



RICCA CHEMICAL COMPANY

Arlington, TX 76012
www.riccachemical.com
An ISO 9001 Registered Company

Carbon Black Testing – Iodine Adsorption Number (ASTM D 1510)

Reagents Required

Iodine Solution, 0.0473 N..... Ricca Chemical Company Cat. No. 3990
Sodium Thiosulfate Solution, 0.0394 N..... Ricca Chemical Company Cat. No. 7930

Stability of Solutions

Our manufacturing specifications for both the Iodine and Sodium Thiosulfate normalities are significantly tighter than the ASTM requirements (Sodium Thiosulfate: 0.03940 ± 0.00008 N; Iodine: 0.04730 ± 0.00003 N). However, our stability studies on Iodine and Sodium Thiosulfate solutions indicate that nonlinear changes to the normalities of both solutions occur during storage even in closed containers. **These changes do not occur at the same rate for both products.** Once opened, both solutions change more rapidly and unpredictably, primarily due to air-oxidation and evaporation. Sodium Thiosulfate may also be affected by bacterial growth, although a preservative is added to minimize this effect. For this reason, ASTM D 1510 requires that the blank be checked daily.

Standardization

We standardize the Sodium Thiosulfate solution against Potassium Iodate that has been validated as a primary standard traceable to Potassium Dichromate, NIST Standard Reference Material 136. ASTM D 1510 also allows the direct use of primary standard Potassium Dichromate for standardization; either standard gives the same normality results to the required degree of precision.

We standardize the Iodine solution against 0.0394 Normal Sodium Thiosulfate as required by ASTM D 1510. Earlier editions of the ASTM test method allowed standardization of the Iodine solution against either Arsenic Trioxide primary standard or against the 0.0394 N Sodium Thiosulfate solution. Beginning with the 2003 edition of the standard (D 1510-03), only the standardization with Sodium Thiosulfate solution is allowed. Our experience shows that the normality obtained by standardization against Arsenic Trioxide does not agree to the required precision with the normality obtained by standardization against Sodium Thiosulfate, even when both are traceable to NIST Standard Reference Materials. Therefore, we do not recommend standardization against Arsenic Trioxide.

Matching Sets

Our policy at one time was to require customers to order matching sets of Iodine and Sodium Thiosulfate solutions for this test. The Iodine solution was standardized against the matching Sodium Thiosulfate solution. This was in an attempt to ensure that the blank titration would always be within acceptable limits. However, after many years of customer feedback, we have determined that requiring the customer to order matching sets does not accomplish the desired effect. The blank value obtained using Sodium Thiosulfate and Iodine solutions from a matching set does not appear to be any better or more consistent than the blank values obtained using unmatched lots. **We no longer require customers to order matching sets of these products.** However, matching sets are still available upon request. Please realize that matching set orders will normally require a longer lead time and still cannot absolutely guarantee that the blank value will be within specification without adjustment. The lot number of Sodium Thiosulfate used for standardization appears on the Certificate of Analysis for the Iodine solution; this allows customers requiring matching sets to confirm the match.

Even when matching sets are ordered, adjustments to normality may have to be made at time of use due to the inherent instability of both of these solutions. Please see "Restandardization and Adjustment of Solutions" below for details.



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Blank Determination

The blank titration is essentially a confirmation of the standardization of the Iodine solution with the Sodium Thiosulfate solution. These products are manufactured to have a blank value of $24.00 \pm 0.03 \text{ cm}^3$ Sodium Thiosulfate solution for 20.00 mL Iodine solution (or $30.00 \pm 0.03 \text{ cm}^3$ Sodium Thiosulfate solution for 25.00 mL Iodine solution) **at the time of manufacturing**. However, due to the instability of these solutions, the blank should be checked daily and the appropriate adjustments made as required by ASTM D 1510.

Please note that it is possible for both solutions to be within ASTM normality specifications and still give an unacceptable blank value. (Values in bold print indicate unacceptable blanks.)

$\text{N Na}_2\text{S}_2\text{O}_3 \backslash \text{N I}_2$	0.04727	0.04728	0.04729	0.04730	0.04731	0.04732	0.04733
0.03932	24.04	24.05	24.05	24.06	24.06	24.07	24.07
0.03934	24.03	24.04	24.04	24.05	24.05	24.06	24.06
0.03936	24.02	24.02	24.03	24.03	24.04	24.04	24.05
0.03938	24.01	24.01	24.02	24.02	24.03	24.03	24.04
0.03940	23.99	24.00	24.01	24.01	24.02	24.02	24.03
0.03942	23.98	23.99	23.99	24.00	24.00	24.01	24.01
0.03944	23.97	23.98	23.98	23.99	23.99	24.00	24.00
0.03946	23.96	23.96	23.97	23.97	23.98	23.98	23.99
0.03948	23.95	23.95	23.96	23.96	23.97	23.97	23.98

mL $\text{Na}_2\text{S}_2\text{O}_3$ solution for 20.00 mL I_2 solution (blank)

Restandardization and Adjustment of Solutions

If the blank determination is not within ASTM specifications ($24.00 \pm 0.05 \text{ cm}^3$ for Procedure A or $30.00 \pm 0.05 \text{ cm}^3$ for Procedure B), the normality of the Iodine solution should be adjusted to bring the blank within acceptable limits.

If the Iodine normality is too high, add water (Ricca Cat. No. 9150) according to the equation:

$$\text{mL H}_2\text{O to add} = (21,000) (\text{volume of Iodine solution in liters}) (\text{Iodine Normality} - 0.0473)$$

If the Iodine normality is too low, add Iodine. Reagent grade Iodine crystals may be used for adjustment, or the Iodine may be more conveniently added as a 0.4728 N Iodine stock solution (Ricca Cat. No. 4023).

If Iodine crystals are used, adjust according to the equation:

$$\text{g Iodine to add} = (127) (\text{volume of Iodine solution in liters}) (0.0473 - \text{Iodine normality})$$

If Iodine stock solution is used, adjust according to the equation:

$$\text{mL Iodine stock to add} = (2350) (\text{volume of Iodine solution in liters}) (0.0473 - \text{Iodine normality})$$

The Sodium Thiosulfate solution is not expected to require adjustment during the labeled shelf life due to the wider acceptance limits for this solution as compared to the Iodine solution.

Reference Document

ASTM Standard D 1510-06a "Standard Test Method for Carbon Black – Iodine Adsorption Number," approved May 15, 2006.

*0.4728 N Iodine solution is a DEA (US Drug Enforcement Administration) regulated product and may not be available from all distributors. Documentation, including an intended use letter, is required for purchase.