Designation: D8451 – 22

Standard Test Method for Calculation of Unfixed Chrome Concentration in Wet Blue[1]

This standard is issued under the fixed designation D8451; 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 procedures to analyze and calculate unfixed chrome concentrations in Wet Blue.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.*

1.4 *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]

D6658 Test Method for Volatile Matter (Moisture) of Wet Blue by Oven Drying

D6659 Practice for Sampling and Preparation of Wet Blue and Wet White for Physical and Chemical Tests

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 *unfixed chrome, n*—chrome that is not chemically bound to hide substance and may be removed by water extraction.

4. Summary of Test Method

4.1 The sample is soaked, static, in deionized water for 20 to 24 h. After 20 to 24 h of static soaking, the mixture is thoroughly mixed, an aliquot filtered (and diluted) if necessary, is analyzed for unfixed chrome content. Atomic Absorption Spectrophotometers using the nitrous oxide/acetylene flame or the air/acetylene flame are acceptable to use for the analysis. Analysis by the ICP (Inductively Coupled Plasma) method is also acceptable.

5. Significance and Use

5.1 This test method measures the amount of unfixed chrome in Wet Blue. Results may vary according to the age of the Wet Blue.

6. Apparatus

6.1 *Leather Cutting Tool.*

6.2 *Styrofoam Cup,* or 250 mL beaker.

6.3 *Beaker,* 250 mL capacity or equivalent.

6.4 *Beaker,* 1000 mL capacity or equivalent.

6.5 *Volumetric Flask,* 200 mL capacity or equivalent.

6.6 *Volumetric Flask,* 1000 mL capacity or equivalent.

6.7 *Stirring Rod.*

6.8 *Magnetic Stirrer.*

6.9 *Atomic Absorption Spectrophotometer.*

6.10 *ICP.*

6.11 *Scale,* accurate to 0.0001 g.

6.12 *Filter Paper,* Whatman 1 or equivalent.

6.13 *Hot Plate,* to be placed inside exhaust or fume hood.

7. Reagents and Materials

7.1 *Purity of Reagents*—Analytical Reagent (AR) Grade shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytic Reagents of the American Chemical Society (ACS), where such specifications are available. 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 determination.

[1] This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.02 on Wet Blue.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

7.2 *Commercial Reagents*—The use of commercially available pre-standardized analytical reagents and solutions is appropriate, providing those reagents and solutions have been prepared in accordance with and conform to the previously mentioned specifications (see 7.1).

7.3 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean deionized water.

7.4 *Commercial Cr Standard,* 1000 ppm.

NOTE 1—Subsections 7.5 to 7.10 only apply when nitrous oxide/acetylene flame is used.

7.5 *Oxalic Acid Anhydrous,* $H_2C_2O_4$ [Formula weight = 90.03] or oxalic acid dihydrate, $H_2C_2O_4{\cdot}2H_2O$ [Formula weight = 126.07].

NOTE 2—For conversion purposes, 1 g of the anhydrous form is equivalent to 1.4 g of the dihydrate form.

7.6 *Potassium Dichromate ($K_2Cr_2O_7$)*—Used in standard Cr_2O_3 solution.

7.7 *Sulfuric acid (H_2SO_4),* 95 to 98 % w/w. Used to prepare standard Cr_2O_3 solution.

7.8 *Sodium Bisulfite ($NaHSO_3$).*

7.9 Using a commercially available 1000 ppm Cr standard solution (7.4), make a 10 ppm Cr solution which will be used to verify the concentration of the prepared Cr_2O_3 standard in 7.10. To convert from Cr to Cr_2O_3, multiply by a factor of 1.462 [A 10 ppm Cr solution equates to a 14.62 ppm Cr_2O_3 solution].

7.10 *Standard Cr_2O_3 solution,* (1000 ppm).

7.10.1 Dry approximately 5.0 g potassium dichromate ($K_2Cr_2O_7$) in 100 °C overnight.

7.10.2 Weight exactly 1.9356 g of oven dried $K_2Cr_2O_7$ and put in a 1000 mL beaker.

7.10.3 Add 500 mL water and stir until dissolved.

7.10.4 Carefully add 5 mL of concentrated sulfuric acid (H_2SO_4) while stirring.

NOTE 3—Perform in fume hood or exhaust hood, especially when adding acids.

