



Application Note AN-I-013

Sulfide in ground and waste water

Fast and economical determination according to ASTM D4658

Sulfide ions are found in ground waters and wastewater derived from both natural sources and industrial processes. Sulfur or sulfate are released into groundwater by processes such as rock weathering. Sulfur-reducing bacteria are then the primary producers of large quantities of hydrogen sulfide by oxidizing organic material with sulfate under anaerobic conditions (in the absence of oxygen or nitrate).

Hydrogen sulfide causes both offensive «rotten egg» odor and corrosion problems (especially when

gaseous) which are even more pronounced at higher temperatures (e.g. hot water). Hydrogen sulfide is toxic, however, normally it is not a health risk at the concentrations found in household tap water. Furthermore, hydrogen sulfide increases the corrosion of water pipes and shortens their lifetime.

To assess the sulfide concentration almost interference free, a fast and inexpensive measurement of sulfide can be performed by using an ion selective electrode.

SAMPLE AND SAMPLE PREPERATION

The method is demonstrated for water samples spiked with different concentrations of sulfide (60, 100, and 240 mg/L sulfide). As sulfide is highly

volatile, it must be preserved under alkaline conditions by using zinc acetate.

EXPERIMENTAL

This analysis is carried out automatically on an 867 pH Module equipped with an Ag/S ion selective electrode, a reference electrode, and a temperature sensor. The sensor is calibrated prior to the analysis. To the prepared sample, sulfide antioxidant buffer is added and stirred for 3 minutes to free bound sulfide. Afterwards, the sensors are placed into the sample and the sulfide concentration is measured.

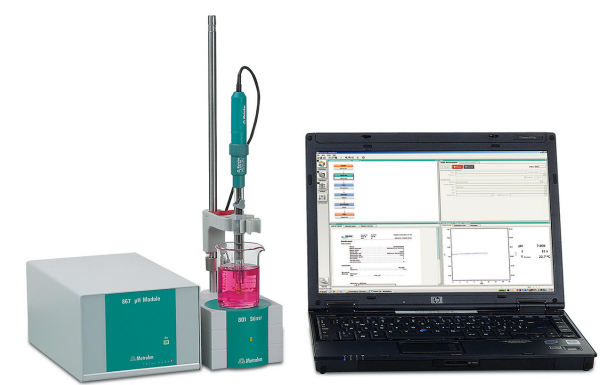


Figure 1. 867 pH Module for precise and reliable ion measurement.

RESULTS

Results are taken after a minimum of 60 seconds when the concentration reading is stable. The

obtained relative standard deviation is smaller than the 15% stipulated by the ASTM standard.

Table 1. Concentration of sulfide in mg/L measured in spiked samples (n = 5).

	Mean S ²⁻ content in mg/L	SD(abs) in mg/L	SD(rel) in %
Sample 1	54.7	2.3	4.2
Sample 2	92.7	4.9	5.3
Sample 3	237.2	1.9	0.8



Figure 2. Example measurement curve of the sulfide content determination.

CONCLUSION

The concentration of sulfide according to ASTM D4658 can be assessed quickly and inexpensively by using ion measurement technique. Concentrations between 0.04 and 4000 mg/L can be determined with the presented technique. Using a fully

automated analyzing system, sample throughput and precision is further increased. The automatic and accurate addition of the solutions save valuable time as the system runs autonomously.

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CONFIGURATION



867 pH Module

Module for pH/ion measurement as supplement to a Titrando or "stand-alone" in combination with a 900 Touch Control.

In addition to measurements of pH, temperature, mV, I_{pol}, U_{pol} and concentration, the pH Module can perform standard additions (manual, dos, autos) and Liquid Handling (add, prep, empty). It enables the use of both conventional and intelligent sensors for measuring. Also integrated in the software is an automatic GLP-compliant pH electrode test.

The pH Module has two USB interfaces for connecting printers, barcode readers or sample changers and four MSB interfaces for stirrers or Dosinos (for the addition of auxiliary solutions or for standard addition).

For use with OMNIS Software, tiamo software, or Touch Control unit. Compliance with GMP/GLP and FDA regulations such as 21 CFR Part 11, if required.



801 Stirrer with stand

Magnetic stirrer including base plate, support rod and electrode holder for supplementing the Titrino plus, Dosimat plus, Titrandos, Sample Processors, 805 Dosimat and 780/781 pH meters as well as the 856 and 867 measuring modules. With permanently attached cable for MSB (Metrohm Serial Bus).

OMNIS

A WHOLE NEW LEVEL OF PERFORMANCE

OMNIS Stand-Alone license

Enables stand-alone operation of the OMNIS software on a WindowsTM computer.

Features:

- The license already includes one OMNIS instrument license.
- Must be activated via the Metrohm licensing portal.



Ion-selective electrode, Ag/S

Silver- and sulfide-selective electrode with crystal membrane.

This ISE has to be used in combination with a reference electrode and is suitable for:

- ion measurements of Ag^+ (10^{-7} to 1 mol/L)
- ion measurements of S^{2-} (10^{-7} to 1 mol/L)
- ion measurements in small sample volumes (minimum immersion depth 1 mm)
- titrations

Thanks to the robust/break-proof plastic shaft made of EP, this sensor is mechanically very resistant.

The polishing set supplied enables easy cleaning and renewing of the electrode surface.



LL ISE reference electrode

Silver / silver chloride reference electrode with double junction system.

This reference electrode is well suited for:

- automated applications
- ion measurements
- surfactant titrations

The ground-joint diaphragm, which is insensitive to contamination, offers a constant and reproducible electrolyte outflow. Additionally, the reference electrolyte is gelified for even better signal stability. The sensor is delivered with $c(\text{KCl}) = 3 \text{ mol/L}$ as bridge electrolyte, which can be freely selected and exchanged as needed.



Pt1000 temperature sensor (installation length 12.5 cm)

Pt1000 temperature sensor (class B) made of glass.

This PT1000 temperature sensor is also available under the article number 6.1110.110 with an installation length of 17.8 cm.