

Sulfide, Standard Methods by Flow Injection Analysis (FIA)

FLOW INJECTION ANALYSIS (FIA) SERIES

Cartridge Part Number: 333176

Channel Part Number: 333175

Scope and Application

This method is used for determining sulfide in drinking water, surface water, and domestic and industrial wastes according to Standard Methods 4500-S2- D. The Method Detection Limit (MDL) is 0.2 µg/L sulfide with an applicable range of 2–5,000 µg/L sulfide. A range of 2–250 µg/L sulfide is achieved using a 200-µL sample loop; using a 100-µL loop attains a range of 50–5,000 µg/L sulfide. The range may be extended to analyze other concentrations by sample dilution or use of a smaller sample loop.

Method Performance

Range	2 - 5000 µg/L
Rate	50 samples / hour
Precision	<1% RSD at mid-point range
Method Detection Limit (MDL)	0.2 µg/L

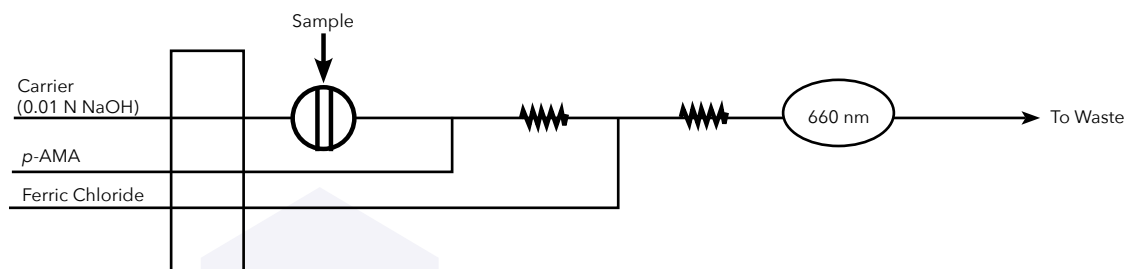


Figure 1. General flow diagram of the FIA system

Reagents and Calibrants

Table 1. Reagents and Calibrants

Chemical Name	CAS #	Chemical Formula	Part Number
p-Aminodimethylaniline (N,N-Dimethyl-1,3-Phenylenediamine Dihydrochloride)	3575-32-4	$C_8H_{12}N_2 \cdot 2HCl$	
Deionized Water (ASTM Type I or II)		H_2O	
Ferric Chloride Hexahydrate	10025-77-1	$FeCl_3 \cdot 6H_2O$	
Hydrochloric Acid, concentrated	7647-01-0	HCl	
Sodium Hydroxide	1310-73-2	NaOH	
Sodium Sulfide Nonahydrate	1313-84-4	$Na_2S \cdot 9H_2O$	
Sodium Thiosulfate Pentahydrate	10102-17-7	$Na_2S_2O_3 \cdot 5H_2O$	
Sulfuric Acid, concentrated	7664-93-9	H_2SO_4	
Zinc Acetate Dihydrate	5970-45-6	$Zn(CH_3CO_2)_2 \cdot 2H_2O$	

Summary of SM 4500-S2-D Method

Sulfide reacts with p-aminodimethylaniline (p-AMA) and ferric chloride to form methylene blue. The absorbance is measured at 660 nm. This method does not detect acid insoluble sulfides. Assure the quality of the analysis through reproducible calibration and testing of the Flow Injection Analysis (FIA) system.

Interferences

Strong reducing agents such as thiosulfate at concentrations above 10 mg/L inhibit color formation. If samples were preserved with zinc acetate and adjusted to pH 9 or higher, the sulfide is complexed as zinc sulfide and coprecipitated with solids.

Samples with background absorbance at the analytical wavelength may interfere.

Turbidity in the sample may interfere. Mark the bottle at the water level. Filter or centrifuge turbid samples prior to preservation with zinc acetate. Allow all solids to settle to the bottom of the bottle. Decant the aqueous portion of the sample to waste, being careful not to discard any solid. Refill the bottle to the mark with 0.01 N NaOH.

Aeration can result in loss of sulfide due to volatilization or reaction with oxygen.

