INCH-POUND

A-A-59893 17 October 2012

COMMERCIAL ITEM DESCRIPTION

1,3,5-TRICHLOROBENZENE (TCB), CAS # 108-70-3

The General Services Administration has authorized the use of this commercial item description, for all federal agencies.

1. SCOPE

1.1 Scope. This commercial item description covers 1,3,5-trichlorobenzene (TCB).

2. SALIENT CHARACTERISTICS

2.1 Material. The material shall be 1,3,5-trichlorobenzene (TCB) meeting the following requirements:

- a) Color: White to off-white
- b) Form: The TCB may be supplied as a cast solid in a suitable container or as a granulated, flaked, or pelletized material
- c) Compound identification: FTIR spectral profile to correspond that of pure material
- d) Purity: 98% minimum, by HPLC or GC area count
- e) Insoluble Material: 0.2 wt% max.
- f) Melting point: 62°C minimum

2.2 Workmanship: The best commercial practice shall be used in the manufacture of this product. Procedures and controls shall be employed to prevent product contamination. There shall be no contamination (such as dirt, paint chips, wood, etc.) that is obvious and readily observable. In addition, the TCB shall be free of contaminants which are hazardous, corrosive, reactive, or metallic.

Comments, suggestions, or questions on this document should be addressed to: Commander, U.S. Army ARDEC, ATTN: RDAR-QES-E, Picatinny Arsenal, New Jersey 07806-5000 or e-mailed to <u>ARDECSTDZN@conus.army.mil</u>. Since contact information can change, you may want to verify the currency of this information using ASSIST Online database at https://assist.dla.mil.

AMSC N/A FSC 6810 DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited

3. REGULATORY REQUIREMENTS.

3.1 Safety and Health Practices. This standard does not purport to address all of the safety concerns, if any, associated with the use of the TCB material. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

3.2 Manufacture Safety Data Statement. Manufacture Safety Data Statement shall be submitted in accordance with requirements as specified in the Code of Federal Regulation, Part 29-1910 (OSHA). (For shipment to foreign users, the Manufacture Safety Data Statement may have to conform to the formatting and legislative requirements of the host country.)

4. QUALITY ASSURANCE PROVISIONS.

4.1 Product conformance. The product provided shall meet the salient characteristics of this Commercial Item Description. The Government reserves the right to require proof of such conformance prior to the first delivery and thereafter as may be otherwise provided for under provisions of the contract. For TCB product which has been sold on the market for less than two years, the manufacturer shall have to demonstrate process stability and to verify product quality by submitting analysis data from the most recent 5 consecutive production batches.

4.2 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of inspections for the verification of all salient characteristics as specified in Section 2 of this CID. The supplier may use his own or any other facility suitable for the performance of the inspection requirements specified herein, unless otherwise specified in the contract or disapproved by the Government. The Government reserves the right to perform any inspections set forth in the CID where such inspections are deemed necessary to assure supplies and services conform to the prescribed requirements.

4.2.1 Bid sample submission. Unless otherwise specified, a sample of 100 grams of TCB shall be submitted for inspection and approval in accordance with the terms of the procurement contract. The sample will be subjected to any or all of the quality inspections listed in this CID for verification of compliance to any or all requirements listed in this CID.

4.2.2 Test and inspection results. The contractor shall provide a summary of product test results (or an appropriate form of document of analysis in accordance with acceptable industry practices) to verify conformance to the salient characteristics requirements. (See 2). The contractor shall maintain a traceable record of inspection test data adhering to general laboratory practices for future reference if required.

4.3 Sampling techniques and inspection procedures. When not specified in this CID, the TCB shall be inspected by using acceptable industry-wide sampling techniques and test procedures such as ASTM and ACS sampling and test procedures. Also, unless otherwise specified herein, all chemicals and reagents shall be ACS or Reagent Grade. Tests for High Performance Liquid Chromatography (HPLC) and the Fourier Transform Infrared Spectroscopy (FTIR) shall be performed as specified below.

