Designation: E 1452 – 92 (Reapproved 1996)

Standard Practice for
Preparation of Calibration Solutions for Spectrophotometric and for Spectroscopic Atomic Analysis[1]

This standard is issued under the fixed designation E 1452; 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 practice covers the preparation, labeling, and storage of calibration solutions employed for calibrating atomic and molecular spectroscopic equipment used for the analysis of metals and alloys.

1.2 *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.* For specific hazards information, see Section 5.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 1193 Specification for Reagent Water
E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals
E 288 Specification for Laboratory Glass Volumetric Flasks
E 319 Practice for the Evaluation of Single-Pan Mechanical Balances
E 969 Specification for Volumetric (Transfer) Pipets

2.2 *National Institute for Standards and Technology (NIST) Documents:*

Circular 602 Testing of Glass Volumetric Apparatus[3]

3. Significance and Use

3.1 Analytical instrumentation is employed extensively throughout industry for quality control, research, and development purposes. Calibration solutions are required for calibrating certain types of analytical atomic and molecular spectroscopic instruments. Therefore, the quality of the analytical results obtained is directly dependent on the accuracy of the calibration solutions.

3.2 It is assumed that the users of this practice will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that the work will be performed in a properly equipped laboratory.

4. Reagents

4.1 *Purity of Reagents*—All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available.[4] Other grades may be used if it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy or stability of the calibration solutions produced. See Practices E 50.

4.2 *Purity of Water*—Unless otherwise indicated, all water used shall be Type II (distilled water) or water of equivalent purity, in accordance with Specification D 1193.

5. Hazards

5.1 All safety precautions referenced in Practice E 50 shall be observed. Generally accepted prudent laboratory practices shall be followed.

5.2 Calibration solutions may be subject to several federal, state, and local laws and regulations, and may be subject to the regulations of some fire departments.

5.3 Other laws or regulations may be applicable, depending on the nature and concentration of the matrix and the element used to prepare the calibration solutions.

6. Calibration of Equipment

6.1 *Balances and Weights*—Calibrate balances and weights periodically, depending on use, or at least once every twelve months. Calibration shall be traceable to NIST. See Method E 319.

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[1] This practice is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.21 on Reference Material Planning, Proficiency Testing and Laboratory Accreditation.

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

[3] Available as Order No. COM 73-10504 from the National Technical Information Services, Springfield, VA 22161.

[4] *Reagent Chemicals, American Chemical Society Specifications*, Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Rosen, J. *Reagent Chemicals and Standards*, D. Van Nostrand Co., Inc., New York, NY, and *United States Pharmacopeia*, United States Pharmacopeial Convention, Inc., Rockville, MD.

6.2 *Glass and Plastic Laboratory Ware*—All glass and plastic volumetric flasks and pipets shall be of Class A, precision grade, within the tolerances established by NIST for the appropriate type of glassware. See Practices E 50, Specifications E 288, and E 969, and NIST Circular 602.[5]

7. Preparation of Calibration Solutions

7.1 *General*—Usually when calibrating an instrument, an analyte stock solution is prepared first. From this solution aliquots are taken by transfer pipets or by weight, and diluted in volumetric flasks to yield a series of calibration solutions which are used to calibrate an instrument in the concentration range of interest.

7.1.1 *Stock Solution*—The concentration of analyte is sufficiently high so that adsorption of the analyte on the container walls is negligibly small compared to the concentration of the analyte, and from which the several needed calibration solutions can be prepared by suitable quantitative dilution.

7.2 *Preparation*—Prepare the stock solution by dissolving an accurately weighed quantity of the element or the appropriate stochiometric quantity of a stable compound of the element, in water, acid, base, or organic solvent, and diluting to a known volume or weight in a suitably sized and calibrated volumetric flask.

7.3 *Purity of Analyte*—The total of all known impurities in the element or compound of the element shall not exceed 0.1 % by weight. If an element is used, its quantity shall be corrected for the total of the metallic impurities and non-metallic elements such as carbon, nitrogen, sulfur, and oxygen, and also for the quantity of such insoluble materials as may be present (see Note 1). The reagents used for dissolution may also contain impurities. If one of the impurities is the analyte, the concentration of the analyte in the stock solution shall be corrected for it. A note should be made also of any impurities that may cause interference in the analysis, and suitable method corrections made. If the stock solution is used to prepare multi-element calibration solutions, corrections should be made for impurities that are among the elements of interest.

NOTE 1—Some finely divided elemental powders may accumulate significant surface oxide. In some cases, it may be possible to remove the oxygen by a suitable controlled atmosphere annealing treatment, although, in general, the use of such powders should be avoided.

7.4 The stock solution shall be considered a *standard stock solution* if all the following conditions are met:

7.4.1 The element or compound is at least 99.9 % pure, and this purity is confirmed with an appropriate analytical procedure. The supplier's certificate of analysis does not satisfy this requirement, unless the supplier is an internationally recognized standardizing agency such as NIST.

7.4.2 The metallic element or compound is weighed on an analytical balance, and the balance and weights are calibrated in accordance with 6.1.

7.4.3 The minimum weight of the analyte or analyte compound or aliquot should be within ±0.05 % relative standard deviation. The minimum weight of the analyte or analyte compound shall be calculated from the number of places to the right of the decimal point that the balance is capable of determining. Compute minimum weight, *w*, as follows:

$$w = [(X_1^2 + X_2^2)^{1/2}/0.05]100 \tag{1}$$

where X_1 and X_2 are the balance limitations for two weighings.

