



## ICP-MS Detector Calibration



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The LGC logo, consisting of the letters 'LGC' in a bold, black, sans-serif font inside a white circle.

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## Introduction

ICP-MS is a powerful elemental analysis technique that relies on the direct measurement of analyte ions. Modern ICP-MS instruments yield detection limits of less than 1 ppt for most elements and can be linear beyond 100 ppm; that's over 9 orders of magnitude of linear dynamic range! This characteristic of today's instruments results from the use of advanced, multimode detectors, termed electron multipliers, which include both analog and pulse counting capability. **Figure 1** shows the electron multiplier, which is positioned after the quadrupole mass filter in a typical ICP-MS.



Figure 1. ICP-MS Electron multiplier

## Basic Principle of ICP-MS Detection

With ICP-MS, the quadrupole scans or jumps from one analyte mass to the next. When the ion beam leaves the quadrupole it is directed into the electron multiplier where it impacts an electrode called a dynode. Upon striking the surface of the first dynode an electron is emitted from the surface. Drawn by an increasingly strong positive charge these electrons cascade downstream through the dynode chain where their effective charge is "multiplied" to create the ICP-MS signal. In a pulsed counting electron multiplier each ion impact of the detector results in a single large pulse of electrons at the end of the detector.

**Figure 2** shows a sketch of a modern discrete dynode electron multiplier, and a graphic that shows the electron multiplication process. The secondary electrons created in a detector can be thought of as a "cloud" of traveling electrons that corresponds with the quantity of analyte being tested. Detector operation is the same regardless of collisions / reaction cells that may be employed or cool plasma modes.

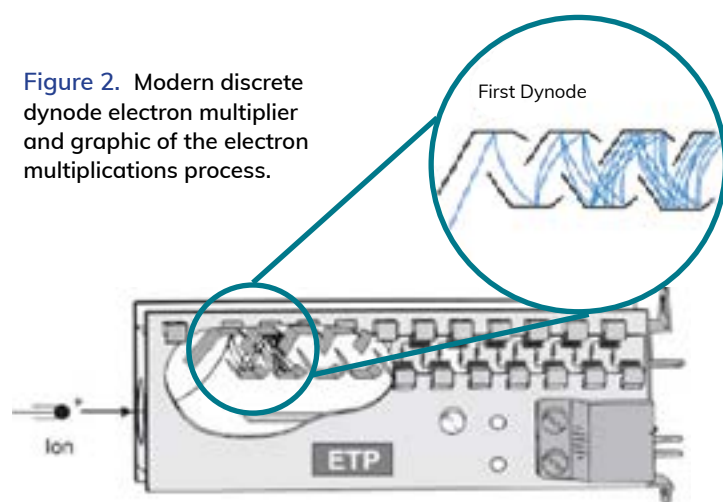


Figure 2. Modern discrete dynode electron multiplier and graphic of the electron multiplications process.

# Linearity

Typically, detectors for analytical instrumentation generate an electronic signal that has a consistent and simple relationship to the quantity of analyte being measured. A “linear” technique is the most desirable for a variety of reasons and implies that the calibration will be a straight line. Analytically, the implication is huge since a linear technique theoretically only requires two calibration standards to define the calibration curve, while non-linear techniques suffer from either a narrow usable range or a large set of standards and complex curve fitting to be usable for quantitative analysis.

## Dual Mode Electron Multipliers

ICP-MS is known for its ability to measure low analyte concentrations. In part, this is the result of an electron multiplier's ability to turn relatively low ion flux into a significant signal pulse at the bottom electrode of the electron multiplier. However as the ion flux increases, these "pulses" become increasingly difficult to distinguish. As a result, the pulse counting detector will start to become non-linear.

While “pulse mode” is excellent for low concentrations, elevated concentrations are typically measured at a dynode midway down the length of the detector. At this point larger signals are measurable and also linear. This measurement mode is typically termed "analog mode." **Figure 3** is a graphic representation of the measurement range and difference in sensitivity of the two detector modes. In rough terms, the difference in sensitivity between the two calibration curves is the detector cross calibration factor.

While these two modes may have individual linear characteristics, they are not well correlated, which means that a calibration curve for each analyte would have a large undesirable break from one mode to the other.