NOTE: The test description described below are not intended to be used as step-bystep laboratory procedures, rather they are intended to define the test methods used to obtain the required characteristics. The user should prepare their own laboratory procedures taking into consideration and complying with all aspects of the following test method descriptions. In preparation of step-by-step laboratory procedures, the user should take into account safety precautions from the manufacturer, industry safety and health practices, and also determine the applicability of safety and environmental regulatory limitations. The test descriptions below do not purport to address all the safety concerns, if any, associated with their use. It is the responsibility of the user of the procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

4.3.1 Compound identification. A sample of TCB is given positive identification by an infrared scan from 4000 to 650 cm⁻¹

4.3.1.1 Apparatus: A Perkin Elmer System One Fourier Transform Infrared (FTIR) spectrometer with a germanium lens and attenuated total reflectance (ATR) attachment, or its equivalent.

4.3.1.2 Sample preparation: Use sufficient sample to cover the lens.

4.3.1.3 Results of examination. The spectral profile must correspond to the one shown below, Fig. 1.



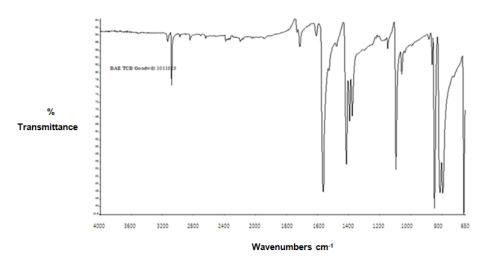


Figure 1

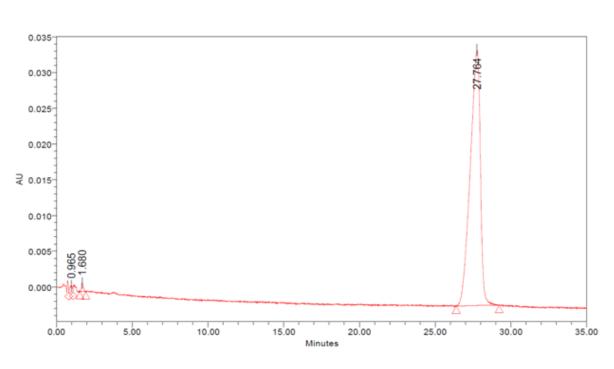
4.3.2 TCB purity: The purity of the TCB shall be determined through High Performance Liquid Chromatography (HPLC)

- 4.3.2.1 Apparatus: A Waters Alliance HPLC with a UV-VIS detector, or its equivalent
- 4.3.2.2 Instrument parameters:
 - (a) Column: Phenomenex Luna 3 micron, C8, 150 x 2.0 mm or its equivalent.
 - (b) Mobile phase (HPLC Grade): actonitrile:H₂O/45:55, 0.3 ml/min, isocratic.
 - (c) Injection volume: 10µL
 - (d) Wavelength: 280 nm
 - (e) Retention time: 27 minutes
 - (f) Run time: 35 minutes

Note: times may vary with instrument type and column plumbing

4.3.2.3 Sample concentration: 1000 ppm in acetonitrile, HPLC or Analytical Reagent Grade.

4.3.2.4 Calculation: The calculation is made by adding together the counts from all the peaks with exception of the solvent front. The count of the TCB peak is then divided by the total count, times 100, to obtain the percentage purity of the TCB. See Fig. 2 below. Purity may also be calculated using a reference standard. The standard must have a certificate of analysis indicating purity of at least 99.0 %. The calculation is made by dividing the sample area count by the standard area count, then multiplying by the nominal value of the standard.



HPLC 1,3,5-TCB

Figure 2

4.3.3 TCB purity, alternate method. The purity of the TCB may be determined through Gas Chromatography (GC).

4.3.3.1 Apparatus: Agilent 6890 Gas Chromatograph or equivalent, with thermal conductivity or mass detector.

4.3.3.2 Instrument parameters:

- (a) Column: DB 5 or equivalent, 30 m x 250 um, 0.25 film thickness, or equivalent
- (b) Helium flow: 1 mL/min
- (c) Oven temp: 70 C initial, 2 min hold, ramp to 230 C @ 20 C/min, hold 10 min
- (d) Injection volume: 1uL
- (e) Injector temperature: 250 C
- (f) Detector temperature: 250 C
- (g) Retention time: 6 minutes

Run time: 20 minutes

Note: times may vary with instrument type and column plumbing

4.3.3.3 Sample concentration: 3 mg/mL in methylene chloride, Analytical Reagent Grade.

4.3.3.4 Calculation: The calculation is made by adding together the counts from all the peaks with exception of the solvent front. The count of the TCB peak is then divided by the total count, times 100, to obtain the percentage purity of the TCB. A correction is made for the material that is insoluble in methylene chloride. Chromatogram is shown below in Fig. 3. Purity may also be calculated using a reference standard. The standard must have a certificate of analysis indicating purity of at least 99.0 %. The calculation is made by dividing the sample area count by standard area count, then multiplying by the nominal value of the standard.



