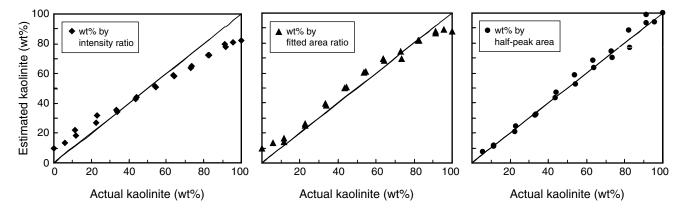




Figure F6. Plots of temporal changes in relative abundances of chlorite and kaolinite within the clay-size fraction and abundance ratio of chlorite to kaolinite in mud and mudstone from ODP Sites 1177 and 1178. Age control from Shipboard Scientific Party (2001a, 2001b).

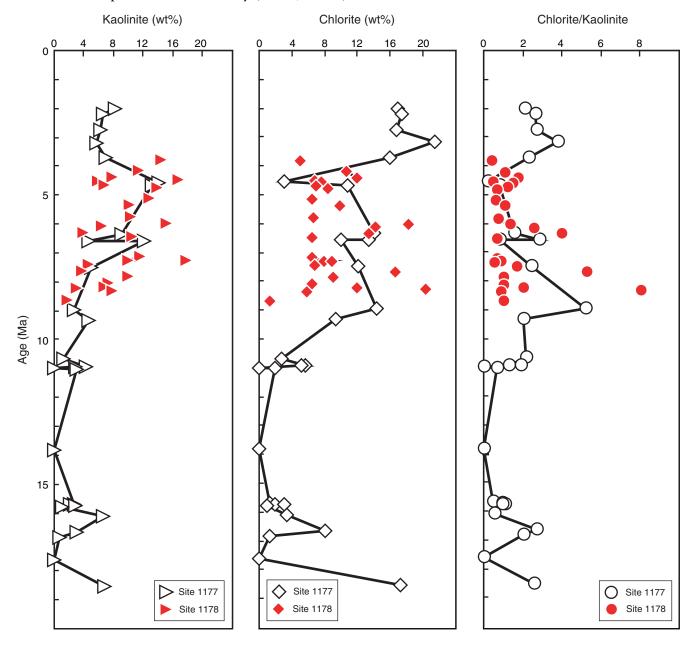


Table T1. X-ray diffraction analysis of standard mixtures of kaolinite and chlorite.

