Government of Canada
Gouvernement du Canada

Canadian General Standards Board
Office des normes générales du Canada

CAN/CGSB-3.0 No. 60.32-2019

Supersedes CAN/CGSB-3.0 No. 60.32-2013

Methods of testing petroleum and associated products

Standard test method for determination of the corrosiveness to silver of gasoline, middle distillate fuels and oxygenated fuels using silver wool: Rapid ultrasonic method

Canadian General Standards Board CGSB

Standards Council of Canada
Conseil canadien des normes

Canada

Experience and excellence
Expérience et excellence
CGSB ONGC

Canadian General Standards Board statement

The CANADIAN GENERAL STANDARDS BOARD (CGSB), under whose auspices this standard has been developed, is a government directorate within Public Services and Procurement Canada. CGSB is engaged in the production of voluntary standards in a wide range of subject areas through the media of standards committees and the consensus process. The standards committees are composed of representatives of relevant interests including producers, consumers and other users, retailers, governments, educational institutions, technical, professional and trade societies, and research and testing organizations. Any given standard is developed on the consensus of views expressed by such representatives.

CGSB has been accredited by the Standards Council of Canada as a national Standards Development Organization. The standards that CGSB develops and offers as National Standards of Canada conform to the requirements and guidance established for this purpose by the Standards Council of Canada. In addition to standards it publishes as National Standards of Canada, CGSB produces standards to meet particular needs, in response to requests from a variety of sources in both the public and private sectors. Both CGSB standards and CGSB national standards are developed in conformance with the policies described in the CGSB Policy and Procedures Manual for the Development and Maintenance of Standards.

CGSB standards are subject to review and revision to ensure that they keep abreast of technological progress. CGSB will review and publish this standard on a schedule not to exceed five years from the date of publication. Suggestions for their improvement, which are always welcome, should be brought to the notice of the standards committees concerned. Changes to standards are issued either as separate amendment sheets, amended standards or in new editions of standards.

An up-to-date listing of CGSB standards, including details on latest issues and amendments, is found in the CGSB Catalogue at our Web site — http://www.tpsgc-pwgsc.gc.ca/ongc-cgsb/index-eng.html along with more information about CGSB products and services.

Although the intended primary application of this standard is stated in its scope, it is important to note that it remains the responsibility of the users of the standard to judge its suitability for their particular purpose.

The testing and evaluation of a product or service against this standard may require the use of materials and/or equipment that could be hazardous. This standard does not purport to address all the safety aspects associated with its use. Anyone using this standard has the responsibility to consult the appropriate authorities and to establish appropriate health and safety practices in conjunction with any applicable regulatory requirements prior to its use. CGSB neither assumes nor accepts any responsibility for any injury or damage that may occur during or as the result of tests, wherever performed.

Attention is drawn to the possibility that some of the elements of this standard may be the subject of patent rights. CGSB shall not be held responsible for identifying any or all such patent rights. Users of this standard are expressly advised that determination of the validity of any such patent rights is entirely their own responsibility.

In this standard, “shall” states a mandatory requirement, “should” expresses a recommendation and “may” is used to express an option or that which is permissible within the limits of this standard. Notes accompanying clauses do not include requirements or alternative requirements; the purpose of a note accompanying a clause is to separate from the text explanatory or informative material. Annexes are designated normative (mandatory) or informative (non-mandatory) to define their application.

For enforcement purposes, standards shall be considered published the final day of the month of their publication date.

Contact the Canadian General Standards Board

To obtain information on CGSB, its services and standards or to obtain CGSB publications, please contact:

web — http://www.tpsgc-pwgsc.gc.ca/ongc-cgsb/index-eng.html

e-mail — ncr.cgsb-ongc@tpsgc-pwgsc.gc.ca

telephone — 1-800-665-2472

mail — Canadian General Standards Board
Gatineau, Canada
K1A 1G6

Standards Council of Canada statement

A National Standard of Canada is a standard developed by a Standards Council of Canada (SCC) accredited Standards Development Organization, in compliance with requirements and guidance set out by SCC. More information on National Standards of Canada can be found at www.scc.ca.

SCC is a Crown corporation within the portfolio of Innovation, Science and Economic Development (ISED) Canada. With the goal of enhancing Canada’s economic competitiveness and social well-being, SCC leads and facilitates the development and use of national and international standards. SCC also coordinates Canadian participation in standards development, and identifies strategies to advance Canadian standardization efforts.

Accreditation services are provided by SCC to various customers, including product certifiers, testing laboratories, and standards development organizations. A list of SCC programs and accredited bodies is publicly available at www.scc.ca.

NATIONAL STANDARD OF CANADA

CAN/CGSB-3.0
No. 60.32-2019

Supersedes CAN/CGSB-3.0
No. 60.32-2013

Methods of testing petroleum and associated products

Standard test method for determination of the corrosiveness to silver of gasoline, middle distillate fuels and oxygenated fuels using silver wool: Rapid ultrasonic method

CETTE NORME NATIONALE DU CANADA EST DISPONIBLE EN VERSIONS FRANÇAISE ET ANGLAISE.

