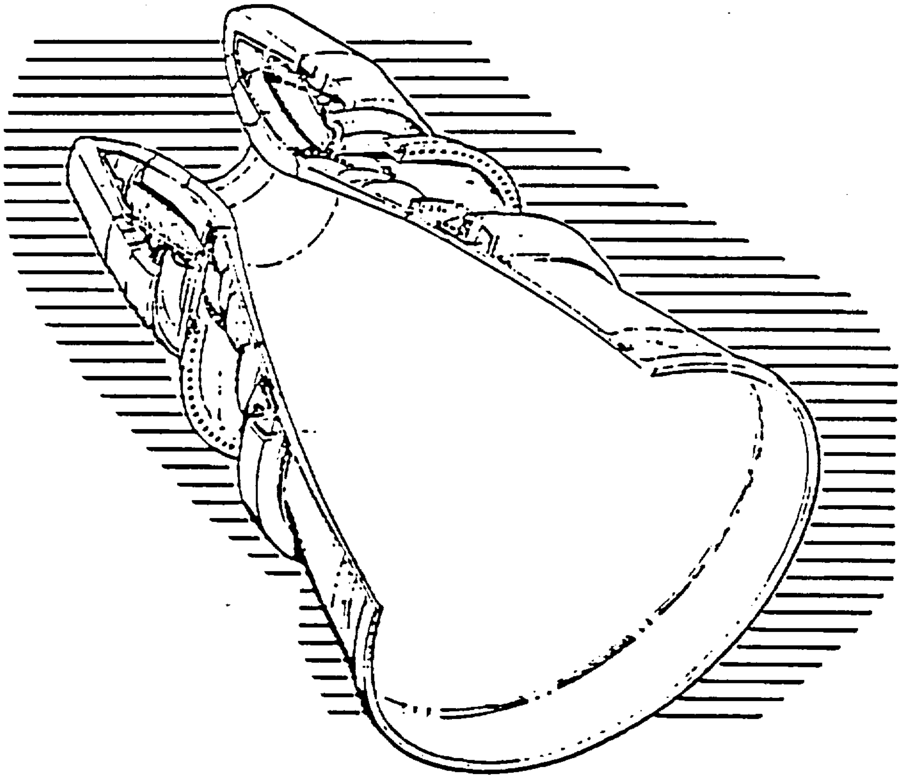
FINAL REPORT

Standardization of the Carbon-Phenolic Materials and Processes

Volume II

Test Methods and Specifications

August 31, 1988



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Final Report

Volume II, Test Methods and Specifications

(for period 1 September 1985 to 31 August 1988)

for

Standardization of the Carbon-Phenolic
Materials and Processes

NASA Grant No. NAG8-545

Prepared for

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PREFACE

The test methods contained in this volume were taken from files of the following:

Marshall Space Flight Center, NASA,
    Huntsville, AL
Avtex Fibers, Inc.
    Front Royal, VA
Highlands, Inc.
    Cheraw, SC
PolyCarbon, Inc.
    Valencia, CA
U.S. Polymeric
    Santa Ana, CA
Borden, Inc.
    Louisville, KY
Fiberite Corporation
    Winona, MN

The test methods were made as generic as possible and would need to be modified as required by other laboratories depending on equipment and personnel. Sincere appreciation is given to all the above identities for the unrewarded assistance.

TABLE OF CONTENTS

MATERIALS

Resins

Cloth

Fillers

PREPREGS

CURED MATERIALS

M-R-1 Chemical Analysis of Resins, Fabrics, Fillers, and Prepregs for Alkali and Alkaline Earth Metals

1. SCOPE

1.1 This procedure describes a method for determining alkali and alkaline earth metal impurities (sodium, potassium, calcium, lithium, and magnesium) in resins, fabrics, and in preimpregnates.

2. EQUIPMENT AND MATERIALS

2.1 Atomic Absorption Spectrophotometer

2.2 Analytical balance, sensitivity 0.0001 gm.

2.3 Volumetric flasks, 100 ml. and 200 ml. (one per sample and blank)

2.4 Graduated cylinders, 10 ml. and 50 ml.

2.5 Hot Plate

2.6 Fume exhaust hood

2.7 Timer

2.8 Gloves; plastic

2.9 Oven, Convection

2.10 Muffle Furnace

2.11 Platinum or ceramic crucibles (30 ml.) one per sample

2.12 Forceps

2.13 Scissors

2.14 Matrix standards (0.5 ppm for Na, K, Ca, Li and Mg)

2.15 Nitric Acid, 6N

2.16 Hydrochloric Acid 6N & 12N

2.17 Hydrofluoric Acid 28N

2.18 Cesium Chloride

2.19 Potassium pyro-sulfate

2.20 Deionized Water

NOTE: Unless otherwise specified, all reagents are to be reagent grade or spectro grade, as applicable.

