



Designation: D 1510 – 02a

Standard Test Method for Carbon Black—Iodine Adsorption Number¹

This standard is issued under the fixed designation D 1510; 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 covers the determination of the iodine adsorption number of carbon black.

1.1.1 Procedure A is the original procedure for this determination.

1.1.2 Procedure B specifies an increased sample mass of carbon black and volume of iodine solution (the ratio is the same as in Procedure A) which results in a simplified formula for the calculation of the iodine number.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 The iodine adsorption number of carbon black has been shown to decrease with sample aging. Standard values for current SRB's may be obtained from the Subcommittee Chairman of D24.61.

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.*

2. Referenced Documents

2.1 ASTM Standards:

D 1799 Practice for Carbon Black—Sampling Packaged Shipments²

D 1900 Practice for Carbon Black—Sampling Bulk Shipments²

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²

D 4821 Guide for Carbon Black—Validation of Test Method Precision and Bias²

3. Summary of Test Method

3.1 A weighed sample of carbon black is treated with a portion of standard iodine solution and the mixture shaken and centrifuged. The excess iodine is then titrated with standard sodium thiosulfate solution, and the adsorbed iodine is ex-

pressed as a fraction of the total mass of black.

4. Significance and Use

4.1 The iodine adsorption number is useful in characterizing carbon blacks. It is related to the surface area of carbon blacks and is generally in agreement with nitrogen surface area. The presence of volatiles, surface porosity, or extractables will influence the iodine adsorption number. Aging of carbon black can also influence the iodine number.

5. Apparatus

5.1 *Vials*, glass, optically clear type, with polyethylene stoppers, 45 cm³.

5.2 *Gravity Convection Drying Oven*, capable of maintaining 125 \pm 5°C.

5.3 *Buret*, either of the following may be used:

5.3.1 *Digital Buret*, 25-cm³ capacity, with 0.01-cm³ increment counter and zero reset control, or

5.3.2 *Buret*, glass 25-cm³, Class A, side-arm filling, graduated in 0.05 cm³ and with automatic zero.

5.4 *Repetitive Dispenser*, 25-cm³ capacity, \pm 0.1% reproducibility and calibrated to within \pm 0.03-cm³ accuracy.

5.5 *Balance*, analytical, with 0.1-mg sensitivity.

5.6 *Centrifuge*, with minimum speed of 105 rad/s (1000 r/min).

5.7 *Volumetric Flask*, 2000-cm³ with standard taper stopper.

5.8 *Funnel*, large diameter, with standard taper joint to fit the 2000-cm³ flask.

5.9 *Glass Bottle*, amber, 2000-cm³, with standard taper stopper.

5.10 *Glass Jug*, approximate capacity 20-dm³.

5.11 *Stirrer*, approximately 300 by 300 mm for mixing.

5.12 *Stirrer*, approximately 100 by 100 mm for titrating.

5.13 *Desiccator*.

5.14 *Miscellaneous Class A Glassware*, and equipment necessary to carry out the test as written.

5.15 *Mechanical Shaker*, with 1 in. stroke length and capable of 240 strokes/min.

6. Reagents

6.1 *Purity of Reagents*—Unless otherwise stated, all chemicals shall be of reagent grade.

6.2 *Iodine*.

6.3 *Potassium Iodide*.

¹ This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.21 on Adsorptive Properties of Carbon Black.

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² *Annual Book of ASTM Standards*, Vol 09.01.

- 6.4 *Potassium Dichromate.*
- 6.5 *Potassium Iodate.*
- 6.6 *Sodium Thiosulfate.*
- 6.7 *n-Amyl Alcohol.*
- 6.8 *Sulfuric Acid.*
- 6.9 *Soluble Starch.*
- 6.10 *Salicylic Acid.*
- 6.11 *Arsenious Oxide, 1.9995 milliequivalent vials.*³
- 6.12 *Water.*

7. Sampling

7.1 Samples shall be taken in accordance with Practices D 1799 and D 1900.

8. Solutions

8.1 Pre-mixed 0.0473 *N* iodine solution and 0.0394 *N* sodium thiosulfate may be purchased from commercial sources. It is recommended that the normality of pre-mixed solutions be verified before use.

