

A NEW TURBIDIMETRIC METHOD FOR THE DETERMINATION OF SULPHATE IN BRACKISH WATERS

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A new turbidimetric method for the determination of sulphate in brackish waters is described. A suspension of barium sulphate is produced in an acidified medium of glycerine d-glucose solution by the addition of barium chloride solution and the turbidity measured with the help of Bausch & Lomb Spectronic '20' Colorimeter. Concentrations upto 160 ppm of sulphate, expressed as Na_2SO_4 , can be directly measured by this method. Phosphate, nitrate, and chromate ions do not interfere, nor do Ca^{2+} , Fe^{3+} , CO_3^{2-} , Ni^{2+} , and Cr^{3+} . The method has been applied to the determination of sulphate in desert soils as well.

The determination of sulphate in brackish waters of the arid regions of Rajasthan is of importance in connection with the survey of water resources of these regions. Various methods exist for the estimation of sulphate. New methods¹ of sulphate estimation (spectrophotometric, indirect flame photometric, nephelometric and chelatometric titration) were reported during 1957-60. Many of them are either time consuming or require complicated apparatus.

Chaudhuri and Purohit² used a medium consisting of glycerine and d-glucose for the phototurbidimetric determination of chloride in brackish water. It was thought that a similar medium might be used for the estimation of sulphate also. Investigation showed that the method gave reliable results³. The method is rapid and well suited for the determination of sulphate in brackish water and desert soil.

MATERIAL AND METHOD

Apparatus—Bausch and Lomb Spectronic '20' Colorimeter.

Materials—Glycerine, *BDH Analar*; d-glucose, *BDH Analar*; barium chloride, *A.R.* (5% solution); sodium sulphate, *BDH Analar* and hydrochloric acid, *A.R.*

Procedure—A solution with the following composition (w/w) was prepared by warming: Glycerine, 50%; d-glucose, 10%; distilled water, 37.5% and hydrochloric acid, 2.5%. The solution was kept overnight. It will be referred to as G-G solution.

Bausch and Lomb Spectronic '20' Colorimeter was set at 450 $m\mu$ to give 100% transmission for the 'blank', which consists of 1 cc. barium chloride solution (5%) and 3 cc. of G-G solution.

A suitable volume, say Z cc., of G-G solution was taken in a test tube to which was added V cc. of brackish water, so that $Z+V = 10$ cc. The solution will be referred to as 'mixed' solution.

3 cc. of the mixed solution was added to 1 cc. of barium chloride solution contained in a 10 cc. spectronic tube, stirred with a glass rod for 2 minutes and set aside for 2 min. or more. The percentage transmission (%T) of the resulting barium sulphate suspension

was measured. The concentration of sulphate, in terms of ppm of sodium sulphate, was read against %T from a standard curve (Fig. 1). The sulphate concentration (Y) of brackish water was calculated in terms of sodium sulphate ppm from the equation:

$$Y = \frac{10 A.X.}{V}$$

where X = ppm as read from the standard curve against %T; A = number of times the brackish water is diluted prior to addition to G-G solution; and V = volume of brackish water or sulphate solution added to G-G solution.

Standard Curve—A standard curve was prepared with sodium sulphate solution using G-G solution as the suspending medium. Fig. 1 shows the percentage transmission with increasing concentration of sodium sulphate. It is seen that the slope of the curve CB changes at B and assumes a new linearity. Experimental results with various water samples have shown that both CB and BA are valid and can be used for their respective concentration range. The linearity breaks down beyond C .

Effect of Temperature—The effect of temperature on percentage transmission (%T) is shown in Fig. 2. It will be seen that %T remains unaffected during the temperature range 20°–35°C. Below 20°C, it decreases and beyond 35°C, it tends to increase with increase of temperature.

Effect of Wave Length—Fig. 3 gives %T through a suspension produced by 1 cc. of sodium sulphate solution (1000 ppm) in G-G solution when the wave length is varied from 375–550 $m\mu$. It will be observed that the percentage transmission increases linearly with increase in wave length. A wave length of 450 $m\mu$ has been chosen for the determination reported in this paper.

Stability of Barium Sulphate Suspension—The percentage of transmission through barium sulphate suspension produced by 0.5, 1.0 and 1.5 cc. of sodium sulphate solution (1000 ppm) remains unaltered between 3 and 30 minutes. During the first 2 minutes (after initial stirring) %T was found to be higher than the constant value by unity.

Influence of other electrolytes—The influence of phosphate and nitrate ions on the determination of sulphate by the turbidimetric method was investigated. The results showed

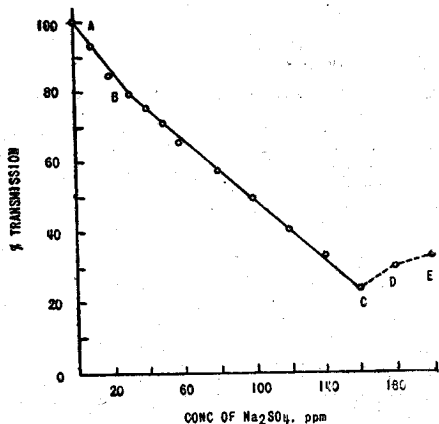


Fig. 1—Percentage transmission with increasing concentration of Sodium Sulphate.

