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CGSB

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No./Nº 122.2-98
April/Avril 1998

WITHDRAWAL

February 2003

CANADIAN GENERAL
STANDARDS BOARD

METHODS OF TESTING PETROLEUM
AND ASSOCIATED PRODUCTS

BENZOTRIAZOLE OR TOLYLTRIAZOLE IN AIRCRAFT DEICING-DEFROSTING FLUIDS (VOLUMETRIC METHOD)

This CGSB test method is hereby withdrawn because the specification 3.856, Fluid, Deicing-Defrosting Concentrate for Aircraft Surfaces, that referenced this test method has been withdrawn.

RETRAIT

Février 2003

OFFICE DES NORMES
GÉNÉRALES DU CANADA

MÉTHODES D'ESSAI DES PRODUITS
PÉTROLIERS ET PRODUITS CONNEXES

BENZOTRIAZOLE OU TOLYLTRIAZOLE DANS LES LIQUIDES DÉGIVRANTS-DÉGELANTS D'AÉRONEFS (MÉTHODE VOLUMÉTRIQUE)

Cette méthode d'essai de l'ONGC est retirée par le présent avis parce que la spécification 3.856, Liquide dégivrant-dégelant concentré pour surfaces d'aéronefs, qui y réfère a été retirée.

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CAN/CGSB-3.0
No. 122.2-98

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No. 122.2-M87

Methods of Testing Petroleum and Associated Products

Benzotriazole or Tolyltriazole in Aircraft Deicing-Defrosting Fluids (Volumetric Method)

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NATIONAL STANDARD OF CANADA

CAN/CGSB-3.0
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METHODS OF TESTING PETROLEUM AND ASSOCIATED PRODUCTS

BENZOTRIAZOLE OR TOLYLTRIAZOLE IN AIRCRAFT DEICING-DEFROSTING FLUIDS (VOLUMETRIC METHOD)

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National Standard of Canada

CGSB Ottawa Canada K1A 1G6	METHODS OF TESTING PETROLEUM AND ASSOCIATED PRODUCTS	CAN/CGSB-3.0
	Benzotriazole or Tolyltriazole in Aircraft Deicing-Defrosting Fluids (Volumetric Method)	**No. 122.2-98**

Supersedes CAN/CGSB-3.0
No. 122.2-M87

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1. SCOPE

1.1 This method covers the determination of benzotriazole or tolyltriazole in the range of 0.4 to 0.6% by mass in aircraft deicing-defrosting fluids.

1.2 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. REFERENCED PUBLICATION

2.1 The following publication is referenced in this method:

2.1.1 Canadian General Standards Board (CGSB)

CAN/CGSB-3.0 — Methods of Testing Petroleum and Associated Products:

No. 0 — Prologue to the Petroleum Test Methods and Table of Contents.

2.2 A reference to a regulation is always to the latest issue. A dated reference is to the issue specified. An undated reference is to the latest issue, unless otherwise specified by the authority applying this method. The sources are given in the Notes section.

3. SUMMARY OF METHOD

3.1 The benzotriazole ($C_6H_5N_3$) or tolyltriazole ($C_7H_7N_3$) in the sample is precipitated with an excess of silver nitrate solution:

(1) $C_6H_5N_3+Ag^+ + NO_3^- \rightarrow C_6H_4N_3(Ag) + NO_3^- + H^+ + Ag^+$(excess)

The precipitate is filtered off and the excess silver nitrate is back titrated with a standardized ammonium thiocyanate solution using an Iron (III) ion indicator to determine the end point:

(2) Ag^+(excess)$+SCN^- \rightarrow AgSCN$

(3) $Fe^{3+}+SCN^- \rightarrow Fe(SCN)^{2+}$(red end-point)

3.2 It is essential that only one triazole compound be present and its identity must be known to the analyst for the calculation of results.

4. SIGNIFICANCE AND USE

4.1 The result of this test determines the percentage by mass of benzotriazole or tolyltriazole present in aircraft deicing-defrosting fluids. Benzotriazole or tolyltriazole is used in these fluids as a fire-retardant.

5. REAGENTS AND MATERIALS

5.1 **Silver Nitrate ($AgNO_3$), Crystal** — ACS reagent grade.

5.2 **Standard Silver Nitrate Solution (0.1 mol/L)** — Refer to par. 6.1.

5.3 **Ammonium Thiocyanate (NH_4SCN), Crystal** — ACS reagent grade.

5.4 **Standardized Ammonium Thiocyanate Solution (0.1 mol/L)** — Refer to par. 6.2.

