

# Flow Solution™ FS3700 Automated Chemistry Analyzer

Sulfate by Flow Injection Analysis (FIA) and Photometric Detection USEPA 375.2 Cartridge Part Number 331386CT

### Scope and Application

This method is used for the determination of sulfate in drinking water, surface water, and domestic and industrial waste according to USEPA method 375.2 and Standard Methods 4500–SO<sub>4</sub><sup>2-</sup> G. 1,2

#### **Method Performance**

Range	1 mg/L – 25 mg/L*	
Rate	40 samples/hour	
Precision	<2% RSD at mid-range	
Method Detection Limit	0.1 mg/L	

<sup>\*</sup>The range may be extended to analyze other concentrations. Analysis of sulfate in ranges up to 300 mg/L will require a different pump tube configuration, smaller sample loop, and a third-order curve fit. Contact OI Analytical for assistance.

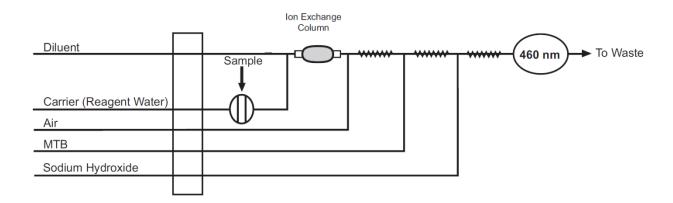


Figure 1. General flow diagram for Sulfate by USEPA 375.2.

#### **Reagents and Calibrants**

Chemical Name	CAS#	Chemical Formula	Part Number
Ammonium chloride	12125-02-9	NH₄CI	
Ammonium hydroxide	1336-21-6	NH₄OH	
Barium chloride dihydrate	10326-27-9	BaCl <sub>2</sub> 2H <sub>4</sub> O <sub>2</sub>	
Bio-Rex <sup>™</sup> 70 Resin, 50–100 dry mesh size, sodium form	81133-22-4		
Brij®-35 (21% Solution)	9002-92-0	(C <sub>2</sub> H <sub>4</sub> O) <sub>n</sub> C <sub>12</sub> H <sub>26</sub> O	326126
Chloroform	67-66-3	CHCl₃	
Ethanol, 95%	64-17-5	C₂H₅OH	
Ethylenediaminetetraacetic Acid Disodium Salt Dihydrate (EDTA)	6381-92-6	C <sub>10</sub> H <sub>14</sub> N <sub>2</sub> O <sub>8</sub> Na <sub>2</sub> •2H <sub>2</sub> O	
Hydrochloric acid	7647-01-0	HCI	
Methylthymol Blue Sodium Salt	1945-77-3	C <sub>37</sub> H <sub>40</sub> N <sub>2</sub> Na <sub>4</sub> O <sub>13</sub> S	
Sodium hydroxide	1310-73-2	NaOH	
Sodium sulfate	7757-82-6	NaSO₄	
Water, deionized (ASTM Type I or II)		H <sub>2</sub> O	

## Summary of USEPA Method 375.2

#### Method

Within the pH range of 2.5–3.0, sulfate ions react with a barium-methylthymol blue (BaMTB) complex to form barium sulfate (BaSO<sub>4</sub>) and free methylthymol blue (MTB). The analytical stream is then made highly basic (pH 12.5–13.0). At this pH, the absorbance maximum for the BaMTB complex is 610 nm, while that of free MTB is 460 nm. Given that the molar concentrations of barium and MTB are approximately equal and that the maximum sulfate concentration to be measured does not exceed the concentration of the BaMTB complex, the sulfate concentration is directly proportional to the free MTB concentration measured at 460 nm.

$$Ba^{2+} + MTB^{6-} \xrightarrow{pH \ 2.5 - 3.0} BaMTB^{4-}$$
 $BaMTB^{4-} + SO_4^{\ 2-} \xrightarrow{pH \ 12.5 - 13.0} BaSO_4 + MTB^{6-}$ 

The sulfate calibration curve is nonlinear. Colovos et al. have attributed this to the formation of a binuclear BaMTB complex and to impurities in commercially available MTB dye.<sup>3</sup>

2

#### Interferences

Multivalent cations such as calcium, magnesium, and aluminum are removed with a cation exchange column. Neutralize samples with pH values less than 2 to prevent the elution of cations from the ion exchange resin.

- Filter or centrifuge turbid samples prior to analysis.
- Other compatible procedures for removing or suppressing interferences may be used, provided they do not adversely affect overall method performance.
- Method interferences can be caused by contaminants in the reagents, reagent water, and glassware, which
  may bias the results. Take care to keep all such items free of contaminants.



Figure 4. Sulfate Calibration Series

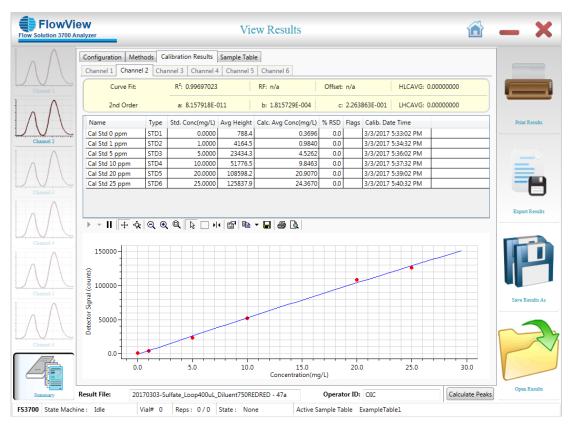


Figure 5. Calibration Curve and Statistics



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