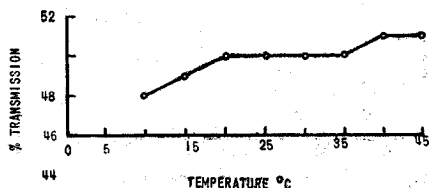


Fig. 2—Effect of temperature on transmission (%T).

TABLE I
SULPHATE CONTENT OF LUNI* BED SOILS

Sulphate content (Gravimetric method) (ppm)	Sulphate content (Turbidimetric) (ppm)
288	300
715	650
4190	4,080
5538	5,600

*Luni is a seasonal river which runs through the district of Barmer (Rajasthan). The samples were collected during the dry season.

that phosphate does not affect the determination of sulphate upto 1500 ppm and nitrate, upto 2000 ppm.

The limits up to which various coloured ions do not interfere with the determination of sulphate were found to be as follows: CrO_4^{2-} , 240 ppm; Cu^{2+} , 1500 ppm; Ni^{2+} , 400 ppm; Fe^{3+} , 200 ppm; Cr^{3+} , 520 ppm, and CO_3^{2-} , 500 ppm.

Sulphate of Salt Solution—The sulphate contents of various salt solutions were determined by the turbidimetric method and compared with the values obtained by the standard gravimetric procedure. The results obtained are given in Table 1. The method is quick and can be adapted for commercial analysis and technical control.

Brackish water samples obtained from various parts of Barmer district, Rajasthan were analysed for the sulphate content by gravimetric as well as phototurbidimetric methods. The results of fifty samples were subjected to statistical analysis.

Paired comparison of values for 20 samples ($\%T > 80$) by the two methods, namely gravimetric and phototurbidimetric, showed that the estimated standard deviation of differences was 9.2. This is not significant; the values of t and P were respectively 0.94 and 0.05.

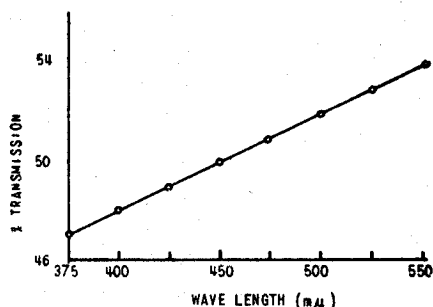


Fig. 3—Variation in $\%T$ with change in wave length (375—550 $m\mu$).

Similarly paired comparison of values for 30 samples ($\%T < 80$) by the two methods gave the estimated standard deviation of differences as 19.7. This is not significant; the values of t and P were respectively 0.61 and 0.05.

In order to further compare the estimate of standard deviation s_1 by the gravimetric procedure with the other estimate s_2 by the turbidimetric method, the F value was determined and found to be 1.24 for 10 d.f. The value of F at $P=0.95$ is 2.98 for $n-1=10$, which shows that there is no real disparity between the two estimates.

TABLE 2

DETERMINATION OF SULPHATE BY GRAVIMETRIC AND TURBIDIMETRIC METHODS

Salt	Concentration of Cation in solution (ppm)	Sulphate value (calc. as Na_2SO_4)	
		Gravimetric (ppm)	Turbidimetric (ppm)
$MgSO_4 \cdot 7H_2O$	176.6	1,041	1,020
$3CdSO_4 \cdot 8H_2O$	873.0	1,113	1,060
$CaSO_4 \cdot 2H_2O$	277.5	994	980
$FeSO_4 (NH_4)_2 SO_4 \cdot 6H_2O$	201.0	1,034	1,040
$K_2SO_4 \cdot Al_2 (SO_4)_3 \cdot 24H_2O$	90.5	1,000	1,018
$CuSO_4 \cdot 5H_2O$	551.3	1,248	1,220
$CoSO_4 \cdot 7H_2O$	469.6	1,149	1,110
$NiSO_4 \cdot 6H_2O$	410.0	1,003	1,020
$Fe_2(SO_4)_3 (NH_4)_2 SO_4 \cdot 24H_2O$	214.7	1,068	1,040

Sulphate in Sea Water—The sulphate in sea water collected from near Bombay was determined by gravimetric and turbidimetric methods. The sample was diluted 4 times and 1 cc. of the diluted solution was used for the determination. The values (calculated as Na_2SO_4) obtained by the gravimetric and turbidimetric methods were respectively 4157 and 4160 ppm.

Sulphate in Desert Soils—Twenty samples of desert soils were collected from the bed of Luni river* (over a stretch of 70 miles). Three soil samples were collected from freshly ploughed agricultural fields at Jodhpur.

Soluble sulphate was extracted by shaking the soil as received (100 gm) with distilled water (250 cc.) for 1 hour in a mechanical shaker.

Sulphate was determined in the extract by the turbidimetric method. Of the 23 samples examined, only four contained sulphate; three samples from agricultural fields were free from sulphates. Their values were compared with those obtained by the gravimetric method. The results of analysis are given in Table 2.

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