3. SAMPLING

3.1 Resins. Obtain representative 25 gm. sample.

3.2 Fabrics. Obtain 16 sq. in.

3.3 Prepregs. Obtain 16 sq. in.

3.4 Fillers. Obtain representative 25 gm. sample.

4. PROCEDURE

4.1 Reagent Preparation

4.1.1 CsCl Stock Solution. Add about 1200 ml. of deionized water to a two liter volumetric flask. Add 253 gm CsCl with agitation. After the CsCl is in solution, allow the solution to cool to ambient temperature and dilute to 2000 ml. with deionized water to yield a 100,000 micrograms/ml. solution.

4.2 Ceramic Crucible Preparation Ceramic crucibles are to be used for atomic absorption spectrophotometric testing of carbonaceous materials. All new crucibles are to be conditioned as below. Crucibles that have been in use for this procedure may be considered clean (proceed to par. 4.2.10).

4.2.1 Label the ceramic crucibles with an appropriate marking agent that will withstand at least 600°C.

4.2.2 Place crucibles on a hot plate in a laboratory fume hood and fill with 6N HCl. Using a medium hot plate setting, evaporate to near dryness then repeat procedure.

4.2.3 Remove crucibles from hot plate and allow to cool for five minutes.

4.2.4 Rinse the crucibles with dionized water at least three times.

4.2.5 Place the crucibles on the hot plate again and fill with 12N HCl. Using a medium hot plate setting, evaporate to near dryness and repeat.

4.2.6 Remove the crucibles from the hot plate and let stand for five minutes.

4.2.7 Rinse copiously with deionized water. Return to hot plate.

4.2.8 Fill crucibles with 6N HCl and again evaporate to near dryness and repeat.

4.2.9 Remove the crucibles from the hot plate and allow to cool for five minutes.

4.2.10 Rinse the crucibles with deionized water copiously.

4.2.11 Place the crucibles in a muffle furnace, pre-conditioned to 600 ± 15°C., for a minimum of three hours.

4.2.12 Remove the crucibles from the muffle furnace and desiccate until used.

4.3 Platinum Crucible Preparation Platinum crucibles are to be used for samples known to contain silica or for samples without a history of successful preparation in ceramic crucibles.

4.3.1 To clean the crucible, fuse about one gram of potassium pyrosulfate in the crucible. Rotate the crucible to distribute the melt along the inside walls.

4.3.2 Pour off the melt and allow to cool to ambient temperature.

4.3.3 Boil the crucibles in deionized water for about thirty (30) minutes. Rinse the boiled crucibles several times with deionized water.

4.3.4 Place the crucibles in a suitable container and fill with 6N HNO_3. Bring acid to a boil and maintain for one (1) hour. Pour off the hot acid and repeat.

4.3.5 Pour off the acid and rinse several times with deionized water.

4.3.6 Ignite the crucible in a muffle furnace (600 ± 15°C) for at least one hour. Pour off the hot acid and repeat.

4.3.7 Pour off the acid and rinse several times with deionized water.

4.3.8 Remove the crucibles from the muffle furnace and desiccate until used.

4.4 Sample Preparation

4.4.1 Using clean plastic gloves and clean scissors, cut the sample into approximate 1/2 inch squares and transfer into an analytically clean and labled sample bottle.

Unused sample will be retained. For multiple samples, every seventh crucible shall be a blank determination.

4.4.2 Assure that the crucibles have been conditioned per par. 4.2 or 4.3, as applicable.

4.4.3 Weigh and record the weight of the crucible to the nearest 0.1 mg.

4.4.4 Add approximately 2 gm. of sample into the crucible using forceps. Weigh exactly and record sample weight to nearest 0.1 mg.

4.4.5 Transfer the crucibles to a convection oven and dry at 125 ± 5°C. for two hours minimum.

4.4.6 Remove the crucibles from the oven and desiccate until ambient temperature is achieved.

4.4.7 Weigh and record the crucibles to the nearest 0.1 mg.

4.4.8 Transfer the crucibles to a muffle furnace and ash at 600 + 15°C. for 16 to 20 hours, or until constant weight is achieved.

4.4.9 Remove the crucibles from the muffle furnace. Desiccate until ambient temperature is achieved. Determine ash content, if required per par. 5.2.

