Application note · ZEEnit 650P/700P

Challenge
Robust and reliable determination of gold in exploration and mining samples

Solution
ZEEnit 650P/700P – graphite furnace atomic absorption spectrometry with powerful Zeeman background correction

Intended audience
Mining companies and exploration teams, geology and geochemistry laboratories, analytical laboratories specializing in trace metal analysis

Determination of gold in aqua regia digestion from ores

Introduction

For the economical and efficient extraction of gold and other precious metals, ore and rock samples intended for mining must first be analyzed for their elemental composition and economic viability. Effective sample preparation is therefore essential in the exploration of mineral raw materials and in the quality control of geological samples. Acid digestion using aqua regia or related HNO_3/HCl-based procedures is an established approach for preparing solid samples and is described in various standards and methods, such as EN ISO 54321 and EPA Method 3051A.

The subsequent analysis of the prepared solutions requires a robust and reliable measurement system. Graphite furnace atomic absorption spectroscopy (GF-AAS) is a suitable and cost-effective technique for quantifying metals in the µg/L range. The complex matrices typically encountered in mining samples place high demands on effective background correction, which can be addressed by Zeeman technology. The ZEEnit 650P/700P series fully meets these requirements.
For automated sample handling, the instrument was equipped with the AS-GF autosampler, which automatically injects sample solutions and additional reagents, such as modifiers, into the graphite tube. It also enables the preparation of calibration standards from a premixed stock solution. Automatic overrange dilution and sample dilution according to predefined dilution factors are also possible.

The performance of the ZEEnit 650P/700P was demonstrated for the determination of gold in ore samples using two certified reference materials (CRMs) in the following measurement series.

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Materials and methods

Reagents

- Certified single element standard solution for gold (1,000 mg/L Au)
- $Pd(NO_3)_2$ matrix modifier (10 g/L Pd)
- $Mg(NO_3)_2$ matrix modifier (10 g/L Mg)
- Concentrated HNO_3 (approx. 60%, purified via sub-boiling distillation)
- Concentrated HCl (approx. 20%, purified via sub-boiling distillation)
- Tergitol™ 15-S-9, used as an additive in the rinsing solution

Samples

- CRM "OREAS 247" (gold ore, certif. value for aqua regia digestion: 42.56 mg/kg)
- CRM "NCS DC 29103" (gold ore, certif. value: 20.0 ± 0.3 mg/kg)

Sample preparation

Microwave-assisted aqua regia digestion was performed using the speedwaveXPERT microwave digestion system (Berghof Products + Instruments) equipped with DAP-60 digestion vessels.

Approximately 0.5 g of each sample was accurately weighed and transferred into the digestion vessel. Then, 7.5 mL of concentrated HCl and 2.5 mL of concentrated HNO_3 were added. The substances were carefully mixed and allowed to stand for at least 15 minutes before the vessel was closed. Microwave heating was performed according to the temperature program shown in Table 1.

After digestion, the vessels were allowed to cool to room temperature. The digests were quantitatively transferred into graduated PP tubes and filled up to 50 mL with deionized water. The mixtures were then centrifuged at 4,000 rpm for 10 minutes, and the supernatants were used for analysis.

For subsequential sample dilution, a matrix-matched acid solution was used, corresponding to the acid composition of the final digest: 7.5 mL concentrated HCl and 2.5 mL concentrated HNO_3 diluted to 50 mL with deionized water.

Each sample was prepared in duplicate to evaluate the reproducibility of the sample preparation procedure.

Instrumentation and method settings

The measurements were performed on the graphite furnace atomic absorption spectrometer ZEEnit 650P with Zeeman background correction, equipped with the autosampler AS-GF. Details regarding instrument settings, method parameters and furnace program are listed in tables 2-4. A matrix modifier solution containing 1 g/L Pd and 0.5 g/L Mg was used.

