Determination of Nb and Ta in weathering soils from Brazilian carbonatite complexes

Tadashi Fujinuki*

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Wet chemical analysis for Nb and Ta was carried out for lateritic soils derived from alkalinecarbonatite complexes and a reasonable result was obtained. This recomended procedure is mainly composed of gravimetric method using cupuferron after separating with anion exchange regin, originally designed as JIS M 8407 (Japanese Industrial Standards Commitee, 1977). The detail of the procedure is shown in Fig. 1.

Soil samples measured here, are characterized by (1) existance of chemically resistant minerals such as Nb-bearing ilmenite and ilmenorutile and (2) high contents of Fe, Ti, Ba, P and Nb. The following treatments, therefore, are carefully carried out for accurate analysis;

1. Sample preparation: Very fine-grained powdered samples (less than $23 \, \mu \text{m}$ in diameter) are prepared using conbination of automatic agate mortar and vibration mill (tungsten carbide).

- 2. Elimination of barium: The residue after treating of EDTA shown in Fig. 1 contains some amounts of Ba as BaSO₄. Most of bariumcomplex remaining as solid at next step in HF+HCl solvent, is excluded by filtration. Barium ion which is passed through the filtration may perfectly be eliminated by anion exchange column.
- 3. Test for this method: The accuracy of the procedure can be totally estimated by two reference samples; MIN-100/69 and MIN-99/ 69 (synthetic mixture of columbite and carbonate. Issured by National Institute of Metallurgy, South Africa).

Table 1 Nb₂O₅ and Ta₂O₅ contents of reference samples and soil samples from Brazilian carbonatites, determined by newly recommended analysis

	Nb ₂ O ₅ wt%	Ta ₂ O ₅ wt%		Nb₂O₅ wt%	Ta ₂ O ₅ wt%		Nb₂O₅ wt%	Ta ₂ O ₅ wt%		Nb ₂ O ₅ wt%	${ m Ta_2O_5}$ wt $\%$
Ar-01F	1.06	0.00	.Cat-06F	0.00	0.00	Sal-03F	0.00	0.00	MIN st.		
Ar-02F	0.00	0.00	Cat-07F	2.60	0.02	Sal-04F	0.00	0.00	100/69	2.65*	0.45*
Ar-03F	0.05	0.00	Cat-7AF	1.76	0.03	Sal-05F	0.10	0.00	-	2.63	0.44
Ar-04F	4.17	0.11	Cat-08F	0.50	0.00	Sal-06F	0.00	0.00		2.64	0.43
Ar-05F	4.59	0.00	Cat-09F	1.02	0.00	Sal-07F	0.00	0.00			
Ar-06F	0.84	0.00	Cat-10F	0.51	0.00	Sal-08F	0.00	0.00	99/69	13.29*	5.64*
Cat-03F	0.48	0.00	Cat-11F	1.29	0.02	Sal-09F	0.00	0.00	_	13.40	5.55
Cat-04F	0.42	0.00	Sal-01F	0.00	0.00	Sal-10F	0.00	0.00		13.28	5.49
Cat-05F	3.44	0.03	Sal-02F	0.08	0.00						

Ar, Araxa; Cat, Catalao; Sal, Salitre. *Recommended values

^{*} Japan Society for Analytical Chemistry, 1–26–2, Nishi-gotanda, Shinagawa-ku, Tokyo 141.

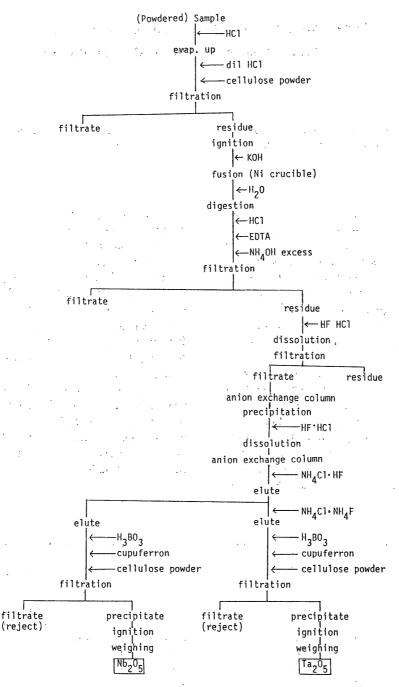


Fig. 1 Procedure for determination of Nb and Ta in lateritic soil samples.

Twenty-six soil samples from lateritized mantle on some alkaline-carbonatite complexes in the Brazilian shield were analyzed by the improved wet method. The results including reference samples are given in Table 1. Data on MIN samples agree well with the recommended

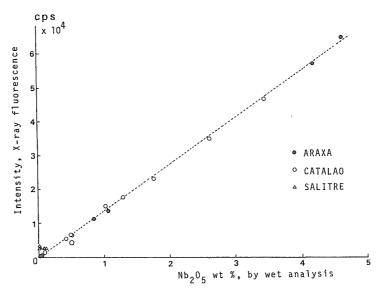


Fig. 2 Comparison of wet chemical analysis and X-ray intensity.

values.

The same rods of the powdered samples were also analyzed by H. GOTO, using XRF, and the result is given in Fig. 2. A good correlation between the wet and XRF analyses may strongly support that XRF method is effective for determination of Nb in lateritic soils, if the samples analyzed here are used as Nb-reference samples.

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Reference

Japanese Industrial Standards Commitee (1977)

Method for determination of niobium and tantalum in ores, JIS M 8407, 7p. Japanese Standards Association. (in Japanese)

ブラジル国カーボナタイト源風化土壌中のニオブとタンタルの化学分析

藤貫 正

要 旨

カーボナタイト-アルカリ岩に由来するラテライト17試料の Nb, Ta の化学分析を実施し、良好な結果を得た.分析手法は、JIS-M-8407に基づき、イオン交換樹脂を使用する重量法である.試料は、難溶性鉱物を含有し、かつ、Fe, Ti, Ba, P および Nb を高濃度に含むため、粉末試料の徴粉化、分離行程における妨害元素の完全除去等分析行程に工夫を加えた.また、同一粉末試料の蛍光X線(XRF)強度を測定し、XRF による確度の高い Nb 分析が可能であることを示した.

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