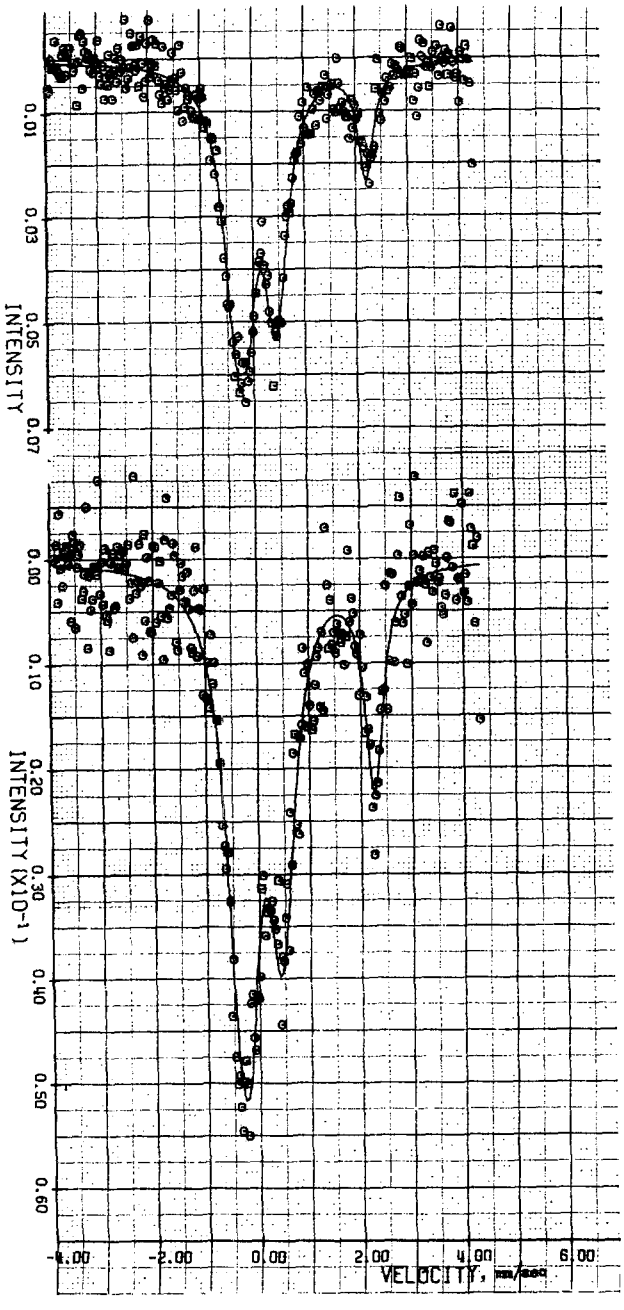

Mössbauer spectra for natural sediments. The top spectrum is before treatment and the lower one after treatment. Spectra taken at room temperature. Both spectra were taken under identical laboratory conditions. One of the peaks of quadruple doublet for Fe^{+2} is masked by the highly prominent Fe^{+3} peak near the centre of the figure.

Mössbauer spectra-data	Untreated sample[a]	Treated sample[a]
Total counts	1.094×10^5	1.093×10^5
Peak A	-0.352 ± 0.009	-0.400 ± 0.013
Peak B	$+0.434 \pm 0.007$	$+0.437 \pm 0.011$
Intensity Fe^{+3}	0.0457 ± 0.001	0.033 ± 0.001
Isomer shift, Fe^{+3}	$+0.041 \pm 0.001$	$+0.018 \pm 0.001$
Quadrupole splitting, Fe^{+3}	0.786 ± 0.001	0.837 ± 0.001
Width	0.561 ± 0.018	0.626 ± 0.027
Peak AB[b]	-0.109 ± 0.016	-0.186 ± 0.018
Peak C	$+2.195 \pm 0.018$	$+2.246 \pm 0.014$
Intensity Fe^{+2}	0.021 ± 0.001	0.021 ± 0.001
Isomer shift, Fe^{+2}	$+1.043 \pm 0.010$	$+1.050 \pm 0.010$
Quadrupole splitting, Fe^{+2}	2.304 ± 0.012	2.500 ± 0.012
Width	0.352 ± 0.035	0.436 ± 0.036
Intensity Fe^{+2}/intensity Fe^{+3}	0.470	0.620

[a] All values, except intensity and counts, are expressed as mm/sec. Isomer shift and quadrupole splitting values have been calculated from peak positions. [b] Peak AB is overlapped between A and B: hence not plotted by computer.

well as in the exchange sites. Since it is not possible to separate the individual clay minerals from the mixture physically, it is difficult to assign specific changes to any particular clay minerals. In view of the known properties of illite and kaolinite, it is most likely that any structural changes in clay minerals due to chemical treatment are probably in the montmorillonite fraction.

From the comparison of spectra for untreated and treated clay mixtures, it appears that the usefulness of chemical pretreatment for mineralogical studies needs to be re-evaluated.

Résumé. A l'aide de l'effet Mössbauer, il a été observé que les méthodes conventionnelles pour enlever les incrustations affectent les silicates quand elles sont appliquées aux sédiments avant leur examen minéralogique.

V. Subramanian

Marine Sciences Centre, McGill University, Station A, P.O. Box 6070, Montreal (Quebec, Canada H3C 3G1), 12 August 1974.

