Designation: ~~C146 – 94a (Reapproved 2014)~~ C146 – 21

Standard Test Methods for Chemical Analysis of Glass Sand[1]

This standard is issued under the fixed designation C146; 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 These test methods cover the chemical analysis of glass sands. They are useful for either high-silica sands (99 % + silica (SiO_2)) or for high-alumina sands containing as much as 12 to 13 % alumina (Al_2O_3). Generally nonclassical, ~~the~~these test methods are rapid and accurate. They include the determination of silica and of total R_2O_3 (see 11.2.4), and the separate determination of total iron as iron oxide (Fe_2O_3), titania (TiO_2), chromium oxide (Cr_2O_3), zirconia (ZrO_2), and ignition loss. Included are procedures for the alkaline earths and alkalies. High-alumina sands may contain as much as 5 to 6 % total alkalies and alkaline earths. It is recommended that the alkalies be determined by flame photometry and the alkaline earths by absorption spectrophotometry.

1.2 These test methods, if followed in detail, will provide interlaboratory agreement of results.

NOTE 1—For additional information, see Test Methods C169 and Practices E50.

1.3 ~~The~~These test methods appear in the following order:

Procedures for Referee Analysis:	Section
Silica (SiO_2)—Double Dehydration	10
Total R_2O_3—Gravimetric	11
Fe_2O_3, TiO_2, ZrO_2, Cr_2O_3, by Photometric Methods and Al_2O_3 by Complexiometric Titration	12 – 17
Preparation of the Sample for Determination of Iron Oxide, Titania, Alumina, and Zirconia	12
Iron Oxide (as Fe_2O_3) by 1,10-Phenanthroline Method	13
Titania (TiO_2) by the Tiron Method	14
Alumina (Al_2O_3) by the CDTA Titration Method	15
Zirconia (ZrO_2) by the Pyrocatechol Violet Method	16
Chromium Oxide (Cr_2O_3) by the 1,5-Diphenylcarbohydrazide Method	17
Procedures for Routine Analysis:	
Silica (SiO_2)—Single Dehydration	19
Al_2O_3, CaO, and MgO—Atomic Absorption Spectrophotometry	20–25
Na_2O and K_2O—Flame Emission Spectrophotometry	26-27
Loss on Ignition (LOI)	28

---

[1] These test methods are under the jurisdiction of ASTM Committee C14 on Glass and Glass Products and are the direct responsibility of Subcommittee C14.02 on Chemical Properties and Analysis.

Current edition approved ~~Oct. 1, 2014~~June 1, 2021. Published ~~October 2014~~July 2021. Originally approved in 1939. Last previous edition approved in ~~2009~~2014 as C146 – 94a ~~(2009).~~(2014). DOI: ~~10.1520/C0146-94AR14.~~10.1520/C0146-21.

~~1.4 *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.*~~

1.4 *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.*

1.5 *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]
C169 Test Methods for Chemical Analysis of Soda-Lime and Borosilicate Glass
C429 Test Method for Sieve Analysis of Raw Materials for Glass Manufacture
D1193 Specification for Reagent Water
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry

2.2 ~~*Other Documents:*~~*NIST Document:*[3]
~~NIST~~SP260 NIST Special Publication 260

3. Significance and Use

3.1 These test methods can be used to ensure that the chemical composition of the glass sand meets the compositional specification required for this raw material.

3.2 These test methods do not preclude the use of other methods that yield results within permissible variations. In any case, the analyst should verify the procedure and technique used by means of a National Institute of Standards and Technology (NIST) standard reference material or other similar material of known composition having a component comparable with that of the material under test. A list of standard reference materials is given in the NIST *Special Publication 260*, current edition.

4. Photometers and Photometric Practice

4.1 Photometers and photometric practice prescribed in these test methods shall conform to Practice E60.

5. Purity of Reagents

5.1 Reagent grade chemicals shall be used throughout. Unless otherwise indicated, it is intended that reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] 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 Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I, II, or III of Specification D1193.

