

LAB-X5000



The LAB-X5000 benchtop analyzer offers many advantages:

- | **Compact and robust:** ideally suited in a lab and near the production line
- | **Easy to use:** reliable results for all operators with minimum training
- | **Reliable:** uninterrupted operation and low cost of ownership
- | **Results in minutes:** fast testing for rapid decision-making and consistent product quality

LAB-X5000 for the rapid elemental analysis of nickel ores

INTRODUCTION

Nickel is one of the most important base metals used in many industry segments. The majority of the nickel is used for the production of stainless steels and non-ferrous alloys. It is also commonly used in the manufacturing of rechargeable batteries for portable computers, power tools, and hybrid and electric vehicles. Nickel is an important element in various coating applications too, as it enhances the bonding between the layers of copper and gold on printed circuit boards.

The most important resources for Nickel are laterites where the main ore minerals are limonite $(\text{Fe,Ni})\text{O}(\text{OH})$ and garnierite $(\text{Ni,Mg})_3\text{SiO}_2(\text{OH})_4$. Magmatic sulfide deposits are also a major source of Ni ore where the principal ore mineral is pentlandite $(\text{Ni,Fe})_9\text{S}_8$. The average Ni ore concentration globally is 1 wt% but new mining techniques have made it possible to extract lower concentrations.

The largest known reserves of nickel ores are in Australia and Indonesia, and the world's largest nickel producer is Indonesia, followed by the Philippines, Russia, New Caledonia and Australia.

It is important to determine the ore composition during the mining and refining steps, as the production process and the ore financial value depend on its grade or quality. X-ray fluorescence (XRF) spectrometry is the preferred method of analysis because of its simplicity, speed and accuracy, and it has been used for many years by the mining industry.

NICKEL ORES ANALYSIS MADE EASY

With the Hitachi High-Tech LAB-X5000 energy-dispersive XRF benchtop analyzer, the analysis of nickel ores couldn't be easier. Once the LAB-X is calibrated, routine analysis is carried out by pouring the finely ground ore in a sample cup, placing the cup in the instrument's analysis port and pressing a button to start the measurement. Preliminary results are displayed within seconds

on the analyzer's large, industrial LCD touchscreen, showing the sample's elemental content. Customized messages can be set up to help operators make fast decisions on process adjustment or stockpiling location.

The LAB-X5000 is calibrated by measuring a series of assayed samples or reference standards. Setting-up samples (drift correction monitors) are measured at the time of the calibration, and again in the unlikely event that the analyzer drifts, so there is no need to source calibration standards again or re-measure them, saving both time and money.

The combination of a high-resolution detector and optimized calibration parameters ensures that you get results you can trust. To compensate for sample inhomogeneity and deliver repeatable results, the LAB-X is equipped with a sample spinner that rotates the sample during the analysis to provide an average reading that is truly representative of its content. A helium purge is used to deliver the best performance for light elements (Na, Mg, P).

The analyzer includes several features that help protect against potential damage caused by sample spills or ambient dust, minimizing downtime and preventing costly repairs: an easy-to-replace safety window is used under the sample cup to retain any potential spill or leak, and the analyzer's turntable only places the sample above the X-ray tube and detector for the duration of the analysis, minimizing the risk of damage or contamination to critical components.

With up to 100,000 results stored on the analyzer itself, operators can view new and old results, print them on the optional integrated printer for a hard-copy record, download them on a USB memory device as a CSV file, and upload them to our ExTOPE Connect cloud service or a local server via Wi-Fi or ethernet for real-time access to the data anytime, anywhere.

SAMPLE PREPARATION

The sample is first dried then ground to obtain a fine powder that will pass through a 75µm sieve (200 mesh). The dried powder is poured into a sample cup fitted with Poly-4 film, filling the cup up to the internal line (approximately 13 ml). After placing a lid on the cup, operators gently tap the cup on a clean surface to compact the powder, and place it over a safety window, also fitted with Poly-4, in the LAB-X analysis port. All that remains is to press the green button on the analyzer to start the analysis.

Note: simple drying and grinding of the sample is sufficient for rapid quality and process control. If better accuracy is needed, ground powders should be pressed into 35 or 40mm-diameter pellets.

PERFORMANCE AND RESULTS

For this application, the LAB-X5000 was calibrated by measuring a series of laterite reference materials to establish the relationship between the elements' content and their X-ray signal.

Ideally assayed production samples should be used for the calibration, so that the mineralogy of the calibration standards matches that of unknowns. The samples' concentrations should evenly span the range of interest for all analytes. This ensures best accuracy.

Table 1 shows the typical calibration performance the LAB-X delivers for the analysis of laterites.

The limits of detection (LOD) were calculated from the results of 10 repeat measurements of a "blank" sample (SiO₂ powder), and the precision from 10 repeats of a series of samples containing the elements of interest.

The analysis time was 220 seconds per repeat.



