TECHNICAL GUIDE

How to Prepare ICP Calibration Standards

For routine ICP analysis, laboratories should use properly documented, commercially prepared standards in the analyte combination, concentrations and matrix required by their method. CPI supplies both ready-to-order and custom-mixed ICP standards to reduce preparation time, minimize the risk of calculation or contamination errors, and support consistent calibration.

However, a laboratory may occasionally need to prepare an interim calibration standard when its normal CPI standard is not available from its on-hand inventory and testing must proceed.

This guide explains the key preparation considerations for those limited situations, subject to the laboratory's validated SOPs, applicable analytical method and quality-system requirements.

About the EPA Method Examples in This Guide

The calibration standards and preparation principles discussed in this guide support a broad range of ICP-MS and ICP-OES methods and laboratory applications. EPA Methods 200.8 and 200.7 are referenced throughout as practical, publicly available examples of how established analytical methods address calibration standards, stock solutions, calibration blanks, matrix consistency and quality control. Their use as examples does not mean that the guidance or CPI products discussed are limited to these methods or to the analysis of water and waste samples.

Requirements vary among regulatory methods, consensus methods, industry procedures and laboratory-developed methods. Laboratories should confirm product suitability and follow the analytical method, instrument instructions, validated SOPs and quality-system requirements applicable to their work.

Technical note

This guide is educational and does not replace a laboratory's validated SOP, instrument manual, SDS, quality system or applicable analytical method. Method-specific requirements for calibration levels, blanks, QC samples, frequencies and acceptance criteria should always be verified against the current official method.

Guide sections

- What ICP calibration standards are
- How to prepare ICP standards
- How to select a working range
- How to calculate dilutions
- Why matrix matching matters
- How working standards should be diluted
- Common preparation errors
- FAQ and CPI product-fit guidance

Accurate ICP calibration starts before the instrument run

ICP-MS and ICP-OES calibration depends on working standards that are prepared accurately, documented clearly and matched to the analytical method. The instrument produces a response; calibration standards provide the known reference points that convert that response into quantitative results.

For trace-element workflows, preparation errors can quickly become analytical errors. An incorrect dilution, contaminated volumetric flask, mismatched acid matrix, unstable element combination, or undocumented stock-standard lot can affect calibration, QC recovery, and reported results.

What is an ICP calibration standard?

An ICP calibration standard is a solution containing known concentrations of one or more elements used to calibrate the instrument response with respect to analyte concentration. EPA Methods 200.8 and 200.7 both define calibration standards as solutions prepared from the dilution of stock standard solutions and used to calibrate instrument response.

Calibration standards may be prepared from single-element standards, multi-element standards, Certified Reference Materials (CRMs), Reference Materials (RMs), or custom inorganic standards. The correct choice depends on the instrument, analytes, method, concentration range, matrix, QC requirements and documentation needs.

How do you prepare ICP calibration standards?

At a high level, ICP calibration-standard preparation follows seven practical steps:

Preparation workflow

Step	Action	Purpose
1	**Confirm the method and SOP**	Identify required calibration levels, blank composition, internal standards, source requirements, QC checks and acceptance criteria.
2	**Select the stock standard**	Use a suitable CRM/RM or other reference material with appropriate analytes, concentration, matrix, traceability and documentation.
3	**Define the working range**	Choose calibration levels that support the reporting range, expected samples, dilution procedure and instrument response.
4	**Match the matrix**	Prepare standards and blanks in a matrix appropriate for the method, analytes and sample-preparation chemistry.
5	**Calculate dilutions**	Use $C_1V_1 = C_2V_2$, dilution factors or gravimetric calculations as specified by the laboratory procedure.
6	**Prepare cleanly**	Use appropriate volumetric equipment, clean containers, high-purity diluent and contamination-control practices.
7	**Verify and document**	Record lot numbers, expiration or validity dates, preparation records, analyst, final concentration, matrix and required QC results.

Step 1: Start with the analytical method

Standard preparation should begin with the applicable method or validated laboratory SOP. Do not start with the concentration printed on a stock-standard label and work backward without first confirming the analytical requirement.