8.2 The solutions may be mixed in the laboratory. For instructions on mixing these solutions, refer to Annex A1.

9. Standardization of Solutions

9.1 *Sodium Thiosulfate, 0.0394 N (±0.00008):*

9.1.1 Use potassium dichromate solution (as follows: see A1.1).

9.1.2 Measure approximately 20 cm³ of 10 % potassium iodide solution into a small graduated cylinder and transfer to a 250-cm³ iodine flask with a ground glass stopper.

9.1.3 Measure approximately 20 cm³ of 10 % sulfuric acid solution (see A1.6) into a small graduated cylinder and add to the KI solution in the iodine flask. The mixture should remain colorless.

NOTE 1—If a yellow color should develop, discard this KI solution.

9.1.4 Using a 20-cm³ pipet, transfer 20 cm³ of standard 0.0394 *N* potassium dichromate solution into the 250-cm³ iodine flask, replace stopper, swirl, and place in the dark for 15 min.

9.1.5 Titrate the contents of the iodine flask against the new sodium thiosulfate solution following 9.1.6 or 9.1.7.

9.1.6 *Digital Buret:*

9.1.6.1 Switch the digital buret to fill mode, fill the reservoir with unstandardized sodium thiosulfate solution, and flush the inlet and delivery tubes.

9.1.6.2 Change to the titrate mode and zero the counter.

9.1.6.3 Add sodium thiosulfate until the contents of the iodine flask is a pale yellowish-green. Wash the buret tip and the walls of the flask with water.

9.1.6.4 Add 5 drops of starch solution to the flask.

9.1.6.5 Continue adding sodium thiosulfate dropwise until the blue or blue-violet color almost disappears.

9.1.6.6 Wash the tip and walls of the flask with water, then advance the counter in 0.01-cm³ increments. Continue this

sequence until the endpoint of the pale sea-green color of chromic sulfate is reached.

9.1.6.7 Record the titration value and repeat from 9.1.4 for a duplicate determination.

9.1.6.8 Calculate the normality of the sodium thiosulfate solutions as follows:

$$N = \frac{20(0.0394)}{T} \quad (1)$$

where:

N = normality, and

T = titration volume, cm³.

9.1.6.9 If *N* is not equal to 0.0394, adjust the solution in the following manner: if the solution is too strong, add water (2.5 cm³/dm³ solution for each 0.0001 *N* over 0.0394); if the solution is too weak, add solid sodium thiosulfate (0.025 g/dm³ for each 0.0001 *N* under 0.0394).

9.1.7 *Glass Buret:*

9.1.7.1 Using a conventional glass buret, fill the buret with unstandardized sodium-thiosulfate solution and flush 2 to 3 cm³ through the tip.

9.1.7.2 Adjust to the mark and titrate to a pale yellowish-green endpoint.

NOTE 2—To achieve maximum performance from a glass buret, it is necessary to use a small magnifier and to read to the nearest 0.025 cm³.

9.1.7.3 Wash the buret tip and the walls of the flask with water.

9.1.7.4 Add 5 drops of starch solution to the iodine flask.

9.1.7.5 Continue adding sodium thiosulfate dropwise until the blue color is just discharged. There will be a residual sea-green color of chromic sulfate.

9.1.7.6 Record the titration value to the nearest 0.025 cm³ and repeat from 9.1.7.1 for a duplicate determination.

9.1.7.7 Calculate the normality of the sodium thiosulfate solution as in 9.1.6.8.

9.2 *Sodium Thiosulfate, 0.0394 N (±0.00008):*

9.2.1 Use potassium iodate/iodide solution as follows:

9.2.2 Pipet exactly 20 cm³ of 0.0394 *N* potassium iodate/iodide solution into a 250-cm³ iodine flask.

9.2.3 Measure approximately 5 cm³ of 10 % sulfuric acid into a small graduated cylinder and add to the iodate/iodide solution.

