

Siwalik rocks were deposited in fore-deeps similar to the present-day Gangetic alluvium, where sedimentation was controlled by fluvial processes. Similar to the shear zones as found in the Gangetic basin certain shear zones might have been active during the sedimentation of the Siwaliks. Gansser (1964; Fig. 1)⁹ has shown certain E-W trending lineaments in the Siwaliks. These might correspond to the shear zones active during sedimentation of the Siwaliks. Krishnan (1961, 1969)^{2,10} also mentions certain longitudinal faults as well, besides the transverse faults, of which some are loci of the earthquakes, occasionally felt in the region. The Bihar earthquake of 1934 with iso seismals (Mercalli scale) trending WNW-ESE, subparallel to the course of Ganga River between Allahabad and Benares suggests presence of zones of disturbance, evidently faults, in the basement below the alluvium, parallel to the trend of the Himalayas.

These weak zones affecting the sedimentation pattern in the Gangetic alluvium would also determine the groundwater pattern in the alluvium. The groundwater development and its potentiality might be different on the two sides of these weak zones, e.g., in the northern and southern blocks of Ganga River.

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1. Wadia, D. N., *Geology of India*, Macmillan & Co., London, 1959, p. 536.
2. Krishnan, M. S., *Mem. G.S.I.*, 1961, 81, 1.
3. Poddar, M. C. (Editor), *O.N.G.C.*, Technical Publication, 1962, 1, 68.
4. Mathur, L. P. and Kohli, G., *6th World Petroleum Congress*, 1963, p. 633.
5. Sengupta, S., *Report of XXII Int. Geol. Congress*, New Delhi, 1964, 11, 334.
6. Mool Chand, Datta, A. N., Chellam, R. S., Ghosh, A. P., Awasthi, A. M., Awasthi, D. N. and Garg, V. C., *Proceedings XXII Int. Geol. Cong.*, 1964, Sec. 2, Pt. II, 260.
7. Fuloria, R. C., "Selected lectures on Petroleum Exploration," *O.N.G.C.*, 1969, p. 171.
8. Misra, R. C., Singh, I. B. and Kumar, S., *Geophytology*, 1971, 1, 151.
9. Gansser, A., Interscience Publishers, London, 1964, p. 289.
10. Krishnan, M. S., Higginbothams & Co., Madras, 1969, p. 536.

ASHING PROCEDURES FOR VOLATILE ELEMENTS IN BIOMATERIALS

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ORGANIC content of biomaterial is destroyed either by ashing in a muffle furnace at different temperatures or wet ashing by the action of various oxidising liquids¹⁻⁵.

It has been suggested that for a proper study of the behaviour of the elements in biomaterials in different ashing techniques, the tracer must be in the same chemical form as that of the stable element, as then only the tracer indicates the proper losses of the corresponding elements. The tracer should therefore be incorporated into the structure of organometallic complexes in the same way as the stable counterpart. To achieve therefore proper exchange of tracer with stable counterparts, the tracer was injected into the pedal sinus of a bivalve *Anadara granosa*, and the losses of iron, strontium, cobalt, zinc, cerium, manganese and cesium, were determined following various ashing procedures and reported in an earlier communication⁵. These studies were further extended to understand the losses of volatile elements like mercury, selenium,

arsenic, antimony, chromium, ruthenium and tungsten following dry and wet ashing techniques and are reported here.

The soft tissues of the experimental animals were dissected after 48-96 hours following tracer injection and transferred into a plastic counting vial and the radioactivity was measured following gamma-ray spectroscopy. For dry ashing, the tissue was transferred into a silica crucible, dehydrated under an infrared lamp and further ashed in a muffle furnace either at 400° C or 700° C for about 24 hours. The ash samples were dissolved in dilute hydrochloric acid, transferred to counting vials and gamma activity measured once again maintaining the geometry. In wet ashing, the samples were transferred into a glass beaker and brought into solution by using oxidising mixtures⁵. The tissues were digested with aliquots of 25 ml of oxidising liquids till the sample was free of carbon. The digested samples were then transferred to counting vials and counted maintaining the geometry.

TABLE I
Physical and chemical characteristics of the isotopes used

Isotope	Half-life	Gamma energy MeV	Chemical form
Cr-51	27.8 days	0.323	Chromium chloride (10-15 mc/g. Cr)
Hg-203	47 days	0.279	Mercuric nitrate (36 mc/g. Hg)
Sb-124	60 days	0.660 0.720 1.729, etc.	Mixture of antimony trichloride and oxychloride in 6 M. HCl (carrier free)
As-76	26.5 hrs	0.560 0.660 1.210 1.440, etc.	Arsenous acid (16 mc/g. As)
Se-75	121 days	0.120 0.140 0.200 0.270, etc.	Sodium selenate in neutral solution (9 mc/g. Se)
Ru-106	365 days	0.510	Ruthenium chloride (carrier free)
W-187	24 hrs	0.072 0.132 0.479, etc.	Sodium tungstate in aqueous solution (200 mc/g.W)

TABLE II
Per cent recoveries of trace elements by dry ashing

Isotope	Per cent recoveries at	
	400° C	700° C
Cr-51	68.4 66.1	48.8 50.2
Hg-203	3.4 8.5 9.9	0.4 0.1 ..
Sb-124	93.1 93.1	66.1 73.6
As-76	19.6 15.3	10.6 8.8
Se-75	0.7 1.1	0.6 0.2
Ru-106	93.6 90.1	26.7 23.3
W-187	94.2	65.6 64.2

The losses of arsenic, mercury, selenium, chromium and ruthenium were further studied following digestion under reflux using water condenser. Generally digestion under reflux was completed in about 8 hours. The aliquots were suitably diluted and counted maintaining the geometry.

