Designation: D5463 – 18

Standard Guide for Use of Test Kits to Measure Inorganic Constituents in Water[1]

This standard is issued under the fixed designation D5463; 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 guide covers general considerations for the use of test kits for quantitative determination of analytes in water and wastewater. Test kits are available from various manufacturers for the determination of a wide variety of analytes in drinking water, surface or ground waters, domestic and industrial feedwaters and wastes, and water used in power generation and steam raising. See Table 1 for a listing of some of the types of kits that are available for various inorganic analytes in water.[2]

1.2 Ranges, detection limits, sensitivity, accuracy, and susceptibility to interferences vary from kit to kit, depending on the methodology selected by the manufacturer. In some cases, kits are designed to replicate exactly an official test method of a standard-setting organization such as the Association of Official Analytical Chemists (AOAC), American Public Health Association (APHA), ASTM, or the U.S. Environmental Protection Agency (USEPA). In other cases, minor modifications of official test methods are made for various reasons, such as to improve performance, operator convenience, or ease of use. Adjustments may be made to sample size, reagent volumes and concentrations, timing, and details of the analytical finish. In yet other cases, major changes may be made to the official test method, such as the omission of analytical steps, change of the analytical finish, omission of reagents, or substitution of one reagent for another. Reagents in test kits are often combined to obtain a fewer number and make the test easier to use. Additives may also be used to minimize interferences and to make the reagent more stable with time. A kit test method may be based on a completely different technology, not approved by any official or standard-setting organization. Combinations of test kits—multi-parameter test kits—may be packaged to satisfy the requirements of a particular application conveniently. The test kits in such combination products may be used to make dozens of determinations of several parameters.

1.3 Test kit reagent refills are commonly available from manufacturers. Refills permit cost savings through reuse of the major test kit components.

1.4 Because of the wide differences among kits and methodologies for different analytes, universal instructions cannot be provided. Instead, the user should follow the instructions provided by the manufacturer of a particular kit.

1.5 A test kit or kit component should not be used after the manufacturer's expiration date; it is the user's responsibility to determine that the performance is satisfactory.

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

1.7 *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.* For specific precautionary statements, see Section 10.

1.8 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D3370 Practices for Sampling Water from Closed Conduits
D4453 Practice for Handling of High Purity Water Samples
D4691 Practice for Measuring Elements in Water by Flame Atomic Absorption Spectrophotometry
D5810 Guide for Spiking into Aqueous Samples
D5847 Practice for Writing Quality Control Specifications

[1] This guide 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 May 1, 2018. Published May 2018. Originally approved in 1993. Last previous edition approved in 2013 as D5463 – 13. DOI: 10.1520/D5463-18.

[2] Test kits for determining inorganic analytes in water are available from various United States and foreign manufacturers, as well as from laboratory supply companies.

[3] 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.

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

TABLE 1 Availability and Types of Test Kits

Analyte	Kit Methodology [A]
Acidity	T
Alkalinity	C, P, T
Aluminum	C, P
Ammonia	C, P
Boron	C, P
Bromine	C, P, T
Cadmium	C
Calcium	P, T
Carbon dioxide	T
Chloride	A, C, P, T
Chlorine	C, P, T
Chlorine dioxide	C, P, T
Chromium (III)	C
Chromium (VI)	C, P, T
Cobalt	C
Copper	C, P, T
Cyanide	C, P, T
Fluoride	P
Hardness	C, GNG, P, T
Hydrazine	C, P
Hydrogen peroxide	C, P, T
Iodine	C, P, T
Iron	C, P
Lead	C, P
Manganese	C, P
Magnesium	C, T
Molybdate	C, P, T
Nickel	C, P
Nitrate	C, P
Nitrite	C, P, T
Oxygen (dissolved)	C, P, T
Ozone	C, P
Permanganate	C, T
pH	C, P
Phosphate	C, P
Silica	C, P
Silver	P
Sulfate	A, C, P, T
Sulfide	C, P, T
Sulfite	C, P, T
Thiocyanate	C
Tin	C
Vanadium	C
Zinc	C, P, T

[A] Kit Methodology: A = appearance /turbidity, C = visual colorimetric, GNG = go no go, P = photometric, and T = titrimetric.

for Standard Test Methods for Water Analysis
E178 Practice for Dealing With Outlying Observations
E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers
E958 Practice for Estimation of the Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129 and Practice D4691.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *analyte, n*—the chemical or constituent being determined.

3.2.2 *carryover, n*—the contamination of a subsequent sample by a previous sample, typically due to incomplete cleaning of a reused test kit component.

3.2.3 *expiration date, n*—a date applied by the manufacturer after which an accurate result is not ensured by the manufacturer.

3.2.4 *finish (usually analytical finish), n*—the analytical methodology used for the measuring step of the analysis.

3.2.5 *kit (or test kit), n*—a commercially-packaged collection of components intended to simplify the analytical-testing function.

3.2.6 *interference, n*—a matrix component's effect that might cause an analytical bias or that might prevent a successful analysis.

3.2.7 *safety data sheet, n*—a federally-mandated, safety-related document that must be made available to kit-chemistry users.

3.2.8 *matrix, n*—sample contents other than the target analyte.

3.2.9 *official method, n*—an analytical test method officially approved by an industry consensus organization (such as ASTM, AOAC, or APHA) or by a government entity such as the USEPA.[4]

3.2.10 *refill, n*—a replacement package of test kit components used in testing.

3.2.11 *spike, n*—a small-volume, high relative concentration aliquot of analyte added quantitatively to a split sample as a quality check.

3.2.12 *split sample, n*—a sample that is split into subsamples that are intended to have the same composition as the original sample.

