

QUANTITATIVE ESTIMATION OF KAOLINITE IN SEDIMENTS BY DIFFERENTIAL INFRARED SPECTROSCOPY

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Abstract—An infrared method has been developed for estimating kaolinite in sediments. Hydroxyl stretching bands of kaolinite in sediments can be recorded by using a differential technique which eliminates the overlapping owing to other mineralic constituents present. By adding known amounts of an appropriate standard to the sample and by measuring intensities of the OH bands before and after the addition it is possible to calculate the proportion of kaolinite in the test sample. The choice of the added standard is made from characteristic features of the hydroxyl stretching bands.

Key Words—Hydroxyl, Kaolinite, Sediments.

INTRODUCTION

Several physical methods are currently used for the quantitative estimation of kaolinite in soils and in sediments, i.e. X-ray powder diffraction, differential thermal analysis (DTA) and thermogravimetric analysis (TGA).

X-ray powder diffraction is an excellent technique for clay mineral identification but it has a limited success when quantification is needed. Since the estimation is based upon the 001 diffraction intensities, the measurements are very sensitive to the crystallinity and particle size of the specimen. It was shown by van der Marel (1960) that the integrated 001 reflection intensity decreased as the surface area of kaolinite increased. Also, Brindley and Kurtosy (1961) emphasized the considerable importance of the sample orientation upon the X-ray intensity measurements. Thus it is critical to refer to a standard having similar characteristics as the test sample for obtaining the correct estimation. Quantitative estimation by X-ray powder diffraction was found to be difficult when kaolinite coexisted along with non-crystalline minerals, i.e. in weathered volcanic ash, because the non-crystalline constituents tend to level off the intensities of the X-ray reflections. Thermal methods, i.e. DTA and TGA are based upon the dehydroxylation reaction on heating kaolinite up to 700°C. The measurement is not affected by the sample orientation; however, the intensity of the endothermal DTA peak (550–700°C) also depends on the crystallinity and particle size of the sample (van der Marel, 1960). Other clay minerals may also dehydrate or undergo some transformations in the same temperature range (Grim, 1968) and there is not always a clear-cut distinction between the water loss due to kaolinite and the other clay constituents.

Current methods are most successfully used when

the proportion of kaolinite in the sediment is large and when the specimen is well crystallized. Scarcity of kaolinite in the test sample and poor crystallinity complicate the choice of a representative standard. In these circumstances a reliable estimation should be based on a fundamental property of kaolinite which can be quantified readily even at low content and in the presence of non-crystalline material, in this connection, the intensity of the hydroxyl stretching band of kaolinite seems to be an adequate parameter to measure because it depends primarily upon the hydroxyl content. A quantitative interpretation of OH stretching bands of solids has been proposed by Rouxhet (1969) who proposed a way to compute the absorption coefficient (K) for the OH bands. The absorption coefficient is proportional to the OH band intensity and can be used to characterize and to compare OH stretching bands of different minerals within the same family (Rouxhet, 1970; Rousseaux et al., 1972).

The hydroxyl stretching spectrum of kaolinite is made up of four discrete bands. The two high-frequency bands (3695 and 3670 cm^{-1} , respectively) correspond to inner-surface hydroxyls which are perpendicular to the ab plane. The medium-frequency band at 3650 cm^{-1} also corresponds to an inner-surface hydroxyl which is nearly parallel to the sheet. Inner hydroxyls absorb at the lower frequency (3620 cm^{-1}). In this study an attempt was made to estimate small proportions of kaolinite in sediments by infrared spectroscopy. The measurement was based on the intensity of the OH stretching bands and the suggested method allowed for the determination in the presence of interfering mineralic components.

MATERIALS

Pure kaolinite specimens and sediment samples are identified in Table 1. P-designated samples were provided and previously studied by Parker (1969). Weathered-andesitic rock derived kaolinites 69–5 and 61–1 were described by Warkentin (1972). The six sediments

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Table 1. Origin of samples, particle size and mineral components identified in the clay fraction*.

