

CHANGE NOTICES ARE NOT CUMULATIVE
AND SHALL BE RETAINED UNTIL SUCH
TIME AS THE STANDARD IS REVISED.

FED. TEST METHOD STD. 191A
July 20, 1978
CHANGE NOTICE 1
March 19, 1985

FEDERAL TEST METHOD STANDARD

TEXTILE TEST METHOD STANDARD

The following changes, which form a part of FED.
TEST METHOD STD. 191A, dated July 20, 1978, are
approved by the Assistant Administrator, Office
of Federal Supply and Services, General Services
Administration, for the use of all Federal
agencies.

REMOVE : Standard Test Method 2015 of July 20, 1978

ADD : Revised Tentative Standard Test Method 2015.1 of March 19, 1985

MILITARY INTEREST:

CIVIL AGENCY COORDINATING ACTIVITIES:

Custodians

Army - GL
Navy - NU
Air Force - 11

GSA - FSS
HHS - NIH

PREPARING ACTIVITY

Army - GL

Review activities

Army - AR, MD, ME, MI, TE
Navy - AS, SH
Air Force - 03, 45, 82, 99

Project No. 83GP-0019

RETAIN THIS CHANGE NOTICE AND INSERT BEFORE THE TABLE OF CONTENTS

FSC 83GP

METHOD 2015.1
March 19, 1985
SUPERSEDING METHOD 2015
DATED July 20, 1978

SODIUM 5-CHLORO-2-[4 CHLORO-2-[3-(3, 4 DICHLOROPHENYL)-
UREIDO]-PHENOXY] BENZENESULFONATE CONTENT

1. SCOPE

1.1 This method is intended for determining the sodium 5-chloro-2 [4-chloro-2-[3-(3, 4-dichlorophenyl)-ureido]-phenoxy) benzenesulfonate content of woolen textile materials that have been treated with this compound as a mothproofing agent (see 7.1 and 7.2).

2. TEST SPECIMEN

2.1 All Wool. When the material to be tested is 100 percent wool, the specimen shall weigh 500 ± 50 mg.

2.2 Polyester and wool blend. When the material to be tested is a blend of polyester and wool, the specimen shall weigh 1000 ± 100 mg.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the procurement document, two specimens shall be tested from each sample unit.

4. APPARATUS AND REAGENTS

4.1 Apparatus.

4.1.1 Electric heater with variable control.

4.1.2 Heat resistant glass flask. A 250 mL round bottom single neck, alkali resistant, heat resistant glass flask.

4.1.3 250 mL trap bulb and connecting arms (see figure 2015A).

4.1.4 Graham condenser (jacket 300 mm long)

4.1.5 Funnel.

4.1.6 Flasks. Six (6) 100 mL volumetric, low actinic red or light sensitive.

4.1.7 Flasks, volumetric 200 mL, 250 mL, 500 mL, 1000 mL.

4.1.8 Pipettes.

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4.1.9 Weighing bottles.

4.1.10 Boiling stones.

4.1.11 Spectrophotometer. A spectrophotometer having a maximum transmission at approximately 550 nanometers.

4.1.12 Silicone stopcock lubricant.

4.1.13 Silicone antifoam.

4.1.14 Analytical balance.

4.1.15 Air oven.

4.1.16 Soxhlet extractor.

4.1.17 Standard laboratory dessicator with drying medium.

4.2 Reagents. All reagents shall be prepared with distilled or deionized water.

4.2.1 3.4 Dichloroaniline Pure ($C_6H_5Cl_2$ MW 162.02) is preferable to store in a refrigerator.

4.2.2 2.5 N potassium hydroxide. Dissolve 165g ($\pm 3g$) potassium hydroxide 85 percent into 1 liter volumetric flask, fill to mark with water.

4.2.3 5.0 N hydrochloric acid. Dilute 570g $\pm 10g$ HCL 35 percent into 1 liter volumetric flask, fill to mark with water.

4.2.4 0.1 N sodium nitrite. Dissolve 6.9g ($\pm 0.1g$) $NaNO_2$ into 1 liter volumetric flask and dilute to mark with water. This solution will stay stable for at least 2 months.