Shake sample solution prior to pouring aliquot and analyze immediately

Figure 2. Sulfide Low Level Calibration Curve

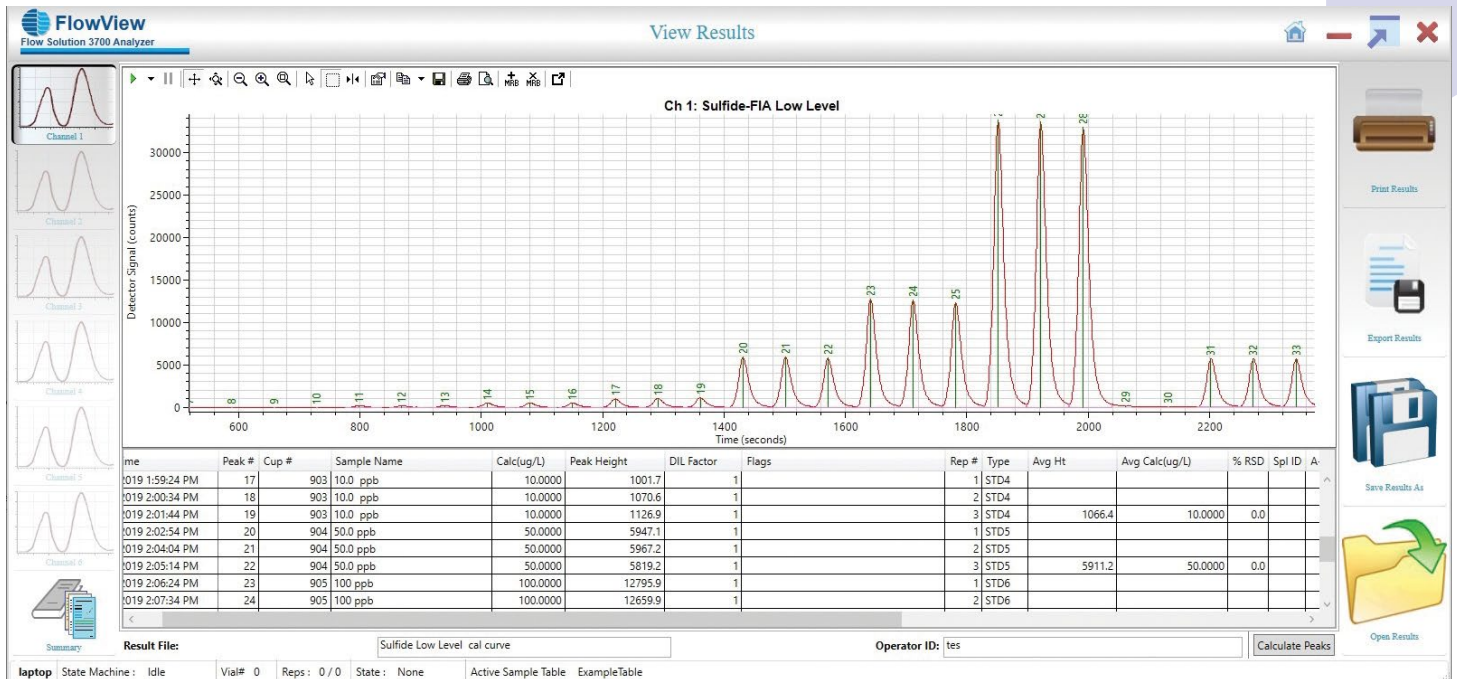


Figure 3. Low Level Calibration Curve and Statistics

