National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2778

Mercury in Crude Oil

This Standard Reference Material (SRM) is intended for use in the evaluation of chemical methods of analysis for mercury in crude oil to support environmental and safety compliance measurements associated with oil refining. A unit of SRM 2778 consists of five amber glass ampoules, each containing approximately 10 mL of crude oil.

Certified Mass Fraction Value: A certified mass fraction value for mercury (Hg) is provided in Table 1 as the total mass fraction of Hg in an oil matrix. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [1]. The Hg mass fraction value in Table 1 is expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty of the certified value. The true value of the analyte is believed to lie within the interval $x \pm U_{95\%}(x)$ with 95 % confidence. To propagate this uncertainty, treat the certified value as a normally distributed, random variable with mean x and standard deviation $U_{95\%}(x)/2$ [2, 3]. Metrological traceability is to the SI-derived unit for mass fraction as realized through the purity determined for the primary chemical standards employed in the NIST mercury calibrator solutions of *SRM 3133* Mercury (Hg) Standard Solution [4].

Table 1. Certified Mass Fraction Value

Mercury (Hg)	38.98 μg/kg ± 1.10 μg/kg

Expiration of Certification: The certification of **SRM 2778** is valid, within the measurement uncertainty specified, until **01 April 2026**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to certification of this SRM was provided by S.J. Christopher and S.E. Long of the NIST Chemical Sciences Division.

Analytical measurements leading to certification were made by C.E. Bryan Sallee and S.E. Long of the NIST Chemical Sciences Division.

Statistical consultation and analyses were performed by B. Toman of the NIST Statistical Engineering Division.

Material acquisition and donation to NIST were coordinated through K. Nadkarni, Chairperson of ASTM International D02.SC 3 Committee on Elemental Analysis of Petroleum Products and Lubricants and B. MacDonald formerly of the NIST Office of Reference Materials.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Certificate Issue Date: 11 October 2019

Steven J. Choquette, Director
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INSTRUCTIONS FOR HANDLING, STORAGE AND USE

Handling: In addition to mercury, the material may contain other constituents of unknown toxicity. Therefore, caution and care should be exercised during SRM handling and use. Refer to Safety Data Sheet for further information.

Storage: Store the SRM in its original packaging, under normal laboratory conditions, and kept away from sunlight and intense sources of heat or radiation.

Use: Prior to removal of test portions for analysis, the ampoule should be mixed thoroughly by carefully inverting and rotating several times, avoiding excessive shaking. A minimum test portion mass of 30 mg should be used for analytical determinations.

MATERIAL SOURCE, PREPARATION AND ANALYSIS[1]

Material Source and Preparation: The source material for SRM 2778 was obtained from ASTM International D02.SC 3 committee on Elemental Analysis of Petroleum Products and Lubricants. Supplemental data related to the source material are listed in Appendix A. The sourced crude oil was siphoned from a 55-gallon drum, approximately 30 cm from the bottom to avoid any sediment present and passed through a drum head filter screen (75 μm) into a clean 55-gallon drum. The filtered material was blended using a paddle mixer, sampled approximately 30 cm from the bottom of the 55-gallon drum, and was dispensed into in 10 mL amber borosilicate glass ampoules.

Homogeneity Testing: Twenty-four ampoules of SRM 2778 were selected for homogeneity assessment of mercury mass fraction via combustion atomic absorption spectroscopy (CAAS), using a random stratified sampling scheme to draw samples from across the full production lot. Single factor analysis of variance statistics (*F*-test) revealed no evidence for mass fraction heterogeneity at the 0.01 significance level for sample test portion sizes of 30 mg.

Certified Mass Fraction Value for Mercury: The certified value for Hg is based on combining the results obtained from two NIST methods, the primary method of isotope dilution cold vapor inductively coupled plasma mass spectrometry (ID-CV-ICP-MS) [5], and CAAS, using a direct mercury analyzer. The certified mercury value and expanded uncertainty were determined using a Bayesian Markov Chain Monte Carlo method consistent with the ISO/JCGM Guide Supplement 1 [6], which accounted for small biases in CAAS-based mass fraction data relative to ID-CV-ICP-MS for paired samples.