4.2.5 0.2 N sulfamic acid. Dissolve 9.7g ($\pm 0.2g$) H_2NSO_3H into 500 mL volumetric flask and fill to mark with water: This solution will stay stable for at least 2 months.

4.2.6 Acetic acid 50 percent. Dilute 500 mL glacial acetic acid with 500 mL water.

4.2.7 1.0 percent N-(1-naphthyl)-ethylenediamine-dihydrochloride. (Dissolve 1g ($\pm 0.02g$) $C_{10}H_7NHCH_2\checkmark 2HCL\checkmark CH_3OH$ into 100 mL volumetric flask and fill to mark with water. This solution has to be stored in a dark glass bottle. Initial solution is water clear and should be renewed as soon as it changes color.

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5. PROCEDURE

5.1 Preparation of standard reference solution.

5.1.1 Dichloroaniline-hydrochloride stock solution. On an analytical balance, weight out 200 mg (± 0.2 mg) pure 3, 4 dichloroaniline and place in a 1000 mL volumetric flask. To that, add 2 mL 5 N hydrochloric acid and 100 mL distilled water. Dissolve by heating slowly in a water bath and fill to the mark with distilled water. The solution should be good for 2 to 4 weeks.

5.1.2 Standard reference solution. Put 25 mL of the standard stock solution into 500 mL volumetric flask and fill to mark with water. Place 5, 10, 15, 20, and 25 mL of this dilution in 100 mL light sensitive volumetric flasks and add to each of them 1 mL 5 N hydrochloric acid.

5.1.3 Diazotization development and spectrophotometric measuring of standard reference solution: Add 5 mL 0.1N sodium nitrate to each of the 100 mL light sensitive volumetric flasks (see 5.1.2). Shake and let rest for at least 2 minutes. Destroy the excess nitrite by adding 5 mL 0.2 N sulfamic acid and shake vigorously (be sure to agitate until bubbling subsides because bubbles in cuvette cause erroneous readings).

After at least 2 minutes, dilute the diazotizing mix with 40 mL acetic acid (50 percent), shake and add 5 mL N-(1 Naphthyl) ethylenediamine dihydrochloride solution (1 percent). Add water up to the 100 mL mark, invert six times (shake) and let rest for 10 minutes (measuring solution). (At 20°C, the final color strength is reached after only 5 minutes; if the solution is not exposed to very bright light the strength of color will not change for at least 12 hours). Place in cuvette and measure absorbance of each dilution with the spectrophotometer and read its absorbance at the maximum (550 nanometers). Draw a calibration curve of these measured absorbance against mg of dichloroaniline and mg moth proofing agent. A sample calibration curve is included (see chart on Figure 2015B).

5.2 Weight of dry specimen: The specimen shall be placed in a weighing bottle, dried in a circulating oven at least 1 hour at a temperature of 221° to 230°F, cooled in a desiccator, and weighed. Repeat this cycle until a weight is obtained that is constant to ± 0.001 g. This is the "weight of dry specimen".

5.3 Testing of specimens containing 100 percent wool. Cut specimen into small pieces and place in a 250 mL round bottom distillation flask with a few boiling stones. Add 180 mL 2.5 N potassium hydroxide solution and 3 drops of antifoam. Carefully grease the joints with silicone grease and assemble the apparatus as shown in Figure 2015C.

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Heat the flask content until it boils and distills about 150 mL (approximately 1 hr.). Use a 200 mL volumetric flask containing 10 mL 5 N hydrochloric acid and having a mark at 150 mL as distilling receiver. Avoid spilling any of the alkaline solution or foam into the receiver. After the distillation, take the 200 mL volumetric flask containing the distillate combined with hydrochloric acid, add distilled water at room temperature up to the 200 mL mark and mix it (=specimen stock solution). Place a 30 mL aliquot of specimen stock solution in 100 mL light sensitive flask and proceed as in (5.1.2) Diazotation.

