

was influenced most by chloride followed by water temperature and primary productivity. Chloride, which has been regarded as an index of organic pollution¹⁷, was recorded in higher concentration in water bodies polluted by community sewage^{7,8,18}. Overall population of rotifers was more both in number and taxa in polluted zone probably due to availability of plenty of bacteria on which they feed^{15,16}. An abundance of *B. angularis* and *B. calyciflorus* was reported from water showing higher chloride by others^{13,14}. These assertions support the present findings that rotifers may be used as potential bio-indicators for monitoring more of pollution than productivity.

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Estimation of arsenic in geological samples by atomic absorption spectrometry

R. K. Tiwari, V. K. Tarsekar and V. W. Khandan

Chemical Division, Geological Survey of India, Nagpur 440 006, India

An indirect method for estimation of arsenic is described. Arsine liberated from sample solution is passed through dilute silver nitrate. The precipitated silver arsenide is filtered, washed with ammonium hydroxide and dissolved in concentrated nitric acid. The amount of silver proportional to arsenic is determined by atomic absorption spectrometry. The method is simple and accurate.

DIRECT determination of arsenic by atomic absorption spectrometry is not accurate and precise due to matrix interferences¹. Hydride generation technique also cannot be applied to samples which contain arsenic in percentage levels. The life of the hollow cathode lamps of arsenic being short² the electrodeless discharge lamp requires a separate power supply³. The resonance line of arsenic (193.7 nm) is nearer to vacuum UV range and therefore difficulties arise from the strong absorption of light by air in the light path and from the flame gases⁴.

Though methods like conventional ICPAES, flow injection/hydride generation ICPAES and nondestructive

activation analysis can be used to determine arsenic in minor/major levels in geological samples, sophisticated instruments are required. Isolation of arsenic from the matrix is essential for accurate determination. There are several ways of isolating arsenic from matrix like solvent extraction^{5,6}, coprecipitation⁷⁻⁹ and widely used arsine generation^{10,11}. These techniques require special reagents and equipment. Arsine is passed through a dilute solution of silver nitrate, the precipitated silver arsenide washed with ammonium hydroxide and the silver in silver arsenide determined by atomic absorption spectrometry.

The reagents used were: 20% KI solution, 1% SnCl₂ (AR grade) in HCl, 1.5% AgNO₃ (AR grade), saturated bromine water, zinc pellets (AR grade), 2% ammonia (AR grade) solution and potassium pyrosulphate (AR grade). Silver nitrate (0.787 g) was dissolved in 50 ml of deionized water diluted to 1 litre with 1% (v/v) HNO₃ and preserved in an amber glass bottle. The resulting solution weighed 500 µg/ml. Standard silver solution of 0.2, 0.5, 1.0, 2.0, 3.0, 4.0 and 5 µg/ml was prepared by simple dilution of stock solution (500 µg/ml) maintaining 1% nitric acid concentration.

Digestion of sample and evolution of arsine

(i) *For sulphide samples*, one gram of sample finely ground to -200 mesh is taken in a 100 ml beaker, and 5 ml of bromine water and 100 ml of concentrated nitric acid are added, the beaker is covered and the water bath kept for an hour, the cover removed and allowed to

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evaporate. The process is repeated with further addition of 5 ml of conc. HCl.

(ii) *For rock samples*, 0.2 gram of samples is taken, mixed with about 1 g of potassium pyrosulphate in the test tube and fused thoroughly. The mass is extracted by adding 5 ml of conc. HCl and about 30 ml of DM water and heated. The resulting solution is transferred to 100 ml volumetric flask and the appropriate aliquot is taken so that arsenic content is below 2000 µg for the experiment. One ml of KI solution is added and left for about 5 min. SnCl₂ solution is added in drops to decolorize the solution. The volume is adjusted to 40 ml maintaining 2 molar acid concentration. AgNO₃ solution (3 ml of 1.5%) is taken in a test tube of 2.5 cm dia and 25 cm long. The solution is diluted to about 75 ml with DM water. Zinc pellets (5 g) are added to the sample solution in bottle and the bottle tightly covered and the arsine passed through silver nitrate solution with the help of passing tube for 50 min. The solution is filtered through fine G-4 sintered crucible using suction pump. The precipitate is washed with 2% ammonia solution until it is free from silver, the test tube washed and passed with 2% ammonia solution. This solution is passed through the sintered crucible. The precipitate is washed twice with demineralized water and the filtrate discarded. The precipitate of silver arsenide adhering to the wall of test tube, the passing tube and the sintered crucible is dissolved with the addition of 2 to 3 ml of concentrated nitric acid and the resulting solution is collected in a 100 ml volumetric flask. The sintered crucible is washed thoroughly with DM water and the solution made up to a known volume. Silver is determined by direct aspiration or after appropriate dilution with AAS in air acetylene flame

Interference due to matrix elements like Cu, Ni, Co in the sample can be eliminated by adding 5 ml of 0.01 M EDTA solution to the sample solution before evolution of AsH₃ (ref. 12). The interference of slow evolution of AsH₃ by the above matrix elements can also be avoided by increasing the acid concentration to 5 M HCl (ref. 13). Hydride-forming elements like antimony (Sb) do not interfere up to the concentration of 2000 µg/g whereas elements like Sn and Bi have no effect on the results obtained.

