METRIC

MIL-DTL-32101 19 September 2002 SUPERSEDING (See 6.7)

### **DETAIL SPECIFICATION**

## CARBON, ACTIVATED, IMPREGNATED, COPPER-SILVER-ZINC-MOLYBDENUM-TRIETHYLENEDIAMINE (ASZM-TEDA)

This specification is approved for use by the U.S. Army Edgewood Chemical Biological Center, Department of the Army, and is available for use by all Departments and Agencies of the Department of Defense.

## 1. SCOPE

**1.1 Scope.** This specification covers activated carbon impregnated with copper, silver, zinc and molybdenum salts and triethylenediamine (TEDA) for use as a sorbent for all known and suspected military chemical weapons agents.

### **2. APPLICABLE DOCUMENTS**

**2.1 General.** The documents listed in this section are specified in sections 3 and 4 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to insure the completeness of this list, document users are cautioned that they must meet all specified requirements documents cited in sections 3 and 4 of this specification, whether or not they are listed.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Technical Director, U.S. Army Edgewood Chemical Biological Center, ATTN: AMSSB-REN-SE-SS, Aberdeen Proving Ground, MD 21010-5424 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

FSC 6850

**<u>DISTRIBUTION STATEMENT A.</u>** Approved for public release; distribution is unlimited.

### 2.2 Government documents.

### 2.2.1 Specifications, standards, and handbooks. None.

**2.2.2 Other Government documents, drawings, and publications.** The following other Government documents, drawings, and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues are those cited in the solicitation.

U.S. ARMY EDGEWOOD CHEMICAL BIOLOGICAL CENTER

DRAWINGS

136–41–352 – Inspection Equipment Apparatus, Agent Testing, Ammonia Content in Charcoal, Q3 Assembly

# **PUBLICATIONS**

## **INSTRUCTION MANUALS**

IM 136-300-34 – Instruction Manual for Installation, Operation, and Maintenance of Apparatus, Agent Testing, Ammonia Content in Charcoal, Q3

(Copies are available from Commander, U.S. Army Edgewood Chemical Biological Center, ATTN: AMSSB-REN-SN, Aberdeen Proving Ground, MD 21010-5424.)

**2.3 Non-Government publications.** The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DOD adopted are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS are the issue of the documents cited in the solicitation (see 6.2).

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM D 1193	 Reagent Water
ASTM D 2854	 Apparent Density of Activated Carbon
ASTM D 2862	 Particle Size Distribution of Granular Activated Carbon
ASTM D 2867	 Moisture in Activated Carbon
ASTM D 3802	 Ball–Pan Hardness of Activated Carbon

(Application for copies should be addressed to ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428–2959.)

**2.4 Order of precedence.** In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

## **3. REQUIREMENTS**

**3.1 Materials.** The active materials on the carbon shall be composed of and limited to copper, silver, zinc and molybdenum salts and TEDA. The base carbon shall be limited to steam activated carbon produced from bituminous coal.

**3.2 Chemical and physical characteristics.** The carbon shall conform to the chemical and physical characteristics specified in table I.

Characteristic	Requi	Requirement	
Characterisuc	Minimum	Maximum	paragraph
Moisture content, percent by weight		2.5	4.3.6.2
Apparent (bulk) density, g/mL		0.68	4.3.6.3
Hardness	85		4.3.6.4
Ammonia, mg/L of air/100 mL carbon		0.0010	4.3.6.5
Copper content, percent by weight		6.0	4.3.6.6
Silver content, percent by weight	0.030	0.1	4.3.6.6
Zinc content, percent by weight		6.0	4.3.6.6
Molybdenum content, percent by weight		2.5	4.3.6.6
TEDA content, percent by weight		3.5	4.3.6.7

TABLE I. Chemical and physical characteristics

**3.2.1 Particle size distribution.** The carbon shall conform to the particle size distribution requirements in table II.

Particle size	Percent by weight of original sample
Passing a 2.36 mm (No. 8) sieve	100
Retained on a 1.70 mm (No. 12) sieve	0-2
Retained on a 1.18 mm (No. 16) sieve	10-30
Retained on a 850 mm (No. 20) sieve	40-65
Retained on a 600 mm (No. 30) sieve	10-35
Passing a 600 mm (No. 30) sieve	2.8 (maximum)*
Passing a 212 mm (No. 70) sieve	0.30 (maximum)

 TABLE II. Particle size distribution

\*2.8 includes the material also passing through the No. 70 sieve

**3.2.2 Gas sorption.** The carbon shall conform to the gas sorption time (life) requirements in table III when the challenge concentration  $(mg/m^3)$  of hydrogen cyanide is 4000, cyanogen chloride is 4000, phosgene is 20000, and dimethylmethylphosphonate is 3000.