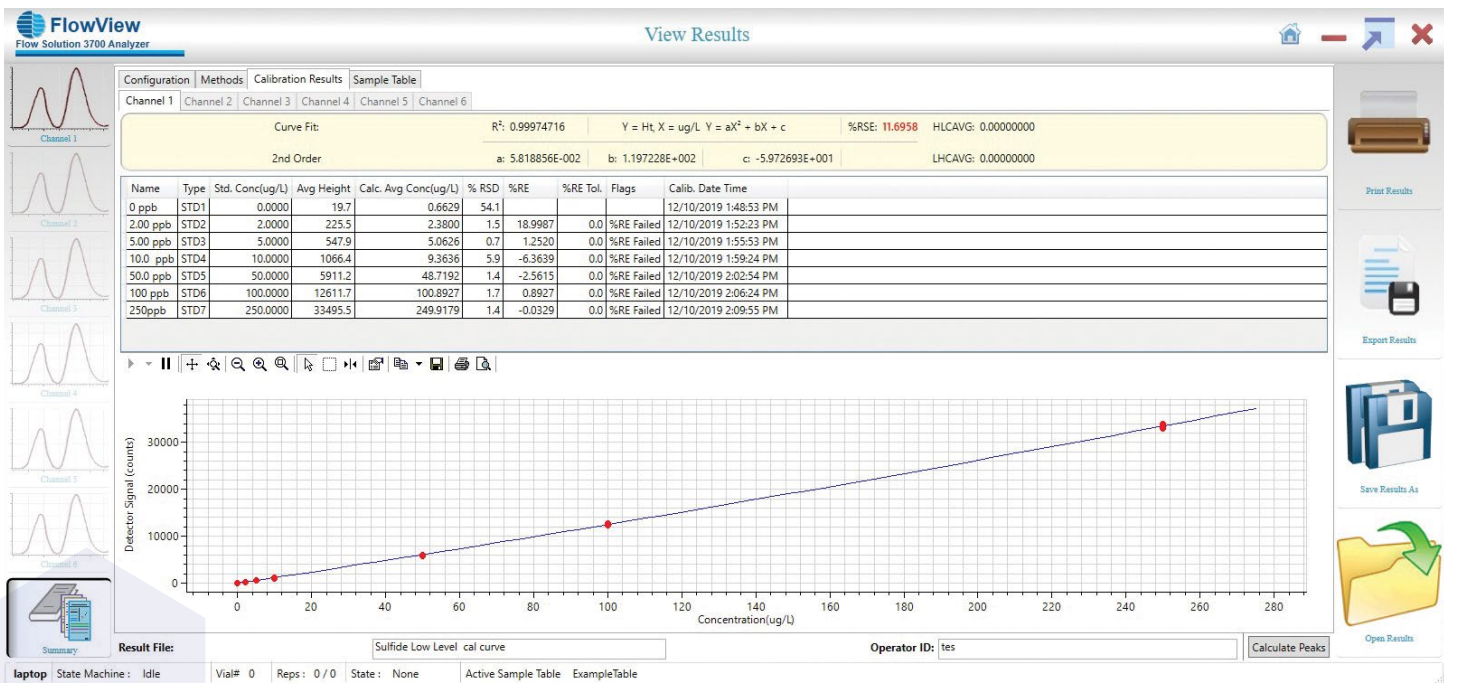
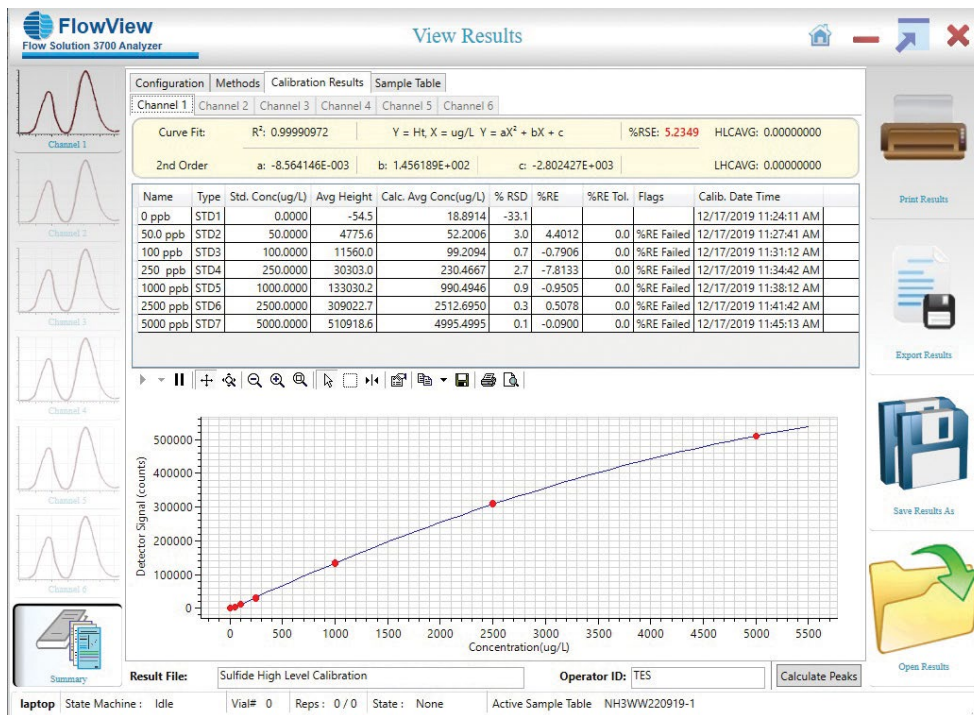


Figure 4. Sulfide High Level Calibration Curve 1



Figure 5. High Level Calibration Curve and Statistics



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