Challenge
The accurate and robust determination of Ag, Au, Cr, Co, Cu, Fe, Mn, and Ni in geological samples after acid extraction

Solution
Quantification of metals using novAA 800 F in flame mode

Intended audience
Industry of mining and metallurgy

Determination of Ag, Au, Cr, Co, Cu, Fe, Mn, and Ni in Mining Samples Using LS-AAS

Introduction

Metals such as silver (Ag), gold (Au), cobalt (Co), chromium (Cr), copper (Cu), iron (Fe), manganese (Mn), and nickel (Ni) play a pivotal role in modern industry, technology and infrastructure. Their extraction from the earth's crust depends on geological availability and economic feasibility.

The concentrations of these elements vary: Iron, for example, is present at a concentration of around 5% by weight, whereas gold occurs at trace levels of approximately 4 µg/kg. Economically viable mining typically requires ore concentrations to be significantly above crustal averages. Copper ores, for example, are considered profitable at concentrations above 0.5%, whereas gold mining may be viable at concentrations as low as a few grams per ton, depending on market prices and extraction costs.

Extraction methods vary by metal and deposit type. Iron and copper are often mined via open-pit or underground operations, followed by crushing, grinding and flotation. Precious metals such as gold and silver may require cyanide leaching or amalgamation. Cobalt and nickel are often obtained as by-products of copper or platinum group metal mining, while chromium is extracted from chromite ores using smelting techniques. Manganese is typically mined from oxide ores such as pyrolusite, often through hydrometallurgical or reductive roasting processes.

Accurately determining metal concentrations in ores is critical for assessing deposit quality and guiding extraction strategies. Elemental analysis must be precise, sensitive and robust when working with different types of material. Flame atomic absorption spectrometry (F-AAS) is a widely adopted technique for this purpose. It reliably quantifies metals in geological samples. Despite its lower sensitivity compared to graphite furnace AAS or ICP-MS, flame AAS remains cost-effective and robust in operation.

F-AAS is a rapid method for routine analysis, especially when dealing with high-concentration samples. Ultimately, flame AAS enables the efficient evaluation of resources and contributes to the sustainable management of mineral assets.

In order to enable flame AAS measurement, the mining samples must first be converted into a solution. Common preparation methods include aqua regia digestion for noble metals or acid leaching using a mixture of hydrochloric and nitric acid. For gold, fire assay remains the benchmark procedure, producing a lead button that is subsequently cupelled and dissolved. These procedures ensure accurate quantification by transforming solid matrices into measurable liquid forms.

This application note explains how to quantify the metals Ag, Au, Cr, Co, Cu, Fe, Mn, and Ni in acidic solutions using the novAA 800 F flame atomic absorption spectrometer. It details the required sample preparation steps and the optimal measurement parameters. The instrument features an eight-position turret for hollow cathode lamps, a fully automated measurement process, and optional optical purging with purified ambient air via the air purge kit (APK). For situations where acetylene is unavailable, the LPG burner head offers an alternative, enabling continued operation despite reduced precision and sensitivity compared to the acetylene–air flame. The system also includes an autosampler with intelligent, automatic sample dilution (AS-FD). Combining outstanding robustness with advanced automation capabilities, the novAA 800 F delivers exceptional performance for routine and demanding applications alike.

Materials and Methods

Reference material

- BAM-U110
- CCU-1c
- CZN-4
- DC29103
- IAEA-457
- AMIS0620
- OREAS 247
- SQC001

Reagents

- Concentrated HNO_3 (65%, p.a.)
- Concentrated HCl (37%, p.a.)
- Cesium chloride-lanthanum chloride solution (100 g/L CsCl, 100 g/L $LaCl_3$)
- $NaSO_4$ (p.a.)
- Certified single element standards for Ag, Au, Cr, Co, Cu, Fe, Mn, and Ni (concentration of the analytes 1,000 mg/L)

Sample preparation

Microwave assisted digestion (aqua regia leaching): Approximately 0.5 g of each sample was accurately weighed into the digestion vessel, to which 7.5 mL of HCl and 2.5 mL of HNO_3 were then added. The mixture was shaken carefully and left to stand for about 15 minutes before the vessel was closed. Subsequent heating was performed using the speedwave EXPERT microwave digestion device with the parameters shown in Table 1.

The vessels were cooled to ambient temperature. The solutions were transferred to polypropylene tubes and topped up to 50 mL with deionised water. For example, the mixture was then centrifuged at 4,000 rpm for ten minutes.