Laboratory number or name	Origin	Particle size (μm)	Minerals in clay fraction†
1. Fireclay type kaolinite			
P2‡	Dorset Ball Clay	0.5–1	K
P5	S. Devon Ball Clay	0.25–0.5	K
P10	S. Devon Ball Clay	0.5–1	K
Yankee Ball Clay	United Mines Corp. U.S.A.	0.2–2	K,I
2. Georgia Kaolin type			
P14	Georgia, U.S.A.	0.5–1	K
P15	Venezuela, Bolivar state	0.5–1	K
hydrite UF	Commercially available	< 0.2	K
hydrite 121	available	10–0.2	K
Rogers	kaolinite	< 2.0	K
Peerless	from U.S.A.	< 2.0	K
3. China clay type			
P21	Cornwall	0.5–1	K
P25	Synthetic, hydrothermal	< 0.5	K
P28	Cornwall	0.5–1	K
Kaolinite Bretonne	Bretagne, France	2–1	K
4. Weathered andesitic rock derived kaolinites			
69–5	Nevis, West Indies	< 2.0	K
61–1	Linstead, Jamaica	< 2.0	K
5. Soil and sediment samples			
1	Antigua, West Indies	0.2–2	K,Mt
4	Antigua, West Indies	0.2–2	K,Mt
9	Antigua, West Indies	0.2–2	K,Mt
6	Western Alberta, Canada	0.2–2	K,I,M-L,Q
N1	Hokkaido, Japan	< 2.0	K,Ch,M,Q,A
C12	Dominica, West Indies	Whole soil	K,Q,C,G,A

* Crystalline components identified by X-ray powder diffraction. Non-crystalline component (allophane) identified by selective dissolution (Rousseaux and Warkentin, 1976).

† Abbreviation of mineral names: K—kaolinite; I—illite; M—mica-type; M-L—mixed-layer; Mt—Montmorillonite; Ch—chlorite; Q—quartz; C—cristoballite; G—gibbsite; A—allophane.

‡ P—designated samples were provided and previously studied by Parker (1969).

contained other minerals associated with kaolinite: samples 1, 4, 6 and 9 had crystalline minerals whereas samples N1 and C12 were predominantly allophane soils derived from weathered volcanic ash.

The clay fraction of all samples except the hydrite kaolinites was separated by centrifugation after dispersion at pH 9.5 using Na_2CO_3 . Sample N1 was dispersed at pH 3 in dilute HCl. The clay fractions recovered were oven-dried and stored in a desiccator over CaCl_2 . The different particle size fractions of hydrite kaolinites were provided by the Georgia Kaolin Co., and no treatment was made on these samples prior to infrared measurements.

METHODS

Infrared measurements

Infrared spectra were recorded with a model 257 Perkin-Elmer grating spectrophotometer. Spectra were

obtained from 1% KBr discs weighing exactly 120 mg; a pure KBr disc also was set in the reference beam. Interference from molecular water absorbed on the sample was eliminated by heating the KBr disc at 130°C under vacuum in a Pyrex cell fitted with CaF_2 windows. Optimum instrument control settings were selected for recording the shape of the hydroxyl bands with an error less than 1%.

In sediments, hydroxyl bands of crystalline clays other than kaolinite may overlap the kaolinite pattern. The pattern was then recorded by using a differential technique which consisted of fitting the reference beam with a heated sample whereas an unheated sample was set in the sample beam. By heating the test sample overnight at 550°C kaolinite was selectively dehydroxylated, but other crystalline clays, i.e. montmorillonite, illite and mica-like clays are much less affected at this temperature. Thus the differential spectrum recorded gave the kaolinite hydroxyl stretching bands alone.

Table 2. Constitutional water content, hydroxyl absorption coefficient, bulk absorption coefficient per hydroxyl and ratio of intensity of HF band to intensity of MF band.

Laboratory number or name	Constitutional water content (%)	K (cm ⁻¹)	κ (cm ⁻¹)	$R = \frac{\text{abs. HF}}{\text{abs. MF}}$
P2*	7.8	287	33	1.0
P5	5.8	241	37	0.8
P10	7.7	337	39	1.33
Yankee Ball Clay	8.4	501	53	1.38
P14	12.0	591	44	1.66
P15	11.5	506	40	1.58
Hydrite UF	13.2	672	46	1.79
Hydrite 121	13.3	586	40	1.76
Rogers	13.1	633	44	2.0
Peerless	13.4	630	42	1.65
P21	11.4	508	40	1.72
P25	11.9	596	45	2.0
P28	11.1	574	56	2.15
Kaolinite Bretonne	12.7	480	34	1.76
69-5	11.4	628	50	1.38
61-1	13.1	690	47	1.62

* Measurements on P-designated samples were done by C. Noël, Catholic University of Louvain.

