Designation: D 1608 – 98 (Reapproved 2003)

Standard Test Method for Oxides of Nitrogen in Gaseous Combustion Products (Phenol-Disulfonic Acid Procedures)[1]

This standard is issued under the fixed designation D 1608; 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.

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

1. Scope

1.1 This test method describes the phenol-disulfonic acid colorimetric procedure **(1)**[2] for the determination of total oxides of nitrogen [nitrous oxide (N_2O) excepted] in gaseous effluents from combustion and other nitrogen oxidation processes.

1.2 It is applicable to a concentration range of oxides of nitrogen as nitrogen dioxide (NO_2) of 5 ppm to several thousand parts per million by volume (four to several thousand milligrams per dry standard cubic metre).

1.3 Since the grab sampling technique used takes a relatively small sample over a very short period of time, the result obtained will be an instantaneous measure of the nitrogen oxides and, therefore, will be representative of the emissions only if the gas stream is well mixed and the concentration constant with time. Multiple samples are recommended.

1.4 The values stated in SI units are to be regarded as standard. The SI equivalents are in parentheses and may be approximate.

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.* (For more specific safety precautionary information see 8.5 and Section 3.)

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[3]

D 1357 Practice for Planning the Sampling of the Ambient Atmosphere[3]

D 1605 Practices for Sampling Atmospheres for Analysis of Gases and Vapors[4]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1356.

4. Summary of Test Method

4.1 The gas sample is admitted into an evacuated flask containing an oxidizing absorbing solution consisting of hydrogen peroxide in dilute sulfuric acid. The oxides of nitrogen are converted to nitric acid by gas phase oxidation due to oxygen in the sample and by the absorbent solution. The resulting nitrate ion is reacted with phenol disulfonic acid to produce a yellow compound (the tri-ammonium salt of 6-nitro-1-phenol-2,4-disulfonic acid), which is measured colorimetrically. Calibration curves, prepared from samples of known nitrate content, are used to determine the amount of nitrate in the sample with results expressed as nitrogen dioxide.

5. Significance and Use

5.1 This test method provides a means to measure the total nitrogen oxides (NO_x) content of gaseous emissions for purposes such as determining compliance with regulations, studying the effect of various abatement procedures on NO_x emissions, and checking the validity of instrumental measurements.

6. Interferences (1, 2)

6.1 Inorganic nitrates, nitrites, or organic nitrogen compounds that are easily oxidized to nitrates interfere with the test method and give erroneously high results. The presence of certain reducing agents, for example, sulfur dioxide (SO_2), may interfere by consuming part of the hydrogen peroxide in the absorbing solution to leave an inadequate amount for reaction with the oxides of nitrogen. Halides lower the results, but interference from halide ion (and lead) are negligible in the concentration usually encountered in combustion sources.

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[1] This test method is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

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

[3] *Annual Book of ASTM Standards,* Vol 11.03.

[4] Discontinued—see *1991 Annual Book of ASTM Standards,* Vol 11.03.

6.2 The role of some of the constituents of combustion effluents as possible interfering substances has not been thoroughly investigated.

7. Apparatus

7.1 The assembled sampling apparatus is shown in Fig. 1.

7.2 *Barometer,* capable of measuring atmospheric pressure to ±250 Pa [±2 mm Hg].

7.3 *Bottles,* 120-mL or 4-oz, glass or polyethylene, with leak-free noncontaminating caps.

7.4 *Evaporating Dishes,* new condition, unetched borosilicate glass or porcelain, about 200-mL capacity. Do not use platinum ware **(7).**

7.5 *Mercury Manometer,* open-end U-tube, 1 mm [or 36 in.] with 1-mm [or 0.1-in.] divisions.

NOTE 1—An unbreakable, roll-up type is convenient for this application.

7.6 *Microburet,* 10-mL capacity, with 0.01-mL divisions.

7.7 *pH Paper,* or litmus paper, covering the range from 7 to 14 pH.