9.2.4 Cap immediately and mix thoroughly. Continue as in 9.1.6-9.1.6.8 or as in 9.1.7-9.1.7.7. (However, note that the endpoint is colorless rather than sea-green in color.)

9.3 *Iodine Solution 0.0473 N (±0.00003)*—This solution may be standardized against standard arsenious oxide (As₂O₃) (see 9.3.1) or against the secondary standard sodium-thiosulfate solution (see A1.3) standardized as in 9.1 or 9.2.

9.3.1 Use arsenious oxide solution as follows:

9.3.1.1 Remove the cap and plug from the arsenious-oxide vial and pour the contents into a titrating vessel. Rinse the vial thoroughly and combine the rinse water with contents in the titration vessel.

9.3.1.2 Add approximately 5 g of sodium bicarbonate to the titration vessel.

9.3.1.3 Place the titration vessel on an ultrasonic stirrer for 5 min or until all solids have dissolved.

³ The sole source of supply of the apparatus known to the committee at this time is Chemical Services Laboratories, P.O. Box 281, Largo, FL 33779. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee ¹, which you may attend.

9.3.1.4 Add 5 cm³ of starch solution.

9.3.1.5 Titrate with iodine solution to a pale blue endpoint that remains blue for 30 s. Record the volume of iodine solution to the nearest 0.01 cm³.

9.3.1.6 Calculate the normality of the iodine solution as follows:

$$N = (S)/(T) \quad (2)$$

where:

N = normality,

S = milliequivalents As I_2 on the vial label, and

T = cm³ of iodine solution

9.3.2 Use sodium thiosulfate solution as follows:

9.3.2.1 Pipet exactly 20 cm³ of iodine solution into a 250-cm³ iodine flask and cap. Continue as in 9.1.6-9.1.6.7 or as in 9.1.7-9.1.7.6.

9.3.2.2 Calculate the normality of the iodine solution as follows:

$$N = \frac{(0.0394)T}{20} \quad (3)$$

where:

N = normality, and

T = cm³ of 0.0394 N sodium thiosulfate solution.

9.3.2.3 If N is not equal to 0.0473 N , adjust solution in the following manner: if the solution is too concentrated, add water (2.1 cm³/dm³ for each 0.0001 N over 0.0473); if the solution is too diluted, add iodine (12.7 mg/dm³ for each 0.0001 N under 0.0473). (This iodine may be more conveniently dispensed from a concentrated solution.)

10. Procedure A

10.1 Dry an adequate sample of carbon black for 1 h, in a gravity-convection oven set at 125°C, in an open container of suitable dimensions, so that the depth of the black is no more than 10 mm. Cool to room temperature in a desiccator before use.

10.2 Weigh a mass of the dried sample into a glass vial as shown by the following table. All masses must be to the nearest 0.0001 g.

Iodine Number	Sample Mass (g)	Ratio I_2 : Sample Mass
0–130.9	0.5000	50:1
131.0–280.9	0.2500	100:1
281.0–520.9	0.1250	200:1
521.0 and above	0.0625	400:1

10.2.1 Use the sample mass determined by the expected iodine number. If the result falls either above or below the range shown for that sample size, retest using the sample mass specified in 10.2 for the range into which it has fallen.

NOTE 3—Unagitated, unpelleted carbon black may be densified, if desired, before drying, prior to weighing.

10.2.2 The sample mass table given in 10.2 pertains to the 25 cm³ iodine solution as given in 10.3. Different volumes of iodine solution and of sample masses are permissible only if the iodine solution to sample mass ratio is kept the same as that given by the table in 10.2. The sample mass must be kept to 1.0000 g maximum. Should the sample mass and corresponding volume of iodine solution be increased, then a glass vial with proper capacity should be used in order to preserve the

efficiency of the shaking.

10.3 Pipet (or dispense from a calibrated repetitive dispenser) 25 cm³ of 0.0473 N I_2 solution into the glass vial containing the sample and cap immediately.