4.4.10 Weigh and record the weights to the nearest 0.1 mg.

4.4.11 Place the crucibles on a hot plate and fill to about 75% of capacity with deionized water. Add 2 ml. of 12N HCl to each crucible.

4.4.12 With the use of moderate heat, effect slow evaporation to near dryness. DO NOT ALLOW THE SAMPLES TO REACH DRYNESS. Repeat par. 4.4.11 and 4.4.12 as needed in order to effect solution. All crucibles must be treated identically.

4.4.13 Quantitatively transfer the solution from the crucibles into analytically clean volumetric flasks of appropriate size (to measure within the linear portion of the standard range).

4.4.14 Add sufficient CsCl stock solution to each volumetric flask to yield a 2000 ppm CsCl concentration when diluted to the mark.

4.4.15 Dilute each flask to the mark and agitate.

4.4.16 Agitate each solution prior to analysis.

4.5 Preparation of Standard

4.5.1 Analysis of USP samples requires the use of a matrix standard below which the Beers Laws relationship is linear. Empirical data has shown that for the alkali metals and alkaline earth metals, the calibration curve appears linear below 0.5 ppm. MATRIX: The matrix contain each of the following elements in deionized water Na, K, Ca, Li, and Mg. The standard is made by adding (Volumetric pipet) 1 ml. of a 1000 ppm stock solution for each of the elements into a 2000 ml. volumetric flask and diluting to volume.

4.6 Spectrophotometer Start Up

4.6.1 Each laboratory shall generate an appropriate written procedure for instrument operation. The following is the U.S. Polymeric procedure.

4.6.1.1 Turn on air and acetylene valves.

4.6.1.2 Make sure air pressure is 40 psi and acetylene pressure is just below 15 psi.

4.6.1.3 Turn spectrophotometer switch on

4.6.1.4 Press oxidant button left instrument panel and turn oxidant knob counter-clockwise until ball float goes down no further. This should be reading 12; if not, adjust knurled knob on nebulizer until a reading of 12 is obtained. Next turn oxidant knob clockwise until a reading of 18 has been achieved.

4.6.1.5 Press fuel/oxidant button and adjust fuel knob until fuel ball float reads 7.

4.6.1.6 Remove nebulizer tube from D.I. H_20 and press flame on button to ignite flame.

4.6.1.7 Adjust fuel knob counter-clockwise until the yellow in the flame just disappears. (Make sure burner head height is 32).

4.7 Analysis for Potassium

4.7.1 Adjust monochrometer to read 766.5 nm; place mode selector to POSITION.

4.7.2 Place MA count selector to the 1.0 position and turn the inner knob completely clockwise.

4.7.3 Aspirate the 0.5 ppm MATRIX Standard and adjust the monochrometer, in close vicinity of the originally set wave length, to obtain maximum deflection on the log scale meter (right instrument panel). This is fine tuning of the wave length.

4.7.4 Aspirate deionized water or nitric acid digestion blank and press auto zero button (you should at this time select the integration period depending on how stable you want the read out to be.)

4.7.5 Start with a slit width of 80. Aspirate the 0.5 ppm MATRIX Standard and use the photomultiplier voltage knob and the inner MA/count knob to obtain a read out of 0.50 on the digital display. If this cannot be done, adjust slit selection either up or down depending on whether you want to increase or decrease the read out, respectively.

4.7.6 Continuing to aspirate the 0.5 ppm standard use the M/A count selector and inner knob to adjust the read out to 50 ppm for the 100 to 1 dilution.

4.7.7 Aspirate deionized water or nitric acid digestion blank and recheck zero.

4.7.8 Aspirate each sample. The read out will give direct ppm to be reported.

4.8 <u>Analysis for Sodium, Calcium:</u>

4.8.1 The procedure here is the same as for Potassium except that the initial monochrometer wave length for sodium is 589.0 nm. and for Calcium 422.7 nm. Calculate the curve for sodium that is linear between 0.1 and 2.5 micrograms per ml..

4.9 <u>Analysis for Magnesium:</u>

4.9.1 Adjust monochrometer to read 285.2 nm.

4.9.2 Place MA/count selector to the 1.0 position and turn the inner knob completely clock wise.

4.9.3 Place mode selector in A/DB position.

4.9.4 Turn lamp turret until Mg lamp locks into position; adjust knurled knob immediately above lamp on turret until lamp current reads 5 MA (right instrument panel)

4.9.5 DO NOT aspirate standard, but fine tune wavelength by achieving maximum deflection on log scale by adjusting monochrometer.