7.8 *Sample Collection Flask, Calibrated*—Two-litre round-bottom glass flask with a short neck 24/40 standard-taper joint, protected against implosion or breakage with tape or foamed plastic, with known volume.

7.9 *Sampling Probe,* borosilicate glass, approximately 6-mm inside diameter, fitted with a 12/5 spherical joint for attachment to the three-way stopcock on the sample collection flask, provided with a filter on the inlet end for removal of particulate matter, long enough to extend from approximately one-third to halfway into the stack or duct (or at least 1 m beyond inside wall of stacks greater than 2 m in diameter), and heated or insulated, or both, sufficiently well to prevent condensation of moisture while purging and sampling.

7.10 *Spectrophotometer,* or filter photometer, capable of measuring absorbance at 405 nm.

NOTE 2—A wavelength of 400 nm was actually used in the Project Threshold tests, but recent work **(3, 6, 7)** has shown that the absorbance peak maximum is actually at 405 nm, which is therefore a better choice. This change should tend to improve the precision and bias of the test method.

7.11 *Squeeze-bulb,* rubber, valved for one-way flow to purge sampling probe.

7.12 *Stirring Rod,* polyethylene, to avoid scratching the evaporating dishes.

7.13 *Stopcock, Three-way, T-bore,* with a 24/40 joint for attachment to the sample collection flask, and a 12/5 spherical joint for attachment to the sampling probe.

7.14 *Stopcock Grease,* high vacuum, high temperature, inert.

7.15 *Thermometer,* dial-type or glass, with 1°C [2°F] divisions and an approximate range from −5 to 50°C [25 to 125°F].

7.16 *Vacuum Pump,* portable, capable of evacuating the sample collection flask to a pressure of about 2.5 kPa [25 mm Hg] or less.

7.17 *Volumetric Flasks,* 50, 100, 1000-mL capacity.

7.18 *Volumetric Pipets,* 1, 2, 3, 4, and 5-mL capacity.

7.19 *Water Bath or Steam Bath,* operating at approximately 100°C [212°F] (an electric hot plate is not suitable because it tends to cause spattering and possible loss of sample).

8. Reagents

8.1 *Purity of Reagents*—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.[5] Other reagents may be used provided it can be demonstrated that they are of sufficiently high purity to permit their use without decreasing the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type III or better of Specification D 1193. Additionally, this test method requires water that is nitrate-free as demonstrated by a low-blank absorbance reading (less than 0.002 nm) in Section 5.

8.3 *Absorbing Solution*—Dilute 3.0 mL of hydrogen peroxide (H_2O_2, 3 %) to 100 mL with sulfuric acid solution (H_2SO_4,

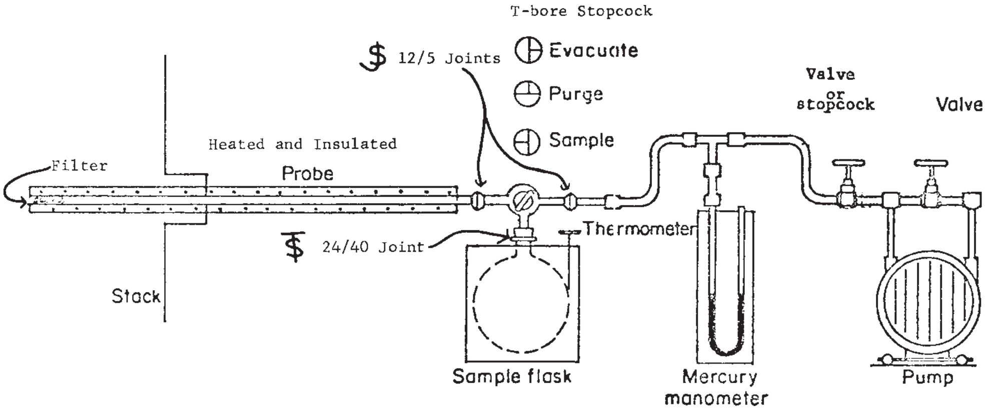

FIG. 1 Diagram of Typical Sampling Apparatus used for Determination of Oxides of Nitrogen by Test Method D 1608

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[5] *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.