Table 1: Digestion parameters

Parameter	Setting/specification
Sample weight	0.5 g
Volume of conc. HCl	7.5 mL
Volume of conc. HNO_3	2.5 mL
Vessel type	DAP-60
Temperature program	5 min ramp to 180 °C 5 min hold at 180 °C 5 min ramp to 200 °C 20 min hold at 200 °C 5 min ramp to 220 °C 10 min hold at 220 °C
Max. power	90%
Final volume	50 mL

Table 2: General instrument setting

Parameter	Specification
Device	ZEEnit 650P/700P
Tube type	PIN-platform
Autosampler	AS-GF
Rinsing solution	1% (v/v) HNO_3, 0.05% (w/v) Tergitol™ 15-S-9
Background correction	Zeeman (2-field mode)
Injection volume sample/standard	20 µL
Injection volume modifier	5 µL (0.1% Pd 0.05% Mg (w/w))

Table 3: Measurement parameters

Element	Optics			ZBG	Evaluation		Modifier
	Wavelength [nm]	Slit width [nm]	HCL current [mA]	Magnetic field [T]	Integration mode	Read time [s]	
Au	242.8	0.8	5	0.8	Area	5.0	$Pd/Mg(NO_3)_2$ *

* Modifier mixture: 1 g/L Pd + 0.5 g/L Mg
ZBG : Zeeman background correction

Table 4: Furnace program

Step		Temperature [°C]	Ramp [°C/s]	Hold [s]	Gas type
1	Drying	80	6	20	Ar
2	Drying	90	3	20	Ar
3	Drying	110	5	10	Ar
4	Pyrolysis	350	50	20	Ar
5	Pyrolysis	850	300	10	Ar
6	AZ *	850	0	6	No gas
7	Atomization	2,050	1,500	5	No gas
8	Clean	2,450	500	4	Ar

* AZ: Auto zero

Calibration

The calibration standards were prepared automatically by the autosampler AS-GF by diluting a pre-mixed stock standard solution. The concentration variation was achieved through the volume adaptation of the stock solution. The blank solution was used to fill up to a constant injection volume of 20 µL.

Diluted aqua regia, analogous to sample preparation (7.5 mL HCl + 2.5 mL HNO_3 in 50 mL deion. water), was used as a diluent and calibration zero. The prepared concentrations are listed in table 5. Figure 1 shows the resulting calibration function.

Table 5: Concentration of the calibration solutions

Standard	Volume of stock solution [µL]	Au concentration [µg/L]
Stock solution	–	50
Cal. zero	0	0
Cal. std. 1	4	10
Cal. std. 2	8	20
Cal. std. 3	12	30
Cal. std. 4	16	40
Cal. std. 5	20	50

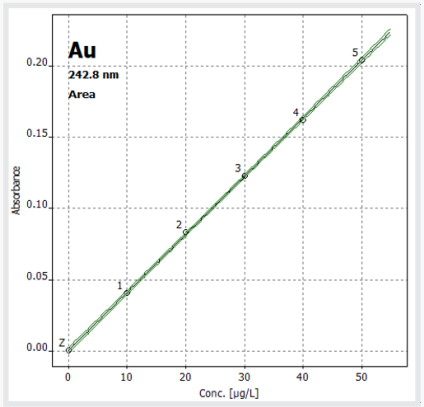


Figure 1: Calibration function (linear, R^2(adj.)=0.9998)

Results and discussion

The prepared aqua regia digests were analyzed after tenfold pre-dilution to adjust the measuring solution to the applied calibration range. The measurement results for both replicates of each certified reference material (CRM), together with the certified values and recoveries, are summarized in Table 6. The measured CRM values are in good agreement with the certified values, with recoveries ranging from 94% to 101%.

The methodological limits of detection and quantification (MLOD/MLOQ) listed were determined using the blank value method, based on threefold and ninefold standard deviations, respectively, of 11 replicate measurements of the reagent blank. These values include the dilution factor resulting from sample preparation (100-fold). Under optimized conditions, the limit of detection of the ZEEnit system is 0.25 µg/L Au for a sample volume of 20 µL.

