



Designation: **D5673—10 D5673 – 15**

Standard Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry¹

This standard is issued under the fixed designation D5673; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers the determination of dissolved elements in ground water, surface water, and drinking water. It may also be used for the determination of total-recoverable elements in these waters as well as wastewater.²

1.2 This test method should be used by analysts experienced in the use of inductively coupled plasma—mass spectrometry (ICP-MS), the interpretation of spectral and matrix interferences and procedures for their correction.

1.3 It is the user's responsibility to ensure the validity of the test method for waters of untested matrices.

1.4 **Table 1** lists elements for which the test method applies, with recommended masses and typical estimated instrumental detection limits using conventional pneumatic nebulization. Actual working detection limits are sample dependent and, as the sample matrix varies, these detection limits may also vary. In time, other elements may be added as more information becomes available and as required.

1.4.1 This method covers the analysis of mine dewatering groundwater and wastewater effluent in the range of 2–120 $\mu\text{g/L}$ dissolved antimony and 3–200 $\mu\text{g/L}$ dissolved arsenic.

1.4.2 This method covers the analysis of metallurgical processing cyanide solutions in the range of 1–500 $\mu\text{g/L}$ dissolved gold.

¹ This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water. Current edition approved Aug. 1, 2010/July 1, 2015. Published September 2010/August 2015. Originally approved in 1996. Last previous edition approved in 2005/2010 as D5673–05/D5673 – 10. DOI: 10.1520/D5673-10.10.1520/D5673-15.

² EPA Test Method: Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma—Mass Spectrometry, Method 200.8.

TABLE 1 Recommended Analytical Mass and Estimated Instrument Detection Limits

| Element | Recommended Analytical Mass | Estimated Instrument Detection Limit, $\mu\text{g/L}$ ^A |
|------------|-----------------------------|--|
| Aluminum | 27 | 0.05 |
| Antimony | 121 | 0.08 |
| Arsenic | 75 | 0.9 |
| Barium | 137 | 0.5 |
| Beryllium | 9 | 0.1 |
| Cadmium | 111 | 0.1 |
| Chromium | 52 | 0.07 |
| Cobalt | 59 | 0.03 |
| Copper | 63 | 0.03 |
| Gold | 197 | 0.01 |
| Lead | 206, 207, 208 | 0.08 |
| Manganese | 55 | 0.1 |
| Molybdenum | 98 | 0.1 |
| Nickel | 60 | 0.2 |
| Selenium | 82 | 5.0 |
| Silver | 107 | 0.05 |
| Thallium | 205 | 0.09 |
| Thorium | 232 | 0.03 |
| Uranium | 238 | 0.02 |
| Vanadium | 51 | 0.02 |
| Zinc | 66 | 0.2 |

^A Instrument detection limits (3σ) estimated from seven replicate scans of the blank (1 % v/v HNO_3) and three replicate integrations of a multi-element standard.

*A Summary of Changes section appears at the end of this standard

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:³

[D1066 Practice for Sampling Steam](#)

[D1129 Terminology Relating to Water](#)

[D1193 Specification for Reagent Water](#)

[D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water](#)

[D3370 Practices for Sampling Water from Closed Conduits](#)

[D5810 Guide for Spiking into Aqueous Samples](#)

[D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

[E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method](#)

[E1763 Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods \(Withdrawn 2015\)⁴](#)

3. Terminology

3.1 ~~Definitions~~—For definitions of other terms used in this test method, refer to Terminology [D1129](#).

3.1 Definitions:

3.1.1 For definitions of terms used in this standard, refer to Terminology [D1129](#).

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calibration blank, n*—a volume of water containing the same acid matrix as is in the calibration standards (see [11.1](#)).

3.2.2 *calibration standards, n*—a series of known standard solutions used by the analyst for calibration of the instrument (that is, preparation of the analytical curve) (see Section [11](#)).

3.2.3 *calibration stock solution, n*—a solution prepared from the stock standard solution(s) to verify the instrument response with respect to analyte concentration.

3.2.4 *dissolved, n=adj*—those elements that will pass ~~capable of passing~~ through a 0.45- μ m membrane filter.

3.2.5 *interference check sample A (ICSA), n*—a solution containing matrix elements at environmental levels that result in interferences on target low level analytes.

3.2.5.1 Discussion—

ICSA is different from the mixed element standards in [8.4](#), which are intended for instrument calibration, not for checking interferences. The interferences formed in the ICP can be corrected for by use of element-specific correction equations ~~or equations~~, collision cell technology with quadrupole-based ICP-MS, or ~~high-resolution~~ high-resolution ICP-MS.

3.2.6 *interference check sample B (ICSAB), n*—the ICSA solution spiked with 20 μ g/L each As and Sb.