TABLE III.	Gas sorption time requirements in minutes
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Agent	Unaged Minimum	Unaged Min Avg	Aged* Minimum	Aged* Min Avg
Hydrogen cyanide (AC)	26	28	_	-
Cyanogen chloride (CK)	44	55	39	45
Phosgene (CG)	19	25	_	_
Dimethylmethylphosphonate (DMMP)	175	—	_	_

\*Age carbon as specified in 4.3.6.8.1.

### 4. VERIFICATION

**4.1 Classification of inspections.** The inspection requirements specified herein are classified as follows:

- (a) First article inspection (see 4.2)
- (b) Conformance inspection (see 4.3)

#### 4.2 First article inspection.

**4.2.1 Sample.** A first article sample shall not be required unless it is specified in the acquisition document (see 6.2). When a first article inspection is required, the quantity sampled shall be manufactured using the same methods, materials, equipment, and processes as will be used during regular production. When a first article is required, sampling from the first article material for inspection and testing shall be the same as that which is required for a regular production lot.

**4.2.2 Inspections to be performed.** As determined by the Government, sample first article items may be subjected to any or all of the examinations and tests specified in this specification.

**4.2.3** Acceptance criteria. If any first article sample item fails to comply with any of the applicable requirements, the first article sample shall be rejected. The Government reserves the right to terminate inspection upon any failure to comply with any of the requirements. The contractor shall obtain written approval from the contracting activity prior to proceeding with regular production.

#### 4.3 Conformance inspection.

**4.3.1 Lotting.** A lot shall consist of a quantity of carbon produced by one manufacturer without change in materials, at one plant, and by one continuous process or in successive increments by the same intermittent process. For the purpose of this specification, the following lot size and weight categories are defined:

- a. Small size lot: total weight is under 250 kilograms (kg).
- b. Medium size lot: total weight is between 250 kg and 9 metric tons.
- c. Large size lot: total weight is over 9 metric tons.

Producing numerous small size lots should be avoided if possible.

### 4.3.2 Sampling

**4.3.2.1 Sampling, small or medium size lot.** Four (4) representative containers of carbon shall be randomly selected from each lot and a specimen of carbon shall be taken from each of those 4 containers and placed in separate clean dry containers in such a way as to minimize the exposure time of the carbon to the atmosphere. The size of each specimen shall be sufficient

to provide all samples required for tests that are to be performed by the manufacturer in accordance with the terms of the contract (see 6.2) and to provide specimens for shipment to the designated government laboratory for required government testing. The containers shall then be sealed with an airtight closure and labeled to identify the manufacturer, lot and container represented. One sample shall be taken from each of the four specimens of carbon for testing as specified in 4.3.6.6 and 4.3.6.7. Two samples shall be taken from each specimen for testing in accordance with 4.3.6.1 through 4.3.6.5. The specimen containers shall be kept sealed except when samples are being removed for testing. A quantity of 4.5 to 5 kg from each of the 4 specimens shall be sealed in their individual containers and shipped to the designated government test laboratory for gas life testing in accordance with 4.3.6.8. One sample shall be taken from each of the 4 specimens for each of the five required chemical agent tests performed by the laboratory in accordance with 4.3.6.8. The five required chemical agent gas sorption tests include four tests on unaged carbon using AC, CK, CG, and DMMP and one test on aged carbon using CK.

**4.3.2.2 Sampling, large size lot.** The number of representative containers of carbon taken from each lot shall be 4 plus one extra container for every 2.25 metric ton increment of production that is over 9 metric tons. Likewise, the number of carbon portions and samples tested shall be as in 4.3.2.1 except there shall be one extra sample for every 2.25 metric ton increment of production that is over 9 metric tons. See table IV.

Increment number	Production or lot size (metric tons)	Base	Sample size Addition	e Total
1	9.01 to 11.25	4	1	5
2	11.26 to 13.50	4	2	6
3	13.51 to 15.75	4	3	7
4	15.76 to 18.0	4	4	8
k	9.01+(k-1)*2.25 to 9.0+k*(2.25)	4	k	4+k

TABLE IV. <u>Sample size for large lots</u>

**4.3.3 Inspection procedure.** The lot represented shall be rejected when nonconformance to a critical characteristic is found except for double sampling provisions in 4.3.6.8. Sample items shall be examined and tested in accordance in 4.3.6. Should any sample tested in accordance with 4.3.6 fail to comply with the requirements in tables I, II and III, the lot shall be rejected.