10.4 Secure the vial in the mechanical shaker and shake for 1 min at 240 strokes/min.

10.5 Centrifuge immediately for 1 min for pelleted black and 3 min for loose black.

10.6 Decant immediately. If more than one sample is being analyzed, the solution should be decanted into small flasks or clean, dry vials and capped immediately.

10.7 Pipet 20 cm³ of solution into a 250-cm³ Erlenmeyer flask and titrate with standard 0.0394 N sodium thiosulfate solution using either the digital or glass buret as follows:

10.7.1 *Using a Digital Buret:*

10.7.1.1 Switch to the fill mode, fill the buret reservoir with solution, and flush the inlet and delivery tubes.

10.7.1.2 Change to the titrate mode, zero the counter, and clean the tip with tissue.

10.7.1.3 Add sodium thiosulfate until the solution is pale yellow. Wash the buret tip and walls of the flask with water.

10.7.1.4 Add 5 drops of starch solution.

10.7.1.5 Continue adding sodium thiosulfate dropwise until the blue or blue-violet color almost disappears.

10.7.1.6 Wash the tip and walls of the flask with water and then advance the counter in 0.01-cm³ increments. Continue this sequence until the endpoint is reached as indicated by a colorless solution.

10.7.1.7 Record the buret reading to the nearest 0.01 cm³.

10.7.1.8 Make a blank iodine determination by pipeting 20 cm³ or dispensing 25 cm³ of 0.0473 N iodine solution into a 125-cm³ Erlenmeyer flask and titrating with 0.0394 N sodium thiosulfate as in 10.7.1 or 10.7.2.

(1) A 25-cm³ blank must be multiplied by 0.8 for use in the formula of 11.1.

10.7.1.9 Make a duplicate blank determination and use the average of the two in the calculations.

NOTE 4—A duplicate blank determination need be run only once each day, unless new solutions are introduced during the day.

10.7.1.10 If both solutions are within acceptable limits, the blank will measure 24.00 ± 0.05 cm³. If not, the normalities of one or both solutions should be rechecked.

10.7.2 *Using a Conventional Glass Buret:*

10.7.2.1 Clean the tip with a tissue. Add sodium thiosulfate until the solution is pale yellow. Wash the buret tip and walls of the flask with water.

10.7.2.2 Add 5 drops of starch solution.

10.7.2.3 Continue adding sodium thiosulfate dropwise until the endpoint is reached as indicated by a colorless solution.

10.7.2.4 Record the titration volume to the nearest 0.25 cm³.

10.7.2.5 Make blank determinations as in 10.7.1.8, 10.7.1.9, and 10.7.1.10.

11. Calculation—Procedure A

11.1 Calculate the iodine adsorption number to the nearest 0.1 g/kg as follows:

$$I = [(B - S)/B] \times (V/W) \times N \times 126.91 \quad (4)$$

where:

- I = iodine adsorption number, grams of iodine/kilograms of carbon black expressed as g/kg,
- B = cm^3 of sodium thiosulfate required for the blank,
- S = cm^3 of sodium thiosulfate required for the sample,
- V = calibrated volume of the 25-cm^3 iodine pipet or dispenser,
- W = grams of carbon black sample, and
- N = normality of the iodine solution, meq/cm^3 , and 126.91 = equivalent mass of iodine mg/meq .

Using the units shown above results in units of milligrams of iodine/grams of carbon black, which is equivalent to grams of iodine/kilograms of carbon black.

12. Procedure B

12.1 Dry an adequate sample of carbon black for 1 h, in a gravity-convection oven, set at 125°C , in an open container of suitable dimensions, so that the depth of the black is no more than 10 mm. Cool to room temperature in a desiccator before use.

12.2 Weigh a mass of the dried sample into a glass vial as shown by the following table. All weights must be to the nearest 0.0001 g.

Iodine Number	Sample Mass
0–130.9	0.8000
131.0–280.9	0.4000
281.0–520.9	0.2000
521.0 and above	0.1000

12.2.1 Use the sample mass determined by the expected iodine number. If the result falls either above or below the range shown for that sample size, retest using the sample mass specified in 12.2 for the range into which it has fallen.