ICS 77.060

Published March 2019 by the
Canadian General Standards Board
Gatineau, Canada K1A 1G6

CANADIAN GENERAL STANDARDS BOARD

Committee on Petroleum Test Methods

(Voting membership at date of approval)

Chair

Wispinski, D.	VUV Analytics (User)

General interest category

Bailey, M.	AmSpec LLC
MacLean, G.	Intertek Caleb Brett
Marotta, L.	PerkinElmer
Ménard, L.	Advanced Engine Technology Ltd.
Pickard, A.L.	Consultant
Poitras, P.	Fuel+ Consulting
Whittington, J.	Anton Paar

Producer category

Boroniec, J.	Suncor Energy Products Partnership
Gupta, R.	Husky Energy
Harvie, S.	Consumers' Co-Operative Refineries Limited
Hiscock, R.	NARL Refining
Lee, I.	Archer Daniels Midland
Mitchell, K.	Canadian Fuels Association
O'Breham, G.	Shell Canada Ltd.
Porter, S.	Renewable Industries Canada
Rockwell, G.	Imperial Oil Ltd.
White, M.	Irving Oil Ltd.

Regulator category

Tardif, M.	Environment and Climate Change Canada

User category

Gaudet, N.	Department of National Defence
Hall, A.	Natural Resources Canada
Hanganu, A.	Bureau Veritas Company
Johnston, J	Innotech Alberta
Levesque, P.	SGS Canada Inc.
Pama, M.	Certispec Services Inc.

Secretary (non-voting)

Lozano, A.	Canadian General Standards Board

Acknowledgment is made for the translation of this National Standard of Canada by the Translation Bureau of Public Services and Procurement Canada.

This National Standard of Canada CAN/CGSB-3.0 No. 60.32-2019 supersedes the 2013 edition.

Changes since the previous edition

- Aligned differing vacuum levels and units for storage of silver wool in the dessicator (See 6.7 and 8.2.3). There is now one single unit and level.
- Clarified angle of insertion of the 16 gauge needle into the lid, the angle of needle insertion is now 30 to 45 degrees relative to the surface of the lid (See 8.2.4.6.1)
- Aligned allowed storage days of silver wool to 120 days (See Figure A.1 and 7.1.1.2)

Contents

Page

1 Scope 1
2 Normative references 1
3 Summary of test method 1
4 Terms and definitions 2
5 Significance and use 2
6 Apparatus 2
7 Materials 4
8 Procedure 5
9 Calculation 8
10 Report 10
11 Precision and bias 10
Annex A (normative) Special care and handling of silver wool 11
Bibliography 13

Figures

Figure 1 — Single cooling coil for small capacity baths 3
Figure 2 — Serpentine cooling coil for large capacity baths 3
Figure 3 — Modified plastic syringe 7
Figure 4 — Procedures for compacting silver wool into a standardized disk 8
Figure 5 — Silver wool colour classification scale 8
Figure 6 — Silver wool corrosion — Rating background 10
Figure A.1 — Suggested label for bulk silver wool container 11

Tables

Table 1 — Silver wool corrosion rating — Colour rating table 9
Table A.1 — Silver wool quantity purchasing guide 12

Supersedes CAN/CGSB-3.0
No. 60.32-2013

Standard test method for determination of the corrosiveness to silver of gasoline, middle distillate fuels and oxygenated fuels using silver wool: Rapid ultrasonic method

1 Scope

This method provides a means of determining the degree of corrosiveness of gasoline and middle distillate fuels to silver or silver alloys used in fuel system components.

This method also applies to oxygenated gasoline and diesel blends, intermediate streams or blending components such as alkylate, reformate, and isomerate.

Units of measure — Quantities and dimensions in this standard are given in SI units with imperial equivalents, mostly obtained through soft conversion, given in parentheses. The SI units are regarded as being official in the event of dispute or unforeseen difficulty arising from the conversion.

The testing and evaluation of a product against this method may require the use of materials and/or equipment that could be hazardous. This document does not purport to address all the safety aspects associated with its use. Anyone using this method has the responsibility to consult the appropriate authorities and to establish appropriate health and safety practices in conjunction with any applicable regulatory requirements prior to its use.

2 Normative references

The following documents contain provisions that, through reference in this text, constitute provisions of this method. The referenced documents may be obtained from the sources noted below.

NOTE The addresses provided below were valid at the date of publication of this method.

An undated reference is to the latest edition or revision of the reference or document in question, unless otherwise specified by the authority applying this method. A dated reference is to the specified revision or edition of the reference or document in question.

2.1 ASTM International

ASTM D5191 — Standard Test Method for Vapor Pressure of Petroleum Products (Mini Method).

2.1.1 Source

The above may be obtained from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, U.S.A., telephone 610-832-9585, fax 610-832-9555, Web site www.astm.org, or from IHS Markit, 200-1331 MacLeod Trail SE, Calgary, Alberta T2G 0K3, telephone 613-237-4250 or 1-800-267-8220, fax 613-237-4251, Web site www.global.ihs.com.

3 Summary of test method

3.1 In an ultrasonic bath, a sample of 0.100 g of silver wool is exposed to 180 mL of fuel sample in a glass test cell for 1h at 50°C.

3.2 Immediately after the test time has elapsed, the test cell is removed from the bath and the fuel sample is decanted. The silver wool is rinsed with wash solvent and then removed from the glass test cell, and blotted dry.

3.3 The wool is then pressed into a disk and rated in accordance with the alphanumeric corrosion rating scale.

4 Terms and definitions

For the purposes of this method, the following terms and definitions apply.