Sample introduction



Starting the analysis

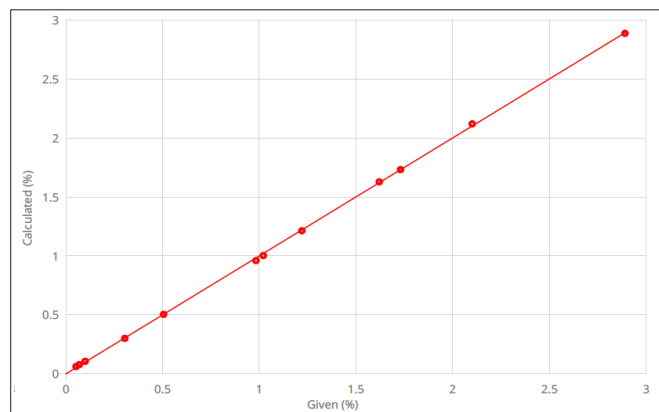
28-09-2023 14:12 Ni ores (powders) Lat 197		
Element	Concentration	Unit
V	506.6	ppm
Cr2O3	0.13	%
MnO	0.901	%
Fe2O3	38.19	%
Ni	0.0606	%
Zn	168.6	ppm

Results screen

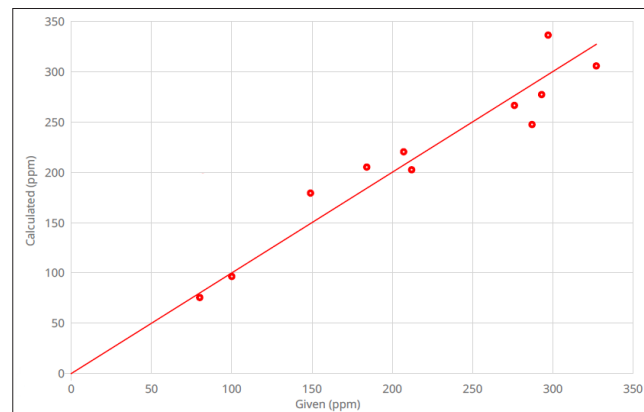
Table 1: Typical calibration performance for laterite ores

Analyte	Concentration unit	Calibration range	Standard error of calibration	Guaranteed limit of detection (3σ)	Limit of quantification (10σ)	Precision (95% confidence)
Na ₂ O	Wt%	0.03 - 0.42	0.05	0.10	0.22	0.07 at 0.22 0.05 at 0.35
MgO	Wt%	0.7 - 27.4	0.5	0.05	0.11	0.04 at 2.5 0.12 at 18
Al ₂ O ₃	Wt%	1.6 - 17.3	0.5	n/a	n/a	0.03 at 4.7 0.05 at 11
SiO ₂	Wt%	22.5 - 47.7	1.5	n/a	n/a	0.05 at 35 0.07 at 45
P ₂ O ₅	Wt%	0.005 - 0.170	0.006	0.008	0.017	0.003 at 0.03 0.004 at 0.17
K ₂ O	Wt%	0.07 - 0.23	0.004	0.011	0.025	0.005 at 0.12 0.007 at 0.23
CaO	Wt%	0.13 - 3.14	0.06	0.005	0.012	0.004 at 0.6 0.009 at 2.4
TiO ₂	Wt%	0.02 - 1.34	0.03	0.002	0.004	0.004 at 0.04 0.008 at 0.30
V	mg/kg	146 - 657	9	3	7	4 at 75 5 at 150
Cr ₂ O ₃	Wt%	0.17 - 1.75	0.08	0.001	0.003	0.008 at 0.9 0.009 at 1.3
MnO	Wt%	0.11 - 1.91	0.01	0.001	0.002	0.002 at 0.2 0.004 at 0.5
Fe ₂ O ₃	Wt%	12.7 - 45.2	0.7	n/a	n/a	0.04 at 19 0.09 at 35
Ni	Wt%	0.05 - 2.89	0.01	0.0002	0.0004	0.004 at 0.5 0.009 at 2.9
Zn	mg/kg	80 - 327	26	16	36	7 at 85 10 at 270

Graph 1: Calibration graph for Nickel



Graph 2: Calibration graph for Zinc



To obtain lower limits of detection (LOD) and quantification (LOQ), longer measurement times can be used. To halve the LOD and LOQ, you need to increase the measurement time by a factor of 4. Alternatively, or additionally, powders can be pressed into pellets.

Note: other elements and oxides can be added to the calibration if needed. Please contact your local Hitachi High-Tech representative for guidance or further information.

SUMMARY

Once calibrated, Hitachi High-Tech's LAB-X5000 is ideal for rapid quality and process control of nickel ores, helping customers ensure that each material batch is within its grade specifications by providing its elemental composition in minutes. Combining simple sample preparation, ease of use and reliability, the LAB-X5000 delivers results fast to keep production running 24/7 and ensure consistent product quality.