5.4 Testing specimens of polyester/wool blends. The specimen shall be extracted with chloroform for 100 cycles in a Soxhlet extractor. Dry the specimen and continue with the procedure described in 5.3

5.5 Calculations.

5.5.1 Specimens containing 100 percent wool. The percent mothproofing agent on the wool fiber shall be calculated from the absorbance measurements as follows:

$$\text{Percent mothproofing agent} = \frac{M \times 100}{W_2 \times .15}$$

Where: M = Amount of moth proofing agent (mg) contained in the measuring solution which equals the measured absorbance. It will be read off the Calibration Curve (see Fig 2015B)

W_2 = weight of dry specimen

.15 = Constant value when using 30 mL aliquot sample

5.5.2 Specimens containing polyester/wool blend. The percent mothproofing agent on the wool fiber shall be calculated from absorbance measurements as follows:

$$\text{Percent mothproofing agent} = \frac{M \times 100 \times 1.09}{W_2 \times .15 \times P}$$

Where: P = Proportion of wool in sample, expressed as a decimal to the nearest 0.01.

6. REPORT

6.1 The percent mothproofing agent content of a sample unit shall be reported as the average of the values obtained for the specimens tested and shall be reported to the nearest 0.1 percent.

6.1.1 The individual values for each individual specimen used to calculate the average shall be reported to the nearest 0.01 percent.

7. NOTES

7.1 This method determines the content as an 80 percent active as is material.

7.2 This mothproofing formulation may be obtained under the trade name of Mitin FF High Cone. from Ciba-Geigy Corp., Dyestuffs and Chemicals Division, Swing Road, Greensboro, NC 27409 or under the name of Intracide M from Dyes and Chemicals Division, Crompton and Knowles Corp., Route 208, Fair Lawn, NJ 07410.

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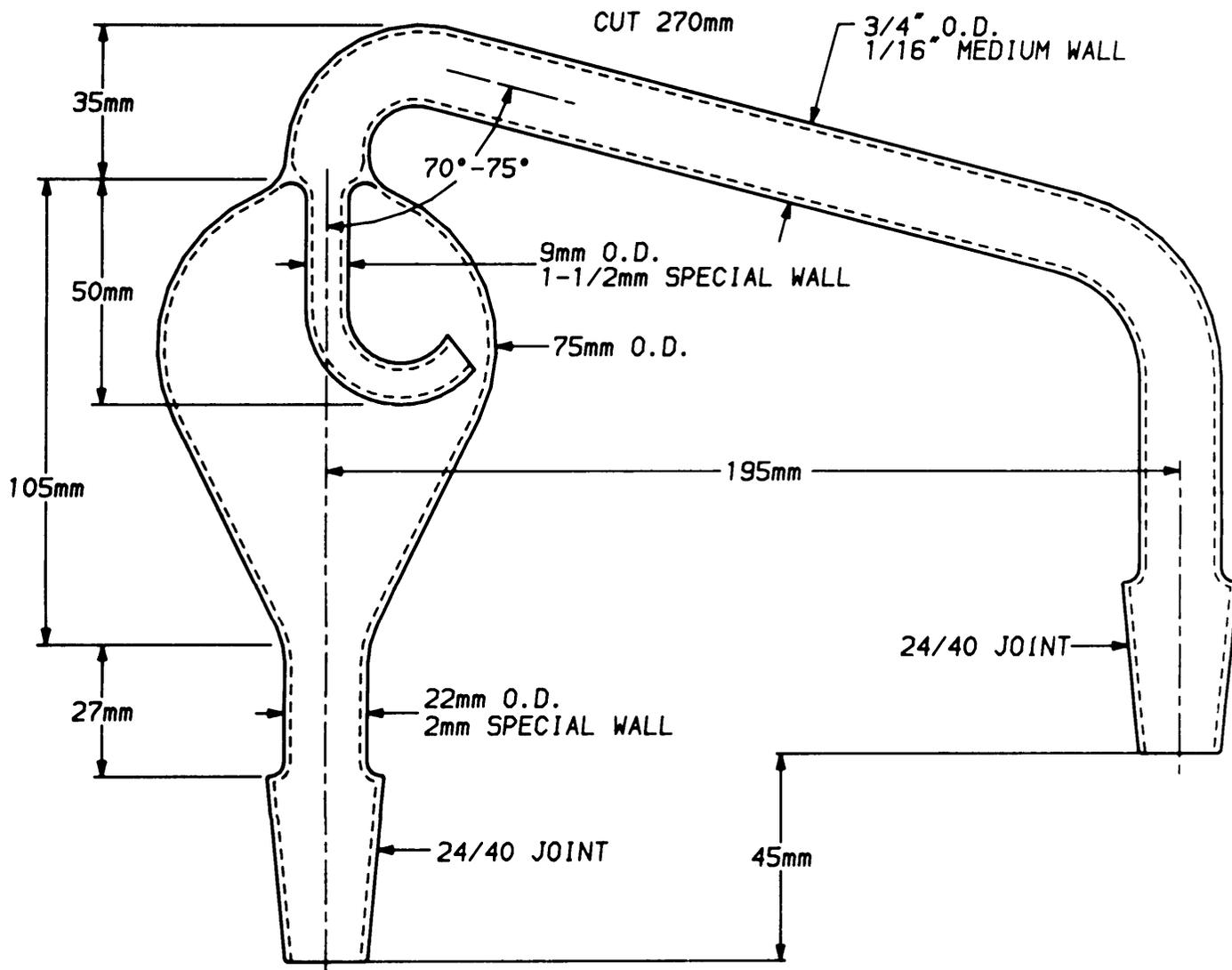