4.1
bulk wool / bulk silver wool
silver wool that has been opened, dated when first used, and from which sub samples (working quantities) are taken.

4.2
working quantity / working (silver) wool
silver wool that is sub sampled from the “bulk wool” and allotted into weekly working quantities.

5 Significance and use

5.1 Fuel wetted fuel system components made of silver or silver alloys can present surface corrosion from certain trace sulphur species (such as H_2S and elemental sulphur, even at a fractional parts per million concentration) occasionally found in gasoline or distillate fuel. Silver or silver-plated bearings used in some fuel pumps and certain silver or silver alloy contacts used in some fuel level sender (or sensor) units are examples of components that can be adversely affected. A layer of silver sulphide on a silver electrical contact can change the resistance, resulting in erroneous electrical signals being transmitted from fuel level sender units to fuel gauges.

5.2 The method provides a relatively rapid means for determining the degree of corrosiveness to silver of gasoline, oxygenated gasoline, middle distillates, and biodiesel fuel blends. The method is also adaptable for product field testing.

6 Apparatus

6.1 Test cells: glass test cells designated as certified or processed. To meet US EPA *Specifications and Guidance for Contaminant-Free Sample Containers,* such as I-Chem Certified 200 Series or equivalent thereof. Designated as 250 mL capacity, wide-mouth, clear glass jars (typical dimensions are 50 mm inner diameter by 127 mm high) with polytetrafluoroethylene (PTFE) lined caps.

6.2 Timer: a bench top type, or digital timer integrated with the ultrasonic bath (see 6.3). The timer shall have a minimum 90 min range and an accuracy of 15 s within 90 min.

6.3 Ultrasonic temperature-controlled bath: a bath with a heater capable of maintaining a temperature of 50 ± 2°C. A bath with industrial stacked transducers and employing a sweep frequency is required. Before use, the bath shall be outfitted or modified as described in 6.3.1 to 6.3.3.

6.3.1 A perforated stainless steel support tray, or mesh basket, is required to keep the test cells off the bottom of the bath, and at the required submerged level (see 8.2.4.7) when the bath is filled to a liquid level required for operation. Consult with the manufacturer or the supplier of the bath.

6.3.2 During normal operation, the bath temperature will have a tendency to increase over time due to the ultrasonic energy input. A cooling coil is necessary to maintain adequate temperature control. A cooling coil fabricated

from thin walled 6.35 mm (¼") stainless steel tubing, bent to fit as appropriate has been found to work well. A simple throttling valve is used to adjust the cooling water flow rate.

NOTE Tubing industry standard nomenclature is expressed in inches.

6.3.2.1 The cooling coil shall be positioned 1 to 2 cm below the surface of the water for maximum effectiveness. Simple wire hangers attached to the sides of the bath have been found to be acceptable. If the coil is placed on the bottom of the bath, density works against fast and efficient cooling.

6.3.2.2 For small baths with capacities of four test cells or less, a single cooling loop is adequate as shown in Figure 1.

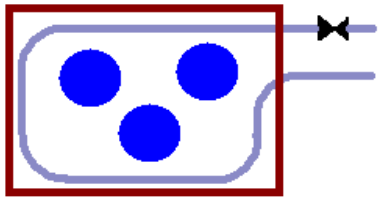


Figure 1 — Single cooling coil for small capacity baths

6.3.2.3 For larger baths with capacities of nine or more test cells, a serpentine cooling coil is recommended. Consider sample placement in the bath before bending the serpentine such that the coil loops demarcate the sample positions as shown in Figure 2. The coil loops can serve as test cell separators.

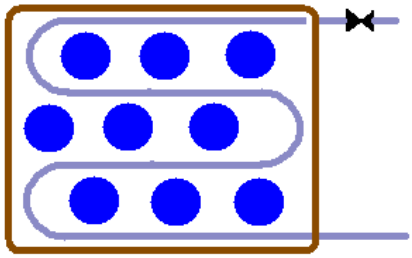


Figure 2 — Serpentine cooling coil for large capacity baths

6.3.3 The bath shall be filled with water to the operating level prescribed by the manufacturer. Between 3 to 6 mg/L of copper sulphate ($CuSO_4 \cdot 5H_2O$) may be added for control of algae growth, if needed.

Caution: Excessive use of $CuSO_4 \cdot 5H_2O$ can lead to staining deposits in the bath.

6.4 Temperature measuring device: a calibrated thermocouple is recommended. Do not use a liquid-in-glass filled thermometer (mercury or alcohol) to measure the bath operating temperature because it can be affected by the ultrasonic energy.

6.5 Analytical balance: with draft shield, capable of measuring to the nearest 1 mg.

6.6 Graduated cylinder: 250 mL capacity, Class B or Class A, "to deliver".

6.7 Vacuum desiccator: approximately 200 to 250 mm inner diameter, with porcelain or polypropylene desiccator plate. Polycarbonate or polypropylene desiccators may be used. A glass vacuum desiccator, although less robust and more expensive, is preferred because some plastics may be gas permeable; whereas, glass is not. Since the desiccator is used to store an oxidative material, a glass desiccator is therefore recommended. It shall be capable of maintaining a vacuum of 84 kPa (25 in. Hg).

6.8 Weighing dish: disposable, polystyrene, 90 to 105 mm square or hexagonal. A 90 mm diameter watch glass may also be used.