---

[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] ~~Standard samples available from the~~ Available from National Institute of Standards and Technology ~~are listed in U.S. Dept. of Commerce, NIST,~~(NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD ~~Special Publication 260~~20899-1070, http://www.nist.gov. ~~(current edition), Washington, DC 20234.~~

[4] *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

6. Concentration of Acids and Ammonium Hydroxide (NH_4OH)

6.1 When acids and ammonium hydroxide are specified by name or chemical formula only, concentrated reagents of the following percent concentrations are intended:

	Sp Gr	%
Hydrochloric acid (HCl)	1.2	36 to 38
Hydrofluoric acid (HF)	1.2	48 to 51
Nitric acid (HNO_3)	1.4	69 to 71
Perchloric acid ($HClO_4$)	1.8	70 to 72
Sulfuric acid (H_2SO_4)	1.8	95 to 98
Ammonium hydroxide (NH_4OH)	0.9	28 to 30

6.2 Concentrations of diluted acids and NH_4OH, except when standardized, are specified as a ratio stating the number of volumes of the concentrated reagent to be added to a given number of volumes of water, as in the following example: HCl (1 + 99) means 1 volume of concentrated HCl (sp gr 1.19) added to 99 volumes of water.

7. Filter Papers

7.1 Throughout these test methods, filter papers will be designated as "coarse," "medium," or "fine" without naming brands or manufacturers. All filter papers are of the double-acid-washed ashless type. "Coarse" filter paper refers to the porosity commonly used for the filtration of aluminum hydroxide. "Medium" filter paper refers to that used for filtration of calcium oxalate, and "fine" filter paper to that used for barium sulfate.

8. Preparation of Sample

8.1 *General Considerations*—The acquisition and preparation of the sample shall follow the principles stated in Test Method C429.

8.2 The laboratory sample is reduced for analysis to 10 to ~~20 g~~ 20 g by use of a small riffle with openings preferably of ~~6.4-mm~~ 6.4 mm (¼-in.) size. The analytical sample is then ground in an agate mortar to pass a ~~150-μm~~ 150 μm (No. 100) sieve.[5] If the laboratory sample as received contains any large particles that are retained on a ~~850-μm~~ 850 μm (No. 20) sieve, these shall be sieved out, crushed (without contamination) so as to pass the sieve, and then mixed back into the laboratory sample before riffling.

9. Precision and Bias

9.1 *Precision*—The probable precision of results that can be expected by the use of procedures described in these test methods is shown in the following tabulation. Precision is given as absolute error and is dependent on the quantity of the constituent present as well as the procedure used.

	~~Probable Precision of Results, Weight %~~	
~~Constituent~~	~~Referee Analysis~~	~~Routine Analysis~~
~~SiO_2 (99 %)~~	~~±0.1~~	~~±0.25~~
~~SiO_2 (85–90 %)~~	~~±0.1~~	~~±0.25~~
~~R_2O_3 (1 %)~~	~~±0.05~~	~~±0.10~~
~~R_2O_3 (10–15 %)~~	~~±0.1~~	~~±0.15~~
~~Al_2O_3 (1 %)~~	~~±0.05~~	~~±0.10~~
~~Al_2O_3 (10–15 %)~~	~~±0.1~~	~~±0.1~~
~~Fe_2O_3~~	~~±0.003~~	~~...~~
~~TiO_2~~	~~±0.005~~	~~...~~
~~ZrO_2~~	~~±0.001 to 0.005~~	~~...~~
~~Cr_2O_3~~	~~±0.0001 to 0.001~~	~~...~~
~~CaO~~	~~...~~	~~±0.001~~
~~MgO~~	~~...~~	~~±0.001~~
~~Na_2O~~	~~...~~	~~±0.001~~
~~K_2O~~	~~...~~	~~±0.001~~

---

[5] Requirements for sieves are given in ~~ASTM~~ Specification E11.

