



Designation: D8186 – 18

# Standard Test Method for Measurement of Impurities in Graphite by Electrothermal Vaporization Inductively Coupled Plasma Optical Emission Spectrometry (ETV-ICP OES)<sup>1</sup>

This standard is issued under the fixed designation D8186; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the measurement of mass fractions of the elements silver (Ag), aluminum (Al), arsenic (As), boron (B), barium (Ba), beryllium (Be), bismuth (Bi), calcium (Ca), cadmium (Cd), cobalt (Co), chromium (Cr), copper (Cu), iron (Fe), potassium (K), lithium (Li), magnesium (Mg), manganese (Mn), molybdenum (Mo), sodium (Na), nickel (Ni), phosphorus (P), lead (Pb), sulfur (S), antimony (Sb), silicon (Si), tin (Sn), strontium (Sr), titanium (Ti), vanadium (V), tungsten (W), yttrium (Y), zinc (Zn), and zirconium (Zr) in graphite.

1.2 Provided that an appropriate validation procedure is carried out, this test method is also applicable to other carbon materials such as coal, coke, carbon black, graphite-felt, graphite-foil, graphite-foam, and fiber reinforced carbon-carbon composites.

1.3 This test method is applicable to element contents from approximately 0.0001 mg/kg to 1000 mg/kg (0.1 ppmw to 1000 ppmw), depending on element, wavelength, measurement parameters, and sample mass.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.

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## 2. Referenced Documents

2.1 *ASTM Standards*:<sup>2</sup>

D1193 Specification for Reagent Water

2.2 *ISO Standards*:<sup>3</sup>

ISO 5725-2 Accuracy (trueness and precision) of measurement methods and results—Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

## 3. Terminology

3.1 *Definitions*:

3.1.1 *ETV, n*—electrothermal vaporization.

3.1.2 *ICP OES, n*—inductively coupled plasma optical emission spectrometry.

## 4. Summary of Test Method

4.1 The ETV unit consists of an electrically heated graphite tube furnace. Graphite boats which fit into the graphite tube are used for inserting the sample, crushed and milled if necessary, into the furnace. Handling of graphite boats is preferably done using an automated system. One end of the furnace, which is sealed with a movable door, is used for inserting the graphite boats (furnace inlet). The other end of the furnace (furnace outlet) is connected via a tube to the injector tube of the ICP-torch. The graphite tube furnace is heated rapidly to a temperature where evaporation of analyte elements takes place. For complete volatilization of analyte elements, a halogenating reaction gas is added to the argon carrier gas stream. The evaporation products containing the analyte elements are transported as dry aerosol with the argon carrier gas stream from the furnace outlet to the ICP-torch where they are excited to emit optical radiation. The emitted radiation is dispersed and detected by a simultaneous spectrometer. The intensity of radiation of emission lines and background (optional) is

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

measured with appropriate detectors. The mass fractions of the analyte elements are calculated by comparing the intensities of the element-specific spectral lines of the sample with calibration samples of known analyte content.

4.2 For ICP OES, sample introduction is usually done by nebulization of liquids. In the case of graphite, sample decomposition prior to analysis is required, for example, by ashing, melt-fusion, or acid/pressure-decomposition. These decomposition procedures are time-consuming, and the possibility of introduction of impurities as well as analyte losses represents a serious source of systematic errors. In ETV-ICP OES, sample introduction by nebulization of liquids is replaced by the electrothermal vaporization of solid samples at high temperatures in a graphite tube furnace, thus eliminating the need for wet chemical sample decomposition prior to analysis. In general, ETV-ICP OES provides a linear working range of up to four orders of magnitude. This range can be expanded for the respective elements by selecting emission lines with different sensitivity or variation of sample mass, or both.

4.3 A prerequisite for ETV-ICP OES is an efficient transport of the gaseous products generated in the graphite tube furnace during the heating step to the ICP-torch. This is achieved by a suitable graphite tube design and gas regime in the transition area between the graphite tube and transport tube as shown in Figs. 1 and 2. See also Refs (1-4).<sup>4</sup> A nozzle-type graphite tube and the use of a bypass-gas in the gap between the graphite tube and transport tube are the key factors for high and reproducible transport efficiencies as well as minimized matrix effects. The temperature of the graphite tube furnace in the evaporation step depends on the analytes to be determined. Release of volatile analytes (such as arsenic, cadmium, potassium, lithium, and sodium) from the graphite matrix starts at 500 °C to 800 °C. To measure all elements listed in 1.1 a temperature of 2600 °C is required.