**4.3.4 Inspection characteristics.** Critical characteristics are characteristics whose nonconformance to specified requirements is likely to result in hazardous or unsafe conditions for individuals who use or maintain the product. Characteristics whose nonconformance to specified requirements is likely to prevent performance of the tactical function of a major end item are also critical characteristics. Major characteristics are characteristics whose nonconformance to specified requirements is likely to result in failure or to reduce materially the usability of the

item for its intended purpose. Minor characteristics are characteristics whose nonconformance to specified requirements is not likely to reduce materially the operation or usability of the item for its intended purpose.

**4.3.5 Classification of characteristics.** Conformance tests are specified in 4.3.6. Acceptance criteria is to accept on no failures and reject on one or more failures for all characteristics (retest per 4.3.6.8 is allowed). Particle size, moisture content, bulk density, hardness, ammonia content, metal content in 4.3.6.6, and TEDA content in 4.3.6.7 are considered major characteristics. Sorbent gas life in 4.3.6.8 is considered a critical characteristic.

**4.3.6 Tests.** Water in accordance with ASTM D 1193, type III and reagent grade chemicals shall be used throughout the tests. Where applicable, blank determinations shall be run and corrections applied where significant. Tests shall be conducted as follows:

**4.3.6.1 Particle size.** Using the sieves specified in table II, determine the particle size distribution of two samples from each specimen container (see 4.3.2) in accordance with ASTM D 2862. Calculate the average weight percents of the particle size distribution for each container based on the average of two samples and compare average weight percents against requirements in table II.

**4.3.6.2 Moisture content.** Determine the moisture content of two samples drawn from each specimen container in accordance with the procedure in 4.3.6.2.1. Calculate the average moisture content for each container based on the average of two samples and compare such average against requirement in table I.

**4.3.6.2.1 Oven drying.** Take two samples of carbon from each of the four specimen containers. The weight of each carbon sample is specified in the oven drying method in ASTM D 2867. Avoid taking samples from the top layer that is exposed to air. Follow the oven drying procedures in ASTM D 2867 except use an oven temperature of 103° to 107° C and a drying time of 3 hours.

**4.3.6.3 Apparent (bulk) density** Determine the apparent (bulk) density of two samples from each specimen container in accordance with ASTM D 2854. Calculate the average bulk density for each container based on the average of two samples and compare such average against requirement in table I.

**4.3.6.4 Hardness.** Determine the hardness (resistance to particle size degradation) of two samples from each specimen container in accordance with ASTM D 3802. Calculate the average hardness for each container based on the average of two samples and compare such average against requirement in table I.

**4.3.6.5 Animonia content.** Determine the ammonia content of two samples from each specimen container using the Q3 Ammonia Content in Carbon Testing Apparatus (Drawing 136-41-352 and IM 136-300-34) or using an equivalent method approved by the Government. Calculate the average ammonia content for each container based on the average of two samples and compare such average against requirement in table I.

**4.3.6.6 Total copper, silver, zinc and molybdenum content.** Determine the total content of each metal on the carbon for each specimen container as follows (see 6.6):

**4.3.6.6.1 Extraction.** Extract the metals from the carbon sample by one of the following procedures:

(a) By acid reflux. Obtain a 5 to 10 g representative sample of carbon using blending and riffling techniques. Grind the sample until 95 percent or more will pass through a 325-mesh screen. Dry the sample in an oven at  $105 \pm 2$ \_C for not less than 3 hours and cool in a desiccator to room temperature. Weigh  $1.0000 \pm 0.0001$  g of carbon into a 250 milliliter (mL) flask. To the flask add 100 mL of reagent water and 65 mL of 20 percent nitric acid solution. Attach the flask to a reflux condenser and boil for 1 hour. Remove the flask from the heater and condenser. Filter the hot solution by vacuum through an 8 to 15 micrometer filter membrane. Rinse the flask with several portions of reagent water. Rinse the carbon on the filter with three 5 mL portions of 20 percent nitric acid solution. Quantitatively transfer the filtrate to a 500 mL volumetric flask and allow to cool to room temperature. Dilute to the mark with reagent water and mix thoroughly. The extract solution may require further dilution depending on the instrumentation used in the analysis (4.3.6.6.2).

