

MEP Application-0589-092015

Sulfur speciation in mining leachates by ion exchange with direct UV detection, using a perchlorate mobile phase.

Branch

Branch 15: Mineral resources

Branch 2: Water, wastewater, air, environmental protection

Keywords

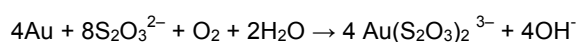
IC / Metrohm 944 Professional UV/VIS Detector Vario/
Metrosep A Supp 5-250/4.0 mm / sulfite, thiosulfate,
thiocyanate, tetrathionate / mining leachates/ leaching
process

Background and purpose:

Health and safety pressures is motivating the gold mining industry to develop a substitute to cyanide for leaching processes. The drive is mainly based on the high toxicity of cyanide.

The chemistry involved in the thiosulfate leaching process is more complex and less robust than the cyanide leaching process, and is therefore more difficult to optimize and more sensitive to operate. The leach process involves a chemical reaction between metallic gold and the thiosulfate anion, with oxygen as the oxidant, ammonium and copper ions as unconsumed catalysts in the reaction.

Thiosulfate forms strong complexes with Au(I), Cu(I), Cd(II), Bi(II), Hg(II) and Fe(III). In the presence of ammonium thiosulfate with oxygen, gold complexes with thiosulfate as follows :



While it's relatively easy, to establish the amenability of an ore to cyanide leaching and optimize the process, the thiosulphate chemistry presents technical challenges and requires careful optimization. Thiosulphate leaching is a sensitive process that requires both dependent and independent optimization of each of the chemical

components of the leach reaction in order to maximize gold recovery and minimize reagent losses.

Summary

The goal of this application is to determine all the common sulfur species, in exception of sulfate via ion exchange chromatography, using perchlorate as a mobile phase and utilizing direct UV absorption as detection technique.

Perchlorate was chosen as the main constituent in the mobile phase, given the fact that the samples might contain relative high concentrations of transition and heavy metals.

Most of the metal perchlorates are highly soluble in water. Hence why this mobile phase was chosen in order to diminish potential precipitation of metals on the anion exchange column.

Samples

The samples were kindly provided by a mining company in Australia.

Instrument

944 Professional UV/VIS Detector Vario	2.944.0010
943 Professional Thermostat Vario	2.943.0210
942 Extension Module Vario ONE/Deg	2.942.1060

Accessories

Metrosep A Supp 5 - 250/4.0	6.1006.530
Metrosep A Supp 4/5 - Guard/4.0	6.1006.500

Instrument picture



Reagents

- Ultrapure water, resistivity >18 MΩ·cm (25 °C), type I grade (ASTM D1193)
- Sodium perchlorate monohydrate, 89152 FLUKA, CAS Number 7791-07-3
- Sodium hydroxide concentrate, 0.1 M NaOH in water (0.1N), 43617 FLUKA, CAS Number 1310-73-2
- Sodium tetrathionate dehydrate, S5758 SIGMA, CAS Number 13721-29-4
- Sodium sulfite ≥98%, S0505 SIGMA-ALDRICH, CAS Number 7757-83-7
- Sodium thiosulfate, purum p.a., anhydrous, ≥98.0%, 72049 SIGMA-ALDRICH, CAS Number 7772-98-7
- Sodium thiocyanate ACS reagent, ≥98.0%, 251410 SIGMA-ALDRICH, CAS Number 540-72-7

Solutions

Eluent	10.0 mM NaClO ₄ / 1.0 mM NaOH in water
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Standard solutions

The multi component standards were made up in ultra-pure water [mg/L] with the concentrations as shown in the table below. The multicomponent standards were made up from the individual 1000 ppm single component standards which were made up from the respective sodium salt.

Std.	1	2	3	4
β(Sulfite)	10	25	50	100
β(Thiosulfate)	10	25	50	100
β(Thiocyanate)	10	25	50	100
β(Tetrathionate)	10	25	50	100

Sample preparation

All the samples were diluted 1:2 in ultrapure water and injected over a 0.45 μ syringe filter.

Analysis

The analysis was carried out fully automated and controlled via MagICNet 3.1

Parameters

Column temperature	Ambient
Flow Mobile phase	0.7 ml/min
Injection volume	20 μl
Absorption wavelength	215 nm

Calculation

Calculation automatically by MagICNet 3.1 based on peak area.

Results.