Review the method or SOP for:

- Required calibration range or calibration levels
- Calibration blank composition
- Required stock or intermediate standards
- Internal standards, if applicable
- Initial and continuing calibration verification requirements
- Interference-check or instrument-check requirements
- Matrix and acid requirements
- Source requirements, such as independent-source QC standards
- Acceptance criteria and corrective-action instructions

EPA Method 200.8 is an ICP-MS method for trace elements in waters and wastes, while EPA Method 200.7 is an ICP-AES/ICP-OES method for metals and trace elements in water and wastes. Their calibration and QC requirements are method-specific and should not be generalized to unrelated ICP methods.

Step 2: Select the appropriate stock standard

A stock standard is a concentrated reference solution used to prepare lower-concentration calibration or working standards. EPA Method 200.8 defines a stock standard solution as a concentrated solution containing one or more method analytes, prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

When selecting a stock standard, evaluate:

- Analytes included
- Concentration of each analyte
- Matrix and acid composition
- Compatibility of the elements in the formulation
- Certificate of Analysis documentation
- Traceability information
- Measurement uncertainty, when applicable
- Expiration or validity date
- Lot number and storage requirements
- Whether a CRM is required by the method or quality system

Product-fit note

For routine methods, a custom formulation can reduce repetitive preparation when the same analyte group, concentration pattern and matrix are prepared repeatedly.

Step 3: Define the working concentration range

The working calibration range should support the concentrations expected in samples after any preparation or dilution. It should also align with the method, reporting limits, instrument response and laboratory quality requirements.

When setting the range, consider:

- Expected sample concentrations
- Required reporting range
- Method detection or quantitation goals
- Sample dilution factors
- Instrument linear dynamic range
- Calibration model and linearity
- QC standard concentrations
- Whether high-level standards may create carryover or memory effects

Step 4: Select and match the matrix

Matrix matching means preparing calibration standards, blanks and QC materials in a matrix appropriate for the method, analytes and sample-preparation chemistry. The goal is to minimize differences between standards and samples that could affect transport, excitation/ionization, stability or analytical response.

EPA Methods 200.8 and 200.7 both define the calibration blank as reagent water acidified with the same acid matrix as the calibration standards. This reinforces an important preparation principle: the blank and standards should be chemically consistent where required by the method.

Matrix-selection checklist

Consideration	Preparation impact
Method requirements	Use the acid composition, calibration blank, internal standard and QC preparation requirements specified by the applicable method or validated SOP.
Analytes and concentration	Confirm that each element is stable in the selected matrix at the target concentration. Compatibility may change at higher concentrations.
Instrument and introduction system	Verify compatibility with sample-introduction components, especially when using chloride-rich or HF-containing matrices.
Sample matrix	When appropriate, prepare standards and blanks in a matrix that reflects sample-preparation chemistry and minimizes mismatch.
Contamination risk	Use appropriate purity grade reagents, clean labware and controlled handling practices for trace-level work.

Should ICP standards be matrix matched?

Yes, when required by the method or necessary for analytical performance. Matrix matching is especially important when samples and standards differ in acid concentration, dissolved solids, salts, viscosity or other components that affect sample introduction and instrument response.

Matrix matching can help reduce problems such as:

- Biased calibration response
- Poor recovery in fortified samples
- Signal suppression or enhancement
- Instability of specific elements
- Unexpected blank or QC failures
- Physical effects in nebulization or transport

Matrix matching is not a substitute for proper interference correction or sample preparation. It is one part of a broader calibration and QC strategy.

Step 5: Calculate the dilution

For simple volumetric dilutions, laboratories commonly use:

$$C_1V_1 = C_2V_2$$

Where:

- C_1 = concentration of the stock or intermediate standard
- V_1 = volume of stock or intermediate standard needed
- C_2 = desired final concentration
- V_2 = final prepared volume

Example calculation

To prepare 100 mL of a 10 µg/L working standard from a 1,000 µg/mL stock standard:

Value	Input	Notes
C_1	1,000 µg/mL	Equivalent to 1,000,000 µg/L
C_2	10 µg/L	Target working concentration
V_2	100 mL	Final volume
V_1	0.001 mL = 1 µL	Direct dilution is impractically small for many routine preparations

Practical preparation point

When V_1 is too small for accurate routine measurement, prepare one or more intermediate standards or use a custom formulation closer to the required working concentration. Avoid forcing a direct dilution that is mathematically correct but operationally unreliable.

Step 6: Decide whether to use direct or serial dilution

Working standards can be prepared by direct dilution from a stock standard, by serial dilution through one or more intermediate standards, or by using a custom-prepared standard closer to the desired working range.