Spectra recorded in transmittance were redrawn in absorbance and integrated absorbances were obtained by measuring the band area.

KBr disc preparation

It was found to be essential to mix sample and KBr homogeneously in order to obtain the true shape of the kaolinite OH bands. KBr was added to the sample in three successive steps and mixing was done in an agate mortar after each addition. Grinding was very mild to prevent abrasion which could have altered the kaolinite sample (Miller and Oulton, 1972). Similarly, the mineral mixtures were prepared by adding 10–30% of kaolinite standard to the test sample prior to dilution in KBr. The KBr used was infrared grade powdered material which was dried at 105°C overnight and then stored in a desiccator over CaCl₂. KBr discs were obtained by pressing the powdered mixture in a Perkin–Elmer die under a pressure of about 875 kg/cm².

The method of known additions

By adding a known weight of kaolinite standard to a sample containing some kaolinite and measuring the OH bands integrated absorbance before and after addition, A_0 and A_i , respectively, one can determine the weight proportion of kaolinite in the test sample.

The integrated absorbance of the OH stretching bands of kaolinite does follow the Lambert–Beer law of adsorption (van der Marel and Beutelspacher, 1976).

Thus one can write

$$A_0 = P_s W_s K_s, \quad (1)$$

where P_s is the kaolinite proportion in the sample, W_s

the sample concentration (%) in the KBr mixture and K_s is a factor related to the hydroxyl absorption coefficient of kaolinite.

Similarly, after addition of a known weight of kaolinite standard to the test sample, the integrated absorbance (A_i) of the kaolinite OH bands was:

$$A_i = (P_s W_s K_s) + (P_t W_t K_t), \quad (2)$$

where P_t is the kaolinite proportion in the kaolinite standard (by definition $P_t = 1$), W_t is the kaolinite concentration (%) added to the test sample and K_t is a factor related to the OH adsorption coefficient of the kaolinite standard.

Assuming that the kaolinite sample and kaolinite standard have the same adsorption coefficient, that is $K_s = K_t$ one has

$$\frac{A_i}{A_0} = 1 + \frac{W_t}{W_s P_s}. \quad (3)$$

By making several standard additions one can measure the A_i and a plot of A_i/A_0 vs. W_t/W_s gives a straight line whose slope is $1/P_s$, the inverse of the kaolinite proportion in the test sample.

Thermogravimetric analysis

Constitutional water content of kaolinite was determined on oven-dried samples by measuring the loss on ignition after heating at 570°C overnight. Thermogravimetric curves of oven-dried sediments were obtained by a static TGA method. The temperature was raised from 110 to 600°C in increments of 50°C. Samples were left in the furnace for 1 day during each step and stored in a desiccator for 2 hr before weighing.

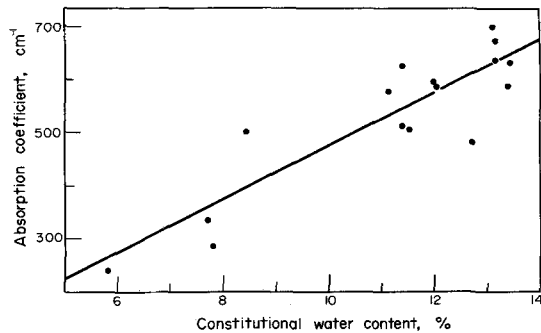


Fig. 1. Correlation between the absorption coefficient and the constitutional water content. $y = -26 + 49.9x$. $r = 0.87^{**}$.

RESULTS

The absorption coefficient of pure kaolinite

Optimum condition settings allowed clear separation of at least three of the four kaolinite bands, i.e. the high-frequency band (HF), low-frequency band (LF) and the band at 3650 cm^{-1} (MF). The band at 3670 cm^{-1} was resolved only for well-crystallized samples.

The absorption coefficient for the OH stretching bands of pure kaolinite specimens was measured according to the method described by Rouxhet (1970). The data are listed in Table 2. The coefficient of variation (CV) of K in Table 2 was 25% which reflects the fairly high variability of the OH absorption coefficient for pure kaolinites.