The results are given in mg/kg

Det. #	SO ₃ ²⁻	S ₂ O ₃ ²⁻	SCN ⁻	S ₄ O ₆ ²⁻
1	<1	55.8	47.3	29.6
2	<1	59.5	49.8	29.7
3	<1	61.1	52.0	26.57
Av	<1	58.8	49.7	28.6
Std. Dev.	-	2.2	1.9	1.4

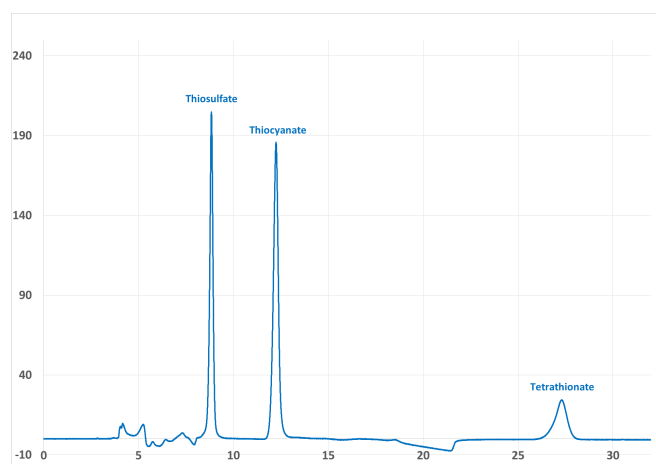
Date

Monday, October 12, 2015

Author

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MEP Instruments / Metrohm Australia