Mixture	Actual abundance (wt%)		Peak intensity (counts/step)		Fitted peak (total count)		Doubled half-peak and composite peak area (total count)			Calculated kaolinite abundances and errors (wt%)					
	Kaolinite	Chlorite	Kaolinite (002)	Chlorite (004)	Kaolinite (002)	Chlorite (004)	Kaolinite (002)	Chlorite (004)	Kaolinite (002) + chlorite (004)	Intensity ratio	Error- intensity ratio	Fitted-area ratio	Error-fitted area ratio	Doubled half- peak area	Error–half- peak area
C-1	91.3	8.7	6,515	1,682	225,627	31,479	127,691	NA	256,933	79.5	11.8	87.8	3.5	99.0	7.7
C-2	82.3	17.7	3,215	1,220	110,471	24,640	62,721	NA	135,634	72.5	9.8	81.8	0.6	88.2	5.8
C-3	73.1	26.9	1,870	1,057	63,138	21,927	35,892	17,663	86,096	63.9	9.2	74.2	1.1	74.6	1.5
C-4	63.6	36.4	1,379	952	47,991	21,213	27,800	18,009	70,457	59.2	4.4	69.3	5.7	68.3	4.7
C-5	53.8	46.2	1,182	1,122	39,371	25,531	23,670	19,264	65,868	51.3	2.5	60.7	6.9	58.7	4.9
C-6	43.7	56.3	577	766	18,730	18,531	11,262	12,259	37,766	43.0	0.7	50.3	6.6	43.4	0.3
C-7	33.3	66.7	467	849	13,643	20,969	8,598	13,289	34,942	35.5	2.2	39.4	6.1	31.9	1.4
C-8	22.6	77.4	223	607	5,485	16,615	NA	9,332	21,961	26.9	4.3	24.8	2.3	20.8	1.8
C-9	11.5	88.5	496	1,761	8,782	54,812	NA	29,134	63,600	22.0	10.5	13.8	2.4	11.8	0.3
K-0	0.0	100.0	69	640	1,673	15,958	NA	8,855	17,143	9.7	9.7	9.5	9.5	-4.8	4.8
K-05	5.9	94.1	107	676	2,713	17,159	NA	9,367	19,723	13.7	7.8	13.7	7.8	7.1	1.2
K-1	11.7	88.3	183	811	4,411	22,114	NA	12,329	26,786	18.4	6.7	16.6	4.9	11.2	0.5
K-2	22.9	77.1	402	868	9,908	27,539	NA	15,310	37,204	31.7	8.7	26.5	3.5	24.4	1.4
K-3	33.8	66.2	551	1,039	16,064	25,867	10,638	16,084	42,769	34.7	0.9	38.3	4.5	32.4	1.4
K-4	44.2	55.8	737	930	23,765	23,039	14,846	16,401	47,516	44.2	0.0	50.8	6.5	46.8	2.6
K-5	54.3	45.7	1,063	1,037	36,160	23,057	20,057	17,279	59,999	50.6	3.7	61.1	6.7	52.2	2.1
K-6	64.1	35.9	1,487	1,065	51,487	24,232	28,895	18,484	76,850	58.3	5.8	68.0	3.9	63.1	1.0
K-7	73.5	26.5	1,620	870	53,500	23,317	30,854	15,270	76,814	65.1	8.5	69.6	3.9	70.2	3.3
K-8	82.6	17.4	2,914	1,106	102,934	22,973	53,545	NA	126,280	72.5	10.2	81.8	0.9	76.7	6.0
K-9	91.5	8.5	6,200	1,731	220,796	34,478	122,081	NA	255,051	78.2	13.3	86.5	5.0	93.2	1.7
K-95	95.8	4.2	5,714	1,358	203,069	25,808	110,104	NA	229,221	80.8	15.0	88.7	7.0	93.7	2.0
K-10	100.0	0.0	7,270	1,552	259,775	36,568	147,478	NA	295,334	82.4	17.6	87.7	12.3	99.8	0.2
										Average:	7.4	•	5.1	_	2.6

NA = Not applicable.



Table T2. X-ray diffraction analysis showing kaolinite and chlorite content in mud and mudstone ($<2~\mu m$ fraction) from ODP Sites 1177 and 1178.

