

# Analysis of Boron in Nickel alloys

ARL ADVANT'X Series with IntelliPower™  
Sequential X-Ray Fluorescence Spectrometer

## Key Words

- ARL ADVANT'X
- IntelliPower™
- Boron
- Nickel
- XRF
- X-Ray Fluorescence



## Introduction

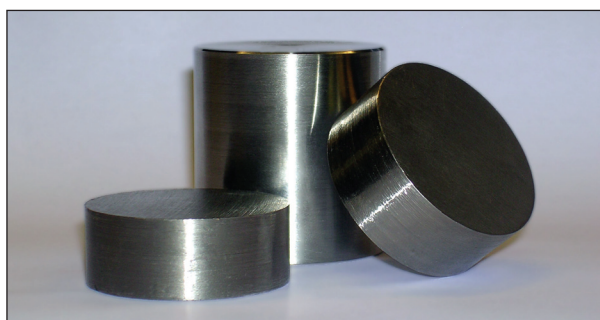
A series of nickel alloys have been used to make a feasibility study on the determination of boron in such alloys.

A calibration curve has been built using the intensities and the concentrations of boron in the nickel alloy samples. The limit of detection can be derived from this calibration curve. Repeatability tests have been performed in order to determine precision data for such analysis.

## Instrumentation

A Thermo Scientific ARL ADVANT'X Series Spectrometer has been used to derive the following results. It is based on the wavelength dispersive X-ray fluorescence technique (WDXRF) that allows analysis of ultra-light elements with the use of multilayer synthetic “crystals”. The geometry of the instrument is optimized to provide the highest sensitivity. It is equipped with an end-window X-ray tube type 5 GN fitted with a 50 μm Be window and a Rh anode.

The AXBeB multilayer crystal, designed specifically for boron analysis is used. This new multilayer crystal provides 1.7 times more sensitivity for boron analysis compared to the former AX20 that was used up to now. The multilayer crystal is used together with the extra-coarse collimator which exploits the full potential of this synthetic crystal. Generator settings are 30 kV/120 mA. The highest mA are beneficial for light elements analysis.



## Results

Calibration work has been performed on a set of 15 samples using optimum analytical conditions. The boron concentration levels span from 0.7% to 3%.

Prior to analysis the samples surface is prepared through grinding with 60 grit abrasive paper. Figure 1 shows a scan over the boron Kα peak in three of the nickel samples.

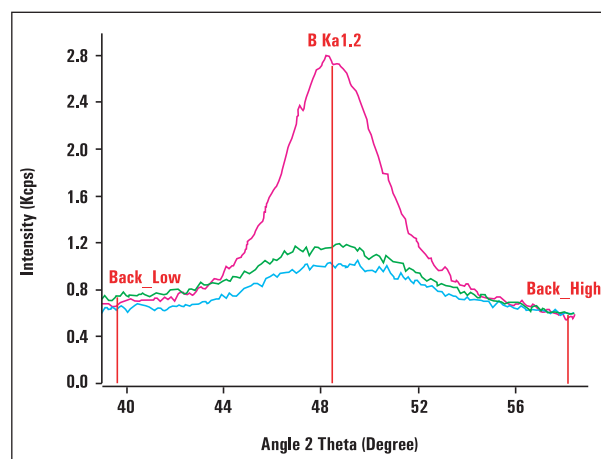


Figure 1: Scan of the boron Kα peak in nickel alloys

Several potential overlaps exist for boron analysis in nickel alloys, notably Mo, W and Cu. Overlap corrections are necessary in order to get a calibration curve providing the highest possible accuracy.

The resulting calibration curve is illustrated in Figure 2. From the sensitivity (slope) and background level shown on this calibration curve, the limit of detection for boron in nickel alloys can be derived:

- 205 ppm in 60 s counting time or
- 160 ppm in 100 s counting time.

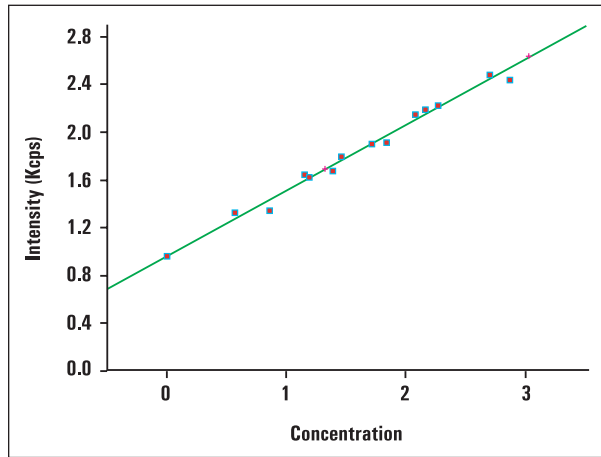


Figure 2: Calibration curve for boron in nickel alloys  
Analysis time: 60 s. Standard error of estimate (SEE) = 0.11 %

### Precision tests

Repeatability tests have been performed in order to show the precision that can be achieved for boron analysis in nickel alloys. Sixty seconds counting time has been used per run and per element. Only the boron data are shown in the following tables.

Table 1	
Run	Boron
1	1.710
2	1.734
3	1.723
4	1.726
5	1.740
6	1.750
7	1.742
8	1.754
9	1.731
10	1.737
11	1.730
Avg	1.734
SD	0.013
RSD	0.720

Table 1: Repeatability on sample A  
SD=Standard Deviation,  
RSD=Relative Standard Deviation

Table 2	
Run	Boron
1	2.790
2	2.815
3	2.807
4	2.795
5	2.820
6	2.837
7	2.817
8	2.820
9	2.823
10	2.815
11	2.832
Avg	2.816
SD	0.015
RSD	0.500

Table 2: Repeatability on sample B  
SD=Standard Deviation,  
RSD=Relative Standard Deviation

### Conclusion

Limits of detection for ultra-light elements are dropping consistently with new spectrometer developments, allowing better analysis of difficult elements. This permits replacement of time consuming wet chemical analyses by faster determination using X-ray fluorescence.

During this study a standard window has been used on the flow proportional counter. An ultra-thin window is also available and would allow even further improvement of the sensitivity.

We used X-ray tube settings of 3.6 kW for these tests, but about 15 % increase of sensitivity would be obtained by working at 4.2 kW.

It should be noted that in a nickel sample, boron X-ray fluorescence radiation escapes from a depth of the order of 0.03  $\mu\text{m}$ . This means that only the surface of the sample is analyzed for boron. Cleanliness of sample preparation, consistency in surface preparation methods and minimized risk of surface contamination are pre-requisites for this analysis. If this level of care is not taken, the results can be substantially in error as contamination will prevent the soft X-rays from escaping from the sample surface and hence they would only be partially measured by the XRF spectrometer.

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