NOTE 5—Unagitated, unpelleted carbon black may be densified, if desired, before drying, prior to weighing.

12.3 Pipet (or dispense from a calibrated repetitive dispenser) 40 cm^3 of 0.0473 N I_2 solution into the glass vial containing the sample and cap immediately.

12.4 Secure the vial in the mechanical shaker and shake for 1 min at 240 strokes/min.

12.5 Centrifuge immediately for 1 min for pelleted black and 3 min for loose black.

12.6 Decant immediately. If more than one sample is being analyzed, the solution should be decanted into small flasks or clean, dry vials and capped immediately.

12.7 Pipet 25 cm^3 of solution into a 250-cm^3 Erlenmeyer flask and titrate with standard 0.0394 N sodium thiosulfate solution as follows:

12.7.1 *Using a Digital Buret:*

12.7.1.1 Switch to the fill mode, fill the buret reservoir with solution, and flush the inlet and delivery tubes.

12.7.1.2 Change to the titrate mode, zero the counter, and clean the tip with tissue.

12.7.1.3 Add sodium thiosulfate until the solution is pale yellow. Wash the buret tip and walls of the flask with water.

12.7.1.4 Add 5 drops of starch solution.

12.7.1.5 Continue adding sodium thiosulfate dropwise until the blue or blue-violet color almost disappears.

12.7.1.6 Wash the tip and walls of the flask with water and then advance the counter in 0.01-cm^3 increments. Continue this

sequence until the endpoint is reached as indicated by a colorless solution.

12.7.1.7 Record the buret reading to the nearest 0.01 cm^3 .

12.7.1.8 Make a blank iodine determination by pipeting 25 cm^3 of 0.0473 N iodine solution into a 125-cm^3 Erlenmeyer flask and titrating with 0.0394 N sodium thiosulfate as in 10.7.1 or 10.7.2.

12.7.1.9 Make a duplicate blank determination and use the average of the two in the calculations.

NOTE 6—A duplicate blank determination need be run only once each day, unless new solutions are introduced during the day.

12.7.1.10 If both solutions are within acceptable limits, the blank will measure $30.00 \pm 0.05\text{ cm}^3$. If not, the normalities of one or both solutions should be rechecked.

12.7.2 *Using a Conventional Glass Buret:*

12.7.2.1 Clean the tip with a tissue. Add sodium thiosulfate until the solution is pale yellow. Wash the buret tip and walls of the flask with water.

12.7.2.2 Add 5 drops of starch solution.

12.7.2.3 Continue adding sodium thiosulfate dropwise until the endpoint is reached as indicated by a colorless solution.

12.7.2.4 Record the titration volume to the nearest 0.25 cm^3 .

12.7.2.5 Make blank determinations as in 10.7.1.8, 10.7.1.9, and 10.7.1.10.

13. Calculation—Procedure B

13.1 Calculate the iodine adsorption number to the nearest $0.1\text{ g}/\text{kg}$. When the correct normalities and sample sizes are used, the formula simplifies to the following:

$$0.8000\text{ g sample: } I = (B - S) \times 10 \quad (5)$$

$$0.4000\text{ g sample: } I = (B - S) \times 20$$

$$0.2000\text{ g sample: } I = (B - S) \times 40$$

$$0.1000\text{ g sample: } I = (B - S) \times 80$$

where:

I = iodine adsorption number, g/kg ,

B = cm^3 of sodium thiosulfate required for the blank, and

S = cm^3 of sodium thiosulfate required for the sample.

14. Report

14.1 Report the following information:

14.1.1 Proper identification of the sample,

14.1.2 Sample mass, and

14.1.3 Result obtained from an individual determination, reported to the nearest $0.1\text{ g}/\text{kg}$.

15. ASTM D24 Standard Reference Blacks

15.1 This procedure may be checked for proper execution by testing the ASTM D24 Standard Reference Blacks. The standard value and range for each, when tested by this test method, is given in Practice D 4821. However, due to the iodine adsorption number of carbon blacks decreasing with age (including SRB's), the preferred technique for monitoring proper test execution is to utilize a statistical control chart (x-chart) to continuously monitor testing as described in D 4821.