6.9 Scissors: stainless steel, surgical-grade.

6.10 Forceps: nominal length 152 mm, stainless steel, surgical-grade, two pair.

6.11 Filter paper: approximately 9 cm diameter, Whatman Grade 1 type or equivalent.

6.12 Plastic syringe: 10 mL capacity, modified as per Figures 3 and 4.

6.13 Wash bottle: 500 mL capacity, solvent venting type.

6.14 Silver wool sample bottles: 60 mL capacity, clear, glass, wide mouth, with caps. Used for keeping working quantities of silver wool. [1, 2]

6.15 Standard illumination: D5000K or D6500K illumination from filtered tungsten-halogen sources or seven-phosphor fluorescent lamps, such as the OTT-LITE, Universal Task Lamp, Model T96G5R from Environmental Lighting Concepts, Inc. [3] or equivalent.

NOTE The type of illumination is important in order to properly perceive colours because ordinary three-phosphor fluorescent lamps have higher blue/green energy, which intensifies these colours, while suppressing reds.

6.16 Rating background: the rating background (see Figure 6) consists of horizontal alternating black and white bars positioned parallel to each other. It is used as a background on which to view the disks and to simulate the colour classification scale (see Figure 5) background.

6.17 Colour classification scale: a series of standard silver wool corrosion images that depict increasing corrosion, alphanumerically rated from A to H2. The scale shall be printed on high quality glossy photo paper. Store the colour classification scale away from direct light and heat to avoid fading. It is suggested that the colour classification scale be kept in a dark brown envelope when not in use.

6.18 Fuel sample bottles: 1 L capacity, amber glass, with screw caps. The caps shall have inert liners such as PTFE. Cork or rubber stoppers shall not be used because they can be sources of contamination.

6.18.1 Plastic bottles are permeable to certain hydrocarbons and shall not be used for fuel sampling and storage. In some cases, plastic bottles retain certain silicon release agents used during the moulding process that can affect the results.

7 Materials

7.1 Silver wool: 99.9% minimum purity, 0.05 mm diameter fibres, Reference Annex A.

7.1.1 The silver wool shall be “bright” when used. Any tarnish or loss of surface shine is unacceptable. The wool should be packaged in a sealed container. Newly purchased wool, if compressed into a disk, should resemble an “A” rating or better. A rating of “A1” indicates it is not fit for use.

NOTE When ordering, it is recommended that the purchaser stipulate that the silver wool be “bright” and shipped in a sealed container, such as a sealed plastic bag or glass bottle.

---

[1] See also A.1.6.2

[2] The bottles should meet “US EPA Specifications and Guidance for Contaminant-Free Sample Containers”.

[3] Environmental Lighting Concept Inc., 1214 West Cass Street, Tampa, Fl, 33606, 1-800-842-8848.

7.1.1.2 As the shelf life of silver wool is limited, any bulk and working wool shall be discarded after four months. Any purchased (unopened) quantities, if present, shall likewise be discarded four months after date of receipt. If upon receipt the wool is discoloured, it shall not be used for testing. A 1 g quantity is sufficient for 8 tests, 5 g is sufficient for 40 to 45 tests, while 25 g is sufficient for 200 or more tests.

7.2 Wash solvent: Isooctane (2,2,4-trimethylpentane), American Chemical Society grade or better.

7.3 Rinsing solvent: 2-Methyltetrahydrofuran or toluene. 2-Methyltetrahydrofuran shall be anhydrous, with purity 99.0% or greater. Toluene shall be pesticide or residue analysis grade or better.

7.3.1 Dispose of rinsing solvent as per site specific requirements or regulations.

7.4 Vacuum grease: silicon type, required for glass vacuum desiccators and for plastic type vacuum desiccators that do not have greaseless seals.

8 Procedure

8.1 Sampling of fuel

8.1.1 Use a clean and dry sample bottle (bottles as per 6.18) for obtaining fuel test samples.

8.1.2 Flush the sample point to prevent contamination of the sample by extraneous materials that can be present in and around the sampling point prior to collecting the sample.

8.1.3 Rinse the sample bottle twice with approximately 25 to 50 mL of sample before filling. Do not touch the bottle to the sample point.

8.1.4 Collect a representative sample of no less than 500 mL. Fill the sample bottle so as to leave between 10 and 15 % headspace. Immediately after sampling, close the bottle using a PTFE lined screw cap. A cap with a polyethylene liner is also acceptable if the sample is to be analyzed within 24 h.

8.1.5 All efforts shall be made to minimize oxidizing the sample during sampling. Minimize aeration of the sample while filling the sample container by allowing the sample to run down the inside wall of the bottle.

8.1.6 Store the sample at 4°C until ready for testing. Allow the sample to warm up to 15°C before measuring the required sample volume for testing. The sample shall be at or above 15°C before it is placed into the ultrasonic bath.

8.2 Testing

8.2.1 The ultrasonic temperature-controlled bath should be placed in a fume hood or other suitable ventilated area that can safely remove any hydrocarbon vapours while not adversely affecting the operation of the bath (strong air current). If samples having a vapour pressure greater than 107 kPa (exerted in a vacuum as per ASTM D5191) are to be tested, use a fume hood with electrical components (such as light fixtures and receptacles) designed to eliminate sparks, i.e. an explosion proof or intrinsically safe fume hood.