4.4 By addition of a halogen-containing reaction gas to the carrier gas, the vaporization temperatures of elements are lowered through the formation of volatile halides. In the case of carbide-forming elements, halogenation is a prerequisite to

vaporize these elements. In addition, aerosols formed by pyrolysis of the reaction gas act as condensation nuclei for sample vapors, which have a positive effect on transport efficiency (see Refs (1-3), (5-7)). To measure all elements listed in 1.1, dichlorodifluoromethane (CCl<sub>2</sub>F<sub>2</sub>) shall be used as reaction gas. Using other reaction gases (for example, CF<sub>4</sub>, CCl<sub>2</sub>F<sub>2</sub>, CHF<sub>3</sub>, CHClF<sub>2</sub>, C<sub>2</sub>H<sub>2</sub>F<sub>4</sub>, SF<sub>6</sub>, and NF<sub>3</sub>) may result in reduced release of some analytes from the graphite matrix.

4.5 The dry aerosol is transported by means of suitable tubing to the injector tube of the plasma torch of the ICP spectrometer where it is excited to emit optical radiation (see Fig. 3).

4.6 A description of possible interferences and their elimination is given in Appendix X2.

## 5. Significance and Use

5.1 The presence and content of various impurities in graphite are major considerations in determining the suitability of graphite for various applications. This test method provides an alternative means of determining the content of trace impurities in a graphite sample which has considerable advantages compared to classical wet-chemical analysis methods.

5.2 The test method provides a standard procedure to measure impurities in graphite and to assure required graphite specifications.

## 6. Apparatus

6.1 Laboratory instruments are required as detailed in 6.2 to 6.8. In the case of the spectrometer (6.2) and the ETV system (6.3), the user shall follow the manufacturer's instructions on use of the apparatus.

6.2 *Inductively Coupled Plasma Optical Emission Spectrometer*—A simultaneous method capable of recording transient emission signals, suited to synchronize data acquisition with an ETV heating cycle.

NOTE 1—In ETV-ICP OES, the evaporating sample may cause a significant alteration to the spectral background near the emission lines, which increases the measurement uncertainty. This effect can be reduced if the spectrometer is capable of measuring the intensity of emission lines and the intensity of spectral background close to the emission lines

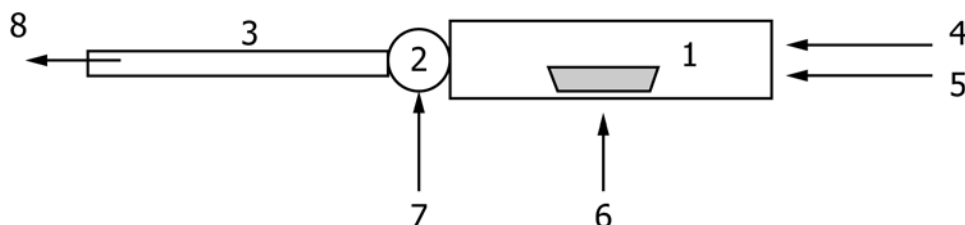


FIG. 1 ETV Unit: Schematic Design of Gas Flows

Key:

- 1 Graphite tube with graphite boat and sample
- 2 Transition area between graphite tube and transport tube
- 3 Transport tube
- 4 Carrier gas (argon)
- 5 Reaction gas (CCl<sub>2</sub>F<sub>2</sub>)
- 6 Furnace shield gas (argon)
- 7 Bypass gas (argon)
- 8 Dry aerosol to injector tube of plasma torch