3.2.7 *instrumental detection limit (IDL), n*—the concentration equivalent to a signal, ~~which that~~ is equal to three times the standard deviation of the blank signal at the selected analytical mass(es).

3.2.8 *internal standard, n*—pure analyte(s) element(s) added in known amount(s) to a solution.

3.2.8.1 Discussion—

~~This~~ The internal standard is used to measure the relative instrument response relative to the other analytes that are components of the same solution. The internal standards must be analyte elements that are not a sample component.

3.2.9 *method detection limit (MDL), n*—the minimum ~~concentration of an analyte~~ analyte concentration that can be identified, measured and reported with ~~99-%~~ 99 % confidence that the analyte concentration is greater than zero.

3.2.9.1 Discussion—

TABLE 2 Recommended Analytical Isotopes and Additional Masses That Are Recommended To Be Monitored

| Isotope | Element of Interest |
|---------------------------|---------------------|
| <u>27</u> | Aluminum |
| <u>121, 123</u> | Antimony |
| <u>75</u> | Arsenic |
| <u>135, 137</u> | Barium |
| <u>9</u> | Beryllium |
| <u>106, 108, 111, 114</u> | Cadmium |
| <u>52, 53</u> | Chromium |
| <u>59</u> | Cobalt |
| <u>63, 65</u> | Copper |
| <u>206, 207, 208</u> | Lead |
| <u>55</u> | Manganese |
| <u>95, 97, 98</u> | Molybdenum |
| <u>60, 62</u> | Nickel |
| <u>77, 82</u> | Selenium |
| <u>107, 109</u> | Silver |
| <u>203, 205</u> | Thallium |
| <u>232</u> | Thorium |
| <u>238</u> | Uranium |
| <u>51</u> | Vanadium |
| <u>66, 67, 68</u> | Zinc |
| <u>83</u> | Krypton |
| <u>99</u> | Ruthenium |
| <u>105</u> | Palladium |
| <u>118</u> | Tin |

TABLE 2 Recommended Analytical Isotopes and Additional Masses That Are Recommended To Be Monitored

| Isotope ^A | Element of Interest |
|---------------------------|---------------------|
| <u>27</u> | Aluminum |
| <u>121, 123</u> | Antimony |
| <u>75</u> | Arsenic |
| <u>135, 137</u> | Barium |
| <u>9</u> | Beryllium |
| <u>106, 108, 111, 114</u> | Cadmium |
| <u>52, 53</u> | Chromium |
| <u>59</u> | Cobalt |
| <u>63, 65</u> | Copper |
| <u>206, 207, 208</u> | Lead |
| <u>55</u> | Manganese |
| <u>95, 97, 98</u> | Molybdenum |
| <u>60, 62</u> | Nickel |
| <u>77, 82</u> | Selenium |
| <u>107, 109</u> | Silver |
| <u>203, 205</u> | Thallium |
| <u>232</u> | Thorium |
| <u>238</u> | Uranium |
| <u>51</u> | Vanadium |
| <u>66, 67, 68</u> | Zinc |
| <u>83</u> | Krypton |
| <u>99</u> | Ruthenium |
| <u>105</u> | Palladium |
| <u>118</u> | Tin |

^A Isotopes recommended for analytical determination are underlined. These masses were recommended and are reflected in the precision and bias data. Alternate masses may be used but interferences must be documented.

This confidence level is determined from analysis of a sample in a given matrix containing the analyte(s).

3.2.10 *quality control reference solution (QCS)*, *n*—a solution with the certified concentration(s) of the analytes, prepared by an independent laboratory, and used for a verification of the instrument’s calibration.

3.2.11 *reagent blank*, *n*—a volume of water containing the same matrix as the calibration standards, carried through the entire analytical procedure.

3.2.12 *stock standard solution*, *n*—a concentrated solution containing one or more analytes, obtained as a certified solution from a reputable source or prepared as described in [Table 4](#).

3.2.13 *total-recoverable*, *n*—*adj*—a term relating to forms of each element that are determinable by the digestion method included in this procedure (see [12.2](#)).