Table 1: Digestion parameters

Parameter	Specification
Sample weight	0.5 g
Volume of concentrated HCl	7.5 mL
Volume of concentrated HNO_3	2.5 mL
Vessel	DAP60
Temperature program	180 °C (5 min ramp, 5 min hold)
	200 °C (5 min ramp, 20 min hold)
	220 °C (5 min ramp, 10 min hold)
	Cool down to ambient temperature
Power	90%
Maximum pressure	40 bar

The supernatant was analysed either directly or after the addition of reagents (e.g. 0.2% (w/w) Cs/La or 0.2% (w/w) Na_2SO_4). If dilution of the sample solutions was required, the same acid mixture used for preparation (7.5 mL HCl and 2.5 mL HNO_3 per 50 mL) was employed.

Depending on the concentration of the sample, a different dilution factor needs to be applied in order to perform the measurement within the ideal calibration range. Table 2 provides guidance on this matter. Typical concentrations of ore samples are listed alongside the recommended dilution factor for the samples after microwave digestion (0.5 g sample; 50 mL final volume). The diluent should be a mixture of 15% v/v HCl and 5% (v/v) HNO_3.

Table 2: Typical ore concentrations and recommended dilution factors

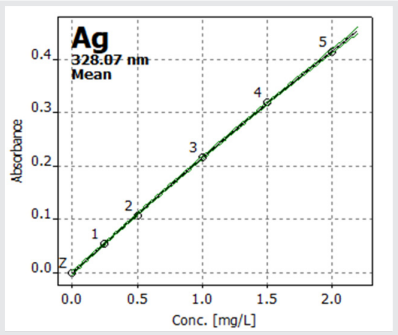
Element (wavelength)	Ore concentration	Dilution factor after microwave digestion
Ag	10–50 mg/kg	none
Au	5–20 mg/kg	none
Co	1–2%	50
Cr	10–25%	1,000
Cu	0.5–5%	150
Fe	60–70%	1,500
Mn	30–45%	1,500
Ni	1–5%	150

Calibration

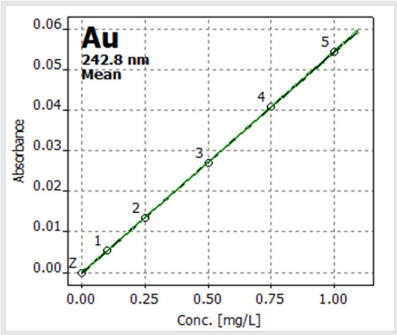
The calibration standards were prepared manually using an acid mixture containing 7.5 mL of HCl and 2.5 mL of HNO_3 per 50 mL (15% (v/v) HCl, 5% (v/v) HNO_3). Single-element standards (1000 mg/L) were used as a stock reference. To minimize matrix effects, buffer solutions were added to the standards and samples, resulting in a final concentration of 0.2% (w/v) $CsCl/LaCl_3$ for Au, Co, Cu, Fe, Mn and Ni analyses, and 0.2% (w/v) Na_2SO_4 for chromium analyses. Silver was analyzed without the addition of buffer solution. The used concentrations for calibrating the measurement are shown in Table 3. Figure 1 lists typical calibration plots.

Table 3: Recommended calibration range [mg/L]

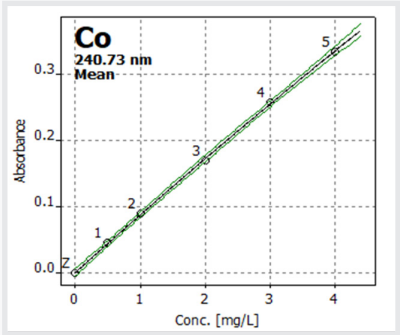
Standard	Ag	Au	Co	Cr	Cu	Fe	Mn	Ni
Matrix	15% (v/v) HCl 5% (v/v) HNO_3	15% (v/v) HCl 5% (v/v) HNO_3 0.2% (w/v) $CsCl/LaCl_3$	15% (v/v) HCl 5% (v/v) HNO_3 0.2% (w/v) $CsCl/LaCl_3$	15% (v/v) HCl 5% (v/v) HNO_3 0.2% (w/v) Na_2SO_4	15% (v/v) HCl 5% (v/v) HNO_3 0.2% (w/v) $CsCl/LaCl_3$	15% (v/v) HCl 5% (v/v) HNO_3 0.2% (w/v) $CsCl/LaCl_3$	15% (v/v) HCl 5% (v/v) HNO_3 0.2% (w/v) $CsCl/LaCl_3$	15% (v/v) HCl 5% (v/v) HNO_3 0.2% (w/v) $CsCl/LaCl_3$
	[mg/L]							
Calibration zero	0	0	0	0	0	0	0	0
Standard 1	0.25	0.1	0.5	0.5	0.5	1	0.5	0.25
Standard 2	0.5	0.25	1	1	1	2	1	0.5
Standard 3	1	0.5	2	2	2	4	2	1
Standard 4	1.5	0.75	3	3	3	6	3	1.5
Standard 5	2	1	4	4	4	8	4	2



