Designation: D7458 – 21

Standard Test Method for Determination of Beryllium in Soil and Sediment Using Ammonium Bifluoride Extraction and Fluorescence Detection[1]

This standard is issued under the fixed designation D7458; 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 test method is intended for use in the determination of beryllium in samples of soil and sediment. This test method can be used for purposes such as environmental remediation projects where beryllium is a contaminant of concern. It is also useful for characterization of levels of beryllium in soil at sites where beryllium is in mining or manufacturing applications, and for determination of background levels of beryllium in soil.

1.2 This test method assumes that samples of soil or sediment are collected using appropriate and applicable methods.

1.3 This test method includes a procedure for extraction (dissolution) of beryllium in dilute ammonium bifluoride, followed by analysis of aliquots of the extract solution using a beryllium-specific fluorescent dye.

1.4 For a 500 mg sample, the lower limit of the working range is approximately 0.04 mg Be/kg (5× dilution) or 0.1 mg Be/kg (20× dilution). The working range extends to concentrations of at least 500 mg Be/kg.

1.5 No detailed operating instructions are provided because of differences among various makes and models of suitable fluorometric instruments. Instead, the analyst shall follow the instructions provided by the manufacturer of the particular instrument. This test method does not address comparative accuracy of different devices or the precision between instruments of the same make and model.

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 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.7.1 For purposes of comparing a measured or calculated value(s) with specified limits, the measured or calculated value(s) shall be rounded to the nearest decimal of significant digits in the specified limit.

1.7.2 The procedures used to specify how data are collected/recorded, or calculated, in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analytical methods for engineering data.

1.8 *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.*

1.9 *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:*[2]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as

[1] This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality.

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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.

Used in Engineering Design and Construction
D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing
D4840 Guide for Sample Chain-of-Custody Procedures
D5730 Guide for Site Characterization for Environmental Purposes With Emphasis on Soil, Rock, the Vadose Zone and Groundwater (Withdrawn 2013)[3]
D6026 Practice for Using Significant Digits and Data Records in Geotechnical Data
D7202 Test Method for Determination of Beryllium in the Workplace by Extraction and Optical Fluorescence Detection
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

2.2 *Other Standards:*
ISO 594-2 Conical Fittings with a 6 % (Luer) Taper for Syringes, Needles and Certain Other Medical Equipment—Part 2: Lock Fittings
EN ISO 8655-2 Piston-Operated Volumetric Pipettes—Part 2: Piston Pipettes[4]

3. Terminology

3.1 *Definitions*—For definitions of common technical terms used in this standard, refer to Terminology D653 and D1356.

4. Summary of Test Method

4.1 This standard test method is used to determine the beryllium content of soil or sediment. Samples are collected in the field using appropriate and applicable procedures, for example, those described in ASTM International standards; see Guide D5730 for listings of appropriate sample collection standards. A 0.5 g aliquot is extracted using 3 % ammonium bifluoride solution heated at 90 °C for 40 hours. The presence of active fluoride ions (HF by dissociation of ammonium bifluoride in acidic medium) enables dissolution of refractory forms of beryllium, including silicates, borosilicates, and oxides. The extraction solution produced from each sample is then filtered and an aliquot of this extract is added to a pH-adjusted detection solution which contains a beryllium-specific fluorescence reagent. The fluorescence of this final solution is then measured on a calibrated fluorometer to quantify the amount of beryllium in the sample. This standard test method is adapted from Test Method D7202.

5. Significance and Use

5.1 Exposure to beryllium can cause a potentially fatal disease, and occupational exposure limits for beryllium in air and on surfaces have been established to reduce exposure risks to potentially affected workers (**1, 2**).[5] Measurement of beryllium in matrices such as soil and sediment is important in environmental remediation projects involving beryllium contamination (**3**) and for establishment of background levels of beryllium at sites where anthropogenic beryllium may have been used (**2, 4-6**). Sampling and analytical methods for beryllium are needed in order to meet the challenges relating to exposure assessment and risk reduction. Sampling and analysis methods, such as the procedure described in this test method, are desired in order to facilitate measurements of beryllium that can be used as a basis for management of remediation projects and protection of human health.

5.2 This test method can be used for purposes such as environmental remediation projects where beryllium is a contaminant of concern. It is also useful for characterization of levels of beryllium in soil at sites where beryllium is in mining or manufacturing applications, and for determination of background levels of beryllium in soil.