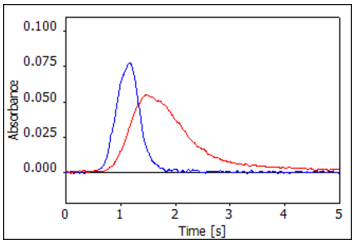
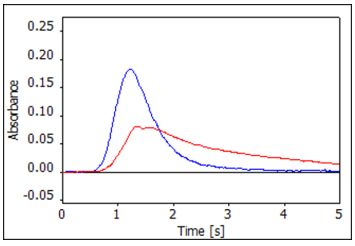
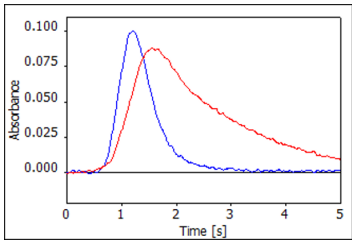
Characteristic analyte signals for an aqueous calibration standard and the analyzed ore samples are shown in Table 7.

Table 6: Measurement results, recoveries and methodological LOD/LOQ

Sample	DF	Certified value ± uncertainty [mg/kg]	Measured value ± SD [mg/kg]	CRM recovery [%]	MLOD [mg/kg]	MLOQ [mg/kg]
OREAS 247 (1)	10	42.56	40.40 ± 0.26	94.9	0.025	0.075
OREAS 247 (2)	10		40.28 ± 0.12	94.6		
NCS DC 29103 (1)	10	20.0 ± 0.3	18.95 ± 0.08	94.8		
NCS DC 29103 (2)	10		20.15 ± 0.03	101		

DF: manual dilution factor
SD: Standard deviation (based on 3 measurement replicates)
MLOD/MLOQ: methodological limit of detection/quantification (based on 11 measurements, 3σ-/9σ criterion)

Table 7: Characteristic gold signals

Element/line	Cal. std. 1 (10 µg/L)	OREAS 247	NCS DC 29103
Au242			

red: background signal
blue: atomic absorption signal

Summary

The ZEEnit 650P and ZEEnit 700P graphite furnace atomic absorption spectrometers provide a robust, reliable, and cost-effective solution for the quantification of gold in digested mining samples. Their powerful Zeeman background correction, with a magnetic field strength of up to 1.0 T, ensures excellent measurement precision and accuracy – even for complex matrices with high background absorption.

For samples with higher analyte concentrations, the calibration range can be significantly extended by physical signal attenuation using the 3-field or dynamic Zeeman mode. This enables precise and flexible analysis across a wider concentration range without compromising analytical performance.

Automated sample handling with the AS-GF autosampler further enhances productivity and ease of use. Variable, automated, and predefined sample dilutions, sample spiking, standard addition procedures, and in-tube enrichment can be conveniently implemented under full software control.

Figure 2: ZEEnit 650P

This makes the ZEEnit 650P/700P series a powerful and efficient solution for demanding precious-metal analysis in mining and geochemical laboratories.

Recommended device configuration

Table 8: Overview of devices, accessories, and consumables

Article	Article number	Description
ZEEnit 650P with camera	813-0650P-2-K	Graphite Furnace AAS with Zeeman background correction
ZEEnit 700P with camera	813-0700P-2-K	Flame- and Graphite Furnace AAS with Zeeman background correction
Chiller, 50 Hz	810-60053-0	Software-controlled cooling system (50 Hz power supply)
Chiller, 60 Hz	810-60052-0	Software-controlled cooling system (60 Hz power supply)
HCL Au	480-450.021C	Coded Hollow Cathode Lamp gold (Au)
Graphite tube, platform	407-152.314	Z-graphite tube PIN-platform - pyrolytically coated
Sample vials (PS) 1.5 mL	407-218.852	Polystyrene-sample vials 1.5mL for autosampler AS-GF

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