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# ESTIMATION OF SULPHIDE IN LAKES/RIVERS WATER AND INDUSTRIAL EFFLUENTS

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A colorimetric method has been developed for the determination of micro-gram amount of sulphide in water. The method is based on staining small strips of filter papers dipped in a concentrated solution of lead acetate and the formation of lead sulphide due to the evolution of the  $H_2S$  from the samples. The method was successfully applied for the micro-estimation of sulphide in water samples of the Kabul river, Kheshki lake and Industrial effluents. The detection limit was as low as 0.01  $\mu$ g/ml and the effect of the interfering radicals on the method are also assessed.

Key words: Sulphides; Water; Effluents.

# INTRODUCTION

Sulphide is often present in ground water, especially in hot springs. Its common presence in waste water comes partly from the decomposition of organic matter, sometimes from the Industrial waste, but mostly from the bacterial reduction of sulphate. The threshold odour concentration of H<sub>2</sub>S in clean water is between 0.025 – 0.50  $\mu$ g/ml. H<sub>2</sub>S is very toxic and has claimed the lives of numerous workers in sewers.

The water of the Kabul river and the Kheshki lake has long been used for irrigation purposes. However, with the setting of industries in Amangarh near Nowshera in mid 1950's the river provided an easy means for the disposal of effluents containing chemicals, some of which besides having deleterious effects on aquatic life have led to the decline of the fish fauna due to the high concentration of sulphide and C.O.D. (Chemical Oxygen Demand).

The determination of sulphide and sulphite in anoxic water remains a challenging excercise because of the ease with which both ions are oxidized by air and because of interferences, notably by other reducing agents [1]. Attempts have been made in the past for the determination of trace amounts of sulphides in water and effluents [2-6].

Efforts were therefore made to develop a colorimetric method using a simple appartus for the determination of sulphides in continuation of our earlier work on the titrimetric determination of sulphide in the presence of other sulphur compounds [7].

## EXPERIMENTAL

Solutions of lead acetate, zinc acetate, sodium sulphide, and sulphuric acid were prepared according to standard methods, and kept in airtight bottles for accuracy

To one litre standard sodium sulphide solution, 10 ml of concentrated sulphuric acid were added, and boiled for 1 hr. Concentrated lead acetate solution soaked paper was used for the detection of lead sulphide. Staining was undertaken in the range 0.01-2.0 mg/ml. The analysis was made by standard methods.

#### **RESULTS AND DISCUSSION**

Precision and limits of detection. The precision of the preposed method was assessed through repeated determination of sulphides. The results were calculated from the intensity of the stains produced on the filter paper strips. These results are shown in Fig. 1. The limits of detection were up to the concentration of 0.01  $\mu$ g/ml, of sulphide.

*Interference.* The effects of various ions on the estimation of sulphide was studied and the results are shown in Table 1. An attractive feature of the method is its relative



Fig. 1. Standards for the estimation of sulphide (µg/ml.).



Fig. 2. Apparatus for the estimation of sulphide.

freedom from interference of sulphite, thiosulphate and sulphate ions in hundredsfold excess.

Sampling and storage. The samples were collected with minimum aeration. 4 drops of 2 N Zinc acetate solution per 100 ml sample, were added to the sample to preserve it for sulphide determination. The bottles were filled completely and were well stoppered.

Table 1. Effect of foreign ions.

Concentration of sulphide					
Added (µg/ml)	Found (µg/ml)	Foreign ions added	Concentration		
2	2		(µg/ml)		
	•	${\stackrel{-2}{\rm SO}}_4, {\stackrel{-2}{\rm S}}_2 {\stackrel{-2}{\rm O}}_3, {\stackrel{-2}{\rm SO}}_3$	1000		
2	1.5	Fe <sup>+2</sup> , Fe <sup>+3</sup> , Mn <sup>+2</sup> , Cr <sup>+3</sup>	100		
2	2	$NO_2^{-1}$ , $I^{-1}$ Mg <sup>+2</sup>	1000		
2	2	$Ca^{+2}$ , SCN <sup>-1</sup> , $C_5H_5O_7^{-3}$	500		

Table 2. Estimation of Sulphide\* in Water/Effluents.