8.2.2 Clean the vacuum desiccator and bottom plate thoroughly prior to storage of silver wool. Apply high vacuum silicone grease sparingly on the glass joints. **Do not** put any desiccant in the bottom of the desiccator.

8.2.3 Preparation of silver wool

The silver wool shall never be handled by direct contact with the skin. Oils and moisture from fingers can produce unpredictable results. Do not leave the silver wool exposed to the atmosphere, as it will begin to tarnish immediately. Store purchased (unopened) wool, bulk wool, and working quantity wool in a desiccator under a vacuum of at least 84 kPa (25 in. Hg). Reference Annex A for further handling instructions.

8.2.3.1 Do not handle the end of the forceps or the cutting blades of the scissors with your fingers. Clean these tools when needed with 2-Methyltetrahydrofuran, or alternatively, with toluene.

8.2.3.2 When allotting bulk silver wool into approximate weekly working quantities remove enough wool from the bulk container to suffice for approximately one month of testing and hermetically seal it in individual weekly 60 mL sample bottles. Each sample bottle is to contain enough wool for about a week's worth of tests. Use forceps to handle the silver wool.

8.2.3.3 Place any remaining bulk silver wool and working quantity of wool back into the desiccator and store under partial vacuum. The bulk silver wool should be stored in its original container.

8.2.4 Conducting tests

Obtain silver wool for testing from previously prepared, or opened, weekly working quantities. Place the wool on a clean filter paper and gently tease apart the strands to create a uniform exposure surface using the forceps. Strand lengths of 3 to 7 cm are recommended. Longer strands may be cut using stainless steel scissors.

8.2.4.1 Weigh out 0.100 ± 0.005 g of the silver wool onto a previously tared weighing dish. Using the forceps, fold and form the bulk of the weighed wool so that it resembles a spherical (fluffed) shape and transfer it into a new test cell (test cells shall not be reused). Place the lid on the test cell. Prepare as many test cells as necessary from the working wool for the current batch of samples.

NOTE Folding and manipulating the wool to resemble a fluffed shape keeps the wool strands apart and off the bottom of the test cell during the test. It may help in producing more accurate results and it creates more uniform disks when pressed.

8.2.4.2 Test cells containing silver wool shall be stored in a vacuum desiccator if they are not to be tested immediately. Ensure that the lids are placed on the test cells loosely before closing the desiccator lid and creating a vacuum; otherwise the lids will "pop off" or become distorted.

8.2.4.3 Store the forceps and the scissors in a clean, well labelled beaker or test cell reserved for this purpose in the vacuum desiccator.

8.2.4.4 Verify that the bath temperature is stable at 50 ± 2°C prior to inserting the test cells and that a small amount of cooling water is flowing. Adjust temperature as necessary.

8.2.4.5 Cooling water flow should be adjusted appropriately taking into account the temperature of the cooling water supply and the size of the bath to be cooled. Flow rates between 30 and 100 mL/min have given acceptable results using tap water.

Caution: The bath water shall be changed out completely if hydrocarbons are spilled into the bath. Even small amounts of certain hydrocarbons will result in rapid build-up of heat in the bath such that temperatures will become uncontrollable within the prescribed limits.

8.2.4.6 Rinse a previously cleaned 250 mL graduated cylinder with two 10 mL portions of the fuel sample. Transfer 180 ± 2 mL of sample to the test cell containing silver wool (prepared in 8.2.4.1) using the graduated cylinder. Immediately tighten the lid and place the cell into the ultrasonic bath. Record the start time.

8.2.4.6.1 Samples below 107 kPa

Testing material having vapour pressure up to 107 kPa (at 37.8°C) using this method requires that a small pinhole be made in the plastic lid and liner of the test cell for venting using a 16 gauge needle or equivalent. The pinhole size needs to be adjusted to ensure adequate pressure release. Inserting the needle at a 30° to 45° angle with respect to the surface of the lid has been found to work well.

8.2.4.6.2 Samples at and above 107 kPa

Some fuel products can have a much higher vapour pressure than would normally be characteristic of automotive gasoline. Samples having vapour pressure above 107 kPa (at 37.8°C) may require additional venting to prevent pressure build-up inside the test cell. Inserting a 13 gauge hypodermic needle at a 45° angle through the plastic cap and liner may be considered. After inserting the needle, ensure that it is not blocked. The needle should be positioned at least 2 cm above the liquid level once the cap is screwed onto the test cell. The needle also allows the connection of a small plastic hose to help in channelling vapours away from the bath for safe venting.

8.2.4.7 Test cells shall be submerged to the level of the fuel sample and be separated from other test cells by at least 4 cm to ensure uniform heating and sonication.

8.2.4.8 If the lids are labelled with the sample ID and the start time, this permits easy identification of the samples without having to pull them out of the bath during sonication.

8.2.4.9 After 1 h in the ultrasonic bath, remove the test cell from the bath and immediately decant the fuel, allowing the silver wool to remain at the bottom of the test cell. Rinse the silver wool with approximately 10 mL of isooctane (wash solvent as per 7.2) using a wash bottle. Repeat the washing step once more.

8.2.4.10 Use clean forceps to gently remove the wool from the test cell and place it on a fresh piece of filter paper. Using a second piece of filter paper, very gently blot the wool dry taking care not to roll or apply any sideways force.

