

ISBN 978-0-626-34264-7

SANS 382:2017

Edition 2

SOUTH AFRICAN NATIONAL STANDARD

Standard test methods for iron in water

This national standard is the identical implementation of ASTM D1068:2015, and is adopted with the permission of ASTM International.

WARNING
This document references other documents normatively.

SANS 382:2017
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Table of changes

Change No.	Date	Scope

National foreword

This South African standard was approved by National Committee SABS/TC 147/SC 04, *Water – Water sampling and analysis*, in accordance with procedures of the SABS Standards Division, in compliance with annex 3 of the WTO/TBT agreement.

This document was approved for publication in February 2017.

This document supersedes SANS 382:2007 (edition 1).

Compliance with this document cannot confer immunity from legal obligations.



Designation: D1068 – 15

Standard Test Methods for Iron in Water¹

This standard is issued under the fixed designation D1068; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 These test methods cover the determination of iron in water. Procedures are given for determining total iron, dissolved iron, and ferrous iron. Undissolved iron may be calculated from the total iron and dissolved iron determinations. The test methods are given as follows:

	Range	Sections
Test Method A—Atomic Absorption, Direct	0.1 to 5.0 mg/L	7 to 16
Test Method B—Atomic Absorption, Graphite Furnace	5 to 100 μ g/L	17 to 26
Test Method C—Photometric Bathophenanthroline μ g/L	40 to 1000 μ g/L	27 to 38

1.2 It is the user's responsibility to ensure the validity of these test methods to waters of untested matrices.

1.3 The chelation-extraction and two former photometric test methods were discontinued. See [Appendix X2](#) for historical information.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that 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 and health practices and determine the applicability of regulatory limitations prior to use. Specific hazards statements are given in [Note 4](#), [11.7.1](#), and [X1.1.2](#).*

2. Referenced Documents

2.1 *ASTM Standards:*²

[D858 Test Methods for Manganese in Water](#)

[D1066 Practice for Sampling Steam](#)

¹ These test methods are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

Current edition approved Oct. 1, 2015. Published October 2015. Originally approved in 1949. Last previous edition approved in 2010 as D1068 – 10. DOI: 10.1520/D1068-15.

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.

- [D1129 Terminology Relating to Water](#)
- [D1193 Specification for Reagent Water](#)
- [D1687 Test Methods for Chromium in Water](#)
- [D1688 Test Methods for Copper in Water](#)
- [D1691 Test Methods for Zinc in Water](#)
- [D1886 Test Methods for Nickel in 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](#)
- [D3558 Test Methods for Cobalt in Water](#)
- [D3559 Test Methods for Lead in Water](#)
- [D3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry](#)
- [D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents](#)
- [D5673 Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry](#)
- [D5810 Guide for Spiking into Aqueous Samples](#)
- [D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis](#)
- [E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry](#)
- [E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers](#)

3. Terminology

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 *total recoverable iron, n*—a descriptive term relating to the iron forms recovered in the acid-digestion procedure specified in these test methods.

4. Significance and Use

4.1 Iron is the second most abundant metallic element in the earth's crust and is essential in the metabolism of plants and animals. If presented in excessive amounts, however, it forms oxyhydroxide precipitates that stain laundry and porcelain. As a result, the recommended limit for iron in domestic water supplies is 0.3 mg/L. These test methods are useful for determining iron in many natural waters.

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

5. Purity of Reagents

5.1 Reagent grade 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.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type I. Other reagent water types may be used, provided it is first ascertained that the water is of sufficiently high purity to permit its use without adversely affecting the bias and precision of the test method. Type II water was specified at the time of round-robin testing of these test methods. In addition, water used in preparing solutions for the determination of ferrous iron shall be freshly boiled and essentially oxygen free.

6. Sampling

6.1 Collect the sample in accordance with Practices D1066 or D3370, as applicable.

6.2 Samples should be preserved with HNO₃ or HCl (sp gr 1.42) to a pH of 2 or less immediately at the time of collection. If only dissolved iron is to be determined, the sample shall be filtered through a 0.45- μ m membrane filter before acidification. The holding time for samples can be calculated in accordance with Practice D4841.