TABLE 3 Common Molecular Ion Interferences

| Molecular Ion | Mass | Element Interference ^A |
|--|------|-----------------------------------|
| NH [±] | 15 | ∞ |
| NH ⁺ | 15 | ∞ |
| OH [±] | 17 | ∞ |
| OH ⁺ | 17 | ∞ |
| OH ₂ ⁺ | 18 | ∞ |
| C ₂ ⁺ | 24 | ∞ |
| CN [±] | 26 | ∞ |
| CN ⁺ | 26 | ∞ |
| CO [±] | 28 | ∞ |
| CO ⁺ | 28 | ∞ |
| N ₂ ⁺ | 28 | ∞ |
| N ₂ H [±] | 29 | ∞ |
| N ₂ H ⁺ | 29 | ∞ |
| NO ⁺ | 30 | ∞ |
| NOH [±] | 31 | ∞ |
| NOH ⁺ | 31 | ∞ |
| O ₂ ⁺ | 32 | ∞ |
| O ₂ H [±] | 33 | ∞ |
| O ₂ H ⁺ | 33 | ∞ |
| ³⁶ ArH [±] | 37 | ∞ |
| ³⁶ ArH ⁺ | 37 | ∞ |
| ³⁶ ArH [±] | 39 | ∞ |
| ³⁶ ArH ⁺ | 39 | ∞ |
| ⁴⁰ ArH [±] | 41 | ∞ |
| ⁴⁰ ArH ⁺ | 41 | ∞ |
| CO ₂ ⁺ | 44 | ∞ |
| CO ₂ H [±] | 45 | Se |
| CO ₂ H ⁺ | 45 | Sc |
| ArC ⁺ , ArO [±] | 52 | Cr |
| ArC ⁺ , ArO ⁺ | 52 | Cr |
| ArN ⁺ | 54 | Cr |
| ArNH ⁺ | 55 | Mn |
| ArO ⁺ | 56 | ∞ |
| ArOH ⁺ | 57 | ∞ |
| ⁴⁰ Ar ³⁶ Ar [±] | 76 | Se |
| ⁴⁰ Ar ³⁶ Ar ⁺ | 76 | Se |
| ⁴⁰ Ar ³⁸ Ar [±] | 78 | Se |
| ⁴⁰ Ar ³⁸ Ar ⁺ | 78 | Se |
| ⁴⁰ Ar ₂ ⁺ | 80 | Se |
| ¹¹⁸ Ta ¹⁶ O ⁺ | 197 | Au |

| Matrix Molecular Ions | | |
|--|----|-------|
| Chloride | | |
| ³⁵ ClO [±] | 51 | V |
| ³⁵ ClO ⁺ | 51 | V |
| ³⁵ ClOH [±] | 52 | Cr |
| ³⁵ ClOH ⁺ | 52 | Cr |
| ³⁷ ClO [±] | 53 | Cr |
| ³⁷ ClO ⁺ | 53 | Cr |
| ³⁷ ClOH [±] | 54 | Cr |
| ³⁷ ClOH ⁺ | 54 | Cr |
| Ar ³⁵ Cl [±] | 75 | As |
| Ar ³⁵ Cl ⁺ | 75 | As |
| Ar ³⁷ Cl [±] | 77 | Se |
| Ar ³⁷ Cl ⁺ | 77 | Se |
| Sulphate | | |
| ³² SO [±] | 48 | ∞ |
| ³² SO ⁺ | 48 | ∞ |
| ³² SOH [±] | 49 | ∞ |
| ³² SOH ⁺ | 49 | ∞ |
| ³⁴ SO [±] | 50 | V, Cr |
| ³⁴ SO ⁺ | 50 | V, Cr |
| ³⁴ SOH [±] | 51 | V |
| ³⁴ SOH ⁺ | 51 | V |
| SO ₂ ⁺ , S ₂ ⁺ | 64 | Zn |
| Ar ³² S [±] | 72 | ∞ |
| Ar ³² S ⁺ | 72 | ∞ |
| Ar ³⁴ S [±] | 74 | ∞ |
| Ar ³⁴ S ⁺ | 74 | ∞ |
| Phosphate | | |
| PO [±] | 47 | ∞ |
| PO ⁺ | 47 | ∞ |
| POH [±] | 48 | ∞ |
| POH ⁺ | 48 | ∞ |

| Background Molecular Ions | | |
|------------------------------|------------|-----------------------------------|
| Molecular Ion | Mass | Element Interference ^A |
| PO ₂ ⁺ | 63 | Cu |
| ArP [±] | 74 | ... |
| ArP ⁺ | 71 | ... |
| Group I, II Metals | | |
| ArNa [±] | 63 | Cu |
| ArNa ⁺ | 63 | Cu |
| ArK [±] | 79 | ... |
| ArK ⁺ | 79 | ... |
| ArCa [±] | 80 | ... |
| ArCa ⁺ | 80 | ... |
| Matrix Oxides ^B | | |
| TiO | 62 to 66 | Ni, Cu, Zn |
| ZrO | 106 to 112 | Ag, Cd |
| MoO | 108 to 116 | Cd |

^A Method elements or internal standards affected by molecular ions.

^B Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes be monitored if samples are likely to contain high levels of these elements. Mo is monitored as a method analyte.