Probable Precision of Results, Weight %

Constituent	Referee Analysis	Routine Analysis
SiO_2 (99 %)	±0.1	±0.25
SiO_2 (85–90 %)	±0.1	±0.25
R_2O_3 (1 %)	±0.05	±0.10
R_2O_3 (10–15 %)	±0.1	±0.15
Al_2O_3 (1 %)	±0.05	±0.10
Al_2O_3 (10–15 %)	±0.1	±0.1
Fe_2O_3	±0.003	. . .
TiO_2	±0.005	. . .
ZrO_2	±0.001 to 0.005	. . .
Cr_2O_3	±0.0001 to 0.001	. . .
CaO	. . .	±0.001
MgO	. . .	±0.001
Na_2O	. . .	±0.001
K_2O	. . .	±0.001

9.2 *Bias*—Standard reference materials or other similar materials of known composition should be analyzed whenever possible to determine the bias of the results.

PROCEDURES FOR REFEREE ANALYSIS

10. Silica (SiO_2) by ~~the~~ Double Dehydration Method

10.1 Weigh 1.000 g of the powdered sample and 2.0 g of anhydrous sodium carbonate (Na_2CO_3) into a clean ~~75-mL~~ 75 mL platinum dish (Note 2); mix well with a platinum or Nichrome[6] wire. Tap the charge so it lies evenly in the bottom of the dish. Cover evenly with an additional 1.0 g of Na_2CO_3. Cover with the platinum lid and heat first at a dull red heat over a clean oxidizing flame; gradually raise the temperature until a clear melt is obtained. Properly carried out, little or no spattering should occur, and the fusion can be performed in 3 to 4 min. When melted, rotate the melt to spread it evenly over the bottom and lower sides of the dish, gradually withdrawing from the flame. Cover and cool to room temperature. During fusion, the dish should be handled at all times with platinum-tipped tongs and the fusion performed with a platinum (preferably 90 % platinum and 10 % rhodium alloy) or silica triangle.

NOTE 2—To obtain accurate repeat weighings, platinum ware must be kept scrupulously clean on the outside of the vessel as well as on the inside. It should be polished brightly with fine, round grain sand and protected from dirty surfaces. It is recommended that porcelain plates be used for cooling fusions, and that platinum be set on paper towels or other clean material during filtration.

10.2 Add 20 to 25 mL of HCl (1 + 1) under the platinum cover and digest on a ~~steam bath or hot plate~~ hot plate or similar evaporation technique until the melt has completely disintegrated; it is also possible to digest the melt in the cold HCl overnight. Police and rinse the lid with a fine jet of water; rinse down the sides of the dish and evaporate to dryness on a steam bath or under an infrared lamp. Keep the dish covered with a raised cover glass during evaporation. When evaporation is complete (absence of HCl), cool, drench the residue with 5 mL of HCl, and then add 20 mL of hot water. Digest for 5 min and filter through a ~~9-cm~~ 9 cm medium filter paper. Catch the filtrate in a ~~250-mL~~ 250 mL platinum dish. Transfer the precipitated silica to the filter with the aid of a policeman and a bit of paper pulp, and wash the precipitate and paper twelve times with hot 2 % HCl. Transfer the paper and precipitate to the dish used for fusion and dehydration and reserve for subsequent ignition. Wipe the stirring rod and the periphery of the funnel with a piece of damp filter paper, and add to the dish containing the precipitate for ignition.

10.3 Evaporate the filtrate to dryness on the ~~steam bath or under an infrared lamp.~~ hot plate or in drying oven. When dry, cool, drench with 10 mL of HCl (1 + 1), and again evaporate just to dryness; then bake in a drying oven at ~~105°C~~ 105 °C for 30 min. Cool, drench with 5 mL of HCl, and add 20 mL of hot water and a small bit of filter pulp. Digest hot for 5 min and filter through a ~~7-cm~~ 7 cm fine paper. Police the dish with the aid of a bit of paper pulp and wash precipitate and paper eight times with hot 2 % HCl. Transfer the paper and precipitate to the dish containing the initial precipitation. Wipe the stirring rod and the periphery of the funnel with a piece of damp filter paper, and add to the dish containing the precipitate for ignition.