NOTE 1—Alternatively, the pH may be adjusted in the laboratory if the sample is returned within 14 days. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. This could reduce hazards of working with acids in the field when appropriate.

6.3 If ferrous iron is to be determined, the sample should be analyzed as soon as possible after collection and contact with atmospheric oxygen should be minimized.

6.4 Additional information on sampling requirements for Test Method C is provided in 33.1.

TEST METHOD A—ATOMIC ABSORPTION, DIRECT

7. Scope

7.1 This test method covers the determination of dissolved and total recoverable iron in most waters and wastewaters.

7.2 This test method is applicable in the range from 0.1 to 5.0 mg/L of iron. The range may be extended to concentrations greater than 5.0 mg/L by dilution of the sample.

7.3 This test method has been used successfully with reagent water; tap, ground, and surface waters; unspecified

³ *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.

wastewaters; and a refinery primary treatment water. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

8. Summary of Test Method

8.1 Iron is determined by atomic absorption spectrophotometry. Dissolved iron is determined by atomizing the filtered sample directly with no pretreatment. Total recoverable iron is determined by atomizing the sample following hydrochloric-nitric acid digestion and filtration. The same digestion procedure may be used to determine total recoverable nickel (Test Methods D1886), chromium (Test Methods D1687), cobalt (Test Methods D3558), copper (Test Methods D1688), lead (Test Methods D3559), manganese (Test Methods D858), and zinc (Test Methods D1691).

9. Interferences

9.1 Sodium, potassium, barium, chloride and sulfate (5000 mg/L each), calcium, magnesium, chromium, manganese, cobalt, nickel, copper, zinc, palladium, silver, cadmium, tin, lead, lithium, mercury, selenium, aluminum, antimony, arsenic, vanadium, boron, and molybdenum (100 mg/L) do not interfere.

9.2 Background correction (or chelation-extraction) may be necessary to determine low levels of iron in some waters.

NOTE 2—Instrument manufacturers' instructions for use of the specific correction technique should be followed.

10. Apparatus

10.1 *Atomic Absorption Spectrophotometer*; for use at 248.3 nm.

NOTE 3—The manufacturer's instructions should be followed for all instrumental parameters. A wavelength other than 248.3 nm may be used if it has been determined to be equally suitable.

10.1.1 *Iron Hollow-Cathode Lamp*—Multielement hollow-cathode lamps are available and have also been found satisfactory.

10.2 *Pressure-Reducing Valves*—The supplies of fuel and oxidant shall be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by suitable valves.

11. Reagents and Materials

11.1 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

NOTE 4—If the reagent blank concentration is greater than the method detection limit, distill the HCl or use a spectrograde acid. (**Warning**—When HCl is distilled an azeotropic mixture is obtained (approximately 6 N HCl). Therefore, when concentrated HCl is specified for the preparation of reagents or in the procedure, use double the volume specified if distilled acid is used.)

11.2 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO₃).

NOTE 5—If the reagent blank concentration is greater than the method detection limit, distill the HNO₃ or use a spectrograde acid.

11.3 *Nitric Acid* (1 + 499)—Add 1 volume of HNO₃ (sp gr 1.42) to 499 volumes of water.

11.4 *Iron Solution, Stock* (1 mL = 1.0 mg Iron)—Dissolve 1.000 g of pure iron in 100 mL of HCL (1 + 1) with the aid of heat. Cool and dilute to 1 L with water. Alternatively, certified iron stock solutions of appropriate known purity are commercially available through chemical supply vendors and may be used.

11.5 *Iron Solution, Standard* (1 mL = 0.1 mg Iron)—Dilute 100.0 mL of the iron stock solution to 1 L with water.

11.6 *Oxidant:*

11.6.1 *Air*; which has been passed through a suitable filter to remove oil, water, and other foreign substances is the usual oxidant.