			Abunda	ince (wt%)		Peak a	area (total c	_		
Hole, core, section, interval (cm)	Depth (mbsf)	Smectite	Illite	Chlorite + kaolinite	Quartz	Half kaolinite	Half chlorite	Total area	Relative abur Kaolinite	ndance (wt%) Chlorite
100										
190-	202.52	22	26	25	7	1 420	N1N 4	£770	0	17
1177A-1R-2, 82	302.52	32	36	25	7	1439	NM 1071	5778	8	17
1177A-2R-2, 111	311.81	22	33	24	21	NM	1071	2678	7	17
1177A-5R-5, 123	343.83	30	38	23	9	NM	1858	4636	6	17
1177A-8R-2, 30	368.80	24	39	27	10	NM	1695	3990	6	21
1177A-10R-2, 118	388.79	31	35	23	10	NM	1707	4399	7	16
1177A-11R-CC, 10	403.75	40	39	17	3	2712	NM	6120	14	3
1177A-12R-1, 44	405.74	30	30	24	16	NM	847	2945	13	11
1177A-16R-3, 119	448.09	34	33	23	10	NM	1956	5533	9	14
1177A-17R-1, 118	454.68	41	35	18	5	NM	2096	5174	5	13
1177A-17R-2, 26	455.26	36	35	22	7	NM	828	2885	12	10
1177A-20R-1, 104	483.44	29	32	17	22	616	NM	2650	5	12
1177A-24R-5, 128	527.68	46	29	17	8	NM	1723	3893	3	14
1177A-25R-6, 105	538.95	44	35	14	7	1197	NM	4731	5	9
1177A-30R-2, 30	580.30	65	20	4	11	NM	950	2477	1	3
1177A-30R-7, 29	587.79	54	26	10	10	NM	999	2977	4	6
1177A-30R-7, 35	587.85	59	24	8	8	NM	1175	3169	3	5
1177A-31R-2, 57	590.27	87	9	0	4	NM	520	1855	0	0
1177A-31R-CC, 8	591.10	70	18	5	8	NM	668	2534	3	2
1177A-40R-1, 115	675.95	75	19	0	6	1015	NM	2782	0	0
1177A-46R-1, 18	732.68	69	19	4	8	995	NM	2513	3	1
1177A-46R-1, 120	733.70	66	19	4	12	627	NM	1898	2	2
1177A-46R-2, 122	735.22	64	15	6	15	687	NM	2161	3	3
1177A-46R-3, 9	735.59	71	20	2	8	NM	703	2336	1	1
1177A-47R-3, 101	746.11	54	21	10	15	NM	702	2962	7	3
1177A-49R-1, 61	761.91	41	30	11	18	320	NM	1439	3	8
1177A-49R-5, 72	768.02	65	22	2	12	NM	255	675	1	1
1177A-52R-2, 56	792.36	91	9	0	0	340	NM	999	0	0
1177A-55R-1, 92	820.12	27	35	24	13	NM	1821	4583	7	17
1178A-9X-5, 129	72.03	50	30	18	2	1559	NM	3840	13	5
1178A-13X-4, 132	109.82	45	27	21	7	NM	1676	5340	10	11
1178A-15X-4, 130	129.00	45	26	19	10	NM	1822	5004	7	12
1178A-16X-4, 128	138.48	47	28	22	4	2526	NM	6400	15	7
1178A-17X-2, 125	144.95	59	21	13	6	1756	NM	6182	5	8
1178A-18X-4, 130	156.46	62	24	13	2	NM	1827	5604	6	7
1178A-19X-3, 125	165.65	43	28	21	8	1762	NM	4875	12	9
1178A-23X-2, 117	201.87	42	18	18	22	NM	568	2283	11	7
1178A-25X-2, 117 1178A-25X-2, 118	221.05	52	24	19	5	1742	NM	5527	9	10
1178A-29X-3, 122	260.93	61	18	16	5	1742	NM	4906	9	7
1178A-23X-3, 122 1178A-31X-4, 116	281.86	27	33	32	8	NM	2405	7073	14	18
1178A-31X-4, 110 1178A-43X-1, 121	392.81	55	22	17	6	2300	NM	6261	10	7
·	404.47	46	30	17	7		1603	5384	9	8
1178B-3R-3, 147 1178A-44X-5, 117		44	21	25	9	NM 21.42	NM		16	9
•	407.69	59	26	23 11	4	2142	2099	5660		7
1178B-5R-1, 114	420.44					NM		5805	4	
1178B-8R-5, 147	456.37	46	27	18	10	NM	595	1915	9	9 12
1178B-12R-4, 112	492.32	35	38	18	9 12	NM	2115	5586	6	12
1178B-13R-2, 80	498.70	38	26	23	13	NM	2886	6265	3	20
1178B-14R-1, 94	506.94	76 60	15	3	6	NM	1130	3648	1	2
1178B-15R-5, 103	522.63	69	16	13	2	NM	2066	7091	7	6
1178B-17R-1, 105	535.85	62	23	13	2	NM	1890	6109	6	7
1178B-19R-2, 144	556.94	44	17	20	20	NM	1848	4175	3	17
1178B-25R-2, 98	614.18	53	19	16	11	NM	1418	5248	9	7
1178B-26R-1, 116	622.56	46	32	17	5	NM	2376	5553	3	14
1178B-27R-1,98	631.98	35	35	20	10	NM	2101	5284	6	14

Values within clay-size fraction are from Steurer and Underwood (2003) and Underwood and Steurer (2003) and were calculated using singular value decomposition normalization factors of Underwood et al. (2003). NM = not measured.