4.9.6 Aspirate deionized water or nitric acid digestion blank and press auto zero button.

4.9.7 Aspirate 0.5 ppm MATRIX standard and set readout to 0.5 in same manner as for previous elements.

4.9.8 Adjust readout to 50 ppm with MA/count selector and inner knob as before with other elements.

4.10 Miscellaneous Notes

4.10.1 The readout will be in direct sample parts per million (ppm) using this procedure.

4.10.2 The calibration (in terms of sample ppm) cannot be considered linear over 50 ppm. If a sample gives a read out of greater than 50 ppm, it must be diluted to give a readout within the operating range. The readout value must, in this case, be multiplied by this additional dilution to give the actual sample ppm.

4.10.3 Because dilute MATRIX standards deteriorate rapidly, a new 0.5 ppm standard must be prepared every two weeks to insure valid instrument calibration.

5. CALCULATIONS

5.1 Moisture

$$\frac{(\text{raw sample weight}) - (\text{dry sample weight})}{(\text{raw sample weight})} \text{ X } 100 = \%\ H_2O$$

5.2 Ash

$$\frac{(\text{ash weight})}{(\text{dry sample wt})} \text{ X } 100 = \%\ \text{ash}$$

5.3 Sodium, potassium, calcium, lithium, or magnesium

$$\frac{(\text{analyte g/ml.}) \times (\text{analyte ml.})}{(\text{sample g})} = \text{ppm sample}$$

6. REPORT

6.1 Moisture/content shall be reported to the nearest 0.10%.(when required)

6.2 Ash content shall be reported to the nearest 0.10% (when required)

6.3 Each alkali and alkaline earth metal contents shall be reported to the nearest 1.0 ppm.

M-R-2 Percent Resin Pickup in Manufacture of Broadgoods

1. SCOPE

1.1 This test method is used to determine the "in-process" resin pickup of broadgoods.

2. EQUIPMENT

2.1 Template

2.2 Balance, graduated in (1) gram increments

3. PROCEDURE

3.1 Using a template, cut a sample from the head of the untreated fabric roll and weigh to the nearest gram. Designate this weight as W_1.

3.2 Treat a four (4) foot piece of fabric to the desired specification.

3.3 Using the template cut a sample from the center of the impregnated fabric, weigh to the nearest gram. Designate this weight as W_2.

4. CALCULATIONS

$$\%\text{Resin Pickup} = (W_2 - W_1)/W_2 \times 100$$

where:

W_1 = weight of template - cut piece of untreated fabric, gm.
W_2 = weight of template - cut piece of treated fabric, gm.

M-R-3 Total Resin Solids Content of Liquid Solutions

1. SCOPE

1.1 This method describes a procedure for the determination of total resin solids content of liquid solutions

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

2.1.1 Balance, analytical, minimum sensitivity 0.001 gms.

2.1.2 Desiccator

2.1.3 Hypodermic syringe, 2 ml. or 5 ml.

2.1.4 Oven, forced air (see 6.1)

2.2 Materials.

2.2.1 Aluminum weighing dish, diameter 2-3/8 inch, depth 5/8 inch, weight approximately 1.4 gms.

2.2.2 Eyedroppers

3. SAMPLING

3.1 Sample size. Unless otherwise specified, use one gram of resin solids for each determination. To find the exact sample size, the formula 1/(total solids)x(1-filler content) will yield the correct weight to use. The following table shall serve as a guide when the anticipated solids are known (unfilled only):

ANTICIPATED SOLIDS	SAMPLE SIZE,GMS.
20%	5.00 ± 0.5
30%	3.30 ± 0.3
40%	2.50 ± 0.2
50%	2.00 ± 0.2
60%	1.70 ± 0.2
70%	1.45 ± 0.1
80%	1.25 ± 0.1
90%	1.10 ± 0.1
100%	1.00 ± 0.1

When the anticipated solids are not known, run one set of solids at the 70% concentration. Once the solids are determined, conduct an additional test using the correct sample size.

3.2 Number of determinations. Unless other wise specified, determine total resin solids in triplicate.

4. PROCEDURE

4.1 Weighing. Weigh an aluminum dish to nearest 0.001 gram. The following procedure shall be used for transferring and weighing the resin.

4.1.1 Low viscosity resins. For low viscosity resins, especially resins using acetone as a solvent, use a syringe. Rapidly fill the syringe and wipe off the outside tip. Weigh syringe and resin to nearest 0.001 gm. Expel the approximate weight of sample (see 3.1) from the syringe into the aluminum dish. Retract the syringe piston and reweigh the syringe to nearest 0.001 gm.