FIG. 2015A. TRAP BULB AND CONNECTING ARM

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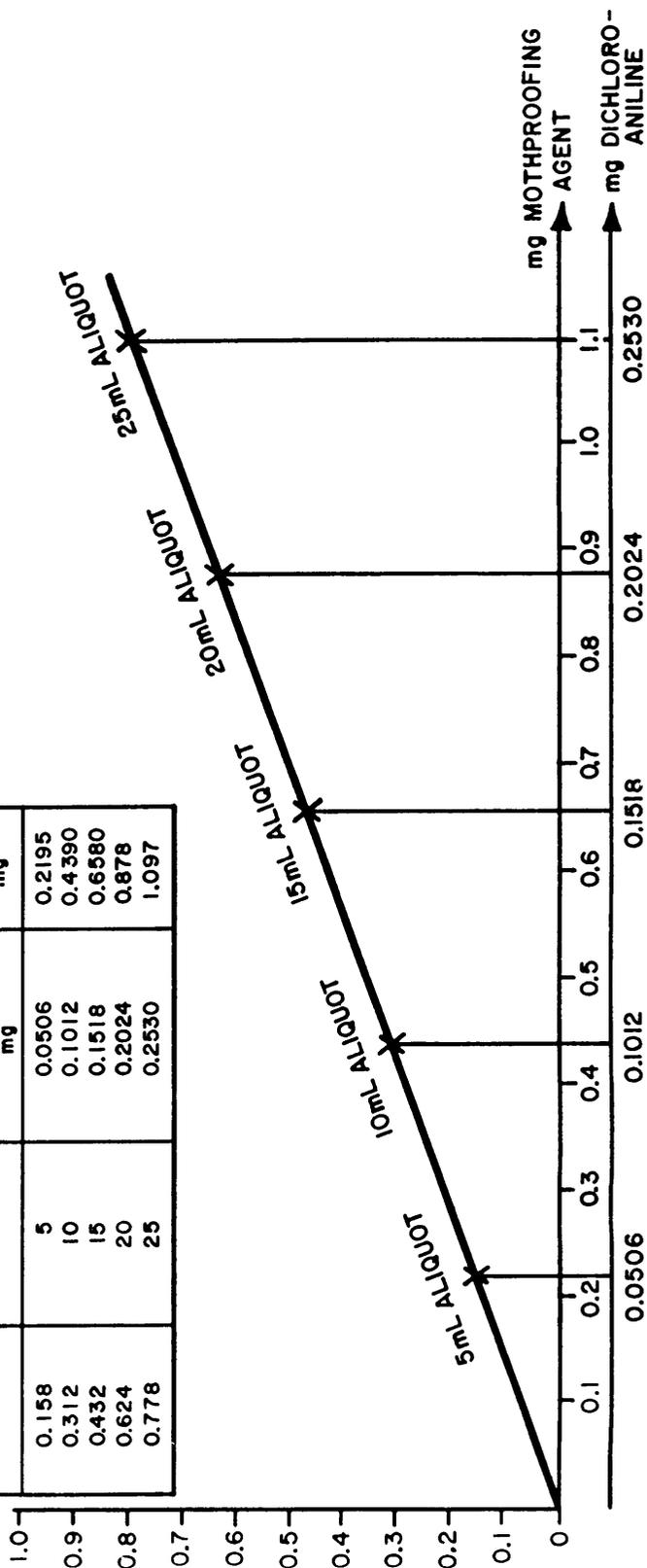
FIGURE 2015B
 DETERMINATION OF MOTHPROOFING CALIBRATION CURVE