10.4 Partially cover the dish with its platinum lid, but leave enough space so air can circulate during ignition. Place the dish in a cold muffle furnace, and bring the temperature to ~~1200°C~~ 1200 °C for 30 min. Carefully and completely cover the dish before

---

[6] Nichrome is a registered trademark of the Driver-Harris Co., 308 Middlesex St., Harrison, NJ 07029.

removing it from the furnace and transfer to a desiccator. Cool to room temperature and weigh the covered dish (W_1). Moisten the silica with 1 to 2 mL of water and add 4 to 5 mL of HF and 0.5 g of oxalic acid crystals. Evaporate to dryness on a ~~sand bath or under an infrared lamp.~~ hot plate or in drying oven. Carefully sublime any remaining oxalic acid, cover the dish with its platinum cover, heat to ~~1000°C~~1000 °C for ~~2 min,~~ 2 min, cool, and weigh (W_2) as before.

10.5 *Calculation*—Calculate the percent of SiO_2 as follows:

$$SiO_2, \% = \frac{(W_1 - W_2) \times 100}{\text{sample weight}} \tag{1}$$

11. Total R_2O_3 by Ammonium Hydroxide (NH_4OH) Precipitation

11.1 *General Considerations*—The weight of sample taken for analysis is governed by the amount of Al_2O_3 known or suspected to be present. Sands low in Al_2O_3 (0.05 to 0.5 %) require a ~~5-~~5 to ~~10-g~~ 10 g sample; sands with larger amounts of Al_2O_3 require a ~~0.5-~~0.5 to ~~1.0-g~~ 1.0 g sample. Usually experience or prior information will indicate a satisfactory sample weight. The total R_2O_3 serves as a check on the sum of the R_2O_3 oxides determined separately. It also helps to identify an unknown sand as a low- or high-alumina type.

11.2 *Procedure:*

11.2.1 Weigh a suitable weight of sample into an ~~80-~~80 to ~~100-mL~~100 mL platinum dish, moisten, and add 10 mL of HF for each gram of sample taken; add 4 mL of H_2SO_4 (1 + 1) and evaporate to the first fuming of H_2SO_4 (Note 3). Cool, carefully wash down the sides of the dish with a minimum of water, and evaporate to the cessation of H_2SO_4 fumes. Cool, add 10 to 15 mL of HCl (1 + 1), 20 mL of hot water, and digest hot until the salts are in solution. If they do not dissolve readily, transfer to a beaker, police the dish, and boil the solution until the sulfates have dissolved (Note 4).

NOTE 3—Some sands may contain small amounts of organic matter as shown by the presence of carbon or carbonaceous material in the concentrated H_2SO_4. If this is the case, add 2 to 3 mL of HNO_3 and 10 to 15 drops of $HClO_4$, and proceed.

NOTE 4—High-alumina sands are generally mixtures of quartz and aluminum silicates of the feldspar group. Some of these silicates can contain barium. If a fine, white, insoluble precipitate persists, it is probably barium sulfate. In this case, partially neutralize the HCl until the solution is about 1 to 2 % acid, add about ten drops of H_2SO_4 (1 + 1) and boil gently for about 30 min. Cool, and after 1 to 2 h, filter the solution through a fine paper. The precipitate may be ignited and weighed and subsequently tested for barium. If the precipitate is not barium sulfate, it should be tested for silica. If the precipitate is neither of these, it can be considered R_2O_3 and added to the R_2O_3 found by ammonia precipitation.

11.2.2 If the expected R_2O_3 is about 10 mg, dilute the sample to about 75 to 100 mL; if much larger, dilute to about 200 to 250 mL. Add approximately 2 g of NH_4Cl, heat to boiling, add three to four drops of methyl red indicator solution and precipitate the R_2O_3 with the addition of NH_4OH (1 + 1). Add the NH_4OH slowly, stirring to obtain a sharp end point; finally add about four drops in excess for small amounts of precipitate and up to eight drops for large amounts. Boil the solution for about 2 min and filter through a coarse paper; there is no need to transfer quantitatively all the precipitate at this time. Wash the precipitate three to four times with hot 2 % NH_4Cl made neutral to methyl red. Transfer the precipitate back into the beaker and add 10 to 15 mL of HCl (1 + 1) and digest to disintegrate the paper and dissolve the precipitate. Dilute to approximately the same volume used for the first precipitation, reprecipitate with NH_4OH, and filter as before. Police the beaker with a bit of paper pulp to ensure complete recovery from the beaker. Wash four to five times with hot 2 % NH_4Cl solution.