4.1.2 High viscosity resins. For high viscosity resins, use an eyedropper. Quickly discharge the sample into the aluminum dish. Immediately weigh the resin and aluminum dish to nearest 0.001 gm.

4.2 Spreading sample. Tilt and rotate the aluminum dish to spread resin over entire bottom of dish. Warm the resin on a hot plate if necessary to accomplish spreading see note 6.2. Add approximately 6 ml. of toluene to SMR dispersions.

4.3 Devolatilization. Unless otherwise specified, place samples in an air circulating oven for time and temperature indicated below, see note 6.3:

RESIN TYPE	TEMPERATURE, oF	TIME, MINUTES
Epoxy	320 ± 5	45 ± 2
Polyester	275 ± 5	20 ± 1
Phenolic	275 ± 5	60 ± 2
Phenyl-Silane	275 ± 5	60 ± 2
Silicones	275 ± 5	180 ± 3
SMR Dispersions	275 ± 5	60 ± 2

4.4 Reweighing. Remove dishes from oven and cool in a desiccator, see note 6.4. Reweigh each aluminum dish and residue to nearest 0.001 gm.

5. CALCULATIONS AND REPORTING

5.1 Calculations. Calculate solids as follows:

5.1.1 Syringe method.

$$\text{Solids, wt. \%} = \frac{W_2 - W_1}{W_4 - W_3} \times 100$$

Where:

W_1 = weight of aluminum dish, gm.
W_2 = weight of dish and residue, gm.
W_3 = weight of syringe, gm., after expelling sample portion
W_4 = weight of syringe and sample, gm.

5.1.2 Eyedropper method.

$$\text{Solids, wt\%} = \frac{W_2 - W_1}{W_5 - W_1} \times 100$$

Where:

W_5 = weight of dish and resin sample, gm.

5.2 Reporting. Unless otherwise specified, round-off and report the average of three determinations to 0.1%. Triplicate determinations should be considered suspect if the range from the average exceeds 0.3% solids.

6. NOTES

6.1 Oven requirements. The oven should have a wattage range of between 1200 and 4000 watts, and an air flow rate between 600 and 1500 feet per minute when measured at the internal ports with an air flow meter (velometer).

6.2 Spreading sample. The sample should remain on the hot plate only as long as is necessary to make the resin liquid enough to spread. A low temperature hot plate should be used, preferably lower than 275°F.

6.3 Placing samples in oven. All aluminum dishes should be placed in the oven at one time. The time required for opening the door, inserting aluminum dish(es) and closing the door should not exceed ten (10) seconds.

6.4 Precautions in removing samples from oven. In removing samples which have bubbled from the oven, precautions should be taken to prevent loss of sample from spattering.

M-R-4 Specific Gravity Determination of Liquids, Solids, and Semi Solids

1. SCOPE

1.1 Scope. This is a method for the determination of the specific gravity of liquids, semi-solids and solids which are neither water soluble nor appreciably absorb water at 25°C.

1.1.1 Method I. A procedure for liquids and materials that flow readily at 25°C.

1.1.2 Method II. A procedure for semi-solids and solids at 25°C.

1.2 Equivalent Method. The procedures described in Methods I and II are equivalent to ASTM-D1963-61.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

2.1.1 Pycnometer, Gay-Lussac Type

2.1.2 Pycnometer, Hubbard Type

2.1.3 Analytical balance, minimum sensitivity 0.0001 gm.

2.1.4 Water bath, capable of maintaining 25 ± 0.2%°C.

2.1.5 Thermometer, 74°C.to 79°C.in 0.1°F. intervals

2.1.6 Forceps, 6 inch

2.1.7 Vacuum chamber

2.2 Materials.

2.2.1 Distilled water, recently boiled

2.2.2 Lint free cloth

2.2.3 Abrasive paper, fine

3. SAMPLING

3.1 Sample Size.

3.1.1 Method I. The sample should be large enough to fill the pycnometer for the required number of determinations.

3.1.2 Method II. For semi-solid materials there should be sufficient sample to fill the pycnometer one-half full for the required number of determinations. For solid materials there should be enough sample for the required number of specimens approximately 3/8 x 3/8 x 1 inch, or an equivalent volume.