TABLE 4 Preparation of Metal Stock Solutions^A

| Element or Compound | Weight, g | Solvent |
|--------------------------------------|-----------|---|
| Al | 0.1000 | 10 mL of HCl (sp gr 1.19) + 2 mL of HNO ₃ (sp gr 1.42) |
| Sb | 0.1000 | 0.5 mL of HCl (sp gr 1.19) + 2 mL of HNO ₃ (1 + 1) |
| As ₂ O ₃ | 0.1320 | 1 mL of NH ₄ OH (sp gr 0.902) + 50 mL of H ₂ O |
| BaCO ₃ | 0.1437 | 2 mL of HNO ₃ (sp gr 1.42) + 10 mL of H ₂ O |
| BeSO ₄ ·4H ₂ O | 1.9650 | 50 mL of H ₂ O, add 1 mL of HNO ₃ (sp gr 1.42) |
| Bi ₂ O ₃ | 0.1115 | 5 mL of HNO ₃ (sp gr 1.42) |
| Cd | 0.1000 | 5 mL of HNO ₃ (1 + 1) |
| CrO ₃ | 0.1923 | 1 mL of HNO ₃ (sp 1.42) + 10 mL H ₂ O |
| Co | 0.1000 | 5 mL of HNO ₃ (1 + 1) |
| Cu | 0.1000 | 5 mL of HNO ₃ (1 + 1) |
| Au | 0.1000 | 20 mL H ₂ O, add 8 mL of HCL = 5 mL HNO ₃ (1 + 1) |
| In | 0.1000 | 10 mL of HNO ₃ (1 + 1) |
| PbNO ₃ | 0.1599 | 5 mL of HNO ₃ (1 + 1) |
| MgO | 0.1658 | 10 mL of HNO ₃ (1 + 1) |
| Mn | 0.1000 | 5 mL of HNO ₃ (1 + 1) |
| MoO ₃ | 0.1500 | 1 mL of NH ₄ OH (sp gr 0.902) + 10 mL of H ₂ O |
| Ni | 0.1000 | 5 mL of HNO ₃ (sp gr 1.42) |
| Sc ₂ O ₃ | 0.1534 | 5 mL of HNO ₃ (1 + 1) |
| SeO ₂ | 0.1405 | 20 mL of H ₂ O |

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| Element or Compound | Weight, g | Solvent |
|--|-----------|---|
| Ag | 0.1000 | 5 mL of HNO ₃ (1 + 1) |
| Tb ₄ O ₇ | 0.1176 | 5 mL of HNO ₃ (sp gr 1.42) |
| TiNO ₃ | 0.1303 | 1 mL of HNO ₃ (sp gr 1.42) + 10 mL of H ₂ O |
| Th(NO ₃) ₄ ·4H ₂ O | 0.2380 | 20 mL of H ₂ O |
| UO ₂ (NO ₃) ₂ ·6H ₂ O | 0.2110 | 20 mL of H ₂ O |
| V | 0.1000 | 5 mL of HNO ₂ (1 + 1) |
| Y ₂ O ₃ | 0.1270 | 5 mL of HNO ₃ (1 + 1) |
| Zn | 0.1000 | 5 mL of HNO ₃ (1 + 1) |

- ^A Metal stock solutions, 1.00 mL = 1000 µg of metal. Dissolve the listed weights of each metal or compound as specified in Table 4, then dilute to 100 mL with water. The metals may require heat to increase rate of dissolution. Commercially available standards of known purity may be used. Alternate salts or oxides may also be used.

3.2.14 *tuning solution, n*—a solution that is used to determine acceptable instrument performance prior to calibration and sample analysis.

3.3 Acronyms:

3.3.1 *ICSA, n*—interference check sample A

3.3.2 *ICSAB, n*—interference check sample B

3.3.3 *IDL, n*—instrumental detection limit

3.3.4 *MDL, n*—method detection limit

3.3.5 *QCS, n*—quality-control reference solution

4. Summary of Test Method

4.1 This test method describes the multi-element determination of trace elements by inductively coupled plasma—mass spectrometry (ICP-MS). Sample material in solution is introduced by pneumatic nebulization into a radiofrequency plasma where energy transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer. The ions transmitted through the quadrupole are detected by a continuous dynode electron multiplier assembly and the ion information processed by a data handling system. Interferences relating to the technique must be recognized and corrected for (see Section 6 on interferences). Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents, or sample matrix. Instrumental drift as well as suppressions or enhancements of instrument response caused by the sample matrix must be corrected for by the use of internal standardization.

5. Significance and Use

5.1 The test method is useful for the determination of element concentrations in many natural waters—waters, metallurgical process cyanide solutions and wastewaters. It has the capability for the determination of up to 2021 elements. High analysis sensitivity can be achieved for some elements that are difficult to determine by other techniques.

6. Interferences

6.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. These interferences can be summarized as follows:

6.1.1 *Isobaric Elemental Interferences*—Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use—use by ICP-MS. All elements determined by this test method have, at a minimum, one isotope free of isobaric