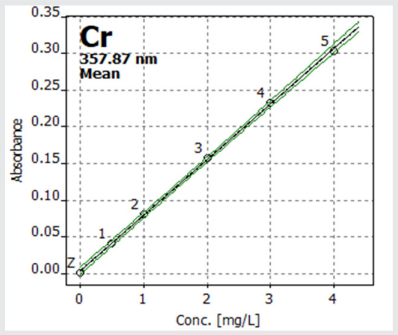
Ag 328.1 nm, nonlinear,
correlation R^2_{adj} = 0.9999



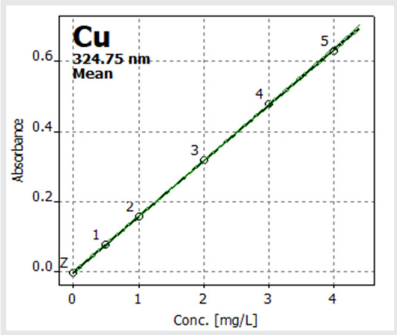
Au 242.8 nm, linear,
correlation R^2_{adj} = 0.99996



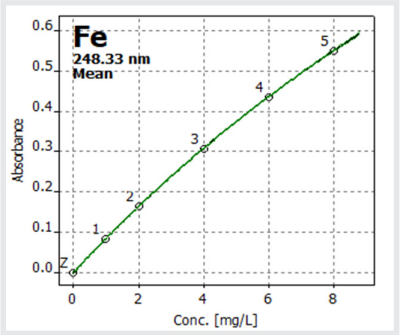
Co 240.7 nm , nonlinear,
Correlation R^2_{adj} = 0.9995



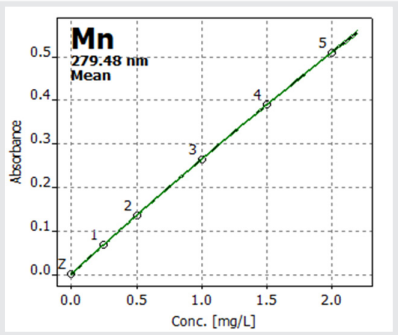
Cr 357.9 nm, linear,
correlation R^2_{adj} = 0.9993



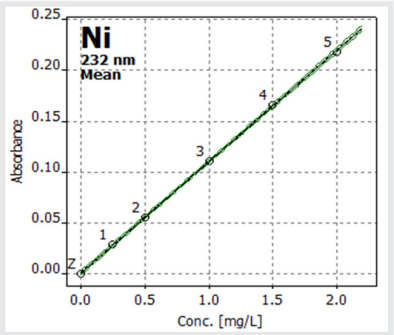
Cu 324.8 nm, linear,
correlation R^2_{adj} = 0.99993



Fe 248.3 nm, nonlinear,
correlation R^2_{adj} = 0.99994



Mn 279.5 nm, nonlinear,
correlation R^2_{adj} = 0.9993



Ni 232 nm, linear,
correlation R^2_{adj} = 0.9998

Figure 1: Typical calibration plot

Instrument settings

The robust and user-friendly novAA 800 F flame atomic absorption spectrometer was used for the determination of metals in geological samples (soil, sediment, and ore). The 100 mm burner head was used for best sensitivity and matrix tolerance. An AS-F autosampler or an AS-FD autosampler with dilution function can be used optionally to automate the measurement. Variable sample dilutions or preparation of the standard addition procedure can be performed automatically with the AS-FD autosampler.

To prevent dust deposits or corrosion of the spectrometer, an optional air purge kit can be used to filter and neutralise the ambient atmosphere. This accessory increases the system's durability in challenging surroundings. When acetylene is unavailable, an LPG burner head provides a practical alternative for the novAA 800 F. Although acetylene remains the preferred choice due to its higher flame temperature and superior performance, LPG ensures continuity of work where supply constraints exist.