Local collection	Sample Date No.		(Sulphide µg/ml.)		Error
angener di gang bendar sa Price sa iga di Kalendar mengena kara sa kara sa ing	a false - Latin		Standard <sup>(2)</sup> method	Present method	
Drain of N. Tanneries across the Railway bridge		30th Apr, 1975	1.00	1.00	0.00
100 feet down stream to the	. 1	4th Nov. 1974	2.00	2.00	0.00
confluence of Kabul river and drain.	2	10th Dec. 1974	0.58	0.50	- 0.08
Confluence of the Kheshki	1	15th May 1975	0.87	1.00	+ 0.13
lake and incomming stream	2	20th Oct. 1975	0.85	1.00	+ 0.15
이가 가장 가지 않는 것이 같아요. 가장 2011년 - 111년 1월 1971년 - 111년 1월 1971년 1월 1971년 1월 1971년 1월 1971년 1월 1971년 1월 1971년 1월 19	3	13th May, 1975	0.66	0.50	- 0.16
Middle of the lake near the	1	15th May, 1975	1.00	1.00	0.00
P.A.F. Boat club	2	20th Aug. 1975	1.10	1.00	- 0.01
	3	2nd Nov. 1975	0.85	1.00	+ 0.15
Channel outlet of the lake used	1	15th May, 1975	0.95	1.00	+ 0.05
for Irrigation	2	20th Oct. 1975	0.85	1.00	+ 0.15

\*Mean of three determinations.

Analysis of rivers/lake waters and industrial effluents. Samples of water and effluents collected periodically from different locations were analysed for the sulphide contents using the foregoing procedure. To check the validity of the method, observations recorded were then compared with the results achieved by using standard method [1]. These observations are shown in Table 2.

# CONCLUSION

The presence of sulphide in rivers, lakes and potable waters has not only caused environmental pollution but also has exercised a deleterious effect on fish and other aquatic life. The present method would be an easy mean to examine the samples of water for sulphide contamination collected periodically from various locations and the drains through which the effluents of industries are indiscriminately discharged into rivers and lakes.

a process for its partification. Oxidation procedure involving the conversion of ferrous from acto ferric state by sunside oxidants life air, nitric acto, radiogen peroxide and persubhare has been tried.

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. Hydroxyfamine hydrochiwins (A.R. 25% w/v) S. welline qualate 0.5% (Fig. said/250 ml. HyO) F. welline disk solution f0 from 100 ml. of 0.21

(1)+

4 0.1% Potassium (transferie) (1.825 prepared from undar grade potestium dich) ornite and standardised against oure area [10, 11], with

Diphenviatame inducator 12 at suppling acta.
Statistics chloride Disserved 2g SuC<sub>1</sub> 2H<sub>2</sub>O crystals in hot cone hydrochloric actal and made up to one little.

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1% stock solution of commercial grade antim was prepared and A1(O<sub>3</sub> Fe<sub>2</sub>O<sub>3</sub>, 1eO, SO<sub>3</sub> and SIO<sub>2</sub> were alterationed both by conventional as well as spectrophotometric methods. The alkeli metals were determined by fluxes photocratic calcuest are inaccession to complexe metric method and water of crystallisation was found where heating at 200°. The results are shown in Table 1

# REFERENCES

- 1. S. Rama Bhat, J.M. Eckert, R. Geyer and N.A. Gibson, Anal. Chim. Acta., **108**, 293 (1979).
- Standard Methods for the Examination of Water and Waste Water, Am. Pub. Health Assoc., Washington, D.C., 16th ed., p. 470 (1985).
- D.J. Hawke, L. Anthony, D.M. Martinson, M. Slater, G. Philip and E. Charlis, Analyst, 110, 269 (1985).
- 4. S. Yanova, Khig, Zdrave Opaz, 26, 429 (1983).
- 5. G.J. Shahwan and E.M. Heithmar, Spectrose, Lett, 17, 377 (1984).
- 6. Y. Xyefeng and X. Xianyun, Uuanjing Kexue, 5, 47 (1984).
- K. Khan, M. Amin and R.A. Shah, Pakistan J. Sc. Ind. Res., 26, 79 (1983).
- 8. Official, Standardized and Recommended Method of Analysis, Soc. Anal. Chem. London, p. 460-65 (1973).

sump of paper 4i is used its a congutent and precipitating agent in sewage treatment faisation [1] is a known as filter atomic, beartes other common names a.g. pickle along cake along and palent stam [3] to prave used for setting dyers [4], the white burlets of stamma is used for dyers pravity pravity for device [4].

The process used for aluminum implanta production as well eachisted and fauly standard among groduluts because of simplicity in 1946 eight muncipal plants produced 12,000 toos of alum for direct use in water purification in the USA in [953 seven plants produced [4,000 toos which is test fract [9] of toos futurginith subplate used in water treatenent [9].

Extensive investigations [6], are an exort on the native and properties of aluminum sulphate and hydroxide from atumna raits. Various hyperness condensation or polymerization products, and a number of cryntalication torms and diansity mineral have been proposed troud time to time [7]. One reason for such diverse views on the subject is due to the widely varying properties of the precipitate with regard to the nature of associated inputs ties, degree of hydration, adsorbability and subjitity [8].

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