5.3 The limit of quantification of this test method varies with the dilution factor (see 13.6.1). For the detection solution containing lysine the detection limit is 0.013 mg beryllium per kilogram of sample, based on a 0.5 g sample (**7**) extracted in a 50 mL extraction solution and analyzed using a dilution factor of 20×. When the lysine-free detection solution is used one may use a 20× dilution factor and obtain the same detection limit or use 5× dilution factor and obtain a detection limit of 0.004 mg/kg of sample.

NOTE 1—Users of this standard are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Interferences

6.1 This test method is specific for beryllium. Other solvated metal ions are either bound by ethylenediaminetetraacetic acid (EDTA) in the detection solution, or they precipitate out due to the high alkalinity of the detection solution. The fluorophore used for detection is specific for the beryllium divalent cation (Be^{+2}).

6.2 If iron or titanium are present in high excess in the sample (typically above 7 %), the resulting measurement solution may appear golden-yellow. In this case the solution is left for two hours or more for the iron or titanium (or both) to precipitate. The solution is then re-filtered using the same procedure as for filtering the extraction solution (after the dissolution step), prior to fluorescence measurement.

7. Apparatus

7.1 *Sampling Equipment*—Use sampling apparatus appropriate for the type of media being collected (for example, soil, rock, sediment, fly ash) and its location (for example, surface, subsurface, vadose zone). Guidance on selection of appropriate sampling apparatus can be found as referenced in Guide D5730.

[3] The last approved version of this historical standard is referenced on www.astm.org.

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

[5] The boldface numbers in parentheses refer to a list of references at the end of this standard.

7.2 *Ultraviolet/Visible (UV/Vis) Fluorometer*—A device with irradiance excitation lamp or light-emitting diode (excitation λ = 365 or 384 nm) and time-integrating visible detector (400–700 nm, λmax ≈ 475 nm).

7.3 *Balance*—Balances shall conform to Guide D4753 and shall have a readability without estimation of 0.1 mg.

7.4 *Oven*—Vented, thermostatically controlled oven capable of maintaining a uniform temperature of 90 °C ± 2 °C throughout the drying chamber.

7.5 *Centrifuge Tubes*—Plastic tubes with a capacity of 15 mL and if needed, 50 mL.

7.6 *Polypropylene Bottles*—Bottles with screw caps having a capacity between 60 mL to 100 mL.

7.7 *Syringe Filters*—Hydrophilic polypropylene or polyethersulfone filters in plastic housings with a pore size of 0.2 to 0.45 µm and having a diameter of 13 mm or 25 mm. A 0.2 µm pore size filter is preferred.

7.8 *Syringes*—Plastic syringes with a capacity of 5 mL or 10 mL.

7.9 *Pipetters*—Mechanical pipetters in an assortment of sizes as needed, with tolerances in accordance with EN ISO 8655-2.

7.10 *Pipet Tips*—Disposable plastic pipet tips in an assortment of sizes sizes as needed, with tolerances in accordance with EN ISO 8655-2.

7.11 *Fluorescence Cuvettes*—Disposable, low fluorescence, 10 mm path length, transparent to UV/Visible radiation cuvettes.

7.12 *Thermometric Device*—A thermometric device capable of measuring the temperature range within which the test is being performed readable to 0.5 °C or better and having an accuracy of at least ±0.5 °C.

7.13 *Miscellaneous Items*—The following items may be needed: plastic beakers, plastic flasks, plastic graduated cylinders, plastic or plastic coated forceps, microfilters, respirators, masks, gloves, lab coats, safety eyewear.

7.14 *pH Meter*—For measurement of pH to within ±0.1 pH unit.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[6] 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent as defined by Type I of Specification D1193 (ASTM Type I Water: minimum resistance of 18 MΩ-cm or equivalent).

8.3 *Calibration Stock Solution*—1000 ppm beryllium in dilute nitric acid.

8.4 Ethylenediaminetetraacetic acid (EDTA) disodium salt dihydrate.

8.5 L-lysine monohydrochloride.

8.6 10-hydroxybenzo[h]quinoline-7-sulfonate (10-HBQS).

8.7 Sodium hydroxide (NaOH).

8.8 *Extraction (or Dissolution) Solution*—Mass fraction 3 % ammonium bifluoride (NH_4HF_2) solution (aqueous) for dissolution of beryllium in collected specimens. The solution may be prepared by dissolving 30 g ± 0.3 g of solid NH_4HF_2 in Type 1 water to a total volume of 1000 mL. **Warning—** Ammonium bifluoride will etch glass, so it is necessary that NH_4HF_2 solutions be contained in plastic labware.