3.2 Number of determinations. Unless otherwise specified, conduct two determinations on each sample.

4. PROCEDURE

4.1 Calibration. If the pycnometer needs cleaning, see 6.1 Calibrate the pycnometer as follows:

4.1.1 Bring the pycnometer to room temperature and weigh to 0.0001 gm. (W_1).

4.1.2 Fill the pycnometer (in such a manner as to prevent the entrapment of air bubbles) with cooled, just previously boiled, distilled water at a temperature of about 20°C. Insert the stopper, taking care that no bubbles are trapped.

4.1.3 Immerse in the water bath at 25 ± 0.1°C.until constant temperature is attained (approximately 20 minutes).

4.1.4 Blot the meniscus of water in the bore so that it is flush with the surface of the stem.

4.1.5 Remove the pycnometer from the water bath and wipe dry with a clean, lint free cloth. Cool the pycnometer slightly to prevent loss of water through capillary and weigh to 0.0001 gm. (W_2).

4.2 Method I.

4.2.1 Materials with a viscosity of 40 stokes or less.

4.2.1.1 Fill a clean, dry Gay-Lussac type pycnometer with the sample, taking care to prevent the entrapment of air bubbles.

4.2.1.2 Proceed as described in 4.1.3 - 4.1.5, recording the weight as W_3.

4.2.2 Material flows readily but viscosity is greater than 40 stokes.

4.2.2.1 Fill a clean, dry Hubbard type pycnometer with the material to be tested, taking care to prevent the entrapment of air bubbles.

4.2.2.2 Proceed as described in 4.1.3 - 4.1.5 recording the weight as W3.

4.3 Method II. This method is for semi-solids at 25 degrees C.

4.3.1 Place the sample in a clean, dry Hubbard type pycnometer and fill about one-half. If the sample is a solid, use a specimen approximately 3/8 x 3/8 x 1 inch or a sample of approximately this volume. Smooth the surface on molded or laminated specimens.

4.3.2 Bring the pycnometer and its contents to room temperature and then weigh with stopper to 0.0001 gm. (W_3).

4.3.3 Proceed as described in 4.1.3 - 4.1.5, (if the sample is porous or powdered, see 6.2), recording the weight as W_4.

5. CALCULATIONS AND REPORTING

5.1 Calculations.

5.1.1 Calibration. Calculate the pycnometer constant as follows:

$$K = W_2 - W_1$$

Where:

K = pycnometer constant
W_1 = weight of pycnometer, gm.
W_2 = weight of pycnometer, plus water, gm.
W_3 = weight of pycnometer, plus material, gm.
W_4 = weight of pycnometer, plus material, plus water, gm.

5.1.2 Method I. Calculate specific gravity as follows:

$$\text{Specific gravity} = \frac{W_3 - W_1}{K}$$

Where symbols are specified in 5.1.1

5.1.3 Method II. Calculate specific gravity as follows:

$$\text{Specific gravity} = \frac{W_3 - W_1}{W_3 + K - W_4}$$

Where symbols are specified in 5.1.1

6. NOTES

6.1 Cleaning. See the cleaning procedure outlined for viscosity tubes.

6.2 Powdered or porous materials. If the material is powdered or porous, add sufficient distilled water to cover the sample. The pycnometer with water and sample should then be placed in a vacuum chamber and the pressure reduced almost to the point of boiling the water, allow to remain at this point for approximately 20 minutes then proceed to 4.1.2.

M-R-5 Viscosity Determination by the Brookfield Methods

1. SCOPE

1.1 This is a method for the determination of viscosity of liquids using a Brookfield Viscometer. (Method I)

1.2 Also included is the Brookfield Thermosel System which is designed for measuring viscosities at temperatures over a range of 100°F to 500°F. (Method II)

2. EQUIPMENT AND MATERIALS

2.1 Equipment

2.1.1 Brookfield Viscometer, Model RVF with 7 spindles (for both methods).

2.1.2 Thermometer, range 74° to 79° in 0.1°F. divisions (for Method I).

2.1.3 Water bath, capable of maintaining 77° ± 0.5°F. (for Method I).

2.1.4 Thermo-container and controller (for Method II).

3. SAMPLING

3.1 Materials having ingredients which tend to settle should be throughly mixed before sampling. A representative one-quart sample is required for each determination.

4. PROCEDURE

4.1 Method I

4.1.1 Sample conditioning. Place approximately one quart of sample in a round container with a sufficient diameter and depth so that the spindle will not be closer than one inch at any point to the container. Place the sample in a water bath and condition to 77° ± 0.5°F, unless otherwise specified.