WEIGHED PORTION 202.4 mg 3, 4 DICHLOROANILINE INTO 1000mL, 25/1000 INTO 500mL = DILUTION

$$\text{SAMPLE CALCULATION: } \left(\frac{202.4 \text{ mg} \times 25 \text{ mL} \times (\text{mL}) \text{ ALIQUOT USED}}{1000 \text{ mL} \times 500 \text{ mL}} \right) \times \left(\frac{702.73}{162.02} \right) = \text{mg MOTHPROOFING AGENT}$$

WHERE 162.02 = MW 3, 4 DICHLOROANILINE; 702.73 = $\frac{562.18 \text{ MW MITIN FF OR INTRACIDE M}}{.8 \text{ (ACTIVE)}}$

ABSORBANCE	ALIQUOT DILUTION (mL)	100mL MEASURING SOLN DICHLOROANILINE mg	MITIN FF (80%) mg
0.158	5	0.0506	0.2195
0.312	10	0.1012	0.4390
0.432	15	0.1518	0.6580
0.624	20	0.2024	0.878
0.778	25	0.2530	1.097



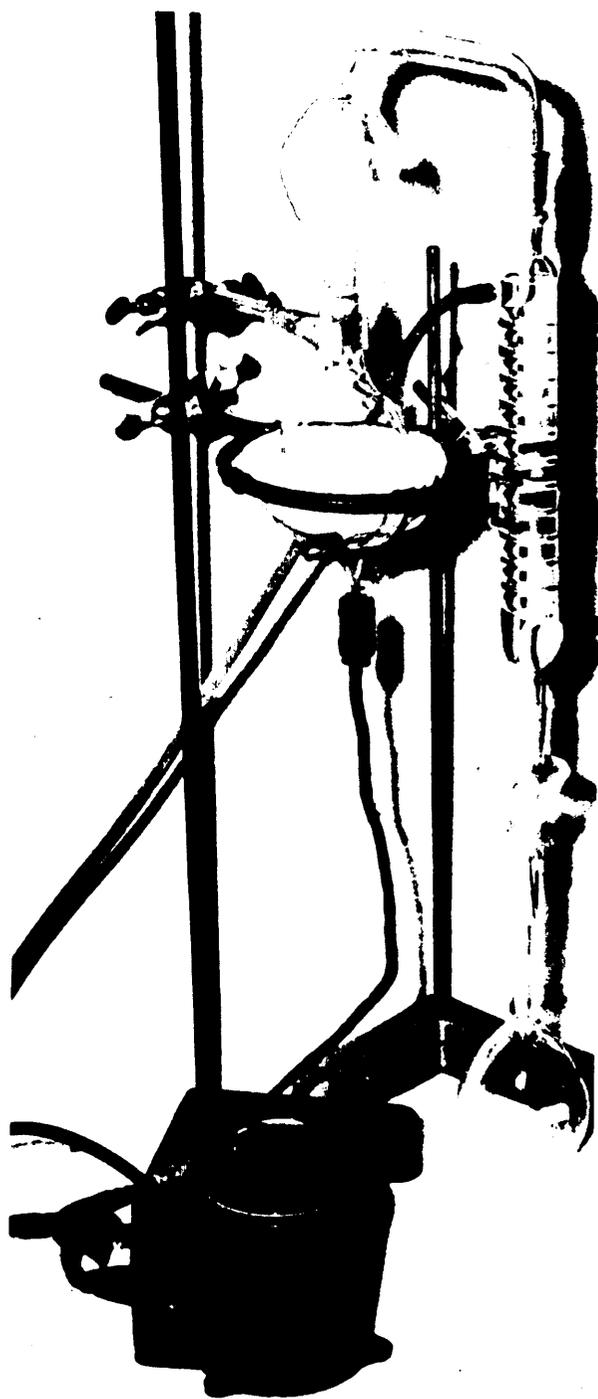


FIG 2015C DISTILLING APPARATUS

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