The absorption coefficient deduced from the band area is proportional to the concentration of hydroxyls generating the band. Figure 1 illustrates this relationship and confirms that K variations are not random but a function of the constitutional water content. The bulk absorption coefficient per hydroxyl, k , calculated from the relation $(K \times 0.9)/(\text{constitutional H}_2\text{O})$ showed a smaller coefficient variation (CV = 14%) (Table 2). The contribution from mineral impurities has been removed from k and its variability could be attributed to crystallochemical and/or crystallographic differences.

The absorption coefficient of kaolinite in sediments

Absorption coefficients were computed from band intensities in the same way as for pure kaolinite (Table

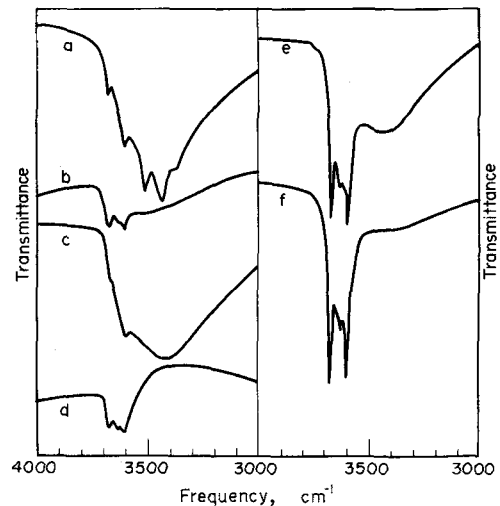


Fig. 2. Infrared spectra of sediments analyzed: a, C12 untreated. b, C12 heated at 300°C . c, N1 untreated. d, N1 differential spectrum, kaolinite component. e, 6 untreated. f, 6 differential spectrum, kaolinite component.

3). For the present working conditions integrated absorbances were measured with a precision of $\pm 10\%$ as determined for sample 6. This precision could have been better if the infrared spectra were recorded in absorbance instead of in transmittance. Then the errors in redrawing the spectra would have been avoided. Improving the technique of mixing would also have increased the precision. The magnitude of K depends upon the kaolinite content of the sample. Calculated k vary within the same range as that observed for pure kaolinite specimens.

The sediments studied contained other crystalline and non-crystalline minerals associated with kaolinite (Table 1) and the OH bands of these components overlap the kaolinite pattern. This interference must be eliminated in order to obtain the true shape of the kaolinite bands; this is prerequisite to a quantitative analysis. When the interfering component dehydroxylated at a lower temperature than kaolinite it was possible to eliminate the interference by heat-treatment alone. This is illustrated by sample C12 containing gibbsite which was decomposed by heating at 300°C for 12 hr.

Table 3. Integrated absorbance, coefficient of absorption, constitutional water content, bulk absorption coefficient per hydroxyl and kaolinite proportion in sediment samples.

Laboratory number	Integrated absorbance (cm^{-1})	K (cm^{-1})	κ (cm^{-1})	Constitutional water content (%)	Kaolinite content (%)
1	11.5	88			12
4	7.9	60			9
6	32.7	251	43	5.2	35
9	16.1	123			18
N1	7	56	32	1.6	10
C12	8	71	29	2.2	14

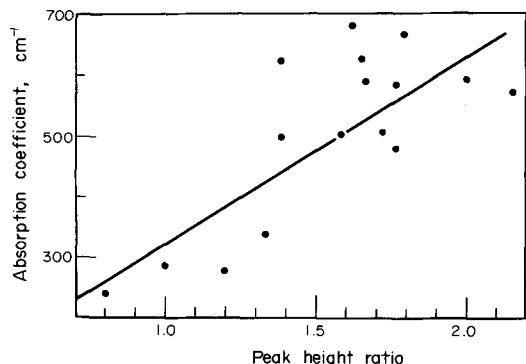


Fig. 3. Correlation between the absorption coefficient and the peak height ratio of the HF band to the MF band. $y = 9.3 + 311.4x$. $r = 0.8^{**}$.

The treatment did not affect kaolinite and the infrared spectrum showed the kaolinite hydroxyl pattern alone (Figure 2). Other clay minerals present in sediments may have a higher dehydroxylation temperature than kaolinite, e.g. montmorillonite, illite or mica-type minerals; kaolinite was then selectively decomposed by heating the sample at 550°C for 12 hr (Murray and White, 1949). The kaolinite pattern was recorded using the differential technique. This procedure was used for samples 1, 4, 6, 9 and N1 (Figure 2). Sample 6 contained a substantial amount of illite which was partly dehydroxylated upon heating at 550°C and the intensity of the hydroxyl band decreased accordingly. However, the error so introduced was small because the illite coefficient of absorption, K , is about three times lower than the average K of kaolinites.