3+997). A fresh solution shall be prepared weekly. Do not expose to excessive heat or direct sunlight for prolonged time.

8.4 *Ammonium Hydroxide (sp gr 0.90)*—Concentrated ammonium hydroxide (NH_4OH). A fresh solution shall be used.

8.5 *Fuming Sulfuric Acid* ($H_2SO_4 \cdot XSO_3$), 15 to 18 weight % free sulfur trioxide. **Caution**—Handle with care.

8.6 *Hydrogen Peroxide (3 %)*—Dilute 10 mL of concentrated H_2O_2(30 %) to 100 mL. A fresh solution shall be prepared each time new absorbing solution is prepared.

NOTE 3—If the strength of the H_2O_2(30 %) is in doubt, test as follows: Weigh accurately about 5 mL of the H_2O_2 solution and dilute to exactly 500 mL. To 20 mL of the dilute solution add 20 mL of H_2SO_4(1+9) and titrate with 0.1 *N* potassium permanganate ($KMnO_4$) solution to a permanent pink color. One millilitre of 0.1 *N* $KMnO_4$ solution = 0.001701 g of H_2O_2.

8.7 *Phenol* (C_6H_5 OH), pure white solid.

8.8 *Phenol Disulfonic Acid Solution*—Dissolve 25 g of phenol in 150 mL of concentrated H_2SO_4(sp gr 1.84) by heating on a steam bath. Cool, add 75 mL of fuming H_2SO_4(15 to 18 % SO_3) and heat on the steam bath at 100°C [212°F] for 2 h. Cool and store in a dark glass-stoppered bottle. The solution should be colorless; it deteriorates on long standing. Discard if a yellow color develops.

8.9 *Potassium Nitrate* (KNO_3)—Dry in an oven at 105 to 110°C for 2 h just before preparation of the standard solution.

8.10 *Potassium Nitrate, Stock Standard Solution (1 mL = 1 mg NO_2)*—Dissolve exactly 2.1980 g of dried KNO_3 in water and dilute to 1 L in a volumetric flask.

8.11 *Potassium Nitrate, Working Standard Solution (1 mL = 100 µg NO_2)*—Dilute 10 mL of the stock standard potassium nitrate solution (1 mL = 1 mg NO_2) to 100 mL with water in a volumetric flask.

8.12 *Sodium Hydroxide Solution (~1 N)*—Slowly add 40 g of sodium hydroxide (NaOH) pellets to 800 to 900 mL of water in a 2-L beaker with stirring until all pellets are dissolved. Dilute to 1 L with water and mix well. Store in an airtight polyethylene or polypropylene bottle.

8.13 *Sulfuric Acid (sp gr 1.84)*—Concentrated H_2SO_4, minimum assay 95 %.

8.14 *Sulfuric Acid (3+997)*—Carefully add 3 mL of concentrated H_2SO_4(sp gr 1.84) to water and dilute to 1 L.

9. Safety Precautions

9.1 Cover the glass sample collection flask, which is evacuated during the sampling procedure, with tape or foamed plastic to avoid injury in case of implosion or breakage.

9.2 The fuming sulfuric acid used in preparing the phenol disulfonic acid reagent is highly corrosive and fumes in moist air. Wear protective gloves, apron, and face shield, and carry out the reagent preparation in a hood.

9.3 Concentrated acids and bases are used throughout the laboratory procedure. Use care when adding them to other solutions to avoid overheating and skin contact.