11.7 *Fuel:*

11.7.1 *Acetylene*—Standard, commercially available acetylene is the usual fuel. Acetone, always present in acetylene cylinders can affect analytical results. The cylinder should be replaced at 345 kPa (50 psig). (**Warning**—“Purified” grade acetylene containing a special proprietary solvent rather than acetone should not be used with poly vinyl chloride tubing as weakening of the tubing walls can cause a potentially hazardous situation.)

11.8 *Filter Paper*—Purchase suitable filter paper. Typically the filter papers have a pore size of 0.45-µm membrane. Material such as fine-textured, acid-washed, ashless paper, or glass fiber paper are acceptable. The user must first ascertain that the filter paper is of sufficient purity to use without adversely affecting the bias and precision of the test method.

12. Standardization

12.1 Prepare 100 mL each of a blank and at least four standard solutions to bracket the expected iron concentration range of the samples to be analyzed by diluting the standard iron solution with HNO₃ (1 + 499). Prepare the standards each time the test is to be performed or as determined by Practice D4841.

12.2 When determining total recoverable iron add 0.5 mL of HNO₃ (sp gr 1.42) and proceed as directed in 13.1 through 13.5. When determining dissolved iron proceed as directed in Note 6, 13.1.

12.3 Aspirate the blank and standards and record the instrument readings. Aspirate HNO₃ (1 + 499) between each standard.

12.4 Prepare an analytical curve by plotting the absorbance versus concentration for each standard on linear graph paper. Alternatively read directly in concentration if this capability is provided with the instrument.

13. Procedure

13.1 Measure 100.0 mL of a well-mixed acidified sample into a 125-mL beaker or flask.

NOTE 6—If only dissolved iron is to be determined, start with 13.5.

13.2 Add 5 mL of HCl (sp gr 1.19) to each sample.

13.3 Heat the samples on a steam bath or hotplate in a well-ventilated hood until the volume has been reduced to 15 to 20 mL, making certain that the samples do not boil.

NOTE 7—When analyzing samples of brines or samples containing appreciable amounts of suspended matter or dissolved solids, the amount of reduction in volume is left to the discretion of the analyst.

NOTE 8—Many laboratories have found block digestion systems a useful way to digest samples for trace metals analysis. Systems typically consist of either a metal or graphite block with wells to hold digestion tubes. The block temperature controller must be able to maintain uniformity of temperature (65°C to 85°C) across all positions of the block. For trace metals analysis, the digestion tubes should be constructed of polypropylene and have a volume accuracy of at least 0.5 %. All lots of tubes should come with a certificate of analysis to demonstrate suitability for their intended purpose.

13.4 Cool and filter the samples through a suitable filter (11.8) (such as fine-textured, acid-washed, ashless paper), into 100-mL volumetric flasks. Wash the filter paper two or three times with water and adjust a volume.

13.5 Utilize sample from 13.4 and determine its absorbance or concentration at 248.3 nm. Aspirate HNO₃ (1 + 499) between each sample.

14. Calculation

14.1 Calculate the concentration of iron in the sample, in milligrams per litre, referring to 12.4.

15. Precision and Bias⁴

15.1 The precision of this test method for 10 laboratories, which include 16 operations within its designated range may be expressed as follows:

Reagent Water Type II:

$$S_T = 0.047 X + 0.053$$

$$S_o = 0.030 X + 0.037$$

Water of Choice:

$$S_T = 0.050 X + 0.114$$

$$S_o = 0.024 X + 0.078$$

where:

S_T = overall precision,

S_o = single-operator precision, and

X = determined concentration of iron, mg/L.

15.2 Recoveries of known amounts of iron in a series of prepared standards were as shown in Table 1.

15.3 The collaborative test data were obtained on reagent water; tap, lake, ground and surface water; unspecified wastewater; and a refinery primary treatment water. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

15.4 This section on precision and bias conforms to Practice D2777 – 77 which was in place at the time of collaborative testing. Under the allowances made in 1.4 of Practice D2777 – 13, these precision and bias data do meet existing requirements of interlaboratory studies of Committee D19 test methods.

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1035. Contact ASTM Customer Service at service@astm.org.