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

[1] Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedures. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

REFERENCES

[1] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136, U.S. Government Printing Office: Washington, DC (2000); available at https://www.nist.gov/sites/default/files/documents/srm/SP260-136.PDF (accessed Oct 2019).

[2] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at available at: https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Oct 2019); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at https://www.nist.gov/pml/nist-technical-note-1297 (accessed Oct 2019).

[3] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at https://nvlpubs.nist.gov/nistpubs/Legacy/SP/nistspecialpublication811e2008.pdf (accessed Oct 2019).

[4] SRM 3133; *Mercury (Hg) Standard Solution (Lot No. 160921)*; National Institute of Standards and Technology; U.S. Department of Commerce: Gaithersburg, MD (22 December 2016).

[5] Christopher, S.J.; Long, S.E.; Rearick, M.S.; Fassett, J.D.; *Development of Isotope Dilution Cold Vapor Inductively Coupled Plasma Mass Spectrometry and Its Application to the Certification of Mercury in NIST Standard Reference Materials*; Anal. Chem., Vol. 73, pp. 2190–2199 (2001).

[6] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to Expression of Uncertainty in Measurement*; Propagation of Distributions Using a Monte Carlo Method; Joint Committee for Guides in Metrology; International Bureau of Weights and Measures (BIPM), Sèvres, France (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Oct 2019).

[7] ASTM D-2 Interlaboratory Crosscheck Program; *Final Report of Results for Crude Oil (CO1207)* (2012).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone 301-975-2200; fax 301-948-3730; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm

Appendix A

The source material for SRM 2778 was used in an ASTM interlaboratory test [7]. Some of the material properties and mass fraction data from the published ASTM report are included as information values in Table 2. NIST makes no claim about the accuracy or applicability of the data in Table 2 to SRM 2778, because the source material for SRM 2778 was further processed and blended at NIST to ensure a homogeneous distribution of mercury in the SRM. The information values in Table 2 cannot be used to establish metrological traceability. These data provide complementary information about the oil matrix and some of the ASTM methods used to measure the source material for SRM 2778, which may be useful to the SRM customer.

Table 2. Information Values from ASTM Interlaboratory Comparison Study

ASTM Method	Measurand	Value	Units
D287	API Gravity	25.09	°API
D6560	Asphaltenes	2.68	%
D4530	Carbon Residue	5.673	%
D1298	Density	0.90306	kg/L @ 15 °C
D5708 Method A	Iron	41.230	mg/kg
D5708 Method B	Iron	42.805	mg/kg
D5863 Method A	Iron	42.65	mg/kg
D5708 Method A	Nickel	51.96	mg/kg
D5708 Method B	Nickel	46.53	mg/kg
D5863 Method A	Nickel	47.04	mg/kg
D5863 Method B	Nickel	51.68	mg/kg
D4629	Nitrogen	4185.6	mg/kg
D5762	Nitrogen	4559.4	mg/kg
D5853	Pour Point	-47.1	°C
D3230	Salts	25.58	lb/1000bbl
D473	Sediment	0.0182	%
D4807	Sediment	0.074	%
D2622	Sulfur	1.093	%
D4294	Sulfur	1.117	%
D664	Total Acid Number	2.177	mg KOH/g
D5708 Method A	Vanadium	44.83	mg/kg
D5708 Method B	Vanadium	41.4	mg/kg
D5863 Method A	Vanadium	42	mg/kg
D5863 Method B	Vanadium	43.8	mg/kg
D323	Vapor Pressure	4.487	p.s.i.
D6377	Vapor Pressure	33.11	kPa
D5191	Vapor Pressure-TVP	4.142	p.s.i.
D445	Viscosity @ 25°C	33.93	mm^2/s
D445	Viscosity @ 40°C	18.742	mm^2/s
D4377	Water by Karl Fischer/Potentiometric Titration	0.62	%
D4006	Water Content	0.5122	volume %
D4928	Water Content	0.612	%