11.2.3 Transfer the precipitate to a clean, tared platinum or porcelain crucible and ignite at a temperature of ~~1200°C~~1200 °C for ~~30 min.~~ 30 min. Unglazed porcelain is best for the ignition as it does not change weight at this temperature. If platinum is used, both outer and inner surfaces should be polished bright. It is also advisable to carry an empty crucible through the ignition cycle to see if a platinum weight change occurs. A slight loss can be considered normal. If a gain in weight occurs, the platinum can be considered dirty and should be repolished and cleaned before reuse. The correct weight can be salvaged by brushing the dish or crucible free of precipitate and reweighing, in which case the original tare weight is not used for computation:

$$R_2O_3, \% = [(\text{weight of precipitate})/(\text{weight of sample})] \times 100 \tag{2}$$

11.2.4 The R_2O_3 contains the Al_2O_3, Fe_2O_3, TiO_2, ZrO_2, and Cr_2O_3 in the ~~sample (phosphoric~~ sample. If present, phosphoric anhydride (P_2O_5) and vanadium pentoxide (V_2O_5) will be included ~~if present, but this is not usual).~~and may impact precision and accuracy. Al_2O_3 is estimated by subtracting the sum of the other oxides from the R_2O_3.

12. Preparation of ~~the~~ Sample for Determination of Iron Oxide, Titania, Alumina, and Zirconia

12.1 *Reagents: Fusion Mixture*—Weigh an approximate 1 + 1 mole portion of lithium carbonate (Li_2CO_3) and anhydrous sodium tetraborate ($Na_2 B_4O_7$), 74 and 201 g, respectively, and mix intimately.

12.2 *Procedure for ~~Low-Alumina, High-Silica~~Low-alumina, High-silica Sands*—Weigh 4 g of sample dried at ~~110°C~~110 °C into a ~~75-~~75 to ~~100-mL~~ 100 mL platinum dish, add 40 mL of HF, and evaporate to near dryness. Wash down the sides of the dish with 10 mL of HF (use a small plastic cylinder or polyethylene dropping pipet) and evaporate to dryness (Note 5). Without any prior heating, evenly cover the residue in the dish with 2 ± 0.02 g of fusion mixture; heat over a gas burner until the residue is in solution in the melt (Note 6). To the fused residue, add 10 mL water and 20 mL of $HClO_4$ (1 + 4); cover and digest hot until the melt is in solution (Note 7). Transfer to a ~~200-mL~~ 200 mL volumetric flask, cool, dilute to the mark, and mix (Note 8). The sample is now prepared for the determination of Fe_2O_3, Al_2O_3, TiO_2, and ZrO_2; the sample for Cr_2O_3 is prepared separately (see Section 17). Prepare a reagent blank with the samples. Aliquots identical to those for Fe_2O_3, TiO_2, and ZrO_2 are used as the photometric reference solutions (Note 9).

NOTE 5—In the procedure for high-alumina sands (12.3), it is preferable to add a few drops of H_2SO_4 with the second addition of HF. This eliminates the chance of volatilizing aluminum and titanium fluorides as the fusion is started.

NOTE 6—The fusion is rapid and can be performed simply as follows: Heat over a Meeker-type burner at a moderate heat until the mixture melts, apply just enough additional heat to give a moderate red heat. No lid is required if the initial heating is not too high. The fusion can be done in ~~2 min~~ 2 min per sample. The dish must be handled with clean platinum-tipped tongs. The only allowable substitute is pure nickel tongs and these must be considered only in an emergency.