Quantitative Separation of Titanium from Numerous Metal Ions by Thin Layer Chromatography

Thin layer chromatography has been extensively used for the separation of organic substances. The use of this technique for the separation of inorganic ions has not been investigated in detail. Seiler and Seiler[1] had applied thin layer chromatography for the separation of cations. Earlier studies in these laboratories have shown that mixed solvent systems are very useful for the qualitative separation of inorganic ions. However, quantitative aspect of the separations is still lacking. It was therefore considered worthwhile to use mixed solvent systems for a

[1] H. Seiler and M. Seiler, Helv. chim. Acta *43*, 1939 (1960).

Quantitative separation of different amounts of titanium from common interfering metal ions

Titanium taken (μg)	Interfering ions added		Titanium found (μg)	Error (%)
172.5	Cu (II)	(200 μg)	168.0	— 2.3
172.5	Fe (III)	(200 μg)	168.0	— 2.3
172.5	A mixture[a]	(Total 200 μg)	168.0	— 2.3
32.0	A mixture[a]	(Total 272.5 μg)	31.0	— 3.2
65.0	A mixture[a]	(Total 272.5 μg)	64.0	— 1.6
80.0	A mixture[a]	(Total 272.5 μg)	78.0	— 2.5
114.0	A mixture[a]	(Total 272.5 μg)	113.0	— 0.9
176.0	A mixture[a]	(Total 272.5 μg)	174.0	— 1.2
346.0	A mixture[a]	(Total 272.5 μg)	345.0	— 0.3

[a] Cu (II), Al (III), Fe (III), V (IV), UO_2 (II), Ni (II), Co (II), Cr (III), Sn (IV), Mg (II), Mo (VI) and Mn (II).

detailed study of 46 common metal ions and to develop some quantitative separations. As a result, titanium has been quantitatively separated from 12 common metal ions. The following report summarizes the results of such a study.

Experimental. Desaga (Made in Germany) TLC apparatus was used for the preparation of thin layers on glass plates. The thickness of the layers for qualitative and quantitative work were 0.25 mm and 0.5 mm respectively. Spectrophotometric studies were made on Bausch and Lomb spectronic-20.

Silica Gel G (E. Merck, Darmstadt) was used for the coating of plates. Other reagents were Anala R grade. Decimolar solutions of nitrates, chlorides or sulphates of metal ions, containing a little acid to prevent hydrolysis, were used for spotting in qualitative studies and for quantitative work 4% metal ions solutions. Titanic chloride (15% W/V) was diluted to get the solution of required strength. It was standardized against ferric ammonium sulphate[2]. Chromotropic acid (1% aqueous solution) was used to detect titanium (v) and usual detectors for the detection of other metal ions.

Preparation of Plates. The calculated amount of silica gel-G (1 g/plate; 16 × 3 cm for qualitative and 2 g/plate for quantitative work) was taken in a conical flask and double amount of water was mixed with it. Mixture was shaken for 5 min. The slurry so formed was poured into the spreader and was applied on the plates with the help of the applicator. The plates were then dried, first at room temperature and then at 100 °C for 1 h.

Procedure. a) Thin glass capillaries were used to spot the test solutions. The solvent system was allowed to ascend 12 cm on the plates. b) The solutions containing known amounts of titanium were spotted along with other metal ions on the plates in the form of uniform streak with the help of lambda pipette. The plates were developed as usual. The developing solvent, methanol + 50% HNO_3 (9:1) was allowed to ascend 12 cm on plates. In order to ascertain the actual position of the respective spots after development, pilot chromatograms were run under similar conditions. 15% HCl solution was used as a blank. Titanium was eluted with 40 ml hot 5% HCl solution in fractions of 10 ml each. The final volume of the filtrate was then reduced to nearly 2 ml by evaporation and then titanium was determined spectrophotometrically using sulphosalicylic acid at 410 nm[3].

Results. a) 46 common metal ions were chromatographed using methanol + HNO_3 and butanol + $HClO_4$ systems in various ratios: Best results were obtained in methanol + 50% HNO_3 (9:1) system. b) Quantitative determination of titanium in presence of common metal ions as impurities. Different amounts of titanium were quantitatively separated from 12 common interfering metal ions. The results are given in the Table.

Discussion. The results (Table) show that the system, methanol + 50% HNO_3 (9:1), is very useful for the separation of titanium from numerous metal ions. It was possible to separate binary mixtures of titanium with 12 interfering metal ions.

Different amounts of titanium (32–346 μg) were separated from Cu, Al, Fe, V, UO_2, Ni, Co, Cr, Sn, Mg, Mo and Mn (Table). The results are reproducible and the error is within the spectrophotometric error range[4].

Zusammenfassung. Es wird die quantitative Abtrennung von Titanium mittels Dünnschicht-Chromatographie aus einem Gemisch von 12 Kationen (Tabelle) beschrieben.

M. QURESHI, J. P. RAWAT, K. N. MATHUR and TRIPAT BINDRA

Chemical Laboratories, Z. H. College of Engineering and Technology, Aligarh Muslim University, Aligarh (India), 11 September 1974.

[2] N. H. FURMAN, *Standard Methods of Chemical Analysis* (Van Nostrand Press, New York 1962), p. 1104.

[3] M. QURESHI, J. P. RAWAT and F. KHAN, Analyt. chim. Acta *41*, 164 (1968).

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