Direct vs. serial dilution

Approach	Best used when	Preparation note
Direct dilution	Useful when the required stock volume can be measured accurately and the final concentration is not extremely far below the stock concentration.	Simple and minimizes intermediate containers but may be unsuitable when the required transfer volume is very small.
Serial dilution	Useful when preparing trace-level standards from high-concentration stock solutions.	Improves practical volume measurement, but each dilution step introduces potential error and must be documented.
Custom working-range standard	Useful when the same working mixture is prepared repeatedly.	Can reduce routine preparation steps and dilution-error opportunities if the formulation is stable and appropriate.

How should working standards be diluted?

Working standards should be diluted quantitatively, in clean and appropriate containers, using the same or appropriate acid matrix, and with volumes that can be measured accurately. Dilution practices should follow the laboratory SOP, method requirements and quality system.

Good practice includes:

- Bring stock standards to appropriate room conditions if required by the SOP.
- Mix stock and intermediate standards thoroughly before use.
- Use calibrated volumetric pipettes, dispensers, balances or flasks as required.
- Avoid pipetting volumes near the lower practical limit of the device.
- Add acid and diluent in an order consistent with the SOP and safety requirements.
- Dilute to volume accurately and mix completely.
- Use clean containers appropriate for trace metals work.
- Label working standards with analytes, concentration, matrix, preparation date, preparer, lot/source and expiration or holding time.
- Document all calculations and preparation records.

Step 7: Prepare the calibration blank

The calibration blank is not simply water. In EPA Methods 200.8 and 200.7, it is reagent water acidified with the same acid matrix as the calibration standards. That matrix consistency is critical because the blank establishes the zero-concentration calibration point under the same chemical conditions used for the standards.

A calibration blank should be prepared with attention to:

- Reagent-water quality
- Acid purity
- Acid concentration

- Container cleanliness
- Potential contamination from labware or handling
- Consistency with calibration standards
- Applicable method and SOP instructions

Step 8: Prepare calibration-verification and QC standards separately

Calibration standards establish the calibration curve. Calibration-verification and QC standards check whether calibration and method performance remain acceptable. They should not be treated as interchangeable unless the applicable method or SOP permits it.

Common QC-related standards include:

- Initial Calibration Verification (ICV) standards
- Continuing Calibration Verification (CCV) standards
- Interference Check Standards
- Instrument Check Standards
- Tuning and Setup solutions
- Laboratory fortified blanks or laboratory control samples, where specified

EPA Method 200.8 defines a Quality Control Sample as a known-concentration solution obtained from a source external to the laboratory and different from the source of calibration standards. That definition should be applied in the context of the method and not assumed to apply identically to every non-EPA method.

Contamination control for ICP standard preparation

Trace-metal calibration is vulnerable to low-level contamination. The lower the calibration range, the more important clean handling becomes.

Common contamination sources include:

- Impure acids or diluents
- Reusable labware that was not cleaned appropriately
- Pipette tips, caps or containers not suitable for trace metals work
- Carryover from high-concentration stock standards
- Dust, fingerprints or exposed work surfaces
- Metal tools or fixtures near the preparation area
- Mislabeling or transcription errors

Preparation record essentials

Record the stock-standard lot, source, certificate reference, concentration, matrix, expiration/validity date, preparation calculation, final volume, diluent, container, preparation date, preparer and any assigned working-standard expiration or holding time.

ICP-MS-specific preparation considerations

ICP-MS can measure elements at very low concentrations, so preparation cleanliness, memory effects, internal standards and matrix effects are especially important.

EPA Method 200.8 notes that instrumental drift and matrix-related suppressions or enhancements must be corrected using internal standards. When internal standards are added manually or online, they should be present consistently in standards, blanks and samples according to the method or SOP.

For ICP-MS workflows, verify:

- Internal-standard elements and concentrations
- Whether internal standards are added online or during preparation
- Potential polyatomic or isobaric interferences
- Dissolved-solids limitations and dilution requirements
- Rinse solution and carryover controls
- Trace-level contamination controls

ICP-OES-specific preparation considerations

ICP-OES calibration preparation must account for the selected wavelengths, expected concentration range, background correction, matrix effects and potential spectral overlap.

EPA Method 200.7 describes multielement determinations by ICP-AES using sequential or simultaneous instruments and notes that background correction and interferences must be considered and addressed appropriately.