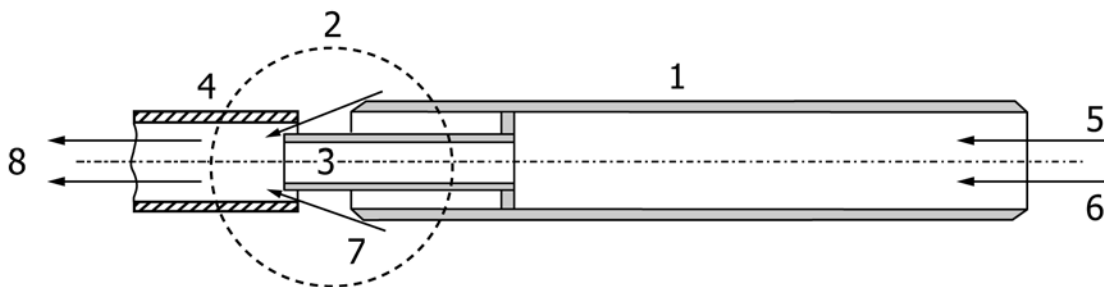


FIG. 2 ETV Unit: Schematic Design of Graphite Tube and Transition Area

- Key:
- 1 Graphite tube
  - 2 Transition area
  - 3 Nozzle of graphite tube
  - 4 Transition tube (alumina)
  - 5 Carrier gas (argon)
  - 6 Reaction gas ( $\text{CCl}_2\text{F}_2$ )
  - 7 Bypass gas (argon)
  - 8 Gas-mixture and dry sample aerosol to injector tube of plasma torch

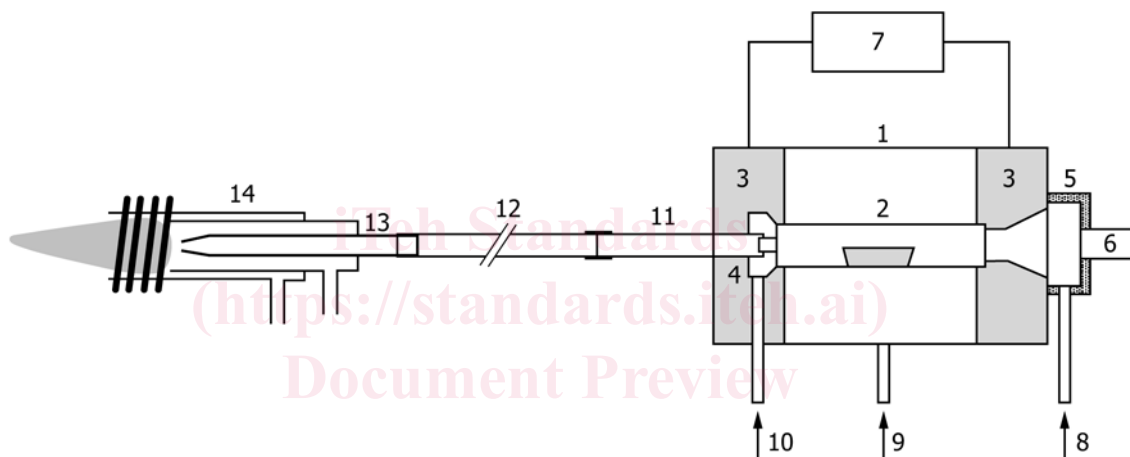


FIG. 3 Schematic Design of ETV Unit and Coupling to ICP

- Key:
- 1 Graphite tube furnace
  - 2 Graphite tube with graphite boat
  - 3 Graphite contacts
  - 4 Transition area
  - 5 Furnace inlet with movable door
  - 6 Pyrometer for online temperature measurement
  - 7 Electrical power supply
  - 8 Carrier gas (argon) and reaction gas ( $\text{CCl}_2\text{F}_2$ )
  - 9 Furnace shield gas (argon)
  - 10 Bypass gas (argon)
  - 11 Transition tube (alumina)
  - 12 Tubing to injector tube of ICP torch
  - 13 Injector tube of ICP torch
  - 14 ICP torch

simultaneously. For method development, it is beneficial if the spectrometer is capable of recording emission signal intensities versus time (so-called “time-scan”).

**6.3 Electrothermal Vaporization System**—With an electrically heated graphite tube furnace, graphite boats, reproducible setting and continuous control of temperatures up to 2600 °C (tolerance  $\pm 50$  °C), programmable temperature versus time runs, controlled gas flows (preferably mass-flow controlled), graphite tube design and transition area between graphite tube and transport tube optimized in terms of dry aerosol formation

and high transport efficiency, heat resistant tubing (up to 150 °C) to connect the ETV system with the ICP-torch, interface for synchronizing the ETV system with the ICP spectrometer.