GC 1,3,5-TCB

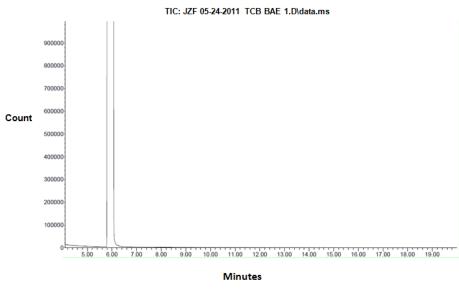


Figure 3

4.3.4 Insoluble material: A weighed sample of TCB is dissolved in methylene chloride and filtered through a tared glass filtering crucible. The crucible is reweighed and the weight gain is considered due to insoluble material.

4.3.4.1 Apparatus and reagents.

- (a) Filtering crucibles, 30 or 50 ml of volume, fine porosity fritted glass.
- (b) Standard laboratory apparatus to weigh the sample, dissolve it in 500 ml methylene chloride, effect an analytical transfer to the filtering crucible, and to wash, dry, and weigh the residue.
- (c) Methylene chloride, Analytical Reagent Grade.

4.3.4.2 Procedure.

 (a) Weigh a 50-g sample to 0.1 mg into beaker capable of holding 500 ml methylene chloride. Add the solvent and stir until the sample is dissolved. Tare a filtering crucible and filter the solution through it using vacuum. Wash

the contents of the crucible with the additional methylene chloride. Dry the crucible at 60°C in an air-flow oven, cools, and weigh.

(b) Calculate the percentage of methylene chloride insolubles as follows:

% Insolubles =
$$\frac{W_{2-}W_3}{W_1} x 100$$

Where W_1 = weight of sample in grams W_2 = weight of crucible and residue in grams W_3 = weight of crucible in grams Correct the percentage purity of the TCB by subtracting insoluble percentage from the purity percentage as calculated in the equation above.

4.3.5 Melting point:

4.3.5.1 Apparatus and materials; Thomas Hoover Uni-Melt 6406H Capillary Melting-Point Apparatus or equivalent, capillary tubes.

4.3.5.2 Procedure: Add ¼ to ½ inch of TCB to capillary tube; place in oil bath. Adjust heating rate to manufacturer's recommended setting. Observe onset and end melting point temperature.

5. PACKAGING. Preservation, packing, and marking shall be as specified in the contract or order (see 6.1.d).

6. NOTES.

6.1 Ordering data. Procurement documents should specify the following;

- a. Title, number, and date of this CID.
- b. Issue of Department of Defense Index of Standards and Specifications (DODISS) to be cited in this solicitation, and if required, the specific issue of the individual documents referenced.
- c. Quantity required.
- d. Packing and marking requirements.

6. 2 Equivalent test method. Prior approval of the Contracting Officer is required for use of equivalent test methods. A description of the proposed method should be submitted to through the Contracting Officer to: Commander, ATTN: RDAR-QAT-P, ARDEC, Picatinny Arsenal, NJ 07806-5000. This description should include, but not be limited to, the procedures used, the accuracy and precision of the method, test data to demonstrate the accuracy and precision and drawings of any special equipment required.

6. 3 Technical agency. ARDEC, Picatinny Arsenal, (RDAR-QAT-P), is the technical agency that is responsible for the preparation and revision of this Commercial Item Description. This CID should not be revised without the expressed concurrence and approval from the Technical Agency. All correspondence to the Technical Agency should be submitted through the contracting officer to: Commander, ARDEC, ATTN: RDAR-QES-E, Picatinny Arsenal, NJ 07806-5000.

Custodian:

Preparing Activity:

Army - AR Navy - SH Air Force - 68 Army - AR (Project 6810-2012-006)

Review activity:

Army – EA, AV, MI, MR, TE Navy – AS Air Force – 03, 11 DLA – GS GSA - FAS

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