5.5 **Ammonium Hydroxide (NH_4OH), Concentrated** — ACS reagent grade.

5.6 **Nitric Acid (HNO_3), Concentrated** — ACS reagent grade.

5.7 **Ferric Ammonium Sulphate ($FeNH_4(SO_4)_2 \cdot 12H_2O$), Crystal** — ACS reagent grade.

5.8 **Indicator Solution, Ferric Alum** — Prepared by dissolving about 80 g of ferric ammonium sulphate crystals ($FeNH_4(SO_4)_2 \cdot 12H_2O$) in 10 mL of concentrated nitric acid diluted to 200 mL with distilled water.

5.9 **Diatomaceous Earth.**

5.10 **Filter Paper** — Whatman No. 5 filter paper or equivalent.

5.11 **Normal Laboratory Glassware.**

6. PROCEDURE

6.1 Preparation of the Standard Silver Nitrate Solution (0.1 mol/L)

6.1.1 Determine the mass of approximately 17 g of silver nitrate crystals into a clean, dry, weighing bottle or dish. Dry the bottle or dish and its content for 1 h in an oven at 105 ± 2°C; store in a desiccator. When cooled, determine the mass of the bottle or dish and its content to the nearest milligram. Carefully transfer the bulk of the solid into a 1-L volumetric flask. Determine again the mass of the bottle or dish to obtain by difference the mass of solid transferred to the volumetric flask. Fill the flask half-full with distilled water and swirl until the crystals are completely dissolved. Dilute to the mark and mix the solution thoroughly by upending the stoppered flask several times.[1] Calculate the molarity of the solution.

6.2 Preparation and Standardization of the Ammonium Thiocyanate Solution (0.1 mol/L)

6.2.1 Dissolve approximately 7.7 g of ammonium thiocyanate crystals in about 1 L of distilled water. Mix well.

6.2.2 Pipet 25 to 30 mL of the standard silver nitrate solution (par. 6.1) into an Erlenmeyer flask and dilute to approximately 100 mL with distilled water. While stirring slowly, add concentrated nitric acid (10 to 15 mL), followed by 1 mL of the indicator solution. Titrate with the ammonium thiocyanate solution (par. 6.2.1) to the pale orange end point. Calculate the concentration of the NH_4SCN solution as follows:

$$NH_4SCN \text{ Concentration, mol/L} = \frac{A}{B} \times M_1$$

where:

A = volume of silver nitrate, mL
M_1 = concentration of silver nitrate, mol/L
B = volume of ammonium thiocyanate, mL

6.3 Determination of the Benzotriazole or Tolyltriazole Concentration

6.3.1 To 50 g of the sample into a beaker add 85 mL of distilled water, 5 mL of concentrated ammonium hydroxide, and 20 mL of the 0.1 mol/L silver nitrate solution.

6.3.2 Stir the suspension well and allow it to stand for 5 min to allow coagulation of the precipitate.

6.3.3 Add approximately 3 to 4 g of diatomaceous earth to aid the filtration and filter the suspension through a No. 5 Whatman filter paper using a Buchner funnel. Exercise care to transfer all solution and precipitate.

6.3.4 Wash the precipitate with three 50 mL portions of distilled water and transfer the filtrate and washings to the beaker.

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[1] *Once prepared, and when not actually in use, $AgNO_3$ solution should be stored in a dark place.*

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6.3.5 Add concentrated nitric acid (10 to 15 mL), stirring slowly, into the filtrate followed by 1 mL of indicator solution.

6.3.6 Titrate the solution with 0.1 mol/L ammonium thiocyanate solution to the pale orange end point.

7. CALCULATION

7.1 Calculate the benzotriazole or the tolyltriazole concentration as follows:

Benzotriazole, % by mass = $[(A \times M_1 - (B \times M_2)] \times 0.238$

or

Tolyltriazole, % by mass = $[(A \times M_1 - (B \times M_2)] \times 0.266$

where:

A = volume of silver nitrate, mL

M_1 = concentration of silver nitrate, mol/L

B = volume of ammonium thiocyanate, mL

M_2 = concentration of ammonium thiocyanate, mol/L

8. REPORT

8.1 Report the benzotriazole or tolyltriazole concentration in percentage by mass.

9. NOTES

9.1 **Source of Referenced Publication**

9.1.1 The publication referred to in par. 2.1.1 may be obtained from the Canadian General Standards Board, Sales Centre, Ottawa, Canada K1A 1G6. Telephone (819) 956-0425 or 1-800-665-CGSB (Canada only). Fax (819) 956-5644.

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