10. Sampling

10.1 Pipet 25.0 mL of absorbing solution into a calibrated sampling flask and attach the three-way stopcock to the flask (see Note 4) with the T-bore in the purge position. Insert the sampling probe tip from approximately one-third to halfway into the stack or duct, or at least 1 m into stacks greater than 2 m in diameter, in such a way as to prevent leakage of air into the stack around the probe. Assemble as shown in Fig. 1, making sure that all ground-glass joints have been properly greased, and that all joints and fittings are tight and leak-free. Turn the flask stopcock to the evacuate position and evacuate the flask to the incipient boiling of the solution. If the incipient boiling of the solution cannot be seen, this will be indicated on the manometer by a low and constant reading (usually around 2.5 to 4.0 kPa [20 to 30 torr or 0.8 to 1.2 in. Hg]. Shut off the pump valve and then the pump. Check for leakage by observing the manometer for any pressure increase. Any increase greater than 1.33 kPa [10 torr or 0.4 in. Hg] over a 1-min period is not acceptable; do not take a sample until the leakage problem is corrected. Turn the flask stopcock to the purge position. Using a pump or a valved rubber squeeze bulb, thoroughly purge the sampling probe and the flask stopcock with the sample gas until the probe and stopcock are warmed to the gas temperature and free of condensate. If condensation remains, heat the probe and purge until the condensation disappears. With the pump valve and pump shut off, turn the flask stopcock to the evacuate position and record the flask temperature, the difference in mercury levels in the manometer, and the barometric pressure. The absolute pressure in the flask is equal to the barometric pressure minus the manometer reading. Immediately, turn the stopcock to the sample position so that the gas enters the flask and the pressures in the flask and the sample line are equalized (usually about 15 s are sufficient). Turn the stopcock to the purge position to seal the flask and allow the gas to remain in contact with the absorbing solution overnight (see Note 5) at room temperature. For further information on sampling, refer to Practice D 1357 and Practices D 1605.

NOTE 4—The end 6 mm [¼ in.] of the male 24/40 standard-taper joint is not lubricated to minimize contact of the gas sample with stopcock grease during absorption.

NOTE 5—If an overnight absorption period is not feasible, the sample can be shaken initially and every 20 min for a 2-h period. The result will not be significantly different than for a static overnight absorption period, provided that the oxygen concentration in the flask is greater than 1 %.

11. Calibration

11.1 *Sample Collection Flask Volume*—Fill the sample flask and stopcock assembly with water up to the stopcock plug. Determine the volume to ±10 mL by measuring either the volume or weight of the water contained in the assembly. Number and record the volume on the flask.

11.2 *Spectrophotometer Calibration*—Prepare a calibration curve to cover a range from about 0 to 125 ppm NO_2, based on 2000-mL samples of dry gas at 21°C [70°F] and 101.33 kPa [760 torr 29.92 in. Hg]. Using a microburet or pipets, transfer 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 mL of the working standard KNO_3 solution (1 mL = 100 µg NO_2) into the 200-mL evaporating dishes and add 25.0 mL of absorbing solution to each. Proceed in accordance with 12.3 to 12.5. Construct a calibration curve by plotting the absorbencies of the solutions at 405 nm against the micrograms of NO_2.

NOTE 6—Higher sample concentrations may be analyzed using this curve by taking smaller aliquots of the absorbing solution or by dilution, or both, of the color-developed solution before reading in the spectrophotometer.

12. Procedure

12.1 After the absorption period is completed, record the barometric pressure and the room temperature where the sample has stood. Connect one arm of the sample flask stopcock to the open-end manometer, turn the stopcock to open the flask to the manometer, and read the difference between the mercury levels in the manometer. The absolute internal pressure in the flask is then the barometric pressure less this difference. Correct the gas volume in the flask as directed in 13.1.

12.2 Transfer the absorbing solution quantitatively from the flask into a 200-mL evaporating dish (see Note 7). Pipet 25.0 mL of unused absorbing solution into another evaporating dish for a blank and add the same amount of water to this dish as was used in transferring the sample. Proceed with the blank in the same manner as directed for the sample.

NOTE 7—If the sample is expected to have a high concentration of oxides of nitrogen, transfer it to a 50-mL volumetric flask instead of the evaporating dish, and dilute to the mark with water. Select a suitable aliquot and pipet it into a 200-mL evaporating dish. Likewise dilute 25.0 mL of unused absorbing solution to 50 mL and pipet an aliquot equal to that of the sample into a 200-mL evaporating dish for a blank.