8.2.4.11 Carefully compact the silver wool into a 1.5 cm diameter standardized disk, using a modified 10 mL plastic syringe (see Figures 3 and 4).

8.2.4.12 View the silver wool simultaneously against both white and black backgrounds using the standard rating background chart (see Figure 6), as darker colours are more apparent against a black background. Examine the wool at various angles in order to perceive subtleties in colour. This examination shall be conducted using a balanced D5000K or D6500K light source as in 6.15.

8.2.4.13 Compare and record the colour(s) of the silver wool to the standard descriptions found in Figure 5 and Table 1.

8.2.4.14 After testing discard the fuel, test cell, cap, and silver wool.

8.2.4.15 Test cells shall not be reused. Silver corrosion additives used from time to time in gasoline or distillate products are highly surface active and can adhere to the test cell walls. This can lead to a false negative where the corrosiveness of the sample is masked by additive memory effect on the test cell.

Figure 3 — Modified plastic syringe

Modified plastic syringe used for creating standardized silver wool disk. This device is created by cutting off the end of the syringe. In addition, the rubber tip of the plunger shall be removed and replaced with a polytetrafluoroethylene cap.

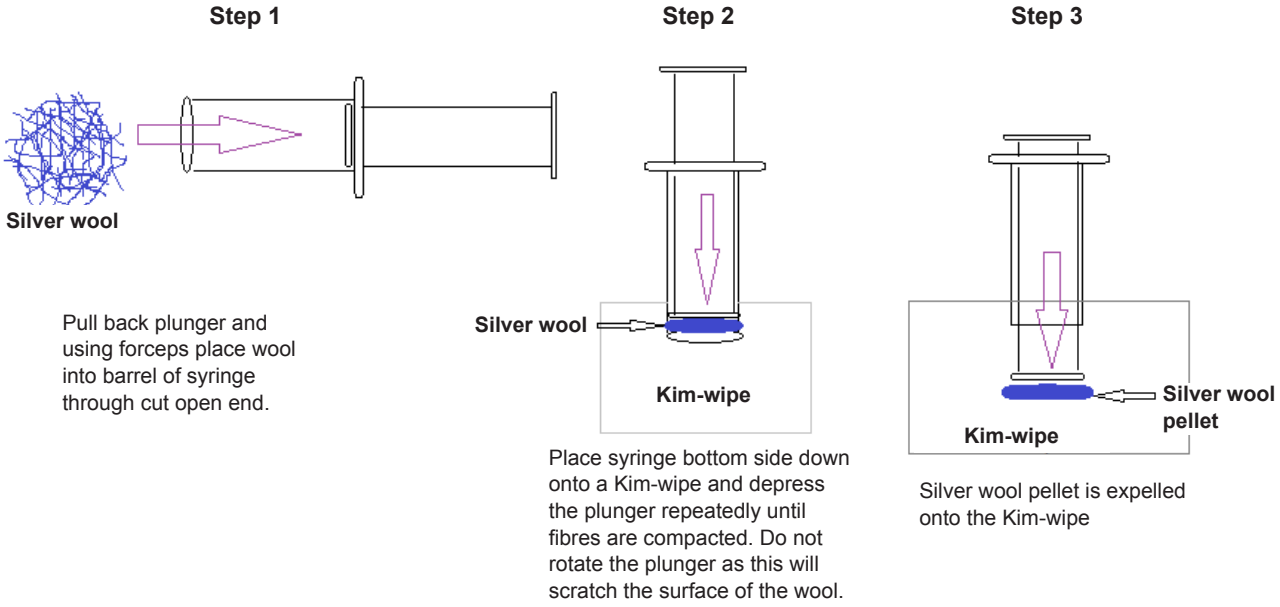
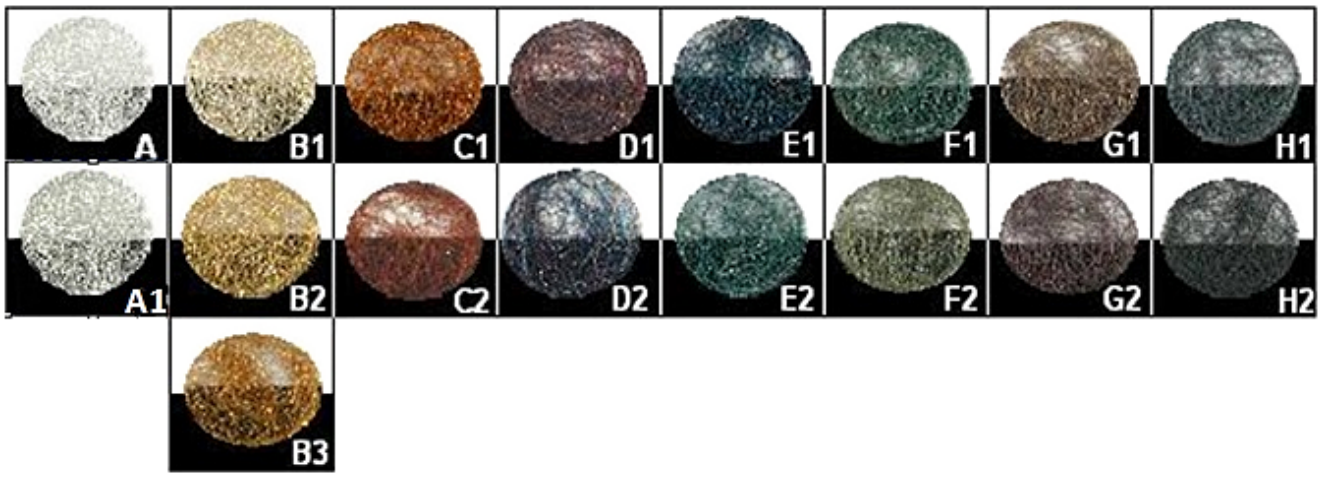


Figure 4 — Procedures for compacting silver wool into a standardized disk

9 Calculation

9.1 Interpretation

9.1.1 The samples will exhibit a continuum of the colours from least to most corroded. A rating of “A” indicates no tarnish or corrosion, while a rating of “H2” indicates severe corrosion.