Table 1. Results of arsenic determination using pure arsenic solutions

Sample	As taken (µg)	As found (µg)	Recovery (%)
1	100	102	102
2	150	150	100
3	200	203	101.5
4	300	305	101.6
5	500	504	100.8
6	1000	1008	100.8
7	1500	1510	100.6
8	2000	2022	101.1
9	3000	3026	100.8

Table 2. Rock sample free from arsenic and standard arsenic solution addition

Sample no	As added (µg)	As found (µg)	Recovery (%)
1894 (41) Bed rock	100	100	100
1894 (42) Bed rock	150	153	107
1894 (46) Bed rock	200	208	104
1894 (4) Bed rock	250	255	102
1894 (56) Bed rock	300	308	102.6
1894 (59) Bed rock	350	308	101
1894 (60) Bed rock	500	510	102
1894 (61) Bed rock	1000	1021	102
1894 (62) Bed rock	1500	1525	101.6
1894 (70) Bed rock	2000	2044	102

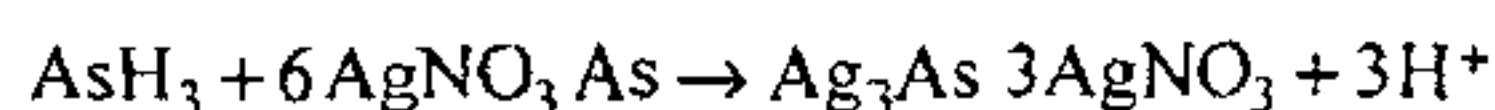
Table 3. Comparison of arsenic values in geological samples with classical wet chemical method (distillation as arsenous chloride and its iodometric titration)

Sample no	Classical method	Present method
2016 (13) Trench	1.84%	1.88%
2052 (87) Core	3.24%	3.26%
2332 (18) Channel	5.58%	5.69%
2332 (8) Channel	6.15%	6.28%
2332 (15) Channel	9.25%	9.47%

Table 4. International standard samples for arsenic analysed by present method

Sample no	Certified value	Present method
IGS 42	0.04%	0.04%
IGS 43	0.11%	0.10%
MP-2	0.20%	0.21%
MP-19	0.84%	0.83%

Results for arsenic (Table 1 to 3) are within experimental errors. Results obtained by wet chemical analysis and by the proposed method (Table 3) show very good agreement. When arsenic present in the sample is less than 50 µg/g the precipitate is less and the results are not reliable. In such cases arsenic is determined by addition technique, i.e. 100 µg of arsenic should be added to the sample and after obtaining the final value, subtract 100 µg from the final value to get the required arsenic in the sample. The experiments show that the values are reliable up to tens of micrograms. When arsine is bubbled through concentrated solution of silver nitrate, the following reaction takes place.



Ag₃As-3AgNO₃ complex breaks with 2% ammonia and AgNO₃ molecule detached from Ag₃As molecule and goes into solution, where Ag₃As (silver arsenide) remained insoluble as black-grey precipitate. In this reaction one arsenic replaces 3 silver atoms. Thus the value obtained should be divided by 4.31 to get the amount of arsenic present.

Table 5. Standard deviation and coefficient of variation of each sample analysed*

First	Second	Third	Fourth	Fifth	Mean	S D	C V
102	108	106	109	111	107	3.43	3.23
215	203	212	208	201	207	6.20	2.99
508	513	515	514	505	511	4.30	0.84
1009	1042	1033	1030	1021	1027	12.54	1.22
2029	2051	2008	2015	2032	2027	16.65	0.82

* Concentration in ppm.

The silver present in solution should be in excess of arsenic to complete the precipitation of silver arsenide. When arsine is bubbled through silver nitrate solution only a part of silver nitrate is utilized for the reaction between arsine and silver nitrate as the contact time is short. For example, 200 µg of arsenic required about 8620 µg of silver for precipitation but the silver in the solution should be approximately 30,000 µg to complete the precipitation. It is clear from Table 4 that the values obtained by the present method are in good agreement with the certified values of international standards. From Table 5 it can be seen that coefficient of variance is higher in the sample containing low arsenic. But samples containing more than 200 ppm of arsenic show low coefficient of variance, confirming the method to be precise and accurate for samples containing higher arsenic.

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Prehistoric flood deposits on the Choral River, Central Narmada Basin, India

Vishwas S. Kale, Sheila Mishra*, V. R. Baker**, S. N. Rajaguru*, Yehouda Enzel** and Lisa Ely**

Department of Geography, University of Poona, Pune 411 007, India

*Department of Archaeology, Deccan College, Pune 411 006, India

**Department of Geosciences, University of Arizona, Tucson, USA

We report here a 5000-year record of large floods on the Choral River in central Narmada Basin. Geomorphic, stratigraphic and hydraulic studies provide evidence of floods with peak discharges greater than 4500 m³ s⁻¹ in the last 5000 years. The flood record from the Choral River is the longest yet discovered from any river basin in India.

RECENT advances in palaeoflood techniques have made it possible to reconstruct chronologies of large floods in bedrock channels using slack-water deposits and other

palaeostage indicators¹. Slack-water deposits consist of silt or sand that generally settle rapidly in backwater locations during large floods¹. These flood sediments selectively preserve records of large floods spanning 10² to >10³ years². Such deposits have been investigated in the central Narmada basin and its tributary, the Choral River.

The site containing prehistoric flood records is located near Barjar (22°21'49"N and 76°02'54"E) on the Choral River (Figure 1a). The river in this reach has developed a small canyon in Vindhyan Quartzites. At several locations, sequences of fine-grained sandy flood deposits have been preserved on the channel margins. Excavation of these deposits and stratigraphical studies revealed at least seven flood units separated by scree deposits, slope wash and charcoal (Figure 1b). Sand and debris from floods were observed in narrow crevices along both the canyon walls, up to 5.9 m above the top of slack-water deposits. A radiocarbon date on charcoal collected from the lowest flood unit of the section is 5170 ± 135 yr BP (δ¹³ C(%) = -27.1; A6859), implying that the overlying flood units were emplaced in the last 5000 years. The occurrence of scree and slope wash