RESULTS AND DISCUSSIONS

Details of the radiotracer used in these experiments with their half-lives, chemical form and

gamma energies are given in Table I. In Table II are given the retention of elements after thermal decomposition at temperatures of 400° C and 700° C. It will be seen from Table II that arsenic-76, mercury-203 and selenium-75 were completely vapourised at 400° C, whereas, the rest of the elements showed no significant losses but vapourised partially at 700° C.

In wet digestion, the obvious advantage is that the decomposition of complex molecules is achieved at lower temperatures than during dry ashing. Under the strong acidic condition most of the organo-metallic complexes dissociate readily. Therefore for volatile elements like ruthenium, selenium, etc., there can be no acceptable dry ashing procedures. The retention of these elements is likely to be increased by wet ashing. It will be seen from Table III, which records the losses on wet ashing, that mercury-203 was completely lost in all oxidising liquids. Selenium-75 losses in oxidation with pure nitric acid were only 10-15% whereas in the other mixtures, the losses were significantly greater extending almost to 99% for the combustion mixture. These significant losses in presence of stronger oxidising agents like H₂O₂ and HClO₄ may be either due to formation of more volatile per or higher oxy-compounds or may be the effect of high temperature sublimation of selenium dioxide in the latter case⁶. The less losses in pure nitric acid alone are understandable, since selenium dioxide is reported to be easily prepared by dissolving

TABLE III

Per cent recoveries of trace elements by wet digestion

Oxidising liquids	Volume used	Per cent recoveries							
		Cr-51	Hg-203	Sb-124	As-76	Se-75	Ru-106	W-187	
HNO ₃	.. 100 ml	100.0	0.9	87.0	75.6	88.9	86.9	100.0	
		100.0	2.6	..	79.2	84.5	..	100.0	
HNO ₃ : H ₂ O ₂ (1:1 v/v)	.. 100 ml	100.0	3.0	95.0	89.3	26.7	84.0	98.5	
		100.0	5.9	..	92.4	32.6	83.7	..	
		93.5	4.6	
Combustion mixture	.. 100 ml	90.8	20.4	86.2	98.3	0.4	..	100.0	
		100.0	24.4	91.1	..	1.0	..	97.8	
HNO ₃ : H ₂ SO ₄ : HClO ₄ : H ₂ O (375: 450: 150: 25)	100.0	20.8	0.6	

TABLE IV

Per cent recoveries of trace elements by wet digestion under reflux

Oxidising liquids	Volume used	Per cent recoveries					
		Cr-51	Hg-203	Sb-124	As-76	Se-75	Ru-106
HNO ₃	.. 50 ml	100	90.3	100	97.1	95.0	100
HNO ₃ : H ₂ O ₂ (1:1 v/v)	50 ml	100	93.5	..	100	89.4	100
Combustion mixture HNO ₃ : H ₂ SO ₄ : HClO ₄ : H ₂ O (375: 450: 150: 25)	50 ml	100	91.3	100	100	94.2	100

selenium in concentrated nitric acid and evaporating to dryness⁷. The recoveries observed in the case of all other elements studied were reasonably good. The results obtained by digestion with different oxidising liquids under reflux are presented in Table IV. It can be seen that all the elements studied were quantitatively recovered under these conditions.

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From these studies, it is evident that dry ashing even at low temperature (400° C) should be avoided especially for extremely volatile elements like mercury, selenium and arsenic. However, dry ashing the biomaterial in a stream of electrically excited oxygen at low temperatures not exceeding 100° C may be usefully carried out where large quantities of environmental samples have to be handled for radiochemical analysis. Wet digestion

under reflux is most suited for the volatile elements like the ones studied here, as losses under these conditions are found to be significantly low.

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1. Strohal, P., Lulic, S. and Jelisaveic, *Analyst*, 1969, 94, 678.
2. Gorsuch, T. T., *Ibid.*, 1959, 84, 135.
3. —, *Ibid.*, 1962, 87, 112.
4. Pijcks, K., Hoste, J. and Gills, J., *Proc. of the International Symposium on Microchemistry*, Pergamon Press, Oxford and New York, 1958, p. 48.
5. Doshi, G. R., Sreekumaran, C., Mulay, C. D. and Patel, B., *Curr. Sci.*, 1969, 38, 206.
6. Kleinberg, J., Argersinger, W. J. (Jr.) and Griswold, E., *Inorganic Chemistry*, Heath and Co., Boston, 1960, p. 1.
7. Gould, E. S., *Inorganic Reactions and Structures*, Holt, Rinehart and Winston, Inc., New York, 1955, p. 1.