If the balance weighs to 0.001 g and two weighings, tare and final, are taken, then:

$$w = \{[(0.001)^2 + (0.001)^2]^{1/2}/0.05\}100 = (0.0014/0.05)100 = 2.800 \text{ g} \tag{2}$$

7.4.4 The solution is diluted in volumetric vessels that meet the requirements of 6.2.

7.4.5 The solution was made within the last twelve months or within the shelf life interval, whichever is shorter.

7.4.6 The standard stock solution is stored at all times in accordance with Section 8.

7.4.7 The concentration has been verified prior to use or sale.

7.5 Stock solutions that do not meet all the conditions of 7.4 shall be analyzed prior to use. The analysis shall be performed using an appropriate analytical method, preferably a gravimetric or volumetric determination that does not depend on instrumental comparison with another solution of the same type.

7.6 Calibration solutions shall be prepared by quantitative dilution of the stock or standard solutions as directed in the analytical method for which they are specified. The frequency of preparation will depend on the shelf life of the calibration solution. At trace level concentrations, depending on the adsorptivity of the analyte and type of container used, it may be necessary to prepare fresh calibration solutions for each series of measurements. Unless already known, shelf life shall be established for representative concentrations of analyte, particularly at trace levels.

8. Storage

8.1 Store light-sensitive metal solutions in opaque containers. Store all stock solutions at 15 to 30°C in closed containers that do not contaminate the solutions, are resistant to the solvent and its solutes, minimize solution evaporation, and minimize adsorption on the walls of the container (see Note 2). It may be advisable to rinse the container several times with small quantities of stock solution to establish equilibrium between the solution and adsorbed analyte.

NOTE 2—Plastic containers breathe and therefore the concentration can change significantly over a year. To avoid this problem, store in a high humidity chamber, or monitor the weight and adjust or assay periodically.

8.2 Shelf life will depend on the properties of the analyte element, the impurities, and the solvent. Thus the amount of analyte in both purchased and "in-house" stock solutions should be analyzed periodically by a suitable analytical method. A record of the analytical results should be kept. Also, the analytical results and their respective dates should be written on a suitable label affixed to the container. In addition,

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[5] For additional information see the following: Federal Fair Packaging and Labeling Act, Food and Drug Administration and Federal Trade Commission; Federal Hazardous Substances Act, Food and Drug Administration, Federal, and State Occupational Safety and Health Acts; Federal and State Right to Know Acts, and DOT Shipping Regulations.

the solution itself shall be inspected for separate phases such as precipitates or separated solvents.

9. Labeling

9.1 If the stock or standard stock solution is prepared in-house, the label should contain the name and concentration of the analyte, the name and concentration of the dissolution reagents, the name of the dilution solvent, and the preparation date. A list of impurity elements and their concentrations should either be on the label or available in a suitable record book. If the solution is supplied to outside users, the label should contain the name and concentration of the analyte, the dissolution reagents, the principal solvent, and a lot number or date of preparation. A list of impurities and their concentrations may either be written on the label or supplied as a separate certificate. The label or certificate should include the name, address, and telephone number of the supplier, as well as the date of the last analysis of the metallic constituents, and the shelf life if known.

9.1.1 For both in-house and outside supplier products, the label shall contain the appropriate safety information. Material Safety Data sheets shall be available.

10. Precautions

10.1 If the stock solution is obtained from an outside supplier other than an internationally recognized standards organization such as NIST, it should be verified for the analyte concentration before using it to prepare calibration solutions.

10.2 If a series of dilutions are needed to prepare calibration solutions of low concentration, it may be advisable to dilute by weight to minimize error propagation.

10.3 Adsorption of the analyte on the walls of the container at low concentrations may lead to significant errors. Precautions should be taken at concentrations below 10 mg/L and especially below 1 mg/L.[6] If the solvent is water, the adsorption of certain elements may be inhibited by adding 5 mL of concentrated nitric acid to a liter of solution.

NOTE 3—**Warning:** Do not employ this approach if the solvent is organic in nature.[7,8]

10.3.1 Another possibility is to prepare the same solution several times in the same container to establish equilibrium between dissolved and adsorbed analyte.

10.4 *Trace Analysis*—If the calibration solutions are to be used for trace analysis, it should be kept in mind that contamination of these solutions may come from the reagents used, from the apparatus used, and from the analyst. Thus to ensure a reliable analysis, reduce contamination by working in a clean environment, use purified reagents, and choose apparatus materials to reduce impurities. Also, analysts should always be aware of their own effect on contamination. Care should be exercised in the selection and cleaning of laboratory ware and personal sources of contamination (for example, smoking and cosmetics) should be avoided.[9]

11. Keywords

11.1 analytical atomic spectroscopy; atomic spectroscopy; calibration solutions; reference solutions; spectrometry; standard solutions; stock solutions

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[6] Robinson, J. W., *Treatise on Analytical Chemistry*, Second Edition, Part 1, Volume 7, John Wiley & Sons, Inc., New York, NY, 1981, pp. 767–769.

[7] Shendrikar, A. D. and West, P. W., "Adsorption Characteristics of Traces of Chromium (III) and VI on Selected Surfaces," *Enelytica Chimica Acta*, Vol 72, No. 1, 1974, pp. 91–96.

[8] Shendrikar, A. D., et al., "Adsorption Characteristics of Traces of Barium, Beryllium, Cadmium, Manganese, Lead, and Zinc on Selected Surfaces," *Enelytica Chimica Acta*, Vol 84, No. 2, 1976, pp. 409–417.

[9] For additional information, see Murphy, T. J., *Accuracy in Trace Analysis*, NBS Special Publication, 422, Vol 1, 1976, pp. 509–539, available from the National Technical Information Services, Springfield, VA 22161 by Order No. PB258091.