DISCUSSION

Usefulness of equation (3) in relation to the variability of K

As shown in Table 2, the coefficient K varies in a rather wide range, this variability may question the usefulness of equation (3) which is based upon the assumption that the test sample and kaolinite standard have the same absorption coefficient. Then the choice of an appropriate standard complicates the determination. For the benefit of this method criteria for the choice of a suitable standard should be deduced from characteristics of the OH stretching bands of the test sample.

Examination of the hydroxyl stretching bands showed large variations of the relative intensities of the HF, MF and LF bands and the variation of the HF band was larger than that of the MF and LF bands. By plotting the ratio R , (peak height of the HF band)/(peak height of the MF band), representing the extent to which the intensities of the two bands vary, vs. the absorption coefficient one gets a linear relationship (Figure 3). This relation allows for a quick estimation of K

Table 4. Kaolinite content as measured by infrared method and calculated from TGA data.

Laboratory number	Kaolinite content	
	Infrared estimation (%)	TGA estimation (%)
6	35.5 ± 4	37.0
N1	10.5 ± 1	11.4
C12	14.0 ± 2	16.0

for kaolinites present in sediments, and also can be used for selecting the standard to be added. The ratio R of the standard should match the R of the test sample; by fulfilling this requirement, equation (3) can be used for estimating kaolinite. The standard error of K predicted from the above empirical relation is 80 cm^{-1} , introducing an error of $\pm 10\%$ in the kaolinite estimation in equation (3).

It is interesting to note that there is a general agreement between the classifications of kaolinites based upon the R ratio defined above and the one found by Parker (1969) who has used the ratio A_{3695}/A_{3620} as a criterion for rating kaolinites. Here also, the R ratio rates the crystallochemical differences of kaolinite since the bands at 3700 and 3670 cm^{-1} correspond to hydroxyls perpendicular to the ab plane whereas the band at 3655 cm^{-1} corresponds to hydroxyls nearly parallel to the same plane (Rouxhet et al., 1976), the coefficient R being then related to populations of hydroxyls associated with different crystallographic sites.

The estimation of kaolinite in sediments

The method of known additions was tested on several kaolinite-containing sediments. Integrated absorbance of kaolinite OH bands increased linearly with addition of the kaolin standard to the test sample (Figure 4). The linearity demonstrates the validity of equation (2) for estimating kaolinite. At least three sources of error affect the precision of the estimation: (1) an error in recording the spectrum which depends upon the instrument used, (2) an error in mixing the kaolinite standard with the test sample and also in preparing the KBr mixture and (3) an error in measuring the area under the hydroxyl bands.

When the kaolinite bands (3695 and 3620 cm^{-1}) were diffuse due to the overlapping of other minerals, i.e. sample N1, Figure 2, it was only possible to estimate the total hydroxyl band intensity of crystalline clays present in the sediment by extrapolation to zero kaolinite addition (Figure 4). Integrated absorbances reported in Figure 4 were the sum of two components, i.e. the kaolinite OH band and the overlapping of other clay minerals present in the sediment.

The overlapping constant was calculated for the sediment sample by subtracting the pure kaolinite intensity (A_0) obtained by differential spectroscopy from the to-

Table 5. Standard clay mixtures: component proportions and kaolinite estimation.

Component proportions			
Kaolinite (%)	Bentonite (%)	Illite (%)	Kaolinite estimation
5	95	—	4.4
10	90	—	9.6
10	—	90	11.0

Clay standards used were: hydrite 121 kaolinite, Volclay bentonite (2 μm), Fithian illite (2–0.2 μm).

tal intensity. It was then possible to calculate by difference the intensities (A_i) of the kaolinite bands which was given different additions of kaolinite standard.

A plot of A_i/A_0 vs. W_i/W_s gave good straight lines when the integrated absorbances (A_i) were calculated in this way. The measured kaolinite contents are listed in Table 3 and the precision of the estimation was calculated by taking 10% as the maximum error made in measuring the OH band intensity.

Results obtained by infrared spectroscopy were compared to kaolinite estimation made by thermogravimetric analysis (Table 4). TGA data were converted into kaolinite content by assuming a 14% constitutional water content for pure kaolinite. The results obtained by two different methods were in very good agreement.