(b) By microwave treatment. Obtain a representative sample of carbon using blending and riffling techniques. Dry the sample in an oven at  $105\pm2$ \_C for not less than 3 hours and cool in a desiccator to room temperature. Weigh 0.2000 g of carbon to the nearest 0.0001 g and place in a lined microwave digestion vessel. Add 10 mL of 20 percent nitric acid and seal the vessel. Place the vessel in a holder in the microwave oven and attach vent tube to the collection container. Operate the microwave for  $20\pm1$  minutes. At the conclusion of the digestion, open the digestion vessel and quantitatively transfer the solution into a 100 mL volumetric flask, dilute to the mark with reagent water and mix thoroughly. The extract solution may require further dilution depending on the instrumentation used in the analysis (4.3.6.6.2).

**4.3.6.6.2 Analysis.** Analyze the extract solution for the concentration of each of the 4 metals by atomic absorption spectrophotometry, inductively coupled plasma or by an equivalent method approved by the Government. Note that the acid content of the standards used in each metal determination must be the same as the acid content of the solution being analyzed. Following the analysis, calculate the weight percent of each of the 4 metal impregnants on the carbon.

**4.3.6.7 TEDA content.** Determine TEDA content of the carbon using the following method or equivalent method approved by the Government.

**4.3.6.7.1 TEDA extraction.** Weigh  $1.000 \pm 0.010$  g of carbon directly into a 15 mL, flat—bottomed flask. Add 10 mL methanol from a measuring cylinder and fit the flask with a reflux condenser. Support the flask and condenser above a hot plate and reflux for 15 to 20 minutes. Remove from the source of heat and allow to cool to room temperature. Remove the reflux condenser and carefully transfer the extract by decanting into a 50 mL volumetric flask. Repeat the extraction process three more times adding the methanol extracts to the volumetric flask. Finally rinse the carbon with 5 mL methanol adding the washing to the volumetric flask.

### 4.3.6.7.2 Preparation of solutions.

(a) **Preparation of tetramethylenediamine (TMEDA) internal standard solution.** Using a dropper pipette, weigh, to the nearest 0.001 g, 0.150 to 0.200 g (X) of TMEDA into a 50 mL volumetric flask. Make up to the mark with methanol and mix thoroughly.

(b) **Preparation of standard test solutions.** Prepare three standard solutions by weighing into three 50 mL volumetric flasks  $0.015 \pm 0.001$  g,  $0.030 \pm 0.001$  g and  $0.045 \pm 0.001$  g TEDA, respectively. Add, by means of a volumetric pipette, 5 mL of TMEDA internal standard solution to each flask and make up to the mark with methanol. Mix thoroughly.

(c) **Preparation of sample test solution.** To the flasks containing the extracts and washing from the carbon samples (4.3.6.7.1), add, by means of a volumetric pipette, 5 mL of TMEDA internal standard solution. Make up to the mark with methanol and mix thoroughly.

**4.3.6.7.3 Gas chromatographic analysis.** Analysis of the sample and standard test solutions shall be made using a gas chromatograph (see 6.3).

**4.3.6.7.4 Calculations.** Measure the areas of TEDA and TMEDA peaks for sample and standard test solutions, as they appear. Determine the ratio of areas, TEDA/TMEDA for each solution. Repeat sample injection twice more ensuring that the area ratios do not vary by more than 5 percent. Determine the average area ratio for each solution. Determine the concentration ratio of TEDA/TMEDA in each standard. (Note that TMEDA concentration in all solutions is X/10 grams per 50 mL). Plot concentration ratios versus area ratios for the three standard solutions using the origin as a fourth data point for calibration. Using linear regression, calculate the slope, M, of the calibration graph. From the area ratio measured for the sample solution, determine the concentration ratio, TEDA/TMEDA as follows:

**Concentration ratio = Area ratio x M** 

Weight of TEDA determined = Concentration ratio x  $\frac{X}{10}$  grams

Percent TEDA on carbon =  $\frac{\text{Weight of TEDA determined}}{\text{original weight of carbon}} \times 100$ 

**4.3.6.8 Standard gas sorption tests.** Gas lives shall be determined using test apparatus approved by the Government and shall be tested under the conditions specified in table V. The tests shall be performed in cylindrical containers using the conditions given in table VI. Fill the test container with the test sample using a procedure such that the packing density is equivalent to that obtained using ASTM D 2854. In addition, the filling procedure used shall be such that segregation of the carbon particles by size does not occur. For each chemical agent and aging condition listed in table III, determine the minimum gas life and average gas life for each sample group. The exact number of samples in a sample group shall be determined from lot size

(see 4.3.2.1 and 4.3.2.2). If all samples meet the minimum gas life requirement and the minimum average gas life requirement for each sample group, the lot shall be accepted. If only one sample in a sample group is responsible for not meeting the minimum gas life requirement or minimum average gas life requirement, a second sample group of the same size from the same container shall be tested. If no more than one sample is responsible for not meeting minimum gas life requirement and the minimum average gas life requirement for the first sample group, and all the samples from the second sample group meet the minimum gas life requirement and the minimum average gas life requirement for each chemical agent, the lot shall be accepted.