7.10.5 Slowly add 5 g of sodium bisulfite ($NaHSO_3$) while stirring.

7.10.6 Place beaker with stirring rod in it on the hot plate.

7.10.7 Add 50 g oxalic acid anhydrous (or 70 g oxalic acid dihydrate) while stirring.

7.10.8 Bring solution to boiling (or near boiling) for 15 min.

7.10.9 Remove from the hot plate and add 300 mL of water while stirring. When cooled to room temperature, transfer solution to a 1000 mL volumetric flask, rinsing beaker completely and into the flask with water.

7.10.10 Bring to volume, 1000 mL, with water and mix well. Verify Cr_2O_3 content according to 7.11. Store according to good laboratory practices.

7.10.11 Pipet 1 mL of the 1000 ppm Cr_2O_3 solution into a volumetric flask and dilute to 100 mL with water. Invert and mix well. Use this as a 10 ppm Cr_2O_3 standard solution.

7.10.12 Pipet 5 mL of the 1000 ppm Cr_2O_3 solution into a volumetric flask and dilute to 200 mL with water. Invert and mix well. Use this as a 25 ppm Cr_2O_3 standard solution.

NOTE 4—Prepare the 10 ppm and 25 ppm Cr_2O_3 standard solutions fresh daily (on day of use).

7.11 *Calibration and Standardization*—Nitrous oxide/acetylene flame.

7.11.1 Optimize the Atomic Absorption Spectrophotometer according to the following instrumental parameters for the Nitrous oxide/acetylene flame:

Wavelength	429 nm
Slit width	0.7 nm

7.11.2 Using water to zero the AA, create a calibration curve with the 10 ppm and the 25 ppm Cr_2O_3 standards from 7.10.11 and 7.10.12, respectively.

7.11.3 Obtain a reading from the AA for the 10 ppm commercial Cr solution. This should be 14.6 ppm (±0.3 ppm) Cr_2O_3, if the standard solution prepared in 7.10 is accurate. If not, the solution will need to be discarded and remade. Once the 1000 ppm Cr_2O_3 standard solution is deemed acceptable, retain this standard stock solution for future use. Verify 7.10 every 6 months with commercial standard (7.9) to ensure standard stock solution is still viable. If the results after six months are not within ±0.3 ppm of the commercial standard, remake your standard stock solution according to 7.10.

NOTE 5—Subsections 7.12 to 7.15 only apply when Air/Acetylene flame or ICP is used.

7.12 *Diluted Nitric Acid*—The concentration of diluted nitric acid is specified as a ratio stating the number of the volume of nitric acid to be diluted with a given number of the volume of water. Example: Nitric acid (HNO_3), 1+1 means to take one volume of concentrated nitric acid and mix with one volume of water. It is the same as a 50 % dilution of the concentrated nitric acid. It doesn't matter what the volume is, as long as the ratio stays the same. **Warning—**When preparing diluted acid solutions always add the acid to the water!

7.12.1 Nitric acid (HNO_3), 1+1.

7.13 Prepare a 100 ppm Cr standard solution by pipetting 10 mL of the 1000 ppm Cr standard solution (7.4) into a volumetric flask and diluting to 100 mL with water. Invert and mix well.

7.13.1 Pipet 1 mL of the 100 ppm Cr solution into a volumetric flask. Add 5 mL 1+1 HNO_3 and dilute to 100 mL with water. Invert and mix well. Use this as a 1 ppm Cr standard solution.

7.13.2 Pipet 4 mL of the 100 ppm Cr solution into a volumetric flask. Add 5 mL 1+1 HNO_3 and dilute to 100 mL with water. Invert and mix well. Use this as a 4 ppm Cr standard solution.

7.13.3 Blank: Dilute 5 mL 1+1 HNO_3 to 100 mL with DI water.

NOTE 6—Prepare the 1 ppm, 4 ppm Cr standard solutions and blank fresh daily (on day of use).

7.14 *Calibration and Standardization*—Air/acetylene flame.

7.14.1 Optimize the Atomic Absorption Spectrophotometer according to the following instrumental parameters for the Air/acetylene flame:

Wavelength	357.9 nm
Slit width	0.7 nm
Type of flame	Reducing (fuel-rich, yellow)