Example determination

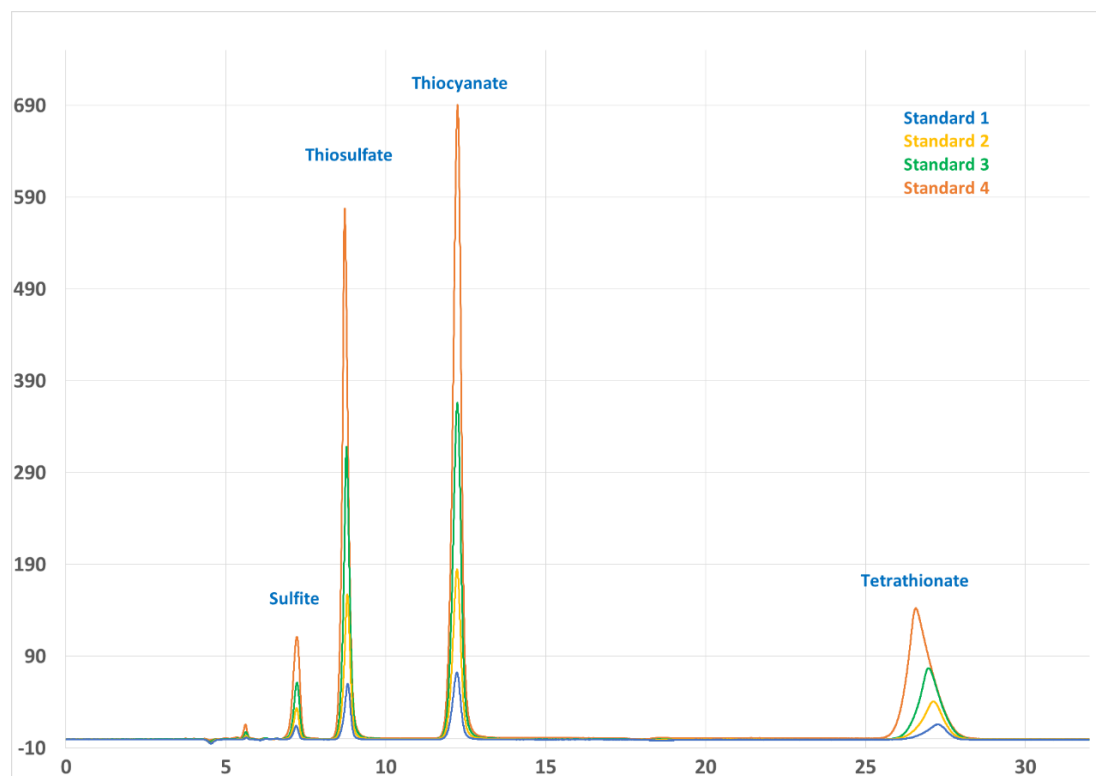


Appendix

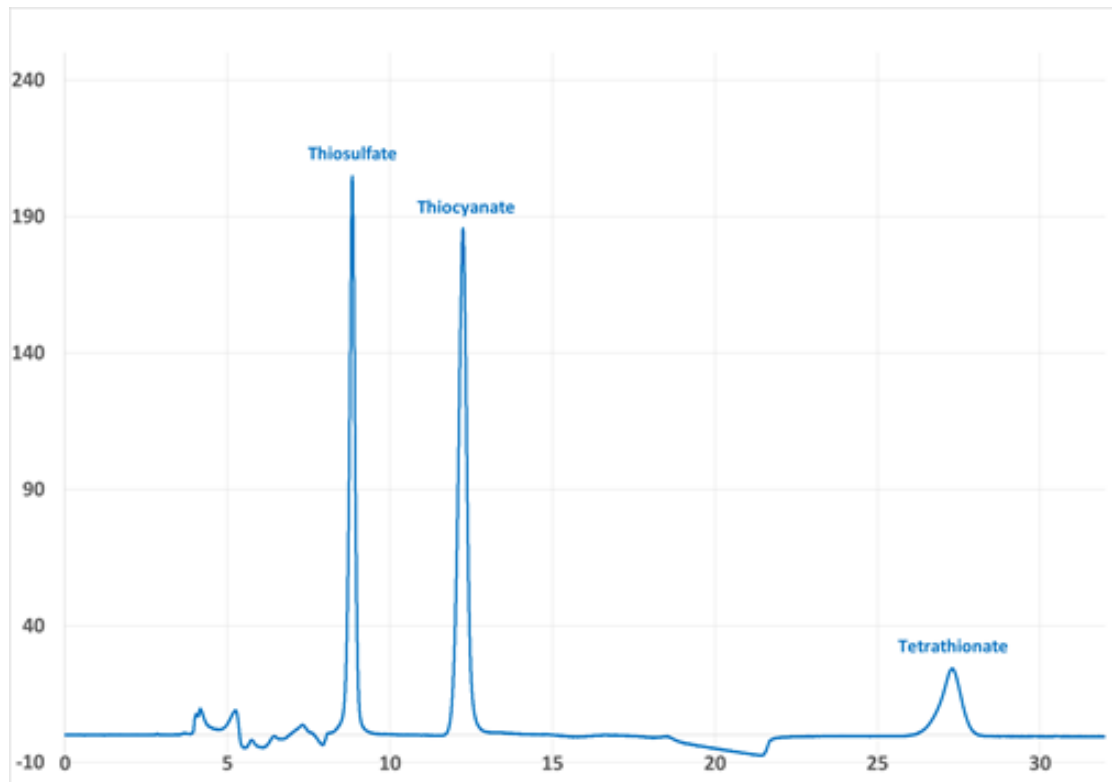
- Overlay of standard chromatograms
- Sample chromatogram
- Calibration curves

References

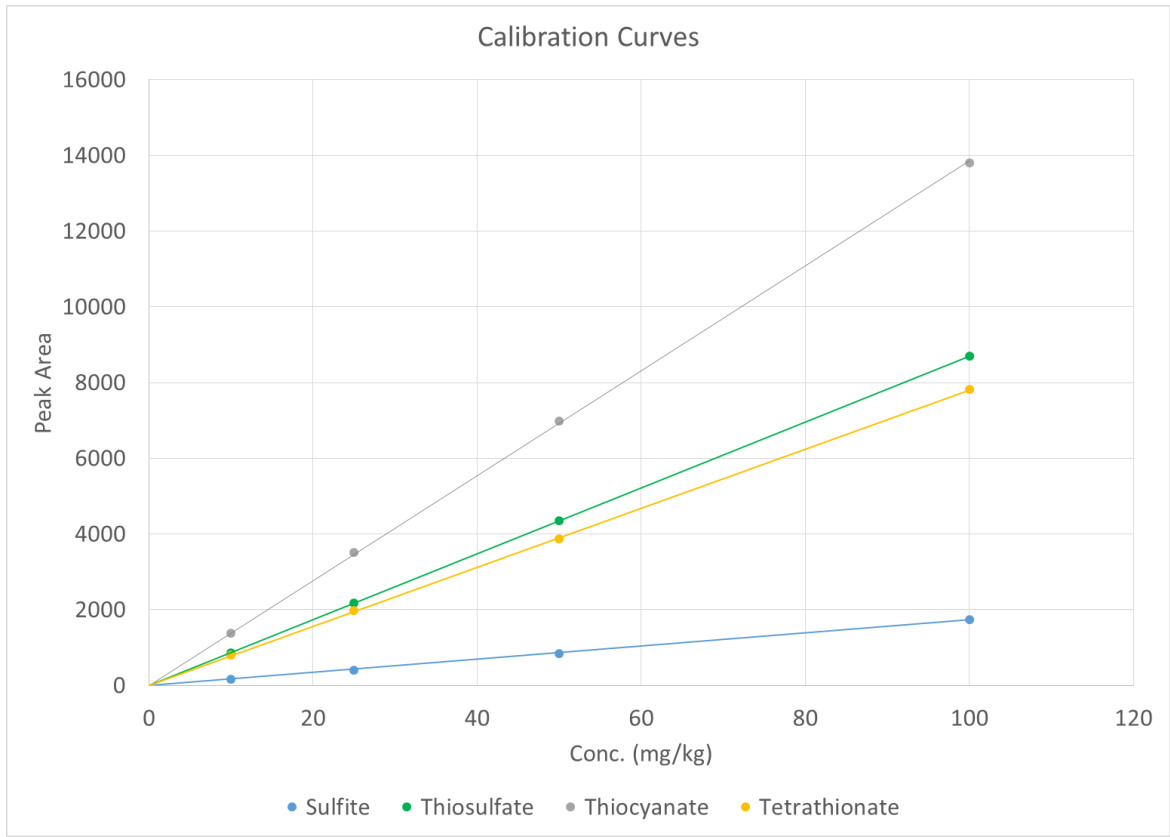
Yen, W.T. & Deschenes, G. & Aylmore, M. (2001) Thiosulfate Leaching as an Alternative to Cyanidation: a Review of the Latest Developments. 33rd Annual Operator's Conference of the Canadian Mineral Processors, Ottawa (Ontario, Canada), 23-25 January 2001.



Appendix A: Overlay of standard chromatograms



Appendix B: Sample chromatogram



Appendix C: Calibration Curves