NOTE 8—To save time, a 15-mL aliquot of the used absorbing solution may be pipetted into the evaporating dish, rather than quantitatively transferring the whole solution. This is permissible if the concentration of nitrogen dioxide (NO_2) is greater than 10 ppm, and if the evacuation of the flask prior to admitting the sample was to the incipient or flash boiling point of the unused absorbing solution. Evacuation up to 1 min after flash boiling appears to result in about 1 % decrease in the volume of the solution.

NOTE 9—If necessary, to immediately reuse the sampling flask or to ship the used absorbing solution back to a laboratory for analysis, the entire used absorbing solution or a 15-mL aliquot may be quantitatively transferred to a leak-free glass or polyethylene bottle until ready for analysis.

12.3 Add NaOH solution to the sample solution to the evaporating dish and to the blank until each is just basic to litmus or pH paper. Do not add any excess NaOH **(7).** Evaporate each to dryness on the water bath and allow to cool. Very carefully add 2 mL of phenol disulfonic acid solution to each residue and triturate thoroughly with a polyethylene rod to ensure complete contact of the residue with the solution. Add 1 mL of water and 4 drops of H_2SO_4(sp gr 1.84) to each and heat on the water bath for 3 min with occasional stirring. Allow the mixture to cool, add 10 mL of water to each, and mix well by stirring. Add 15 mL of fresh, cool NH_4OH dropwise to each, with constant stirring. Test with litmus paper to make sure an excess of the NH_4OH is present.

12.4 Filter the solutions through 7-cm, rapid, medium-texture filter papers (see Note 10) into 50-mL volumetric flasks. Wash the evaporating dishes three times with 4 to 5 mL of water and pass the washing through the filters. Make up the volumes of the solutions to 50 mL with water and mix thoroughly.

NOTE 10—Reference **(7)** recommends discarding any sample for which a significant precipitate is present or the evaporating dish is visually etched. However, the need for this has not been demonstrated. The same grade filter paper should, however, be used both in preparing the calibration curves and in running the samples. It has been found that some yellow color is retained on the paper when filtering more concentrated samples and this factor must be taken into account by use of the sample type of filter paper throughout, or by continued washing until no color is retained in any case. Alternatively, the ammoniacal solutions may be centrifuged, instead of filtered, after dilution to 50 mL.

12.5 Read the absorbance of the sample solution against the blank in suitable equipment for measurement at 405 nm. If the absorbance falls beyond the range of calibration, thinner cells may be used or a suitable aliquot selected. Dilute the aliquot and the blank to the same volume and read the absorbance of the sample aliquot against that of the blank aliquot.

12.6 Convert the photometric readings to micrograms of NO_2 by means of the calibration curves.

NOTE 11—The calibration curves must not be assumed to be usable over any protracted length of time. One or more standards should be run along with the samples each time a set is run, or at least every few days if samples are being run daily.

13. Calculations

13.1 *Gas Sample Volume Correction*—Correct the volume of the dry gas sample (after removal of absorbed constituents) to 21°C [70°F] and 101.33 kPa [760 torr or 29.92 in. Hg].

13.1.1 Calculate the volume of gas sample as follows:

$$V_c = (V_f - V_a)(P_f/T_f - P_i/T_i)(21 + 273.2)/101.33$$

where:

V_c = corrected volume of sample gas, mL,
V_f = volume of sample collection flask, mL,
V_a = volume of absorbing solution used, 25 mL,
P_i = initial absolute pressure in flask prior to sampling, kPa,
P_f = final absolute pressure in flask after absorption period, kPa,
T_i = initial absolute temperature of flask prior to sampling, K (°C + 273.2), and,
T_f = final absolute temperature of flask after absorption period, K (°C + 273.2).