NOTE 2—Reproducibility of analysis results can be improved using an automated handling system for the graphite boats (autosampler). An autosampler also increases sample throughput and saves working time.

**6.4 Balance**—Capable of weighing to the nearest 0.01 mg.

**6.5 Tweezers.**

### 6.6 *Microspatula*.

6.7 *Crusher or Mill*—Material adapted to the analytical task.

6.8 *Drying Apparatus*—Suitable for contamination-free drying of calibration solutions pipetted into the graphite boats, a drying temperature of maximum 100 °C, and heating of graphite boats from both sides.

6.8.1 Ensure that possible contamination originating from laboratory instruments has no effect on the accuracy of the analysis results.

## 7. Reagents and Materials

7.1 *Purity of Reagents*—Reagents of known analytical grade shall be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without compromising the accuracy of the determination.

7.2 *Sample Boats*—Made out of low-porosity or pyrolytically-coated high-purity graphite, the size adapted to the graphite tube of the ETV-furnace.

NOTE 3—With low-porosity or pyrolytically-coated graphite sample boats, diffusion of calibration solution through the sample boat can be avoided.

7.3 *Reaction Gas*—Dichlorodifluoromethane (CCl<sub>2</sub>F<sub>2</sub>).

NOTE 4—The use of ozone-depleting substances such as CCl<sub>2</sub>F<sub>2</sub> is restricted under the Clean Air Act. For laboratory and research purposes, however, the use of CCl<sub>2</sub>F<sub>2</sub> is still allowed. Complete thermal decomposition of CCl<sub>2</sub>F<sub>2</sub> is achieved in the hot graphite tube furnace and in the inductively-coupled plasma.

7.4 *Water*—Comply with grade II of ASTM Specification D1193.

7.5 *Calibration Solutions*—Aqueous single- or multi-element calibration solutions, prepared by dilution of commercially available standard-stock solutions with water to the required concentration.

7.6 *Calibration Samples*—With defined mass fractions of impurities, preferably certified reference materials (CRM).

NOTE 5—A commercially available CRM is listed in Appendix X3.

7.7 *Argon*—Purity  $\geq 99.996$  (volume fraction).

## 8. Sampling and Sample Preparation

8.1 Sampling shall be representative of the graphite grade lots and billets. If the dry state of the graphite is not secured, the sample must be dried at 110 °C  $\pm$  5 °C until there is no change in mass and then stored in a desiccator. Inhomogeneous sample materials must be homogenized.

8.2 Graphite blocks shall be crushed or sawed into small pieces which fit into the graphite boats or milled to graphite powder. Alternatively, a powder sample may be drilled out of the graphite block (graphite foil and graphite felt shall be ripped into small pieces, for example, using tweezers, which fit into the graphite boats). Standard apparatus and procedures for sample preparation may be used provided that no contamination occurs which affects the accuracy of the determination. Special attention should be paid to contamination control if high-purity graphite materials are analyzed.

## 9. Preparation of Apparatus

9.1 *ICP OES*—Consult the manufacturer's instructions for the operation of the inductively coupled plasma optical emission spectrometer.

9.2 *ETV System*—Consult the manufacturer's instructions for the operation of the electrothermal vaporization system. Before use, the graphite boats must be cleaned by thermal-halogenation in the graphite tube furnace of the ETV system at a temperature not lower than the vaporization temperature used for sample analysis.

9.3 *Operating Parameters for ICP OES and ETV System*—For materials other than graphite, these parameters must be evaluated as part of method development for each specific material. Appropriate operating parameters shall be established using calibration samples, preferably certified reference materials. The release behavior of each analyte shall be investigated by recording the intensity of the used emission lines versus time (so called "time-scan").

NOTE 6—For operating parameters for the analysis of graphite, see 11.6.

## 10. Calibration

10.1 Calibration shall be performed prior to each measurement cycle. Calibration solutions and calibration samples with defined analyte concentrations and contents, respectively, shall be measured applying the same measurement parameters and procedure as for the unknown sample (see Section 11). For each element, the calibrated range shall be adjusted to the content of this element in the unknown sample. This is achieved by using (i) different volumes of the same calibration solution, (ii) different masses of the same calibration sample or calibration solutions, and (iii) calibration samples with different analyte element concentrations.

