

P-C-111D  
 AMENDMENT-2  
November 19, 1982  
 SUPERSEDING  
 AMENDMENT-1  
 1 June 1978



FEDERAL SPECIFICATION

CARBON REMOVING COMPOUND

This amendment, which forms a part of P-C-111D, dated February 11, 1977, is approved by the Commissioner, Federal Supply Service, General Services Administration, for the use of all Federal agencies.

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Table I, delete in its entirety and substitute the following:

TABLE I. Chemical requirements

<u>Characteristics</u>	<u>Requirements</u>	<u>Test Paragraphs</u>
Chlorine derivatives	Negative	4.5.1
Aromatics, more volatile <sup>1/</sup>	Negative	4.5.2
Water, percent by volume of compound	55 ± 3	4.5.3
Monoethanolamine, percent by volume	21.0 min.	4.5.5
Ethylene glycol monoethyl ether	Negative	4.5.5
Ethylene glycol monobutyl ether, percent by volume	9.0 min.	4.5.5
Diethylene glycol monomethyl ether, percent by volume	5.0 min.	4.5.5
Diethylene glycol monobutyl ether, percent by volume	3.0 min.	4.5.5
Phenolic and cresylic acids or their salts	Negative	4.5.18

<sup>1/</sup> Benzene, toluene, xylene and highflash naphtha with flashpoint in range of 100-110°F are not permitted. Certain high flash naphthas with relatively high flashpoint (140°F) are permitted.

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Paragraph 3.4, delete in its entirety and substitute the following:

"3.4 Flashpoint. The flashpoint of the compound shall be above 210°F when tested as specified in 4.5.7 and in accordance with FED-STD-313."

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Paragraph 4.4.2, line 3, change "4.5.4" to "4.5.5".

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Paragraph 4.5.4, delete in its entirety.

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Paragraph 4.5.5, delete in its entirety and substitute the following:

"4.5.5 Clycol ethers and monoethanolamine.

4.5.5.1 Ethylene glycol monoether, ethylene glycol monobutyl ether, diethylene glycol monomethyl ether, diethylene glycol monobutyl ether, and monoethanolamine.

4.5.5.1.1 Preparation of sample. Pipette 20 mL of the carbon removing compound and 2 mL of hexyl carbitol (internal standards) into a 100 mL beaker. Mix with a glass stirring rod. Add 10 g of anhydrous  $K_2CO_3$  and stir until dissolved. This material will at first become pasty; but, with a few minutes of further stirring, will go into solution. Transfer the solution to a 125 mL separatory funnel and let separate. Discard the lower layer and transfer the upper layer to a 50 mL glass-stoppered erlenmeyer flask. Add 10 mL of n-butanol to the flask and mix gently. Add 20 g of anhydrous  $K_2CO_3$  and shake vigorously for 2 minutes. Let settle and decant into a centrifuge tube. Stopper and centrifuge until solution is clear.

4.5.5.1.2 Column preparation. Prepare a 2-column system as follows:

4.5.5.1.2.1 Precolumn. Pack a 4-inch long by 1/8 inch outside diameter, teflon-coated, stainless steel column with 20% SE30 on 60-80 mesh chromosorb WAW.

4.5.5.1.2.2 Second column. Pack a 6-foot long by 1/8 inch outside diameter, teflon-coated, stainless steel column with 10% Reoplex 400 on 80-100 mesh chromosorb WHP.

4.5.5.1.3 Apparatus. The apparatus shall be a gas chromatograph equipped with a thermal conductivity detector and controls for obtaining the following operating conditions.

Thermal conductivity detector, °C	300
Injection port, glass lined, °C	250
Carrier gas (helium), mL/min	25
Column heating rate, °C/min	1
Initial column temperature °C	125
Final column temperature, °C	165
Length of run, min.	40

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4.5.5.1.4 Analysis of sample. With a 10 uL syringe, inject a 2uL sample into the injection port of the gas chromatograph. Identify peaks on the chromatogram from predetermined retention times. The following retention times are given as approximate: ethylene glycol monoethyl ether - 2.0; ethylene glycol monobutyl ether - 3.9; diethylene glycol monomethyl ether - 9.1; diethylene glycol monobutyl ether - 17.5; monoethanolamine - 4.6.

Calculate concentrations in percent by volume using the relationship  $C = \frac{ABF}{D}$

Where A is the area under the peak in question, B is the percent of internal standard, D is the area under the internal standard peak, and F is the correction factor determined from solutions with known concentrations.

Correction factors should be checked periodically with a sample on hand for this purpose. The 4-inch precolumn should be replaced when necessary to maintain reproducibility and to prevent serious peak tailing.

The chromatogram shall be examined to determine the absence of ethylene glycol monoethyl ether."

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Add the following paragraph:

"4.5.18 Phenolic or cresylic acids or their salts. To 5 mL of the compound in a 25 mL graduated cylinder, add 1 mL Claissen's alkali. (Claissen's alkali: Dissolve 350 g potassium hydroxide in 250 mL water and dilute to one liter with methyl alcohol.) Shake the cylinder thoroughly. Add 5 mL ethyl ether and shake again. Allow to separate into 2 sharp layers. The upper ether layer shall be removed with a dropping pipette and discarded. Wash the bottom layer with 5 mL ethyl ether and separate as before.

Acidify the lower layer with 6 mL of 6N hydrochloric acid, add 5 mL of ethyl ether, and shake the cylinder. Transfer the top ether layer to a test tube. Wash the ether layer with 5 mL water, transfer ether to another tube, and wash with 3 mL of 3 percent aqueous sodium bicarbonate solution. The ether layer shall be transferred to another tube and evaporated over a steam bath. To the residue add 2 mL of water. The tube shall be shaken thoroughly. Draw off 2 drops of the saturated water into another tube and add 3 drops of ferric chloride solution, the tube being shaken and the color observed after the addition of each drop. (Ferric chloride solution: Dilute 3 drops of 10% aqueous ferric chloride to 1 mL with water.) Any color other than yellow or orange yellow shall be considered as a positive test for phenolic or cresylic type acids."

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**MILITARY INTERESTS:**

Custodians

Army -- MR  
Navy -- AS  
Air Force - 11

Review activities:

Army -- MD, ME, AR  
Air Force -- 99

Agent:

Army -- ME

**CIVIL AGENCY COORDINATING ACTIVITIES:**

GSA - FSS

**PREPARING ACTIVITY:**

Army -- MR

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