ORDERING INFORMATION

The instrument configuration and accessories needed to produce the data in this application note are:

- | LAB-X5000 with Pd-target tube with Helium purge (P/No. 10022338 with integrated printer, 10027133 without printer); includes 3 safety windows
- | Sample spinner (P/No. 10009405)
- | Re-usable sample cup outers, box of 10 (P/No. 10001172)
- | Disposable sample cup inners and lids, box of 100 each (P/No. 10011741)
- | Poly-4 sample film, 100m roll (P/No. 10000780)

Setting-up samples (SUSs):

- | It is recommended to use a SiO_2 powder as the low SUS for all elements (high SUS for SiO_2), and to use 1 or 2 samples as the high SUS(s) for all elements (low SUS for SiO_2). These should be prepared as pressed pellets for durability. Powders and pellets should be kept in a dessicator, so they remain free of moisture.

Visit www.hitachi-hightech.com/hha for more information.

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ADDENDUM 1: CALIBRATION PARAMETERS

The performance shown in this application note was achieved on the LAB-X5000 calibrated using the parameters in the tables below.

Table 2: Analytes and conditions

Analyte	Condition	Measurement time (seconds)	Region of interest (keV)	Used in calibration
Na ₂ O	5kV 600µA open filter Helium ON Sample spinner ON	120	Default (0.96 - 1.12)	Yes
MgO			Default (1.17 - 1.33)	Yes
Al ₂ O ₃			Default (1.41 - 1.57)	Yes
SiO ₂			Default (1.66 - 1.83)	Yes
P ₂ O ₅			Default (1.93 - 2.10)	Yes
K ₂ O			Default (3.22 - 3.41)	Yes
CaO			Default (3.59 - 3.79)	Yes
Pd			Custom (2.79 - 2.89)	No
TiO ₂	20kV 80µA A6 filter Helium OFF Sample spinner ON	100	Custom (4.47 - 4.57)	Yes
V			Custom (4.89 - 5.01)	Yes
Cr ₂ O ₃			Default (5.30 - 5.52)	Yes
MnO			Default (5.78 - 6.01)	Yes
Fe ₂ O ₃			Default (6.28 - 6.52)	Yes
Ni			Default (7.35 - 7.60)	Yes
Zn			Default (8.50 - 8.77)	Yes
Ba			Custom (4.74 - 4.88)	No

Table 3: Regression

Analyte	Correction model applied
Na ₂ O	Additive correction: Mg
MgO	-----
Al ₂ O ₃	Additive correction: Si
SiO ₂	Absorption/Enhancement: Al
P ₂ O ₅	Additive correction: Ca (corrects the overlap from Ca escape peak)
K ₂ O	Additive correction: Pd (corrects the overlap from Pd tube line)
CaO	-----
TiO ₂	Additive correction: Fe
V	Additive correction: Ba
Cr ₂ O ₃	Additive correction: V
MnO	Additive correction: Cr
Fe ₂ O ₃	-----
Ni	Absorption/Enhancement: Fe
Zn	Absorption/Enhancement: Ni

Additional information:

Estimated LOD for Ba: 280 mg/kg (LOQ 620 mg/kg), due to strong overlap with Ti Ka line.

Estimated LOD for Co: 400 mg/kg (LOQ 900 mg.kg), due to strong overlap with Fe Kb line.

ADDENDUM 2: PERFORMANCE IN AIR PATH

For performance comparison, especially for the light elements, the calibration was also created in air path.

The same parameters (Table 2) were used, but the 5kV condition used air path and a measurement time of 200 seconds.

Table 4: Typical calibration performance (air path)

Analyte	Concentration unit	Calibration range	Standard error of calibration	Guaranteed limit of detection (3 σ)	Limit of quantification (10 σ)	Precision (95% confidence)
Na ₂ O	Wt%	0.03 - 0.42	0.07	0.65	1.40	n/d
MgO	Wt%	0.7 - 27.4	0.7	0.25	0.55	0.32 at 2.5 0.55 at 18
Al ₂ O ₃	Wt%	1.6 - 17.3	0.5	n/a	n/a	0.04 at 4.7 0.09 at 11
SiO ₂	Wt%	22.5 - 47.7	1.5	n/a	n/a	0.12 at 35 0.20 at 45
P ₂ O ₅	Wt%	0.005 - 0.170	0.006	0.014	0.031	0.007 at 0.03 0.008 at 0.17
K ₂ O	Wt%	0.07 - 0.23	0.004	0.013	0.030	0.006 at 0.12 0.008 at 0.23
CaO	Wt%	0.13 - 3.14	0.06	0.009	0.020	0.008 at 0.6 0.011 at 2.4
TiO ₂	Wt%	0.02 - 1.34	0.03	0.002	0.004	0.004 at 0.04 0.008 at 0.30
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MnO	Wt%	0.11 - 1.91	0.01	0.001	0.002	0.003 at 0.2 0.005 at 0.5
Fe ₂ O ₃	Wt%	12.7 - 45.2	0.7	n/a	n/a	0.04 at 19 0.09 at 35
Ni	Wt%	0.05 - 2.89	0.01	0.0002	0.0004	0.004 at 0.5 0.009 at 2.9
Zn	mg/kg	80 - 327	26	16	36	7 at 100 10 at 270

Note: the same regression models (shown in Table 3) were applied.

The precision for Na was not determined as Na cannot be determined at these levels in air path.