10.2 Calibration shall be done using:

(a) Calibration samples of the same material as the unknown sample, preferably certified reference materials (CRM, see Appendix X3) or matrix-adapted synthetic calibration samples.

(b) Aqueous single- or multi-element calibration solutions which are pipetted into the graphite boats and then dried in the drying apparatus (6.8) (see (8, 9) and Appendix X4). Calibration using dried calibration solutions shall be validated by the analysis of suitable CRMs or by comparison of the analysis results with those obtained by independent analysis methods.

(c) A mix of (a) and (b).

10.2.1 The calibration with aqueous standard solutions can be matrix-adapted using materials of the same composition but with analyte contents known, based on independent methods, to be negligible to those expected in the unknown sample. This so-called blank sample is weighed into the graphite boat (same mass as unknown sample), and the aqueous standard solution is added and then dried in the drying apparatus.

10.3 Depending on the sample material, release of the elements boron and silicon in the graphite tube furnace and the efficiency of their transport to the ICP may be reduced, which would lead to a result that lies below the actual value (see Section 15). To avoid this systematic error, the elements boron and silicon shall be calibrated using calibration samples of the



same material (with respect to raw materials, manufacturing, and morphology) as the unknown sample. If such calibration samples are not available, the trueness/percentage recovery of results must be validated using independent test methods. Trueness is a measure of how close the measured element content is to the real element content in the graphite sample. Percentage recovery is an alternative measure of trueness defined as (measured element content/real element content ×100).

10.3.1 The release and transport of boron can be improved by addition of a suitable matrix modifier to the sample. Aqueous solutions of potassium hydroxide (KOH) or sodium hydroxide (NaOH), for example, show this effect. The modifier solution is pipetted onto the graphite sample in the graphite boat and then dried using the drying apparatus (6.8).

10.4 The data obtained by measurement of calibration samples shall be used to establish a calibration function for each element. The procedure for this shall follow the instructions of the manufacturer of the spectrometer. Usually, the operating software of the spectrometer allows different regression models. For each element, the regression model should be selected according to the response of the emission signal to the absolute element mass vaporized in the vaporization step of the ETV program. As ETV-ICP OES usually provides a linear working range of up to four orders of magnitude, linear regression can be applied in most cases to establish the calibration function.

## 11. Procedure

11.1 Before analysis, a cleaning cycle shall be performed with empty graphite boats (see 9.2).

11.2 Calibration samples and unknown samples (prepared as described in Section 8) shall be weighed into cleaned graphite boats. The mass shall be recorded. Calibration solutions shall be pipetted into the cleaned graphite boats and dried at a temperature below the boiling point of the calibration solution in the drying equipment (6.8). The pipetted volume shall be recorded.

11.3 If an automated sample changer is used, the graphite boats shall be placed on the sample rack in the following order: calibration solutions – calibration samples (both in ascending analyte mass) – unknown samples.

11.3.1 Depending on analyte element, analyte content, selected analyte emission lines, and graphite boat size, a sample mass between 1 mg to 100 mg and 1 mg to 200 mg shall be used for graphite powder and small graphite pieces, respectively. Based on experience, the recommended sample mass is as shown in Table 1.

TABLE 1 Recommended Sample Mass

Graphite Purity	Ash Content, ppmw	Graphite Powder, mg	Graphite Pieces, mg
High	<5	100	200
Medium	<300	30	30
Low	<1000	10	10

11.4 At the selected sample mass, the measured emission intensities of the analytes shall be within the linear range of the calibration functions.

11.5 The blank value shall be measured using empty and cleaned graphite boats. A graphite boat containing a calibration sample or a dried calibration solution shall be used to verify that the measuring position for the selected emission lines and background is at optimum position.

11.6 The graphite boats are inserted into the graphite tube furnace of the ETV-unit by means of tweezers or an automated sample changer. To avoid errors due to memory effects, the calibration samples shall be measured in ascending analyte concentrations. For analysis, the temperature-time program of the ETV-unit and the registration of the emission signals at the spectrometer shall be started simultaneously. At the end of the analysis program the graphite boat is removed from the graphite tube furnace and the next graphite boat is inserted. This procedure is repeated until all calibration samples and unknown samples are measured.