**4.3.6.8.1** Artificial aging. Prior to aging, the carbon shall be equilibrated for 16 hours at a relative humidity (RH) of  $80\pm 3$  percent, temperature of  $24\pm 3$ \_C, and a flow rate approximately the test flow rate. After equilibrating, place the carbon in open containers and age for 7 days in air at  $80\pm 3$  percent RH and a temperature of  $45\pm 1$ \_C. After aging, the carbon shall be re—equilibrated at  $80\pm 3$  percent RH at room temperature before testing with CK as specified in table V.

Gas	Concentration* (mg/m <sup>#</sup> )	Endpoint (mg/m <sup>#</sup> )	Relative Humidity (percent)	Temperature (° C)
AC	$4000 {\pm} 400$	5**	80±3***	24±3°C
СК	$4000 {\pm}400$	5	80±3***	$24\pm3^{\circ}C$
CG	$20000 \pm 1000$	8	$50 \pm 5^{****}$	24±3°C
DMMP	$3000{\pm}400$	0.04	<15****	24±3°C

### TABLE V. Standard gas sorption test conditions

\* The variations to the nominal concentrations as indicated above will be allowed in conducting the test. However, the gas life results obtained for the test shall be corrected to the nominal values using the following equation:

Corrected gas life = (Measured gas life) 
$$\begin{pmatrix} 1 \\ -2 \end{pmatrix}$$

- \*\* The endpoint time is the time when the effluent concentration rises to the specified endpoint concentration (5 mg/m<sup>3</sup> for AC and CK).
- \*\*\* Carbon shall be equilibrated at 80±3 percent relative humidity and 24±3° C for 16 hours at approximately test flow rate
- \*\*\*\* "As received" carbon will be used for these tests.

### TABLE VI. Test parameters for gas sorption measurements

Inside diameter of container, minimum, centimeters	4.0
Linear flow, centimeters per second	$5.90\pm0.05$
Bed depth, centimeters	$2.0 \pm 0.1$

### 5. PACKAGING

**5.1 Packaging.** For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of materiel is to be performed by DoD personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity within the Military Department or Defense Agency, or within the Military Department's System Command. Packaging data retrieval is available from the man-

aging Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

# 6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

**6.1 Intended use.** The carbon covered by this specification is intended for use in military nuclear biological chemical filters.

6.2 Acquisition requirements. Acquisition documents should specify the following:

- (a) Title, number, and date of this specification
- (b) Issue of DODISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced
- (c) Whether first article testing is required
- (d) Contractor and government responsibilities for inspection and testing (see 6.5)
- (e) Address(es) for shipment of samples for Government testing and time allowed for the Government to complete inspection/testing and report results
- (f) Packaging requirements.

**6.3 Gas chromatograph.** The column description and parameters provided below have been found to achieve good gas chromatographic separations for the TEDA content test:

25 meter, 0.32 mm id BP 10 capillary column Split-splitless injector, at 160\_C, operated in splitless mode Flame ionization detection; detector temperature 200\_C Inlet pressure 6 psi Carrier gas – helium GC oven: 65\_C for 1 minute 65\_C-170\_C at 30\_C/minute 170\_C for 2 minutes Injection volume: 1 microliter

**6.4 Storage of containers with carbon.** After containers are opened and the seal broken, the specimen containers should be stored in a controlled storage area with a relative humidity of 40 percent or less.

**6.5 Responsibilities for inspection and testing.** The contractor will normally be responsible for all inspections and tests except for the chemical agent gas life tests (4.3.6.8); however, the government has the right to conduct any or all of the inspections and tests at any time whether or not it has responsibility for them under the contract. Both the government and the contractor should perform all inspections and tests for which they are assigned responsibility under the contract regardless of what inspection and testing the other party may perform for their own purposes.

**6.6 Alternative verification methods.** Alternate verification methods including supporting rationale may be submitted by the contractor to the procuring contracting officer for evaluation and approval by the Government.

**6.7 Supersession.** This specification supersedes Purchase Description EA–DTL–1704A, dated 22 January 1999.

### 6.8 Subject term (key word) listing.

Sorbent

Custodian: Army – EA Navy – SH Air Force – 68 Preparing activity: Army – EA (Project 6810–1703)

Review activities: Air Force - 99 DLA - GS

Industry associations: Calgon Carbon, Inc.

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