15.2 If the values for the Standard Reference Blacks do not fall within the accepted ranges, then new solutions should be

prepared and retests made on the reference blacks.

16. Precision and Bias

16.1 These precision statements have been prepared in accordance with Practice D 4483. Refer to this practice for terminology and other statistical details.

16.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials used in the particular interlaboratory program described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method. Any appropriate value may be used from Table 1.

16.3 A type 1 interlaboratory precision program was conducted. Both repeatability and reproducibility represent short term (daily) testing conditions. The testing was performed using two operators in each laboratory performing the test once on each material on each of two days (total of four tests). The number of participating laboratories is listed in Table 1.

16.4 The results of the precision calculations for this test are given in Table 1. The materials are arranged in ascending “mean level” order.

16.5 *Repeatability*—The **pooled relative** repeatability, (*r*), of this test method has been established as 2.49 %. Any other value in Table 1 may be used as an estimate of repeatability, as appropriate. The difference between two single test results (or determinations) found on identical test material under the repeatability conditions prescribed for this test will exceed the repeatability on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results that differ by more than the appropriate value from Table 1 must be suspected of being from different populations and some appropriate action taken.

NOTE 7—Appropriate action may be an investigation of the test method procedure or apparatus for faulty operation or the declaration of a significant difference in the two materials, samples, and so forth, which generated the two test results.

16.6 *Reproducibility*—The **pooled relative** reproducibility, (*R*), of this test has been established as 5.21 %. Any other value in Table 1 may be used as an estimate of reproducibility, as appropriate. The difference between two single and independent test results found by two operators working under the prescribed reproducibility conditions in different laboratories on identical test material will exceed the reproducibility on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results produced in different laboratories that differ by more than the appropriate value from Table 1 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

16.7 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.

17. Keywords

17.1 carbon black; iodine adsorption number

TABLE 1 Precision Parameters for D 1510 Iodine Adsorption Number, (Type 1 Precision)

Units		g/kg				
Material	Number of laboratories	Mean Level	<i>Sr</i>	(<i>r</i>)	<i>SR</i>	(<i>R</i>)
SRB D6 (N762)	19	26.5	0.42	4.51	0.77	8.28
SRB F6 (N683)	19	33.1	0.48	4.10	0.67	5.75
SRB E6 (N660)	19	35.3	0.54	4.31	0.69	5.56
SRB C6 (N326)	18	82.4	0.36	1.24	0.97	3.34
SRB B6 (N220)	20	117.9	0.76	1.82	1.81	4.34
SRB A6 (N134)	19	137.2	1.00	2.06	2.19	4.52
Average		72.1				
Pooled Values			0.63	2.49	1.33	5.21

ANNEX

(Mandatory Information)

A1. SOLUTIONS

NOTE A1.1—The designated iodine solution strength for running the iodine test is 0.0473 *N*, which may be made up directly from the solid or from a stronger (10 X) solution. Both test methods are described.

A1.1 Iodine Solution, 0.4728 *N*:

A1.1.1 Weigh 1140 g of potassium iodide (KI) into a 1000-cm³ beaker.

A1.1.2 Add about 850 g of KI to a clean 2000-cm³ volumetric flask through a large diameter funnel (with standard-taper-joint).

A1.1.3 Add enough water to cover the KI. Swirl to dissolve, then allow to stand until the solution attains ambient temperature.

A1.1.4 Add about 50 g of the remaining KI to a 250-cm³ beaker and enough water to dissolve it.

A1.1.5 Add 200 to 300 cm³ of deionized water to the balance of the KI in the 1000-cm³ beaker.

A1.1.6 Weigh 120.0000 g of iodine (I₂) into a tall-form weighing bottle fitted with ground glass stopper.

A1.1.6.1 Use only a porcelain spoon to transfer iodine crystals. Place the cap on the weighing bottle when making weighings. Good results can be obtained by cleaning and drying the weighing bottle and allowing it to stand in the open for 1 h before using. This allows the moisture adsorbed on the surface to come to equilibrium.