This method was tested on sediments in which the kaolinite was well crystallized. For soils containing fire clays the method could be used with equal success providing that the R of the standard matches the R of the test sample.

Testing the method against standard mixtures

The infrared method effectiveness was tested against standard mixtures of kaolinite–bentonite–illite. Proportions of kaolinite and bentonite or illite in the mixture are listed in Table 5. No special reason has determined the choice of hydrite 121 kaolinite for making up the synthetic mixtures but this particular kaolinite was in abundant supply when the experimental work was done. The measured kaolinite contents were close to the proportions of kaolinite in the mixture (Table 5) with about 10% error. This agreement demonstrates the effectiveness of the method for estimating kaolinite in sediments.

CONCLUSION

The method described for estimating kaolinite was based on a fundamental property of the mineral, i.e. the hydroxyl stretching vibration in the infrared region. From characteristics of the hydroxyl pattern of the test sample it is possible to choose an appropriate standard for the addition. The kaolinite in test sample and standard should have a similar absorption coefficient. The precision of the estimation, calculated as 10%, corre-

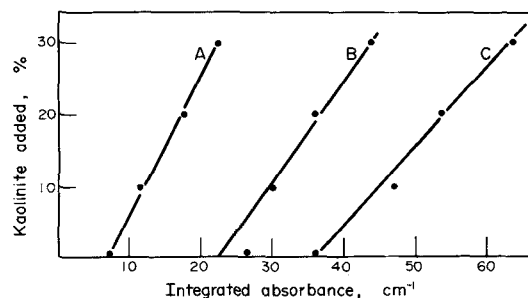


Fig. 4. Integrated absorbance of hydroxyl band vs. the percentage of kaolinite added to the test sample. A, C12, whole sediment. B, N1 < 2 μm . C, 6, 2–0.2 μm .

sponded to a maximum error in measuring the OH band intensity.

In sediments containing substantial proportions of non-crystalline minerals, the precision on the kaolinite estimation could be better than that of other conventional techniques, i.e. X-ray powder diffraction, DTA or TGA because the intensity of OH bands primarily depends upon the constitutional water content and is much less affected by crystallinity.

Heating the sample is the only treatment required in the method when the hydroxyl pattern of other minerals interferes with kaolinite. This makes the method attractive for estimating kaolinite in tropical soils in which the amount of sesquioxides can be substantial.

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Резюме- Инфракрасный метод был разработан для оценки количества каолинита в осадках. Гидроксильные растянутые полосы каолинита в осадках могут быть обнаружены с дифференциальной техники, которая устраняет влияние других присутствующих минеральных образований. Добавляя к образцу известное количество надлежащего эталона и замеряя интенсивности полос OH до и после добавления эталона, удается вычислить долю каолинита в изучаемом образце. Выбор добавляемого эталона зависит от характерных черт гидроксильных растянутых полос.

Kurzreferat- Eine Infrarotmethode für die Bestimmung von Kaolinit in Sedimenten ist entwickelt worden. Hydroxylstretschwingungen von Kaolinit in Sedimenten können, durch den Gebrauch einer Differentialtechnik, angezeigt werden, die das Überlappen durch andere anwesende, mineralische Bestandteile eliminiert. Indem bekannte Mengen eines angemessenen Standarts zu der Probe gegeben werden und durch Messung der Intensität der Hydroxylschwingung vor und nach der Zugabe des Standarts, ist es möglich, die Menge von Kaolinit in der Testprobe zu errechnen. Die Wahl des zugegebenen Standarts, wird mit Hilfe der charakteristischen Eigenschaften der Hydroxylstretschwingungen getroffen.

Résumé- Une méthode à l'infra-rouge a été développée pour l'estimation de kaolinite dans des sédiments. Des bandes d'allongement hydroxyles de kaolinite dans des sédiments peuvent être enregistrées par l'emploi d'une technique différentielle qui élimine toute superposition due à d'autres constituents minéraux présents. Par l'addition de quantités connues d'étalon approprié à l'échantillon, et en mesurant les intensités des bandes OH avant et après l'addition, il est possible de calculer la proportion de kaolinite dans l'échantillon expérimental. Le choix de l'étalon ajouté est dicté par les phénomènes caractéristiques des bandes d'allongement hydroxyles.