4. Summary of Guide

4.1 Analytical test kits simplify the operational procedures necessary to perform an analysis. This guide includes general considerations relating to the procedures to be followed in order to ensure an accurate determination. This guide also describes, in general terms, the characteristics of some kit types and kit components and includes some comments on their capabilities, benefits and, where appropriate, their limitations.

5. Significance and Use

5.1 Inorganic constituents in water and wastewater must be identified and measured to support effective water quality monitoring and control programs. Currently, one of the simplest, most practical and cost effective means of accomplishing this is through the use of chemical test kits and refills. A more detailed discussion is presented in ASTM STP 1102.[5]

[4] Other documents: *Official Methods of Analysis of the Association of Official Analytical Chemists*, 15th ed., AOAC, Arlington, VA, 1990. Changes are published in annual supplements. *Standard Methods for the Examination of Water and Wastewater*, 17th ed., APHA, AWWA, and WPCF. Washington, DC, 1989. *Methods for the Chemical Analysis of Water and Wastes*, USEPA, Cincinnati, OH, March 1983.

[5] Spokes, G. Neil, and Bradley, Julie A., "Performance Testing of Selected Test Kits for Analysis of Water Samples," *ASTM STP* 1102, ASTM, Philadelphia, PA, 1991.

5.2 Test kits have been accepted for many applications, including routine monitoring, compliance reporting, rapid screening, trouble investigation, and tracking contaminant source.

5.3 Test kits offer time-saving advantages to the user. They are particularly appropriate for field use and usually are easy to use. Users do not need to have a high level of technical expertise. Relatively unskilled staff can be trained to make accurate determinations using kits that include a premixed liquid reagent, premeasured reagent (tablets, powders, or glass ampoules), and premeasured sample (evacuated glass ampoules).

6. General Considerations

6.1 *Personnel*—The selection of a test kit and determination that the test kit analysis is appropriate should be conducted by a responsible chemist. The development of suitable protocols and conditions for safe use should be conducted by the responsible chemist with the assistance of an industrial hygienist. The kit user may be a relatively unskilled staff person but must be trained to an appropriate level of proficiency.

6.2 *Completeness of Kits*—The kit's components may or may not be complete for the required determination. The user must assemble all instruments and materials necessary for the determination. For example, if the test kit is used for field screening to indicate the need for samples requiring a high accuracy measurement, the user may need to provide a means of preserving a sample for later measurements at a laboratory.

6.3 *Protocol Established by a Responsible Chemist*—A responsible chemist must determine whether the sample can be analyzed correctly by a particular kit chemistry. The responsible chemist should determine whether matrix factors, interferences, and temperature are handled correctly by the kit chemistry. Questions to be answered include the following: Has the kit chemistry previously given satisfactory results under the proposed conditions? What changes have occurred that must be taken into account? For example, the chemist should consider seasonal changes, new interferences, sample pH changes, new dischargers upstream, and new process wastes in the sample. The responsible chemist must also decide whether the proposed kit chemistry is applicable to the particular circumstances. For example, it is necessary to determine whether the test range is appropriate, ensure that a colorimetric test kit that compensates for color is used with a highly colored sample, and ensure that a colorblind user is able to run a test requiring visual color comparisons accurately. The chemist must also ensure that an officially approved kit chemistry is used when an official method is required.

6.4 *Technical Support*—In case of difficulties, many kit manufacturers may provide technical assistance.

7. Interferences

7.1 Kit chemistries that are based on an official test method are subject to the same interferences as that test method. If the kit manufacturer uses a revised version of the official test method, the revision may increase or decrease interference effects.

7.2 Sample carryover effects may occur if a common sampling cup or tube is used. Appropriate care is necessary under such conditions in order to prevent sample carryover. The carryover may be prevented or reduced by either cleaning the reused item or rinsing with fresh sample several times. Aggressive cleaning action may be necessary after a sample containing a high concentration is tested.

7.3 Careful note should be made of the manufacturer's comments concerning interferences, and appropriate action should be taken.

7.4 Temperature may affect kit performance.

8. Apparatus

8.1 *Colorimetric Determinations*—Many procedures depend on color determination with a color comparator, photometer, or spectrophotometer. The manufacturer may offer a color comparator for visual comparisons based on liquid, glass, plastic, or printed color standards. The manufacturer may offer a photometer or may recommend the use of a spectrophotometer for photo-electric color determinations. The manufacturer's photometer may be based on optical filters using either colored glass or plastic, or on interference filters or LEDs. The filter bandwidth may be wide (up to 100-nm full width half maximum height) for colored glass or plastic filters and LEDs or quite narrow (10 nm) with interference filters. The laboratory spectrophotometer may have a 1- to 20-nm bandwidth and is typically more accurate than a kit photometer or colorimeter. Refer to Practices E275 and E958 for additional discussion of colorimetry.

NOTE 1—Visual comparator kits may require the use of a particular type of background illumination. The user should use the light source that produces the correct color or spectrum of background illumination, as specified by the manufacturer.

NOTE 2—Color standards may not be permanent; reference should be made to the manufacturer's recommendations.

8.2 *Titrimetric Determinations*—Many procedures depend on measuring the volume of a standard solution required to react with an analyte completely. The manufacturer may offer a buret, digital titrator, drop-test, or calibrated sample container to dispense and measure the volume of a standard solution. A buret or digital titrator typically provides more accuracy than a drop-test or calibrated sample container.

9. Reagents and Materials

9.1 *Purity of Reagents*—Reagent grade or better chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6] However, these reagents may not be of sufficient purity in some cases due to the sensitivity of the technique. It is the responsibility of the

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.