A1.1.7 Transfer the iodine to a funnel and wash through with the remaining KI solution into a 1000-cm³ beaker.

A1.1.8 Wash out the weighing bottle into a 2000-cm³ volumetric flask using the KI solution in the 250-cm³ beaker. Continue washing until no color remains.

A1.1.9 Place the flask on a magnetic stirrer and stir at medium speed for 30 to 60 min.

A1.1.10 Adjust the volume to the mark, invert the flask once or twice, and then replace on the stirrer and continue stirring at medium speed for at least 2 h.

NOTE A1.2—Allow for volume of spin-bar.

A1.1.11 Transfer the solution to the amber bottle and let stand overnight. Shake briefly and stir for 5 min before removing any solution.

NOTE A1.3—The procedure described in A1.1 produces 2000 cm³ of stock solution at a normality of 0.4728. Exact volumetric dilution of 10:1 will result in the production of 20 dm³ of working solution at 0.0473 *N*. Standardization will always be necessary and may be done with arsenious-oxide or a sodium-thiosulfate secondary standard. Both will be described.

A1.2 Iodine Solution, 0.0473 *N*:

A1.2.1 Apparatus:

A1.2.1.1 Beaker, 1-dm³(1-L),

A1.2.1.2 Glass Jug, 20-dm³ (20-L), with mark at 16-dm³ (16-L),

A1.2.1.3 Beaker, 250-cm³ (250-mL),

A1.2.1.4 Weighing Bottle, tall-form with ground glass stopper,

A1.2.1.5 Funnel,

A1.2.1.6 Magnetic Stirrer, and

A1.2.1.7 Stir-Bar.

A1.2.2 Reagents:

A1.2.2.1 Potassium Iodide (KI), reagent grade,

A1.2.2.2 Iodine (I₂) reagent grade, and

A1.2.2.3 Water.

A1.2.3 Procedure:

A1.2.3.1 Weigh 912 g of KI into the 1-dm³ (1-L) beaker.

A1.2.3.2 Prepare a concentrated KI solution in the 20-dm³ (20-L) jug.

A1.2.3.3 Using a large diameter funnel, take about 700 g of KI from the 1-dm³ (1-L) beaker and add it to a clean 20-dm³ (20-L) glass jug.

A1.2.3.4 Add enough water to cover the KI. Swirl to dissolve and let stand until the solution reaches room temperature.

A1.2.3.5 Prepare a KI solution in the 250-cm³ (250-mL) beaker (later used to wash the weighing dish for iodine).

A1.2.3.6 Take about 50 g from the KI remaining in the 1-dm³ (1-L) beaker, add it to a 250-cm³ (250-mL) beaker, and add enough water to dissolve it.

A1.2.3.7 Prepare a KI solution in the 1-dm³ (1-L) beaker (later used to wash the funnel used for iodine).

A1.2.3.8 The 1-dm³ (1-L) beaker, initially used to weigh 912 g of KI, now contains roughly 162 g of KI. To this quantity, add roughly 200 to 300 cm³ (200 to 300 mL) of water.

A1.2.3.9 Weigh 96.000 g of iodine into a tall-form weighing bottle with ground glass stopper.

A1.2.3.10 Add the iodine to the 20-dm³ (20-L) jug using a funnel; wash through with the KI solution contained in the 1-dm³ (1-L) beaker.

A1.2.3.11 Wash out the weighing bottle with the KI solution contained in the 250-cm³ (250-mL) beaker.

A1.2.3.12 Add water to the 20-dm³ (20-L) jug in increments of 2–3 dm³ (2–3 L), mixing vigorously between the addition until the final volume is almost reached (about 15 dm³ (15 L)). Let stand for 30 to 60 min.

A1.2.3.13 Adjust the volume to the mark 16-dm³(16-L) with water. Add a stir bar, place the jug on the magnetic stirrer, and stir overnight.

NOTE A1.4—It is important that the I₂ be entirely dissolved before making up to the final volume. If this is done too early, the remaining undissolved I₂ will dissolve extremely slowly.