The specifications of the instrument and the associated measurement parameters are summarized in Table 4. Table 5 outlines the detailed settings applied during the analytical procedure. For background correction, a deuterium hollow cathode lamp (D_2-HCL) was employed, ensuring optimal light throughput and effectively minimizing signal noise. This setup supports high analytical precision, particularly in complex sample matrices.

Table 4: General instrument parameters

Parameter	Specification
Device	novAA 800 F
Burner type	100 mm
Burner position	0°
Flame type	Air/Acetylene
Measuring time	3 s*, 5 repetitions
Background correction	D_2-HCL
Rinsing solution	3% (v/v) HCl and 1% (v/v) HNO_3
Accessories	AS-F(D), air purge kit

*Measuring time of 5 s was used for analyzing Au

Table 5: Analysis parameters

Element	Wavelength [nm]	Lamp current [mA]	Slit [nm]	Background correction	Fuel flow [L/h]	Auxiliary oxidans [L/h]	Burner height [mm]
Ag	328.1	3	0.5	on	40	75	5
Au	242.8	4	0.8	on	40	150	6
Co	240.7	5	0.2	on	55	75	6
Cr	357.9	5	0.8	on	120	150	7
Cu	324.8	2	1.2	on	45	75	5
Fe	248.3	5	0.2	on	90	150	6
Mn	279.5	4	0.2	on	70	75	7
Ni	232.0	4	0.2	on	60	75	5

Results and Discussion

Table 6 shows achievable limits of detection and quantification under the used conditions, using an 11-fold blank value measurement and the 3σ or 9σ standard deviation criterion. The method-related limits of detection and quantification refer to the solid sample, taking into account the preparation process involving weighing 0.5 g of the sample and filling to a volume of 50 mL.

Table 7 shows the measurement results for certified reference materials (ore, soil, and sediment). These values demonstrate that the method presented in this application note is accurate and therefore suitable for the correct and precise determination of geological samples.

The primary line provides the optimal signal-to-noise ratio when using hollow cathode lamps. If the metal content of the samples is high, secondary absorption lines with lower sensitivity can be used as an alternative, for which lower sample dilution factors are needed. Alternative lines are listed in Table 8. The calibration range must be adjusted according to sensitivity. Turning the burner head to the 90° position enables a working range that is ten times more concentrated, thus allowing for a dilution that is ten times lower.

Table 6: Achievable limits of detection (LOD) and limits of quantification (LOQ) of the device and method-related limits of detection (MLOD) and limits of quantification (MLOQ)

Element	Wavelength [nm]	LOD [mg/L]	LOQ [mg/L]	MLOD* [mg/kg]	MLOQ* [mg/kg]
Ag	328.1	0.003	0.009	0.3	0.9
Au	242.8	0.014	0.042	1.4	4.2
Co	240.7	0.010	0.030	1	3
Cr	357.9	0.011	0.033	1.1	3.3
Cu	324.8	0.008	0.024	0.8	2.4
Fe	279.5	0.013	0.039	1.3	3.9
Mn	232	0.006	0.018	0.6	1.8
Ni	248.3	0.011	0.033	1.1	3.3

* Considering a sample mass of 0.5 g and a final dilution volume of 50 mL (dilution factor 100)

Table 7: Measurement results for certified reference materials

Element	CRM	Pre-dilution factor	Measurement value [mg/kg]		Target value [mg/kg]	
Ag	AMIS0620	pure	51.4	± 0.5	52	± 5
	SQC001	pure	49.9	± 0.3	46.2	± 7.7
	CCU-1c	pure	117	± 0.7	129	± 2
	CZN-4	pure	48.6	± 1.3	51.4	± 0.7
	DC29103	pure	17.6	± 3.3	18	± 0.6
Au	OREAS 247	pure	43.8	± 0.6	42.96	± 2.15
	DC29103	pure	20.8	± 0.7	20	± 0.3
Co	AMIS0620	pure	1,139	± 9	1,137	± 69
	SQC001	pure	259	± 2	257	± 4
	CZN-4	pure	90.6	± 0.7	93.5	± 3.7
Cr	AMIS0620	pure	99.8	± 0.8	104	± 30
	SQC001	pure	159	± 1	159	± 4
Cu	AMIS0620	50	9,855	± 145	9,708	± 1,734
	SQC001	pure	157	± 2	151	± 3
	CCU-1c	1,000	253,750	± 3,177	256,200	± 1,200
	DC29103	10	1,276	± 8	1,200	± 100
	BAM-U110	pure	271	± 1	263	± 12
	IAEA-457	pure	372	± 4	365	± 19
Fe	AMIS0620	200	105,550	± 705	106,900	±2,800
	SQC001	10	5,741	± 38	5,752	± 84
	OREAS 247	50	32,840	± 171	33,200	± 1,600
Mn	AMIS0620	50	11,825	± 115	12,700	± 1,000
	SQC001	2	440	± 4	435	± 14
	BAM-U110	2	635	± 5	621	± 20
	IAEA-457	2	412	± 2	427	± 30
Ni	AMIS0620	pure	79	± 0.8	83	± 6
	SQC001	pure	182	± 2	183	± 4
	BAM-U110	pure	106	± 1	101	± 5
	IAEA-457	pure	52.9	± 0.6	53.1	± 2.7
	OREAS 247	pure	45.1	± 0.7	45.9	± 2.3