4.1.2 Determination of viscosity.

4.1.2.1 Level the viscometer by means of tripod screws and bubble level.

4.1.2.2 Select a spindle consistent with expected viscosity and screw it into the viscometer using counter-clockwise rotation.

4.1.2.3 Immerse the spindle into the liquid to the depth where the notch on the stem is even with the liquid level.

4.1.2.4 Unless otherwise specified, rotate the spindel at 20 RPM, depressing the clutch. Release the clutch after one revolution and read the outer dial as soon as equilibrium is established.

4.1.2.5 If dial reading is less than 10, repeat test using a smaller numbered spindle; if greater than 90, repeat using a larger numbered spindle. Make two readings.

4.1.2.6 Turn viscometer off, remove sample and clean spindle thoroughly.

4.2. Method II

4.2.1 Set Up Viscometer
Set up the viscometer stand connecting the black 3-1/4 inch coupling post to the rack. Screw both posts into the base leaving the cock nut loose. Put the three (3) leveling screws into position on the base. Attach the viscometer to the stand inserting it into the clamp, and position it reasonably level and centered between the stand legs. Lock the posts tight to the stand base with the lock nut on the coupling post. Raise the viscometer to the highest position on the stand. Check the power switch off and plug the viscometer power cord into a 15 amp., 115 volt, 60 cycle A.C. electrical service.

4.2.2 Set Up Controller
Set controller on a level surface adjacent to thermo-container. Insert the male plug (three-prong) from the thermo-container braided cord into the socket on the back of the controller (turn and lock connection). Insert the four inch stainless steel probe (Resistance Thermometer) into the hole in the thermocontainer located directly above the braided cord. Plug the other end of the probe into the connector located on the back of the controller. To remove probe, depress spring clip and carefully slide probe out. The procedure should not be followed if the thermo-container is at a temperature above 100^{o}F.

NOTE: If the probe is NOT inserted in the thermo-container and the controller is turned on, the thermo-container will be destroyed.

4.2.3 Familiarization
The operator should become familiar with the alignment procedure so that he can safely align and

operate the system at elevated temperatures. The system is designed to operate in the temperature range from ambient to a maximum of 500°F. (260°C.). Precise control of test sample temperature is possible in the 100° to 500°F. range with accuracy of ± 0.5% of the controller set point. That is; at 500°F set point, the test sample temperature will be held to ± 2.5°F.

4.2.4 Determination of Viscosity

4.2.4.1 Remove the insulating cap and spindle.

4.2.4.2 Raise the viscometer to the highest level on the stand.

4.2.4.3 Remove the sample chamber, using the extracting tool, and place it in the auxiliary holder.

4.2.4.4 Using a syringe, graduate cylinder or other suitable measuring device, pour into the sample chamber the volume of liquid sample designated on the data sheet. DO NOT OVERFILL! The liquid level should intersect the spindle shaft at a point approximately 1/8 inch above the upper "Concical Body" - "Spindle Shaft" Interface.

4.2.4.5 Using the extracting tool, put the loaded chamber back into the thermo-container.

4.2.4.6 Lower the viscometer and align the thermo-container.

4.2.4.7 Insert the spindle into the liquid in the chamber and couple it to the viscometer (observe left hand thread).

4.2.4.8 Replace the insulating cap.

4.2.4.9 Set the control knob on the controller to the desired set point temperature at which viscosity measurements are to be made. The Controller scale reads directly in the 0-500°F. range with 1°F. increments.

4.2.4.10 For temperatures between 100° and 250°F., set the control knob initially 20°F. LOWER than the desired equilibrium temperature (for temperatures between 250° and 500°F., set control knob on temperature desired.) This will prevent temperature overshoot in the thermo-container and reduce the lag time to reach spindle, chamber and sample temperature equilibrium. As the temperature rises in the thermo-container, the power output level light will go from full ON to dimmer and then full OFF. When the temperature in the thermo-container has dropped to the initial set point (after about 5 minutes), the output level light will start to glow. Advance the set point knob in consecutive steps up to the desired setting. About 30 minutes should be allowed for the system to come to temperature equilibrium. EQUILIBRIUM CONDITIONS WILL BE DENOTED BY A STEADY GLOW OF THE OUTPUT LEVEL LIGHT ON THE CONTROLLER, STABILIZED DEVIATION METER AND BY A STEADY DIAL READING ON THE VISCOMETER.

4.2.4.11 Turn ON the Viscometer and leave it running during the equilibrium period. The rotating spindle provides agitation which reduces temperature equilibrium time and temperature gradients within the test sample.