8.9 *Detection Solution*—63.4 µM 10-hydroxybenzo[h] quinoline-7-sulfonate (10-HBQS) (**8, 9**) / 2.5 mM ethylenediaminetetraacetic acid (EDTA)/50.8 mM lysine monohydrochloride (pH adjusted to 12.8 with NaOH): The aqueous detection reagent is prepared by the addition of 12.5 mL of 2.5 mM ethylenediaminetetraacetic acid (EDTA) disodium salt dihydrate and 25 mL of 107 mM L-lysine monohydrochloride to 3 mL of 1.1 mM 10-hydroxybenzo[h]quinoline-7-sulfonate (10-HBQS). The pH is adjusted to 12.85 with addition of sodium hydroxide and Type 1 water added to a total of 50 mL.

8.9.1 An alternative preparation of dye solution without lysine may be made by adding 1.104 g of EDTA and 64 µmoles of the 10-HBQS dye in 900 mL of water. After a clear solution is obtained, 114.5 mL of 2.5 M NaOH is added and mixed to obtain the final dye solution.

8.9.1.1 Check the pH of the dye solution by using the pH meter; the measured pH should be 12.5 to 13.3. If the pH requires adjustment to fall within the desired pH range, add 2.5 M NaOH dropwise until the measured pH is satisfactory.

NOTE 2—The pH of the dye solution was measured by Adams et al. (**7**) as 13.2. The dye solution without lysine may be used for all analytical purposes and also provides superior detection limits.

9. Hazards

9.1 Ammonium bifluoride is highly corrosive, and is very toxic when in contact with the skin. Effects to the skin, including irritation and burns, may not be felt for several hours. Avoid exposure by contact with the skin. Use suitable personal protective equipment (including impermeable gloves and eye protection) when working with NH_4HF_2. See Appendix X1 for further pertinent safety information.

9.2 Hydrofluoric acid can cause serious medical issues. Upon contact with skin, it easily and quickly enters through the skin and into the tissues in the body where it damages cells. Upon inhalation, it can damage lung tissue and cause swelling and fluid buildup in the lungs. The seriousness of chemical

[6] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

exposure depends on the amount, route, and length of time of exposure. Use appropriate protective equipment as stated in 9.1.

10. Sampling and Test Specimens

10.1 *Sample Collection*—Collect samples in accordance with applicable standards for the type of media of interest (for example, soil, rock, sediment, fly ash) and the location being sampled (for example, surface, vadose zone). Refer to Guide D5730 in selecting applicable standards for sample collection.

10.2 *Sample Transport*—If applicable (that is, if samples are transported to a different location prior to sample preparation and analysis), follow sampling chain-of-custody procedures to document sample traceability. Make sure that the documentation that accompanies the samples is suitable for a chain of custody to be established in accordance with Guide D4840.

10.3 Wear appropriate personal protection during sample aliquoting, specimen preparation, and analysis activities. Perform these activities in a clean area that is well removed from possible beryllium contamination.

10.4 *Specimen Preparation*—Obtain a 0.5 g specimen (aliquot) that is representative of the sample (**10-13**) (Note 3). Measure and record the mass of the specimen to the nearest 0.1 mg and put it into a polypropylene bottle. Close the lid and gently tap the lid to make sure that all of the specimen (aliquot) falls to the bottom of the bottle. The bottle size is typically 10 mL to 50 mL larger than the liquid to be added.

NOTE 3—The specimen size of 0.5 g is typically necessary to sufficiently account for the heterogeneous nature of soil and sediment samples (**14**). In cases where the matrix is sufficiently homogenous to allow a smaller specimen size (for example, 50 mg), the amount of extraction solution may be reduced (for example, to 5 mL instead of 50 mL).

11. Preparation of Apparatus

11.1 *Fluorometer Set-Up*—Set up the fluorometer for excitation radiation from 360 nm to 390 nm (peak wavelengths are 365 nm or 384 nm) and measurement of emission in a spectral window selected from a range of (at least) 440 nm to 490 nm. Allow appropriate warm-up of the system prior to analysis in accordance with the manufacturer's instructions.

NOTE 4—For fluorescence measurement, an emission band pass filter with peak transmission wavelength at ~475 nm and with a full width at half maximum (FWHM) of less than ±20 nm have been shown to be effective (**4, 5**).