CRM = certified reference material
RSD = relative standard deviation, obtained from duplicate sample digestions

Table 8: Primary and secondary lines of the analytes

Element	Primery line [nm] (sensitivity)	Secondary line	Sensitivity [%]
Ag	328.1 (100%)	338.3	53%
Au	242.8 (100%)	267.6	50%
Co	240.7 (100%)	252.1	46%
Cr	357.9 (100%)	427.5	27%
Cu	324.8 (100%)	327.4	50%
Fe	248.3 (100%)	302.1	27%
Mn	279.5 (100%)	280.1	44%
Ni	232 (100%)	341.5	33%

Summary

Fast, reliable and cost-effective determination of chromium, cobalt, copper, gold, iron, manganese, nickel and silver in ore and soil samples is achieved using the novAA 800 F. The system is designed for straightforward operation and smooth workflows, even with complex sample matrices. Accessories such as the autosampler AS-FD with an auto-dilution function, an air purge kit for spectrometer protection, and an LPG burner head for alternative fuel provide flexibility and ensure consistent, long-term performance.

After acid digestion, the novAA 800 F provides precise quantification down to the lowest mg/kg range, thanks to its excellent detection capability. Validation with certified reference materials confirms the system's accuracy and reliability, ensuring dependable results for routine and advanced applications alike.

Figure 2: novAA 800 F

Recommended device configuration

Table 9: Overview of devices, accessories, and consumables

Article	Article number	Description
novAA 800 F	812-08000-2	Compact high-performance atomic absorption spectrometer with flame
novAA 800 F 100 V	812-08000-9	Compact high-performance atomic absorption spectrometer with flame technology working with 110 V
Burner head 100 mm	810-60056-0	Burner head for best sensitivity in the acetylene/air flame
Burner head LPG	810-60059-0	Burner head for determining elements that can easily be atomized using a LPG flame
Air purge kit (APK)	810-60506-0	Optional spectrometer purging with air for increased resistance to corrosive laboratory conditions
AS-FD	810-60501-0	Autosampler for flame mode with dilution function
AS-F	810-60500-0	AS-F autosampler for flame mode
Air compressor, 50 Hz	810-60055-0	Air compressor, 50 Hz, 230 V – to supply AAS with water- and oil-free compressed air
Air compressor, 60 Hz	810-60506-0	Air compressor, 60 Hz – to supply AAS with water- and oil-free compressed air
Air compressor, 110V	810-60276-0	Air compressor, 50/60Hz, 110 V – to supply AAS with water- and oil- free compressed air
Ag-HCL	480-450.051C	Hollow cathode lamp chrome (Ag) equipped with RFID chip
Au-HCL	480-450.021C	Hollow cathode lamp manganese (Au) equipped with RFID chip
Co-HCL	480-450.013C	Hollow cathode lamp cobalt (Co) equipped with RFID chip
Cr-HCL	480-450.012C	Hollow cathode lamp nickel (Cr) equipped with RFID chip
Cu-HCL	480-450.014C	Hollow cathode lamp cupper (Cu) equipped with RFID chip
Fe-HCL	480-450.026C	Hollow cathode lamp zinc (Fe) equipped with RFID chip
Mn-HCL	480-450.032C	Hollow cathode lamp cadmium (Mn) equipped with RFID chip
Ni-HCL	480-450.036C	Hollow cathode lamp lead (Ni) equipped with RFID chip

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