NOTE 7—Some samples may develop a cloudiness or precipitate after solution of the fusion or transfer to the volumetric flask. Tests have shown this will not affect results for Fe_2O_3, TiO_2, or Al_2O_3. After diluting to the mark of the flask and mixing, the precipitate is allowed to settle; sample aliquots are pipeted without disturbing the precipitate. The precipitate is probably a fluoborate.

NOTE 8—An aliquot of this solution can now be used for the Cr_2O_3 analysis (Section 17).

NOTE 9—Use of a predetermined amount of buffer for the determination of Fe_2O_3 and TiO_2 obviates the use of indicators and speeds the analysis when a group of samples must be analyzed. Preparation for this is made as follows: Weigh 2 g of fusion mix into a ~~250-mL~~ 250 mL beaker, add 100 mL of water and 20 mL of the HCl (1 + 4), cover, and boil for several minutes to eliminate CO_2. Cool and transfer to a ~~200-mL~~ 200 mL volumetric flask, dilute to the mark, and mix. Transfer a ~~25-mL~~ 25 mL aliquot to a ~~150-mL~~ 150 mL beaker and dilute to about 70 to 80 mL. Add from a ~~100-mL~~ 100 mL buret (which is used for dispensing) enough 2*M* sodium acetate solution to give a pH of 3.1 (make measurements with a pH meter). Record the volume used for the determination of iron. Continue adding sodium acetate until a pH of 3.8 is reached; record for the determination of titanium.

12.3 *Procedure for ~~High-Alumina, Low-Silica~~High-alumina, Low-silica Sands*—The method and technique is identical to 12.2 with the exception of weights and volumes. Weigh 2 g of sample dried at ~~110°C~~110 °C into a ~~75-mL~~ 75 mL platinum dish and add 20 mL of HF; evaporate to near dryness. Wash down the sides of the dish with 5 mL of HF as in 12.2 and evaporate to dryness. Add 3 g of fusion mix and fuse as in 12.2. Add 15 mL of water and 26 mL of HCl (1 + 4) and digest until in solution. Transfer to a ~~100-mL~~ 100 mL volumetric flask; cool, dilute to the mark, and mix (Note 7). The amounts of predetermined buffer should be nearly the same as for 12.2; however, test the pH before proceeding (Note 8).

13. Iron Oxide (as Fe_2O_3) by ~~the~~ 1,10-Phenanthroline Method

13.1 *Reagents:*

13.1.1 *Hydroxylamine Hydrochloride* (10 % weight/volume in water)—Filter if necessary.

13.1.2 *1,10-Phenanthroline*—The solution may be prepared from the monohydrate or the hydrochloride. The latter is readily water soluble; the monohydrate requires heating. Dissolve 12.0 g of the monohydrate by adding to 800 mL of hot water, stir and heat until in solution, cool and dilute to 1 L; store in a dark bottle or in a dark place. If the hydrochloride is used, dissolve 13.0 g in 200 to 300 mL of water and dilute to 1 L; protect from light during storage. Two millilitres of either solution will complex 1.2 mg. This will cover the absorbance curve for the area of interest depending on instrumentation.

13.1.3 *Sodium Acetate (Buffer) Solution* (2*M)*—Dissolve 272 g of sodium acetate ($CH_3COONa·3H_2O$) per litre of aqueous solution prepared. Filter before use if necessary. Since sodium acetate solutions tend to develop mold growth with age, a preservative can be used; 0.025 g of para-chlorometaxylenol per litre has been found satisfactory for this purpose.

13.2 *Fe_2O_3 Procedure (For All Sands):*

13.2.1 For sand with an iron content between 0.01 and 0.12 % Fe_2O_3, pipet an aliquot equivalent to 0.5 g (25 mL) into a ~~100-mL~~ 100 mL volumetric flask if the Fe_2O_3 is between 0.10 and 0.24 %, transfer the aliquot to a ~~200-mL~~ 200 mL volumetric flask (Note 10). If the Fe_2O_3 is higher than 0.24 %, a proportionally smaller aliquot will be necessary. By choice of volume and size of aliquots, a single standard curve should be adequate for the percentages of iron normally encountered in glass sand.