NOTE This scale should be printed on high-quality glossy photo paper for accurate colours.

Figure 5 — Silver wool colour classification scale

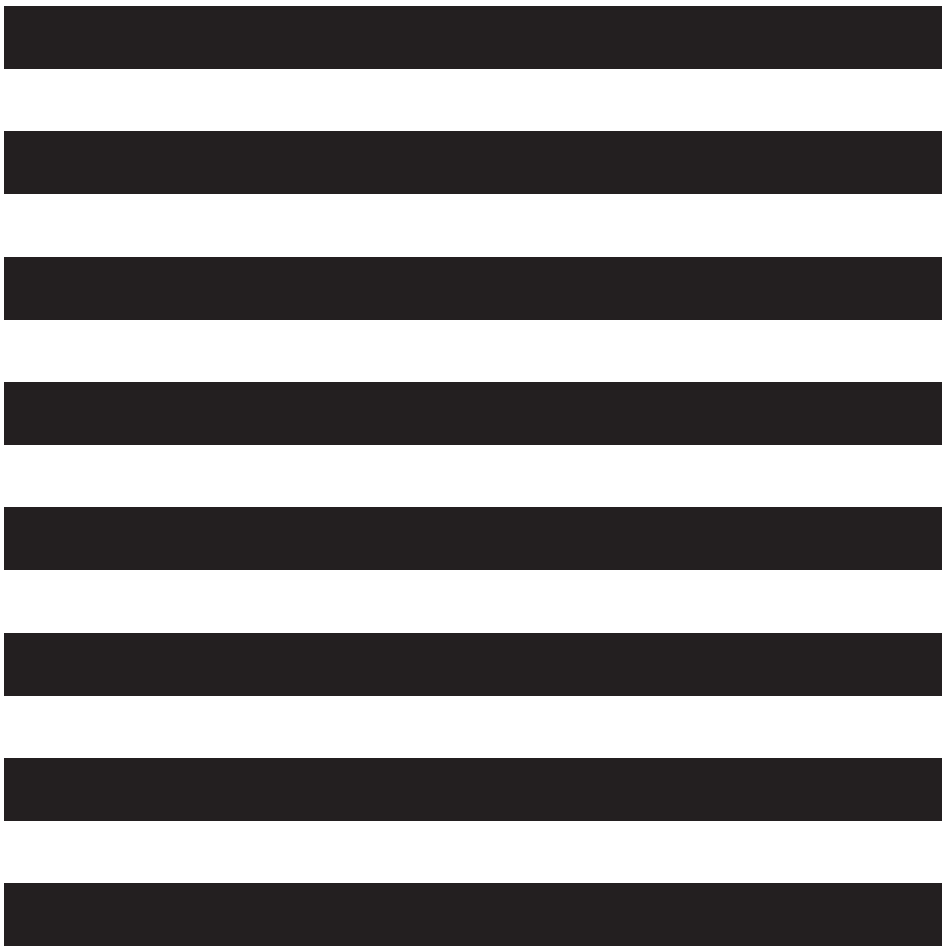
9.1.2 Record the corrosion rating of the silver wool using the standard descriptions given in Table 1. The colour chart in Figure 5 shall be used as a guide. If a rating falls in between two levels, always rate to the more corroded level.

9.1.2.1 Exercise caution when using the colour chart. It is intended as a guide to the most common colour successions seen with most standard fuels. There are some colours that have been observed with intermediate streams such as coker naphtha (high olefinic) that are not part of the standard colour scale. For these cases the user is advised to consider time studies.

9.1.2.2 If it is difficult to differentiate between ratings, it may be necessary to rerun the test by preparing four sample jars and inspecting a jar at 15 min increments until all four jars have been inspected over a 60-min period. Inspect the first jar after 15 min, the second after 30 min, the third jar after 45 min and the fourth jar after 60 min. In this way it is possible to differentiate between ratings, for example C2 and G1.

Table 1 — Silver wool corrosion rating — Colour rating table

Silver wool rating	Description
A	No corrosion, crisp, bright and shiny surface — same as unexposed wool
A1	Loss of surface shine or brightness with no other visible colour change — lustreless, matt appearance, dull
B1	Light gold
B2	Gold
B3	Dark gold
C1	Copper
C2	Reddish copper, red oxide, reddish brown
D1	Peacock red (red with a combination of gold, blue, and/or copper fibres)
D2	Peacock blue (blue with a combination of red fibres which may cause a dark purple appearance)
E1	Blue
E2	Greenish blue, teal
F1	Green, pine colour
F2	Yellowish green, olive green
G1	Orange-pink (salmon coloured), burnt-orange
G2	Pinkish purple (plum or wine coloured)
H1	Bluish or purplish grey
H2	Charcoal or black



NOTE This rating background should be printed on high-quality glossy photo paper to enable accurate comparison of compacted disks to that of the colour classification scale in Figure 5.

