Designation: D 5463 – 03

An American National Standard

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

This standard is issued under the fixed designation D 5463; 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 *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 precautionary statements, see Section 10.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[3]

D 1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits[3]

D 1193 Specification for Reagent Water[3]

D 3370 Practices for Sampling Water from Closed Conduits[3]

D 4453 Practice for Handling of Ultra-Pure Water Samples[3]

D 4691 Practice for Measuring Elements in Water by Flame Atomic Absorption Spectrophotometry[3]

D 5810 Guide for Spiking into Aqueous Samples[3]

D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis[4]

E 178 Practice for Dealing with Outlying Observations[5]

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers[6]

E 958 Practice for Measuring Practical Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers[6]

[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 June 10, 2003. Published July 2003. Originally approved in 1993. Last previous edition approved in 1998 as D 5463–98.

[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] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 11.02.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] *Annual Book of ASTM Standards*, Vol 03.06.

***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.

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, refer to Terminology D 1129 and Practice D 4691.

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.2 *carryover*—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*—a date applied by the manufacturer after which an accurate result is not ensured by the manufacturer.

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

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

3.2.6 *interference*—an effect of a matrix component that might cause an analytical bias or that might prevent a successful analysis.

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

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

3.2.9 *official method*—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.[7]

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

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

3.2.12 *split sample*—a sample that is split into sub-samples 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.[8]

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

[7] 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.

[8] 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.

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 E 275 and E 958 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.[9] 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 manufacturer to provide reagents and accessory solutions of sufficient quality to meet the performance specification claims of the test kit. In addition, the manufacturer should specify acceptable conditions of storage and provide expiration dates, where appropriate. It is the responsibility of the kit user to ensure that no unacceptable deterioration has occurred in transit or due to improper storage conditions and that the kits are not used improperly after their expiration dates (see 9.4).

9.2 *Purity of Water*—Water must be of sufficient purity that it does not interfere with the test. Manufacturer's instructions should be followed. Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III or better of Specification D 1193.

9.3 *Kit Components and Packaging*—The test kit components and packaging are usually designed carefully by the manufacturer to facilitate quick and easy determination of the analyte. Pre-mixed liquid reagents eliminate the need for making up reagents and offer the benefits of simplicity of use, reduced need for operator measurements, and good immunity to environmental effects. Test kits with unit dose disposable

[9] *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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

reagent packs offer the further benefits of simplicity of use and reduced need for operator measurements. In particular, tablets, foil packs, powders, and glass ampoule reagent packaging techniques eliminate the need for making up reagents and then measuring the reagent volume. Glass ampoules with reagents packaged under vacuum also offer immunity to reagent oxidation and make the measurement of sample volume unnecessary. Test kit reagent refills may often be obtained from manufacturers. Refills save the expense of purchasing an entire kit by permitting the reuse of major kit components.

9.4 *Storage*—Users should follow the manufacturer's instructions for acceptable storage conditions. Some kit components can be refrigerated to prolong usability. If the user is uncertain about past storage, a quality control check sample (see 13.1.1) should be used to determine the acceptability of a test component.

9.5 *Kit Expiration*—The user should not use kit components after their expiration date.

10. Safety Precautions

10.1 The majority of kit test methods use chemicals that have some type of hazard associated with them. The responsible chemist/industrial hygienist should ensure that proper heed is taken of warnings in the instructions. Material safety data sheets must be obtained for each kit, and all appropriate care should be taken based on the manufacturer's warnings. Protective clothing and safety eyewear may be required, depending on the hazards posed by sample and kit. A ventilation hood may be required in the laboratory. Ventilation of the work area is rarely a problem in the field, except when working in confined spaces such as in manholes. Kits involving instruments should not be used where flammables are in use unless previously certified as acceptable by the responsible safety officer.

10.1.1 Test kits containing premixed reagents, including the unit dose type of test kit, greatly reduce the need for operator contact with chemicals and therefore improve safety compared with the officially approved laboratory test methods.

10.1.2 The responsible chemist/industrial hygienist must take all necessary steps to ensure that the operator performs tests using the kits in a safe manner. Protective clothing and safety eyewear should be used as necessary. Used test kit components must be disposed of in accord with federal, state, and local laws. The responsible chemist/industrial hygienist should provide written instructions where special disposal techniques are required.

10.2 *Safety Training*—The responsible chemist/industrial hygienist must ensure that the operator is trained properly in the use of the kit, with due regard to the kit's safety hazards.

10.3 *Right-to-Know and Other Laws*—The responsible chemist/industrial hygienist must ensure that the test user is trained in safe use of the test kit and is informed properly regarding the various hazards associated with the kit chemicals. The manufacturer will provide the required information through a material safety data sheet.

11. Samples and Sampling Procedures

11.1 *Sample Collection*—Unless otherwise specified, collect all samples in accordance with Specification D 1192 and Practices D 3370.

11.2 *Sample Handling*—Follow the kit manufacturer's recommended sample handling procedures, when available. Contamination and loss are of prime concern for the determination of trace metals. Environmental dust and impurities in apparatus components that contact the sample are potential sources of contamination. Containers can introduce errors in the measurement of trace metals by contributing contaminants through leaching or surface desorption or by depleting the concentration of the analyte through adsorption. Refer to Practice D 4453 for the handling of ultra-pure water samples.