11.6.1 Predefined ETV program for the determination of all the elements indicated in the Scope:

Gas flow rates:	Furnace shield gas (argon)	500 mL/min
	Carrier gas (argon)	150 mL/min
	Bypass gas (argon)	350 mL/min
	Modifier gas (CCl <sub>2</sub> F <sub>2</sub> )	2.0 mL/min
Step 1 (pretreatment)	Ramp	25 °C to 450 °C, 7 s
	Hold	450 °C, 10 s
Step 2 (vaporization)	Ramp	450 °C to 2600 °C, 5 s
	Hold	2600 °C, 20 s
Step 3 (cooling)		

11.6.2 Spectrometer integration interval: 17 s to 45 s after starting the furnace program (total signal integration time of 28 s).

11.6.3 The cooling time depends of the cooling system of the furnace. The graphite boat should be changed at a temperature below 200 °C.

11.6.4 Coarse-grained samples or pieces may lead to a delayed thermal release of some analytes which can be recognized by a tailing of the transient analyte emission signals. Tailing can be reduced by: (1) increase of the vaporization temperature and/or vaporization time, (2) addition of a matrix modifier, and (3) milling of the sample.

11.7 Multiple measurements shall be carried out for each sample, with a minimum of three replicates. If the single values are deviating from each other by more than a given degree, depending on the repeatability given in Section 15, the analysis shall be repeated. If the repeatability is still not sufficient, the sample shall be homogenized. In the case of very low analyte contents near the limit of detection, sample homogenization may be omitted.

## 12. Wavelength, Limits of Detection, and Working Range

12.1 The selected wavelength of the analyte elements shall be free of interferences.

NOTE 7—Recommended wavelengths and limits of detection are given

in [Table X1.1](#) of [Appendix X1](#).

NOTE 8—A discussion of possible interferences and their elimination is given in [Appendix X2](#).

12.2 The limits of detection given in [Table X1.1](#) are only valid for graphite. The upper working range is limited by a decrease of the slope of the calibration function to 80 % of its initial value. If applicable, the working range can be extended by using less sensitive emission lines.

### 13. Calculation

13.1 The mass fractions of the analyte elements in the sample shall be calculated using background-corrected net-intensities of the emission lines, calibration functions, blank value, and sample mass.

### 14. Report

14.1 The mass fractions of the analytes in the sample shall be expressed as an average of the single values of a minimum of three replicate measurements.

Key:

$p_j$  = Number of participating laboratories for element j  
 $n_j$  = Total number of accepted measurements for element j  
 $a_j$  = Total arithmetic average for element j  
 $s_{rj}$  = Repeatability for element j  
 $r_j$  = Repeatability limit for element j,  $r_j = 2.8 \times s_{rj}$   
 $V_{rj}$  = Relative repeatability for element j in %  
 $S_{Rj}$  = Reproducibility for element j  
 $R_j$  = Reproducibility limit for element j,  $R_j = 2.8 \times S_{Rj}$   
 $V_{Rj}$  = Relative reproducibility for element j in %

## 15. Precision and Bias

15.1 For evaluation of precision data of ETV-ICP OES, an Interlaboratory Study (ILS) with 12 participating laboratories was carried out on a medium purity graphite powder sample. A comprehensive description of this ILS can be found on the website of the Federal Institute for Materials Research and Testing (BAM).<sup>5</sup> The ETV unit as illustrated in [Figs. 1-3](#), which is commercially available, was used by all participating laboratories to generate precision data given in [Table 2](#). It is emphasized that, if an ILS is based on different parameters (for example, a different graphite sample, ETV unit, ICP OES, or participating laboratories), then the quoted levels of precision reported in [Table 2](#) may not be achieved.

<sup>5</sup> The certificate and certification report for CRM BAM-S009 is available at the website of the Federal Institute for Materials Research and Testing (BAM), Berlin, Germany: <https://rrr.bam.de/RRR/Navigation/EN/Reference-Materials/RM-Certificates-reports/Special-materials/special-materials.html>.