A1.3 Sodium Thiosulfate, 0.0394 *N*:

A1.3.1 Add about 4 dm³ of water to a 20-dm³ glass jug (calibrated for 16 dm³) that has been placed on a magnetic stirrer.

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NOTE A1.5—Calibration of the 20-dm³ jug, either by weight or by measuring 2-dm³ increments from a graduated cylinder, should be done as carefully as possible in order to minimize the number of standardization titrations.

A1.3.2 Weigh 156.5 g of sodium thiosulfate (Na₂S₂O₃·5H₂O) and wash through the filling funnel into the jug.

A1.3.3 Add 80 cm³ of n-amyl alcohol to the jug.

A1.3.4 Stir until the crystals are dissolved.

A1.3.5 Add water to the 16-dm³ mark.

A1.3.6 Place the jug on the magnetic stirrer and stir for 1 to 2 h.

A1.3.7 Age for 1 to 2 days, then stir for 0.5 h before using.

A1.4 *Potassium Dichromate, 0.0394 N (Primary Standard):*

NOTE A1.6—Potassium dichromate solution is a *primary* standard in this test method and as such it is mandatory that all precautions be taken to ensure its accuracy.

A1.4.1 Dry an adequate quantity of potassium dichromate for 1 h in an oven set at 125°C. Allow to cool to ambient temperature in a desiccator.

A1.4.2 Weigh 0.9660 g of the dried potassium dichromate (K₂Cr₂O₇), then using a standard taper-joint funnel, add this to a 500-cm³ volumetric flask.

A1.4.3 Add about 200 cm³ of water to the volumetric flask.

A1.4.4 Place on the magnetic stirrer and stir until all crystals are dissolved.

A1.4.5 Add water to the mark and then add additional water to compensate for the volume of the magnetic spin-bar.

A1.4.6 Stopper the flask and age for 30 min prior to using.

A1.5 *Potassium Iodine (KI), 10 % Solution:*

A1.5.1 Weigh approximately 10 g of potassium iodide into a small stoppered flask or bottle.

A1.5.2 Fill a graduated cylinder with 90 cm³ of water and transfer to the small stoppered flask or bottle.

A1.5.3 Mix thoroughly until dissolved.

NOTE A1.7—It is possible for the KI solution to be oxidized and release iodine. Discard the KI solution if any yellow color is observed.

A1.6 *Sulfuric Acid, 10 % Solution:*

A1.6.1 Fill a graduated cylinder with 90 cm³ of deionized water and transfer to a 250-cm³ Erlenmeyer flask.

A1.6.2 Measure about 7 cm³ of concentrated sulfuric acid into a small graduated cylinder or beaker.

A1.6.3 Very carefully pour the acid into the 250-cm³ flask of water and swirl gently to mix. Rinse the graduate with diluted acid from the 250-cm³ flask.

A1.6.4 Allow the acid to cool to ambient temperature before using.

A1.7 *Soluble Starch, 1 % Solution:*

A1.7.1 Into a 100-cm³ beaker weigh about 1 g of soluble starch and 0.002 g of salicylic acid.

A1.7.2 Add enough water to make a thin paste while stirring with a stirring rod.

A1.7.3 Add about 100 cm³ of water to a 250-cm³ beaker and bring to a boil on a hotplate.

A1.7.4 Add the starch paste to the water, stir, and continue to boil for 2 to 3 min.

A1.7.5 Allow the starch solution to cool. Add 2 to 3 g KI and stir to dissolve.

A1.8 *Potassium Iodate/Iodide Solution, 0.0394 N (Primary Standard):*

A1.8.1 Dry an adequate quantity of analytical grade potassium iodate for 1 h in an oven set at a temperature of 125°C. Allow to cool to ambient temperature in a desiccator.

A1.8.2 Dissolve 45 g of potassium iodide in about 200 cm³ of water contained in a 1000-cm³ volumetric flask.

A1.8.3 Weigh 1.4054 g of the freshly dried potassium iodate and add to the iodide solution in the 1000-cm³ flask.

A1.8.4 Make up to the mark and cap the flask.

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