12. Calibration

12.1 *Preparation of Calibration Standards*—Using calibration stock solution and detection solution, prepare at least four standards covering the concentration range of interest. Record the concentration of each standard.

NOTE 5—For example: To measure from about 1 mg to 80 mg of beryllium in samples, calibration standards from 0 parts per billion (ppb) to 800 ppb are recommended (see Table 1). Alternatively, a different range of calibration standards, covering the range of interest, may be used provided that a linear calibration curve can be produced.

12.2 *Calibration and Specifications:*

TABLE 1 Preparation of Calibration Standards

Specimen	Final Concentration of Beryllium (ppb) in Calibration Standard Solutions	Corresponding Amount of Beryllium (Be) in Sample (mg/kg)[A]
0.1 mL of 0 ppb standard + 1.9 mL of detection solution (DF = 20)	0.0	Corresponds to 0 mg/kg beryllium per sample when DF = 20
0.1 mL of 10 ppb standard + 1.9 mL of detection solution (DF = 20)	0.50	Corresponds to 1 mg/kg beryllium per sample when DF = 20
0.1 mL of 40 ppb standard + 1.9 mL of detection solution (DF = 20)	2.0	Corresponds to 4 mg/kg beryllium per sample when DF = 20
0.1 mL of 200 ppb standard + 1.9 mL of detection solution (DF = 20)	10.0	Corresponds to 20 mg/kg beryllium per sample when DF = 20
0.1 mL of 800 ppb standard + 1.9 mL of detection solution (DF = 20)	40.0	Corresponds to 80 mg/kg beryllium per sample when DF = 20
0.4 mL of 0 ppb standard + 1.6 mL of detection solution (DF = 5)	0.0	Corresponds to 0 mg/kg beryllium per sample when DF = 5
0.4 mL of 1 ppb standard + 1.6 mL of detection solution (DF = 5)	0.20	Corresponds to 0.1mg/kg beryllium per sample when DF = 5
0.4 mL of 4 ppb standard + 1.6 mL of detection solution (DF = 5)	0.80	Corresponds to 0.4 mg/kg beryllium per sample when DF = 5
0.4 mL of 20 ppb standard + 1.6 mL of detection solution (DF = 5)	4.0	Corresponds to 2 mg/kg beryllium per sample when DF = 5
0.4 mL of 80 ppb standard + 1.6 mL of detection solution (DF = 5)	16.	Corresponds to 8 mg/kg beryllium per sample when DF = 5

[A] Incorporating sample dilution factor for 50 mL of extraction solution and 0.5 g of sample; note that volumes other than 50 mL and/or a different sample size will require a different appropriate dilution factor.

12.2.1 *Calibration Blank and Calibration Stock Standard Solutions Preparation*—Calibration blank is prepared by adding the 0 ppb standard and the detection solution at a volume proportion of 1:19 into a cuvette suitable for fluorescence measurements. Calibration standard solutions are also made in a similar fashion where the calibration standard and the detector solution are mixed in a volumetric ratio of 1:19 (a 20× dilution) and in a volumetric ratio of 1:4 in case 5× dilution is used. At least four standard measurement solutions, plus a blank, must be made for calibration. Make sure that these are mixed fully.

12.2.2 For routine beryllium analyses, calibration stock standard solution concentrations of 0 ppb, 10 ppb, 40 ppb, 200 ppb and 800 ppb are used. When lower detection limits are necessary, concentrations of 0 ppb, 1 ppb, 4 ppb, 20 ppb, and 80 ppb are recommended; see Table 1 for an example.

NOTE 6—The 0 ppb, 1 ppb, 4 ppb, 20 ppb, and 80 ppb standards may be made by diluting the 0 ppb, 10 ppb, 40 ppb, 200 ppb, and 800 ppb standards in 3 % ammonium bifluoride extraction solution, for example taking 0.1 mL of 800 ppb standard and mixing it with 0.9 mL of 3 % ammonium bifluoride will result in a standard with 80 ppb beryllium. Concentrations may be adjusted based on anticipated beryllium content. Solutions are preferably made up in 3 % ammonium bifluoride; alternatively, commercially available solutions using 1 % ammonium bifluoride may be used. Use of either 1 % or 3 % ammonium bifluoride has been determined to be acceptable (**5**).

