The samples (L1 and L2) have yielded a rich assemblage of calcareous nannofossils consisting of *Braarudosphaera africana* Stradner, *B. bigelowi* (Gran and Braarud), *B. discula* Bramlette and Riedel, *B. Lakhpatensis* n. sp., *Chiasmolithus gigas* Bramlette and Sullivan, *Coccolithus pelagicus* (Wallich), *Cyclococcolithina formasa* (Kamptner), *Discoaster* sp., *D. barbadiensis* Tan Sin Hok, *D. saipanensis* Bramlette and Riedel, *D. lodoensis* Bramlette and Riedel, *D. sublodoensis* Bramlette and Sullivan, *D. tani* Bramlette and Riedel, *D. tani nodifer* Bramlette and Riedel, *D. trinus* Stradner, *Lophodolithus reniformis* Bramlette and Sullivan, *Reticulofenestra bisecta* (Hay, Mohler and Wade), *R. umbilica* (Levin) Martini and Ritzkowski, *Thoracosphaera* sp., *T. deflandrei* Kamptner and *Triquetrorhabdulus inversus*. Bukry and Bramlette. The present nannoplankton assemblage has been referred to an informal *Discoaster tani nodifer* zone which is correlatable with the upper part of Martini's NP 16, *Discoaster tani nodifer* Zone (late Middle Eocene; Martini[4]).

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December 3, 1977.

1. Sen Gupta, B. K., *Jour. Geol. Soc. India*, 1964, 5, 138.
2. Samanta, B. K., *Micropaleontology*, 1970, 16(2), 185.
3. Sen Gupta, B. K., *Jour. Pal.*, 1965, 39(1), 86.
4. Martini, E., *Proc. 2nd Plank. Conf. Roma*, 1971, p. 739.

RAPID ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD FOR ESTIMATION OF (REACTIVE & TOTAL) SILICA, IRON, ALUMINIUM AND TITANIUM IN BAUXITE

DISCOVERY of huge bauxite reserves in the East Coast of India and the exploration programme taken up by the Geological Survey of India during the field season 1975-76 aimed at assessing the extent and grade of bauxite has necessitated the analysis of large number of samples in different laboratories of Geological Survey of India. Conventional methods of bauxite analysis are very slow and naturally cannot keep pace with the progress of the drilling programme envisaged in this time bound project. It has therefore become necessary to devise rapid methods of analysis involving the use of instrumental methods.

Though Atomic Absorption Spectrophotometry (AAS), X-ray-Fluorescence (XRF) and Neutron-Activation techniques are ideally suited to provide the desired rapidity coupled with high precision and accuracy, it is only with regard to the AAS technique that facilities can easily be created at a reasonable cost, while the other two require a more expensive and sophisticated set up. Bowman and Willis,[1] who had earlier attempted bauxite analysis by the AAS technique, preferred sodium-carbonate-borax fusion in a platinum crucible to either triacid treatment (H_2SO_4 + HNO_3 + $HClO_4$) or alkali fusion in a nickel crucible for decomposition of bauxite. Langmyhr and Paus[2] resorted to HF treatment of siliceous material in a teflon bomb prior to AAS analysis of Si, Al, Fe & Ti. Such methods of decomposition involving the use of platinum ware, etc., cannot answer the need of routine analysis on a large scale.

Detailed studies carried out by the authors have resulted in a rapid method, involving fusion of bauxite with potassium pyrosulphate in a hard glass pyrex test-tube and dissolution of the fused mass in 2N HCl before subjecting to AAS analysis. This method proved to be so fast that a team of five analysts could process at the rate of 1000 samples a month. Nearly 7500 samples from the East Coast Bauxite project were analysed by this method during the period August 1976 to April 1977.

A distinct advantage of this method lies in the determination of reactive silica along with the other four constituents. Silica brought into solution by pyrosulphate fusion corresponds to the reactive component of the total silica which according to Schellman[3] is mostly contributed by the clay minerals present in bauxite. Information about its actual content in bauxite is a vital necessity as it has a significant bearing in Bayer's extraction process. While conventional methods[4] reported in literature are cumbersome, our method provides a neat and rapid means of estimating the same. Three samples from Dr. Schellman, BGR, Hanover- analysed for reactive silica by the AAS method gave values which compare very well with conventional analysis. (Probe No. 1166, AAS 2·36%, Conv.—2·42%. Probe No. 1111, AAS—0·84%, Conv. —1·00%; Probe No. 619. AAS—5·62%, Conv.—5·42%).