**TABLE 2 Precision and Bias Data**

Element	Precision									Bias	
	$p_j$	$n_j$	$a_j$ mg/kg	$s_{rj}$ mg/kg	$r_j$ mg/kg	$V_{rj}$ %	$S_{Rj}$ mg/kg	$R_j$ mg/kg	$V_{Rj}$ %	Certified Values <sup>A</sup> mg/kg	Values for Information <sup>A</sup> mg/kg
Silver (Ag)	4	24	0.0023	0.0006	0.0016	25.7	0.0009	0.0024	38.3		0.0018
Aluminum (Al)	12	70	0.27	0.04	0.12	16.4	0.09	0.25	32.9	0.27	
Arsenic (As)	3	18	0.015	0.002	0.006	14.4	0.004	0.010	23.1		0.016
Boron (B)	8	48	0.22	0.05	0.13	21.2	0.10	0.27	43.3	0.83	
Barium (Ba)	10	60	0.77	0.08	0.22	10.2	0.11	0.31	14.5	0.80	
Beryllium (Be)	6	36	0.00047	0.00018	0.00050	38.0	0.00034	0.00094	72.2	0.00050	
Bismuth (Bi)	3	18	0.020	0.009	0.026	45.6	0.012	0.035	61.3		0.016
Calcium (Ca)	12	70	4.44	0.55	1.55	12.4	1.43	4.01	32.2	5.1	
Cadmium (Cd)	6	36	0.0021	0.0006	0.0018	30.1	0.0027	0.0076	130		0.0022
Cobalt (Co)	11	66	0.151	0.014	0.038	9.0	0.015	0.043	10.1	0.143	
Chromium (Cr)	12	70	1.36	0.14	0.39	10.2	0.30	0.84	22.1	1.39	
Copper (Cu)	11	66	0.065	0.007	0.029	10.7	0.010	0.029	15.8	0.067	
Iron (Fe)	12	70	27.1	2.7	7.5	10.0	3.9	10.8	14.3	28	
Potassium (K)	10	60	1.05	0.18	0.51	17.2	0.26	0.72	24.5	1.04	
Lithium (Li)	8	48	0.023	0.005	0.014	21.1	0.007	0.021	31.8	0.022	
Magnesium (Mg)	11	66	0.142	0.029	0.081	20.4	0.036	0.100	25.1	0.135	
Manganese (Mn)	11	66	0.094	0.011	0.030	11.4	0.014	0.039	14.7	0.094	
Molybdenum (Mo)	9	54	0.19	0.02	0.05	9.5	0.08	0.21	39.6	0.20	
Sodium (Na)	10	60	0.32	0.06	0.16	17.5	0.10	0.29	33.0	0.32	
Nickel (Ni)	11	64	5.7	0.4	1.1	6.6	0.5	1.4	8.7	5.6	
Phosphorus (P)	9	54	0.26	0.03	0.09	12.5	0.08	0.23	32.4	0.26	
Lead (Pb)	9	54	0.044	0.012	0.035	28.4	0.037	0.104	85.2	0.052	
Sulfur (S)	9	52	10.9	1.7	4.7	15.5	2.2	6.3	20.5	10.7	
Antimony (Sb)	4	24	0.033	0.009	0.025	26.4	0.020	0.056	60.2		0.022
Silicon (Si)	10	60	12.1	1.6	4.5	13.3	2.4	6.7	19.7	41	
Tin (Sn)	5	30	0.16	0.04	0.10	22.1	0.06	0.16	36.7		0.16
Strontium (Sr)	9	50	0.32	0.04	0.12	13.5	0.05	0.15	17.3	0.32	
Titanium (Ti)	11	66	8.1	1.2	3.3	14.7	1.5	4.1	18.3	8.6	
Vanadium (V)	11	64	1.33	0.27	0.75	20.2	0.32	0.90	24.2	1.30	
Tungsten (W)	9	54	3.4	0.4	1.1	11.7	1.1	2.9	30.9	3.0	
Yttrium (Y)	10	60	0.048	0.008	0.024	17.6	0.014	0.040	29.4	0.049	
Zinc (Zn)	11	66	0.059	0.008	0.023	14.1	0.014	0.039	23.6	0.070	
Zirconium (Zr)	11	66	0.66	0.11	0.31	16.9	0.13	0.36	19.5	0.81	

<sup>A</sup> See certificate for CRM BAM-S009 Medium Purity Graphite Powder (see Footnote 5).