11.3 *On-Site Sampling*—Test kits that permit on-site analysis offer great advantages to their users, inasmuch as error-prone sample preservation techniques are often unnecessary. However, the value of an analytical result is only as good as the sample that is obtained, and the usual care is required to ensure the representativeness of the sample.

11.4 *Sample Containers*—Take care to collect a representative sample in clean containers that will not cause contamination in any significant way.

11.5 *Sample Size*—The sample size shall be sufficient to complete the determinations. A larger sample may be necessary when sample processing or multiple determinations, or both, are required.

11.6 *Sample Preservation and Storage*—The kit method ordinarily permits an immediate measurement. If the test result is equivocal or the responsible chemist directs that a preserved sample be obtained, preserve a sample in accord with the applicable approved procedure.

12. Calibration and Standardization

12.1 Kit chemistry manufacturers may provide a precalibrated color comparator or colorimeter. Alternatively, a spectrophotometric analytical finish may be recommended. A titration kit test method may require a drop count determination or titration scale reading. The drop count or scale reading may be multiplied by one factor to generate an analytical result. The calibration accuracy depends on the accuracy of the system design and ensuing manufacturing processes. All kits that lead directly to an analytical result rely on an internal calibration. Manufacturers will offer to provide a calibration chart in some cases. Wherever possible, known reference samples or standards should be analyzed to validate the results from test kits.

13. Quality Control

13.1 *Quality Control*—Test kit quality control is the responsibility of the manufacturer. The manufacturer may be requested to furnish supporting quality control data. The user should verify that the final color resulting when using a colorimetric test kit is the same as that of a calibration sample. For more information on Quality Control Specifications, see Practice D 5847.

13.1.1 *Quality Control Check Samples*—Kit quality may be determined by occasionally running a blank sample, a known mid-range sample, a known upper mid-range sample, and a

higher than a full scale sample. The user should verify independently that the kit calibration and recovery are satisfactory for the intended application, with due regard for interferences typically found in the user's samples.

NOTE 3—In some cases, it is not possible to establish an analytical standard solution that can be routed to a user laboratory (for example, dissolved oxygen in water). In that event, it may be possible to use a surrogate compound instead of the analyte. In the event of difficulty in obtaining a standard, the manufacturer should provide the necessary product support information.

13.1.2 *Split Samples*—Split samples should be analyzed frequently. As a recommended minimum, analyze one set of split samples every 10 samples (see 14.4).

13.1.3 *Laboratory Spikes*—Prepare and analyze laboratory spikes. The spiked sample should contain approximately double the anticipated quantity of target analyte in the test sample, and the spike should be of comparatively small volume. The spike should not be so great as to exceed the range of the test kit method.

13.1.4 *Field Spikes*—Field spikes may be prepared in the laboratory and added to split samples in the field.

13.1.5 For more information on spikes, see Guide D 5810.

14. Procedure

14.1 *Sample Pretreatment*—Sample pretreatments specified by the kit manufacturer must be conducted prior to the use of the kit. The responsible chemist must ensure that any necessary sample pretreatments are conducted prior to use of the kit. Failure to conduct the sample pretreatments can result in the generation of meaningless test results. The manufacturer should be able to provide the necessary information, but such procedures are often not addressed adequately in manufacturer's test kit instructions.

14.2 Prepare quality control check samples in the laboratory.

14.3 Perform sample analysis procedures in accord with the kit manufacturer's instructions.

14.4 The frequency of quality control check samples should be based on a statistical plan established to achieve a desired level of confidence. An alternative plan is to check at least one calibration standard after every 10 samples and at the end of the analyses. Analyze the quality control check samples, split samples, and spiked samples as directed by quality control procedures.

15. Calculation

15.1 In most cases, kit test methods provide instructions that lead to direct numeric results. The user should follow the manufacturer's instructions.

15.2 *Units*—The numeric results obtained from the test kit should be stated clearly in commonly used units, for example, mg/L, µg/L, ppm (parts per million), or ppb (parts per billion). If a result is presented as ppm or ppb, it should specify whether this is a weight or volume specification. Note that ppm in gas analyses is almost always volume per volume, whereas ppm in dissolved gas results usually means mg/L, for example, dissolved oxygen in water. The numeric results should specify the measurement units where chemical species may vary, for example, nitrate as nitrogen versus nitrate as nitrate. In the use of test kits for process control, the analyte may be measured in terms of a treating chemical, not in mg/L of a particular ion.

15.3 The user should refer to Practice E 178 to deal with outlying observations.

16. Precision and Bias

16.1 The accuracy and precision of a test kit method should be determined by the user for the specific application. The reference method or official method may be used to confirm the accuracy of a test kit method.

17. Keywords

17.1 inorganic analysis; kits; test kits; wastewater; water

SUMMARY OF CHANGES

Committee D19 has identified the location of selected changes to this standard since the last issue (D 5463–98) that may impact the use of this standard.

(1) The QC Section 13 was modified.