

Determination of Cd, Cr, Ni and Pb in Grains using the Agilent 280Z Zeeman GFAAS



Elements: Cd, Cr, Ni, Pb

Matrix: Water

Modifiers:

Pb: $\text{NH}_4\text{H}_2\text{PO}_4 + \text{Mg}(\text{NO}_3)_2$

Cd: $\text{NH}_4\text{H}_2\text{PO}_4$

Cr, Ni: $\text{Pd}(\text{NO}_3)_2$

Instrumentation: Agilent 280Z Graphite Furnace AAS

Standards:

GB 5009.12–2010 for Pb,

GB 5009.15–2014 for Cd,

GB 5009.123–2014 for Cr,

GB 5009.138–2003 for Ni.

Stable, reliable and precise GFAAS

The Agilent 280Z GFAAS with Zeeman background correction offers some key features for determining trace elements in food products such as grains, including:

- **High sensitivity:** Combining an Agilent GTA 120 Graphite Tube Atomizer with the 280Z GFAAS and Agilent UltrAA lamps improves the signal-to-noise ratio, ensuring high sensitivity and low detection limits.
- **Increased accuracy:** Agilent UltrAA lamps reduce baseline noise and produce high, sharp emission intensities, resulting in excellent calibration linearity. Zeeman background correction handles the spectral interferences and high background absorbances associated with complex samples.
- **Reduced running costs:** The GTA 120 offers outstanding furnace performance and low running costs. The fast heating and short run time of the GTA 120 requires less argon, and extends tube lifetime.
- **Ease-of-use:** The Agilent PSD 120 autosampler automates standard preparation, standard additions, and dilution of overrange samples. Accurate volumes are delivered to the furnace and, when coupled to pre-emptive sampling, optimum analysis times are achieved.

- **Plug-and-play:** The Agilent plug-and-play lamps are automatically recognized by the instrument, allowing the recommended operating conditions to be automatically selected, simplifying method development.

Analysis example

Two wheat flour samples were analyzed in accordance with Chinese methods: GB Method 5009.12–2010 for Pb, GB 5009.15–2014 for Cd, GB 5009.123–2014 for Cr, and GB 5009.138–2003 for Ni.

Three replicate digests of the wheat flour standard reference materials (SRMs) were measured. The results were in good agreement with the certified values, confirming the accuracy of the method. The results are shown in Table 1.

Table 1: Results for the analysis of NIST 1567b Wheat Flour (Pb and Cd) and DUWF-1 (Cr and Ni) Durum Wheat Flour by GFAAS.

Element	MDL* (n=7) (ppb)	Certified Value (ppb)	Measured Value (mean, n=3) (ppb)	Standard Deviation (ppb)	Recovery (%)
Pb	2.6	10.4 ± 2.4	10.9	1.4	105
Cd	0.37	25.4 ± 0.9	23.4	0.22	92
Cr	1.5	23.0 ± 9.0	21.5	2.1	93
Ni	6.0	170 ± 80	172	7.5	101

*Method Detection Limit (MDL) based on 10 µL sample and 0.3, 0.05, 0.1, and 0.5 ppb standard solution for Pb, Cd, Cr, and Ni, respectively, for each of the GB methods tested.

Calibration and stability

A representative calibration curve is shown in Figure 1. All elements displayed excellent calibrations with correlation coefficients greater than 0.999. A 5 ppb Pb standard was analyzed periodically over 7 hours to determine the long-term stability of the instrument (Figure 2).

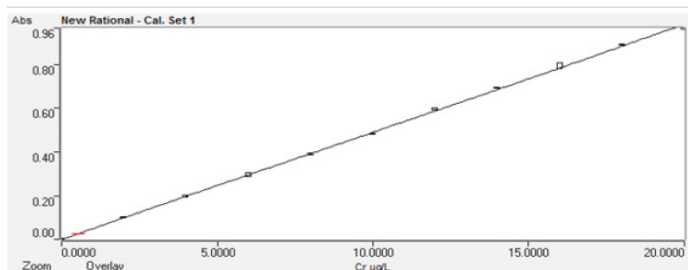


Figure 1. Calibration curve for Pb from 0 to 20 µg/L. All standards were prepared by the PSD 120 sample preparation system.

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Printed in the USA, March 6, 2019
5991-8844EN

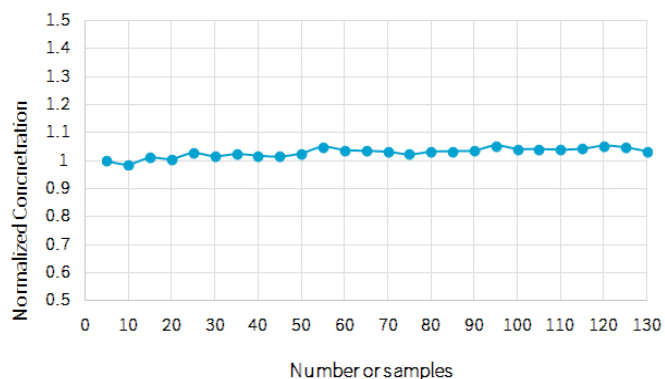


Figure 2. Long-term stability plot for a 5 ppb Pb standard measured periodically during the analysis of a wheat flour digest run over a 7-hour period.

Results

The results of the analysis demonstrate:

- Outstanding performance of the 280Z Graphite Furnace AA spectrometer, fitted with a GTA 120 atomizer. The method delivered good accuracy and precision of target analytical values for Pb, Cd, Cr, and Ni.
- The method detection limits exceeded the values specified in the GB methods.
- Excellent linearity was achieved across the calibration range for Pb, Cd, Cr, and Ni. All standards were automatically prepared using the PSD 120 sample preparation system.
- Excellent long-term stability, with 1.6% RSD over a 7-hour period and only 4% recovery deviation from the initial reading.

Download the full application note

[Determination of Pb, Cd, Cr and Ni in Grains Based on Four Chinese National Methods via Zeeman GFAAS](#), Agilent publication number 5991-8144EN.