Figure 6 — Silver wool corrosion — Rating background

10 Report

10.1 Report the silver wool corrosion rating, as determined in 9.1.2. Report an unusual colour that does not align with the standard colour classification scale.

11 Precision and bias

11.1 Repeatability and reproducibility

It is not possible to specify the precision of this test method because the test method results cannot be analyzed by standard statistical methodology.

11.2 Bias

This test method has no bias because the silver wool corrosion rating is defined only in terms of this test method.

Annex A
(normative)

Special care and handling of silver wool

A.1 The following deals with common sense practices that shall be followed to ensure reliable results when using silver wool. The wool is made from high purity silver and is quite reactive to many compounds that are frequently found in a petroleum laboratory environment. The practices described here are in addition to those stated in the procedure section of the method.

Caution: Minimize exposure of silver wool to the atmosphere. Silver wool that has been exposed to moist air, ozone, or other reactive species such as HCl fumes will lose surface brilliance and shine. The silver wool will appear matt or dull without any hint of discolouration. In severe cases it will start to show tones of grey. Using discoloured silver wool will compromise the results by lowering the corrosion “severity” of the rating, thereby giving a false pass (versus a specification limit).

A.1.1 The inspection process and the splitting of the bulk wool into working quantities shall be done in an area that is clean and free of most airborne reactive compounds such as, but not limited to, hydrogen sulphide, sulphur dioxide, volatile thiols, volatile organic and inorganic sulphides, hydrochloric acid, ammonia, ammonium chloride, hydrofluoric acid, and ozone (corona discharge from photocopiers).

A.1.2 Good analytical practice requires that on receipt of new silver wool it be immediately inspected for suitability for use. The silver wool shall be uniformly bright and shiny throughout with no hint of discoloration. A label should be affixed to the container stating the date of receipt, the date of inspection, and the date the silver wool from this container was first put into use. This is the bulk silver wool, different from the working silver wool as explained below. A suggested label is shown below.

Silver wool for use with CAN/CGSB-3.0 No. 60.32		
Bulk	**Date (yyyy-mm-dd)**	**Initials**
Date received		
Date inspected and approved for use		
Date first used		
Discard after	This silver wool is no longer fit for purpose 120 days after receipt or if discoloured in any way before that time.	

Figure A.1 — Suggested label for bulk silver wool container

A.1.3 The bulk wool container should be opened only long enough to remove sufficient wool to prepare the necessary working quantities. It should then be resealed and returned to the desiccator.

NOTE The bulk silver wool is best kept in its original container if it can be properly sealed under vacuum.

A.1.4 Sub sampling of the bulk wool into working quantities is necessary in order to minimize corrosion of the bulk wool as a result of exposure to the atmosphere. Working quantities of silver wool are obtained from the bulk wool and individually sealed based on the quantity of tests expected to be performed. Determining working quantities of wool required for tests is predicated on knowing how many samples are expected to be tested weekly over a finite period of time (usually one month). Thereby, enough working quantity of silver wool is obtained from the bulk

quantity to suffice for approximately four weeks' worth of tests. The working quantities are allotted into approximate weekly test portions and individually sealed.

A.1.5 The working wool container that is "in use" during a particular week shall be stored in the desiccator, under vacuum. Therefore, this container is no longer hermetically sealed but sufficiently closed to prevent the silver wool from falling out or contaminants from falling in. Since the container will be stored under vacuum it needs to "breathe." The wool in the "in use" container shall be discarded after seven days if it is not entirely consumed.

A.1.6 Silver wool should be purchased in quantities that are in balance with the number of tests done, or expected to be done, over a four-month period. Wool can be purchased in quantities of 1, 5, and 25 g, representing approximately 8, 42, and 220 tests. Use the following Table A1 as a guide.

Table A.1 — Silver wool quantity purchasing guide

Tests per week	Quantity purchased	Working quantity
1 or less	1 g (as required)	1 g (leave in original container, discard after 120 days)
2 or less	1 g (2 X 1 g)	0.25 g
3 to 4	5 g	0.50 g
5 to 8	5 g (2 X 5 g)	0.83 g
10 or more	25 g (as required)	As required

A.1.6.1 When working with dual package such as 2 X 1 g, it is best to split one package at a time, using the original container as one of the working containers.

A.1.6.2 When properly separated, a 25 g bulk package can be divided into two bulk quantities. The split quantity shall be placed in a clean and easily sealable container such as an I-Chem sterile and contaminant free 60 mL glass wide mouth bottle. Each bulk quantity shall be inspected separately before being put into use.

A.1.7 Silver wool that is past its "discard after" date (as in A.1.2) shall not be used for this procedure.

Bibliography

[1] CAN/CGSB-3.5 — *Automotive Gasoline*

[2] CAN/CGSB-3.511 — *Oxygenated Automotive Gasoline Containing Ethanol (E1-E10 and E11-E15)*

[3] CAN/CGSB-3.517 — *Diesel Fuel*

[4] ASTM D4057 — *Standard Practice for Manual Sampling of Petroleum and Petroleum Products*