For ICP-OES workflows, verify:

- Calibration concentrations appropriate for the selected wavelength and linear range
- Background correction requirements
- Potential spectral overlap
- Acid matrix and dissolved-solids compatibility
- Interference-check requirements
- Blank and calibration-verification requirements

Common ICP calibration-standard preparation mistakes

Mistake	Better practice
Using water as the blank when an acid matrix is required	Prepare the calibration blank in the same acid matrix as the calibration standards when required by the method.
Making a mathematically correct but impractical dilution	Use intermediate dilutions or a custom formulation when the required transfer volume is too small for reliable preparation.
Assuming all elements can be combined	Confirm multi-element compatibility, concentration and matrix stability before combining analytes.
Ignoring stock-standard matrix	Account for the matrix introduced from the stock standard when preparing final working standards.
Using expired or undocumented standards	Verify certificate, lot, storage and validity information before preparation.
Treating QC standards as calibration standards	Keep calibration, verification, interference and tuning functions distinct unless the method states otherwise.

Failing to document calculations	Record enough information for another qualified analyst to reconstruct the preparation.

When a custom ICP standard can reduce preparation work

If a laboratory repeatedly prepares the same multi-element mixture, a custom standard can reduce routine preparation steps while matching the method's analytes, concentrations, matrix and volume.

A custom standard may be useful when:

- The same mixture is prepared frequently
- Several stock standards are combined during each preparation
- The method requires unusual concentration ratios
- The laboratory wants to reduce pipetting or transcription steps
- The final matrix or analyte combination is not available as a catalog item
- The lab wants to order only the volume it needs

A custom formulation still needs technical review. The requested analytes, concentrations and matrix must be chemically appropriate and stable for the intended use.

FAQ

Should ICP standards be matrix matched?

Yes, when required by the method or needed to minimize response differences between standards and samples. At minimum, blanks and standards should follow the method's specified acid matrix.

How should working standards be diluted?

Working standards should be diluted using validated volumetric or gravimetric procedures, appropriate clean containers, high-purity diluents, the required acid matrix and practical transfer volumes. Very small transfer volumes should usually be avoided through intermediate dilutions or custom formulations.

Can I prepare ICP calibration standards from a 1,000 µg/mL stock?

Yes, if the stock standard is appropriate for the method and documentation requirements. Use quantitative dilution calculations and ensure the final concentration and matrix are suitable.

Can I combine multiple ICP elements into one standard?

Often, but not always. Element compatibility, concentration, acid matrix, oxidation state and long-term stability must be evaluated before preparing a multi-element standard. If you need multiple elements in one standard a custom standard is recommended.

Is an ICP calibration blank the same as reagent water?

Not necessarily. EPA Methods 200.8 and 200.7 define the calibration blank as reagent water acidified with the same acid matrix as the calibration standards.

What documentation should be kept for prepared working standards?

Keep the calculation, source standard lot numbers, certificate references, matrix, final concentration, preparation date, preparer, final volume, container, expiration or holding time and any applicable QC results.

Choose standards around the method, not around convenience

The best calibration standard is not simply the most concentrated stock solution or the largest multi-element mix. It is the formulation that fits the method, instrument, analytes, concentration range, matrix, QC requirements and documentation expectations.

CPI International supports ICP-MS and ICP-OES laboratories with:

- ICP Calibration Standards
- ICP Quality Control Standards
- ICP Tuning and Setup Solutions
- Single Element ICP Standards
- Custom Inorganic Standards

Explore CPI's ICP Standards or configure a Custom Inorganic Standard for your analytical workflow.

Source notes for technical review

This guide avoids assigning universal calibration frequencies, acceptance limits or QC requirements as those details are method-specific.

U.S. EPA Method 200.8 page: https://www.epa.gov/esam/epa-method-2008-determination-trace-elements-waters-and-wastes-inductively-coupled-plasma-mass

U.S. EPA Method 200.8 PDF: https://www.epa.gov/sites/default/files/2015-06/documents/epa-200.8.pdf

U.S. EPA Method 200.7 page: https://www.epa.gov/esam/method-2007-determination-metals-and-trace-elements-water-and-wastes-inductively-coupled

U.S. EPA Method 200.7 PDF: https://www.epa.gov/sites/default/files/2015-06/documents/epa-200.7.pdf