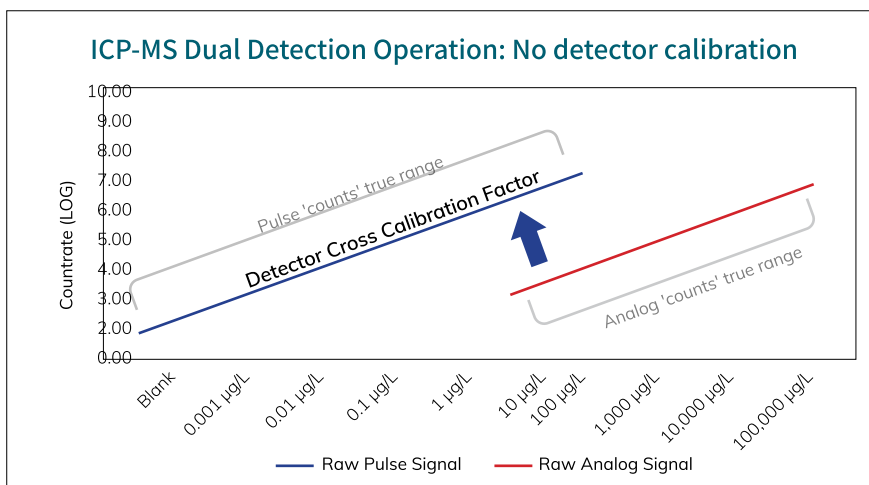
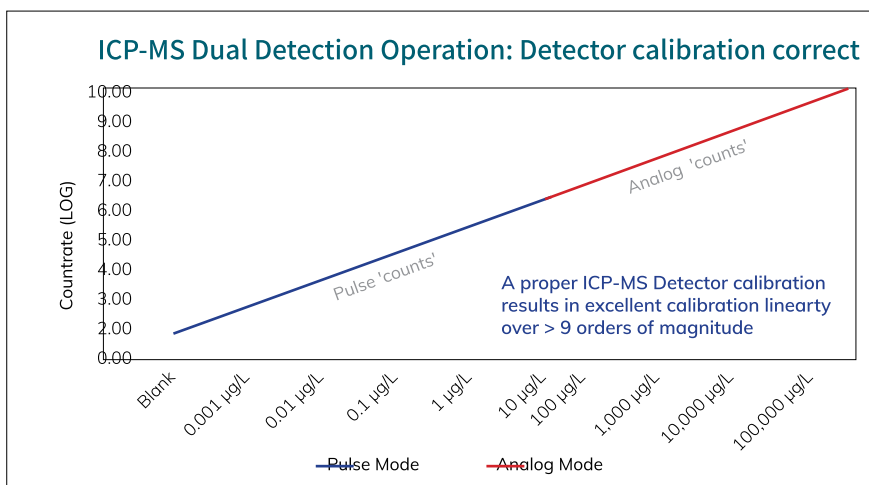


Figure 3. Pulsed counting and analog modes.

Detector cross calibration is used to align these modes into a single linear signal response (termed “cross-cal”, “P/A calibration”, or “dual detector calibration”) and allows the full linear, dynamic range of the instrument to be exploited.

Detector calibration is done with measurement of actual instrument raw data and is also mass dependent; hence the data used for detector calibration must cover the mass range for the analytical test and analytes involved.

# Performing Calibration

Instrument manufacturers typically recommend the use of specialized detector calibration standards. These are also useful for compliance with performance qualification (PQ). Usage is simple: merely deliver the detector calibration solution at the manufacturer's recommended concentration (usually a wide range is allowed) for the special tuning process designed for detector calibration (e.g. "P/A, Dual detector, detector cal"). It should be a normal part of system setup and maintenance. Frequency of re-calibration varies depending on the analytical requirements, but a daily update is optimal. New detectors and those nearing the end of their usable lifetime require more frequent calibration.

## Outcome of Incorrect or Non-Updated Detector Calibration

If detector calibration is not performed, or if the calibration is performed incorrectly or with inadequate solutions, system linearity suffers. As a result, analysts may experience accuracy errors and calibration curve problems.

## VHG™ Detector Calibration and other Tuning Solutions

VHG™, part of the larger LGC Standards family, manufactures a complete line of detector calibration, tuning, and wavelength calibration standards for most commercially available ICP/ICP-MS instruments. Many detector calibration standards are listed in the table below; for a full list of ICP-OES and ICP-MS Start Up Solutions, visit [lgcstandards.com/VHGAqueousInorganic](http://lgcstandards.com/VHGAqueousInorganic).

| Detector Calibration & Tuning Solutions |   |               |                                       |     |                    |   |
|---|---|---------------|---------------------------------------|-----|--------------------|---|
| Product                                 | Elements  | Conc. (µg/mL) | Matrix                                | mL  | Product No.        | Suitable for                                |
| Dual Detector Solution                  | Al, Ba, Ce, Co, Cu, In, Li, Mg, Mn, Ni, Pb, Tb, U, Zn                                 | 200           | 2% HNO <sub>3</sub>                   | 250 | VHG-LSUSPENXDD-250 | PerkinElmer® Cell ICP-MS: NexION™           |
| P/A Tuning Mix 1                        | Tb, Y   | 2.5           | 20% HCl, tr. HF                       | 100 | VHG-LDPA1-100      | Agilent® 7500, 7700, 7800, 7900, 8800, 8900 |
|   | <sup>6</sup> Li, Al, Bi, Ba, Co, Cr, Cu, In, Ir, Lu, Mn, Na, Th, Ti, Tl, Sc, Sr, U, V | 5             |                                       |     |                    |   |
|   | Ge, Mg, Mo, Ni, Pb, Pd, Ru, Sb, Sn  | 10            |                                       |     |                    |   |
|   | As, Be, Cd, Zn  | 20            |                                       |     |                    |   |
| P/A Tuning Solution 1                   | Y, Yb   | 2.5           | 2% HNO <sub>3</sub>                   | 100 | VHG-LAGPATSOL1-100 | Agilent® 7500, 7700, 7800, 7900, 8800, 8900 |
|   | Al, Ba, Bi, Co, Cr, Cu, In, <sup>6</sup> Li, Lu, Mn, Na, Sc, Sr, Th, Tl, U, V         | 5             |                                       |     |                    |   |
|   | Mg, Ni, Pb  | 10            |                                       |     |                    |   |
|   | As, Be, Cd, Zn  | 20            |                                       |     |                    |   |
| P/A Tuning Solution 2                   | Ir, Ti  | 5             | 10% HCl, 1% HNO <sub>3</sub> , tr. HF | 100 | VHG-LAGPATSOL2-100 | Agilent® 7500, 7700, 7800, 7900, 8800, 8900 |
|   | Ge, Mo, Pd, Ru, Sb, Sn  | 10            |                                       |     |                    |   |

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