13.2.2 To the sample in the flask, add 1 mL of hydroxylamine hydrochloride and the predetermined amount of buffer, dilute to ¾ the volume of the flask, and add either 1 or 2 mL of 1,10-phenanthroline, depending on the iron present, mix, dilute to the mark, and after 5 min, measure the absorbance at 508 nm on a suitable (spectro) photometer. The reagent blank is used as the reference solution.

13.2.3 *Calculation*—Convert the photometric reading to milligrams of Fe_2O_3 by means of the standard curve, and calculate the percent Fe_2O_3 as follows:

$$\%\ \mathrm{Fe_2O_3} = \frac{A \times B \times 100}{C \times D \times 1000} \tag{3}$$

where:

~~A = milligrams of Fe_2O_3 from the calibration curve;~~
A = milligrams of Fe_2O_3 from the calibration curve,
~~B = total volume from 12.2, mL;~~
B = total volume from 12.2, mL,
~~C = sample weight from 12.2, g; and~~
C = sample weight from 12.2, g, and
D = millilitres of aliquot from 13.2.1.

NOTE 10—If color is developed in a volumetric flask other than ~~100-mL~~ 100 mL volume, then this must be taken into account in the calculation in 13.2.3.

13.3 *Preparation of ~~the~~ Standard Curve for Standard Iron Solution*—Weigh 0.4911 g of ferrous ammonium sulfate into a ~~1-L~~ 1 L volumetric flask, dissolve in water, add 8 to 10 mL of HCl, dilute to the mark and mix; 1 mL = 0.1 mg of Fe_2O_3; (the fact that the iron may slowly oxidize is of no consequence as it is subsequently reduced when developing the complex). Prepare a series of ~~100-mL~~ 100 mL volumetric flasks containing 0, 1, 2, 3, 4, 5, and 6 mL of the standard iron solution, dilute to 20 to 30 mL, and proceed as described in 13.2. The zero iron solution is the photometric reference. ~~Plot on linear graph paper~~Create linear plot of absorbance versus concentration in milligrams of Fe_2O_3.

14. Titania (TiO_2) by ~~the~~ Tiron Method

14.1 *Reagents:*

14.1.1 *Buffer* (2*M Sodium Acetate)*—See 13.1.3.

14.1.2 *Acetate Buffer* (pH 4.5)—To 1 L of 1*M* sodium acetate solution add 390 mL of glacial acetic acid. Adjust to a pH of 4.5 with either solid sodium acetate or glacial acetic acid using a pH meter.

14.1.3 *Thioglycolic Acid ($CH_2SHCOOH$, Reagent, Assay 96 to 97 %)*—Prepare a 20 % v/v solution; keep refrigerated.

14.1.4 *Tiron Reagent ~~(Disodium-1,2-di-Hydroxybenzene-3,5-Disulfonate)~~ ~~—~~(Disodium-1,2-di-Hydroxybenzene-3,5-Disulfonate)*—Prepare a 5 % w/v solution. Filter if necessary. The solution should be nearly colorless. Protect from light in storage.

14.1.5 *Titanium Dioxide, Standard Solution* (1 ~~mL = 1.0-mg~~ mL = 1.0 mg TiO_2)—Weigh 1.0026 g of National Institute of Standards and Technology SRM No. 154b titanium dioxide and prepare 1 L of solution as directed by the certificate furnished with the material for use as a standard for colorimetry. (If an older supply, Nos. 154 or 154a, is available, use the appropriate weight as determined from the certified percentage of TiO_2.)

14.1.6 *Titanium Dioxide, Dilute Standard Solution* ~~(1 mL = 1.0-mg~~(1 mL = 1.0 mg TiO_2)—Pipet 50 mL of the ~~1.0-mg~~ 1.0 mg TiO_2/mL standard solution into a ~~500-mL~~ 500 mL volumetric flask, add 15 mL of H_2SO_4, and dilute to about 400 mL; mix by swirling. Cool to room temperature, if necessary; dilute to volume and mix.