13.2 Calculate the concentration of NO_2 in parts per million by volume (ppm), dry basis, as follows:

$$NO_2\text{, ppm} = (24.1\ W \times 10^3)/46V_c$$

where:

V_c = corrected volume of sample gas, mL,
W = weight of NO_2 found in gas sample, µg,
24.1×10^3 = standard molar gas volume at 21°C [70°F] and 101.33 kPa [760 torr or 29.92 in. Hg], mL, and
46 = formula weight of NO_2.

NOTE 12—Parts per million by volume of NO_2 may be converted to milligrams per cubic metre (mg/m^3) or to pounds per cubic foot (lb/ft^3) by multiplying by 1.91 and by 1.19×10^{-7}, respectively.

14. Precision and Bias (3, 4, 5)

14.1 In an ASTM-sponsored collaborative effort, laboratories, which had not worked under such conditions before, performed a series of pilot-plant tests indicating a correlation between the precision and the square root of the mean concentration of the NO_x over the range from 20 to 2000 ppm.

The measures of precision used presented standard deviations for the components of variance between-laboratories (S_R) and within-laboratories (S_W) obtained from the analysis of variance technique. These correlations are shown in Fig. 2 **(5).**

14.2 These same laboratories, working in the field at an oil-fired power plant and cement kiln, showed a mean between-laboratory standard error of 14.5 ppm over NO_x concentrations in the range from 90 to 260 ppm. The mean between-laboratory standard error obtained from the field data is significantly lower than the comparable standard error measure calculated from the pilot plant work. Presumably, the increased experience of the operators contributed to the improved precision.

14.3 To use these data to determine the 95 % confidence limits (95 % CL) of the mean of *n* replicate analyses by a single laboratory, the mean level, *m,* is calculated and the appropriate value of S_W is found from Fig. 2. The 95 % CL of the mean is $m \pm (1.95/\sqrt{n}) \times S_W$.

14.4 The 95 % CL of the estimate that the mean of *n* replicate analysis by one laboratory represents the mean of a similar number of analyses by a large number of laboratories is calculated from the standard error, S_T, which in turn, can be calculated from the data in Fig. 2 using the equation:

$$S_T = \sqrt{(S_B{}^2 + S_W{}^2)/n)}$$

If the experience of the laboratories with the method is appropriate and the NO_2 levels is similar to the field tests, the relation can be used:

$$S_T = 14.5 \text{ ppm } NO_2$$

Finally, the 95 % CL of a single laboratory's data with respect to a mean value determined by many laboratories is $\pm 1.96 \times S_T$.

14.5 In the same collaborative study, it was shown that the test method had no significant bias over the range studied. Thus, it can be stated that the accuracy of a single laboratory's analysis is $\pm 1.96 \times S_T$ from 20 to 2000 ppm of NO_2.

14.6 Analysis of standard nitrate solutions following each test indicated that variations introduced by the color formation and measurement portion of the test method accounts for roughly 2/3 of the total observed variability. In addition, predominantly negative biases were associated with analysis of these standards ranging from −3.6 to −7.7 % of the true value. Although not statistically significant, the pilot plant spiking tests also produced a negative bias of −3.25 %, suggesting that