As silica present in the form of quartz and sillimanite cannot get extracted by pyrosulphate fusion, separate alkali fusion in a nickel crucible was suggested for the estimation of total silica in bauxite.

A check analysis for total SiO_2, Fe_2O_3, Al_2O_3 and TiO_2 in respect of three samples by the conventional

TABLE I

Estimation of Al_2O_3, Fe_2O_3, TiO_2 and total SiO_2 in bauxite

Sample No.	Al_2O_3%			Fe_2O_3%			TiO_2%			Total SiO_2%		
	Conv.	AAS	XRF	Conv.	AAS	XRF	Conv.	AAS	XRF	Conv.	AAS	XRF
A	34·17	34·13	35·22	18·56	18·20	17·36	1·30	1·44	1·38	30·40	30·47	30·75
B	*54·34	54·17	54·65	*23·21	22·93	22·74	*2·40	2·41	2·24	*7·36	7·91	7·72
C	48·88	48·21	48·92	15·20	15·38	15·68	8·03	8·44	8·00	4·18	3·85	3·83

A   — East Coast Bauxite.
B   — BXN Standard sample from Nancy, France.
*   — Internationally certified values.
'C   — Standard sample from BALCO.
Conv. — Data furnished by G.S.I. Central Headquarter Lab., Calcutta.
XRF   — Analyst, Dr. M. S. Rao of X-ray Lab. of A.M.S.E. Wing.

and XRF methods revealed close agreement with AAS analysis as can be seen from the data in Table I. Precision and accuracy of the new method appear to be quite good as evidenced by the standard deviation values of 0·09 for reactive SiO_2, 0·14 for total SiO_2, 0·30 for Al_2O_3, 0·19 for Fe_2O_3 and 0·07 for TiO_2.

While full paper with relevant details will be published separately, a brief account of the procedure adopted is given below:

PROCEDURE A

Estimation of Reactive Silica, Alumina, Iron and Titania

The dried bauxite sample (0·1 g) was weighed into a dry pyrex test-tube and fused with 1 gm of A.R. potassium pyrosulphate on a Meker Burner till the decomposition is complete. The fused mass was dissolved by digestion with 10 ml of 2N HCl. After settling down, the supenatant liquid was carefully transferred to a 50 ml volumetric flask. The residue remaining at the bottom was treated with 2 ml of 30% KOH solution and finally dissolved in 2N HCl and added to the main solution and made up to the mark. While this solution could directly be used for estimating reactive silica and titanium, a 1 : 10 dilution was necessary for estimating Al_2O_3 and Fe_2O_3. Blank correction for silica is necessary as traces of it is likely to be contributed by the test-tube during fusion.

PROCEDURE B

Estimation of Total Silica

0·1 gm of dried bauxite was fused with 0·5 to 1·0 gm of A.R. KOH in a nickel crucible and the melt after digestion with water on a waterbath was transferred to a 100 ml volumetric flask, dissolved in 2N HCl and made up to the mark. This solution was used for estimation of total silica.

Equipments Used

Varian Techtron Model AA–1200 and Perkin–Elmer Model 403 AAS were used in concentration mode

with requisite scale expansion maintaining the prescribed instrument parameters in respect of hollow cathode lamp current, slit width, wavelength, etc.

Flame Conditions

Nitrous oxide–acetylene flame was used for the determination of SiO_2, Al_2O_3 and TiO_2 and air-acetylene flame for Fe_2O_3. Fuel-oxidant ratio was so adjusted as to give maximum sensitivity.

Mode of Comparison with Standards

Concentrations of Al_2O_3 and Fe_2O_3 in samples were arrived at by comparison with the standards. Aluminium standards in the range of 50 to 110 ppm at 10 ppm interval were used whereas in the case of Fe_2O_3, standards in the range of 5 to 65 ppm at 5 ppm interval were used for comparison. Concentration of total SiO_2 and TiO_2 in the samples were arrived at by the standard addition method as solution composition is found to affect absorbance considerably.

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1. Bowman, G. A. and Willis, J. V., *Anal. Chem.*, 1967, **39**, 1210.
2. Langmyhr, F. J. and Paus, P. E., *Anal. Chim. Acta*, 1968, **43**, 508.
3. Schellman, W., *Mining Magazine*, July 1975, **133** (1).
4. *I.S.I. Bulletin*, 1962, No. 2000.