12.2.3 *Instrument Calibration:*

12.2.3.1 Using the calibration standard solutions prepared above, calibrate the instrument for fluorescence intensity versus the concentration of beryllium. A calibration curve using linear regression shall be obtained between the fluorescent intensity and the concentration of beryllium. Samples shall not be left in the instrument for longer than necessary for measurement (see Note 7). The instrument should be programmed to display the concentrations (in ppb) of the calibration solutions. The correlation coefficient should be equal or greater than 0.999.

NOTE 7—Leaving samples in the instrument for longer than necessary can cause changes in sample temperature and consequent change in signal intensity.

12.2.3.2 Verify calibration by measuring an intermediate concentration standard, which should yield a value of within 10 % of the known value. The calibration shall be verified at least once every two hours (for example, after completing the measurement of the unknowns) to make sure that calibration still holds (Note 8).

NOTE 8—Changes in temperature can cause a drift in the readings; thus, it is important to verify calibration periodically.

12.2.4 The calibration of fluorescence intensity due to the amount of beryllium present can be accomplished in either of two ways: by examining instrument response due to *(a)* the concentration of beryllium in calibration solutions, or *(b)* in terms of the amount of beryllium in the media; see Table 1.

NOTE 9—The intensity calibration on the instrument may have been carried out in terms of absolute intensity or one of the following if the instrument automatically prepared a correlation using linear regression fit of concentration of beryllium in calibration standards, concentration of beryllium in calibration standard solutions or in terms of amount of beryllium in the medium (soil, rock, sediment, or fly ash). Table 1 shows a correlation between various standards, calibration standard solutions and the amount of beryllium in the sampling medium.

13. Procedure

13.1 Remove the lid of the polypropylene bottle containing the specimen (aliquot). Use a graduated cylinder to measure 50 mL ± 0.5 mL of extraction solution and then add it to the bottle.

13.2 Put the lid back on tightly and swirl the bottle to make sure that the specimen is completely wetted.

13.3 Preheat a laboratory oven to 90 °C ± 2 °C. Place the bottle in the oven for 40 h ± 1 h.

NOTE 10—The 40-hour heating step may be reduced by using a more concentrated solution: up to mass fraction 10 % of NH_4HF_2 can be used. After extraction using concentrations at or above mass fraction 5 % NH_4HF_2, dilution with water is necessary to maintain a pH of 12 or higher when mixed with the dye solution; this pH is necessary to achieve quantitative recovery (**7, 8**). Method evaluation by using more concentrated NH_4HF_2 should consider the sample media, particle physical characteristics (such as shape and size) and the inertness of beryllium-containing compounds in the specimens being analyzed.

13.4 Remove the bottle from the oven and allow it to cool to ambient temperature.

13.5 *Filtration*—It is not necessary to filter all of the specimen and extraction solution since each analysis only needs 0.1 mL of solution. Therefore, to filter the solution, attach a 25 mm diameter syringe filter to a 5 mL or 10 mL syringe with a lock fitting (as described in ISO 594-2) and pour a small amount of approximately 5 mL of the solution into the syringe. Force the solution through the syringe filter over an inert microfilter into a 15 mL centrifuge tube (see Note 11).

NOTE 11—The filtration process given in 13.5 is one way of filtering the solution. Other methods can be used provided they meet the intended results of the filtration process given in this standard.

13.6 *Preparation of Measurement Solution:*

13.6.1 Pipet 100 µL of the filtered solution into a fluorescence cuvettes. Add by pipetting 1.9 mL of detection (dye) solution to the cuvette and make sure these are mixed well by vigorous shaking. This proportion of detection solution to filtered solution represents a 20× dilution factor (DF). Either of the two detection solutions, with or without lysine, may be used (**7, 9**). Record which type of detection is solution used.

13.6.1.1 For specimens where lower detection limits are needed, pipet 400 µL of filtered solution extract into a fluorescence cuvette. Then add 1.6 mL of lysine free detection (dye) solution to the cuvette and make sure these are mixed well by vigorous shaking. This proportion of detection solution to filtered solution represents a 5× dilution factor (DF) (**7**). A 5× dilution factor must not be used with the lysine containing detection (dye) solution when using the mass fraction 3 % ammonium bifluoride extraction solution since the pH of the mixture of the specimen and the detection (dye) solution will be too low for analytical determination of beryllium (**7, 9**).

13.6.2 Make sure that the measurement solution is colorless, or near colorless. If the solution is golden yellow, wait for two hours for the solution to clear, and then re-filter the solution, as described in 13.5, into a clean 15 mL centrifuge tube.

NOTE 12—If iron is present in high excess (typically more than 20 µM)