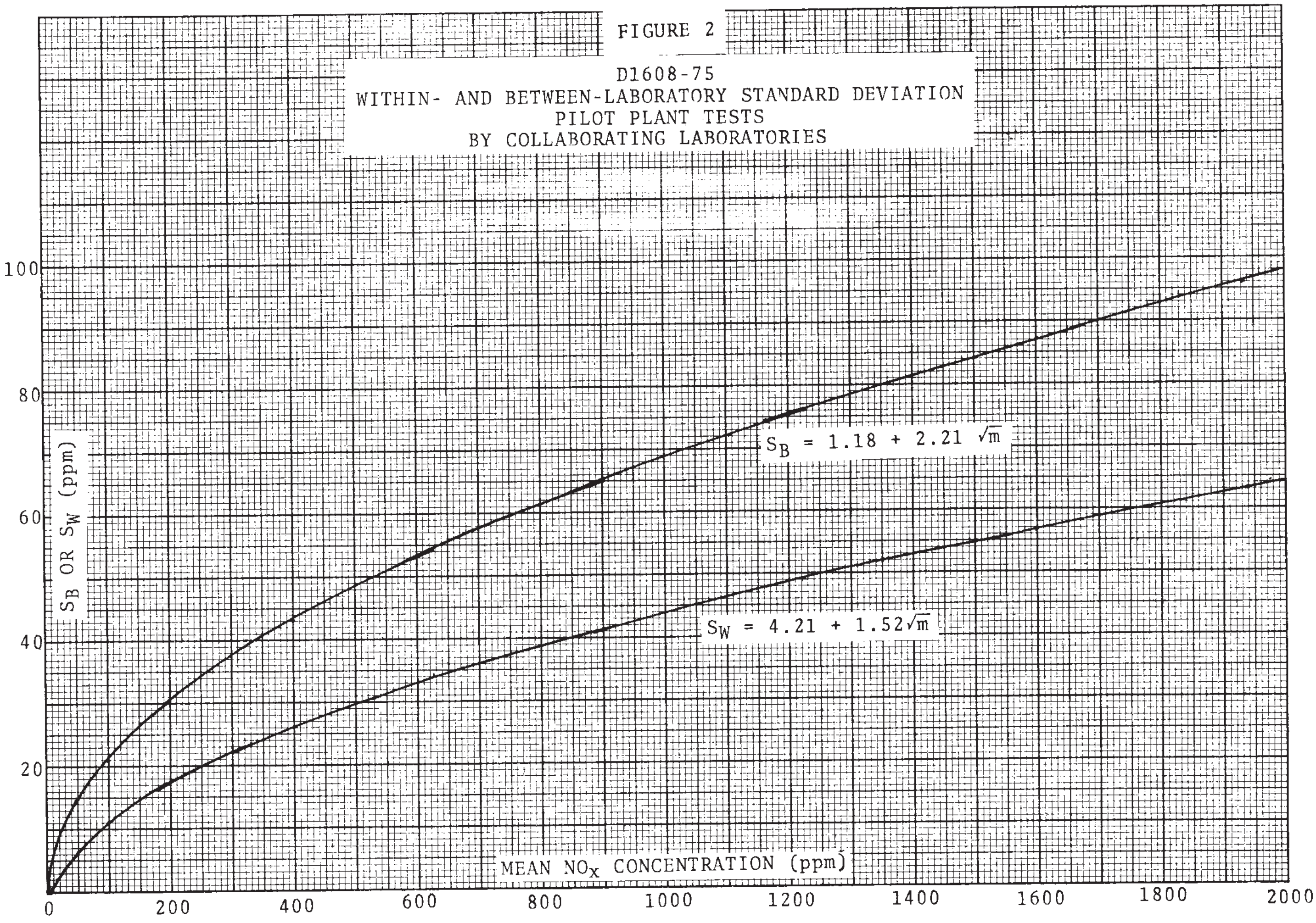

FIG. 2 Within- and Between-Laboratory Standard Deviation Pilot Plant Tests by Collaborating Laboratories

there was an influence of a negative bias in the chemical portion of the method on the overall results.

14.7 Additional collaborative data concerning precision and accuracy of the phenoldisulfonic acid test method, as applied in analysis of effluents by the EPA Method 7, are given in Refs **(3)** and **(4).** Generally, these studies showed significantly better precision than indicated by Fig. 2 at NO_x concentrations lower than 400 ppm. The difference in findings may well represent differences in experience of the collaborating laboratories since the field data of the ASTM teams, which were obtained after the initial pilot plant work, was within the range of significance of the EPA correlations.

15. Keywords

15.1 combustion sources; emissions; EPA Method 7; Method 7; oxides of nitrogen; phenol-disulfonic acid procedure; stack emissions; total oxides of nitrogen

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