4.2.4.12 Turn on the controller and refer to 4.2.4 above prior to taking viscosity measurements.

4.2.4.13 After the thermo-container, spindle, chamber and test sample has reached temperature equilibrium, viscometer readings are taken at different speeds and viscosities are obtained using the ranges supplied on the data sheet.

5. CALCULATIONS AND REPORTING

5.1 Calculations for Method I

Calculate viscosity as follows:

Viscosity in centipoise = RF

Where: R = average dial reading
F = factor from the following chart

Spindle No.	20 RPM Factor	10 RPM Factor	4 RPM Factor	2 RPM Factor
1	5	10	25	50
2	20	40	100	200
3	50	100	250	500
4	100	200	500	1,000
5	200	400	1,000	2,000
6	500	1,000	2,500	5,000
7	2,000	4,000	10,000	20,000

5.2 Calculations for Method II

Speed (RPM)	Spindle Number 21	27	28	29
100	5	25	50	100
50	10	50	100	200
20	25	125	250	500
10	50	250	500	1M
5	100	500	1M	2M
4	125	625	1.25M	2.5M
2.5	200	1M	2M	4M
2	250	1.25M	2.5M	5M
1	500	2.5M	5M	10M
0.5	1M	5M	10M	20M

5.3 Reporting for Both Methods I and II

Report viscosity in centipoise or poise (as appplicable) to three significant figures. The test results should be considered suspect if the range for duplicate readings exceeds 0.2%.

M-R-6 Gel Time Determination in an Oil Bath of Liquid Resins

1. SCOPE

1.1 Scope. This method describes two procedures for the determination of gel time by immersing the sample in an oil bath.

1.1.1 Method I. This method is in general applicable to phenolic resins.

1.1.2 Method II. This method is in general applicable to silicone resins.

1.2 Equivalent methods. Method I is equivalent to ASTM-D1472-62. Method II is equivalent to the Dow Corning gel method for Dow Corning 2106 resin.

2. EQUIPMENT

2.1 Equipment.

2.1.1 Jar, Pyrex, 6-inch diameter, 8-inch height, or similar container

2.1.2 Stirrer, electrical with rheostat

2.1.3 Test tubes; Method I, 13mm. x 120 mm.; Method II, 25 mm. x 100 mm.

2.1.4 Stirring rods: Method I, wire, .06 to .08 inch diameter with 0.30 to 0.40 inch loop perpendicular to stem; Method II, glass rod, 1/4-inch diameter with flattened paddle of 0.5 to 0.6 inch diameter at the end.

2.1.5 Silicone oil, Dow Corning F-1-D173, 100 cs. or equivalent

2.1.6 Clamps and ringstand

2.1.7 Thermoregulator with range to 480°F., control-sensitive to ± 1°F.

2.1.8 Thermometer, 20° to 600° F, 1°F intervals

2.1.9 Timer

2.1.10 Balance, minimum sensitivity 0.1 gm.

2.1.11 Heater, 250 W., immersion type

3. SAMPLING

3.1 Sample size. Method I requires 2 ± 0.1 gm. for each determination. Method II requires 12 ± 0.1 gm. for each determination.

3.2 Number of determinations. Unless otherwise specified, run duplicate determinations.

4. PROCEDURE

4.1 Preheating bath. Preheat the oil bath to 338° ± 1.5°F. for Method I or to 480° ± 2° F. for Method II. If necessary, add oil to bring the oil level to within 1 inch of the top. Using an electric stirrer, stir bath thoroughly and continually for at least 5 minutes prior to testing and throughout the test to assure even heat dissipation.

4.2 Method I.

4.2.1 Weighing sample. Weigh a 2 ± 0.1 gm. sample into a test tube. Place the wire stirrer, looped end down, into the test tube.

4.2.2 Immersion. Lower the test tube into the holder to be sized to hold the tube loosely suspended by the rim and shall be positioned so that the rim of the test tube is 1.5 ± 0.25 inches above the surface of the oil. The stopwatch is started immediately upon immersion.

4.2.3 Stirring Sample. Agitate the resin with rapid vertical strokes, with not more than 1/4 inch strokes for the first 40 seconds. After 40 seconds, agitate at the rate of 4 strokes every 5 seconds.

4.2.4 End point. The end point is defined as the time when there is no motion of the test tube in relation to the stirrer, i.e. when the tube moves up and down with the stirrer. CAUTION: Be sure the holder is loose enough to permit this.

4.3 Method II.

4.3.1 Weighing sample. Weigh a 12 ± 0.1 gm. sample into a test tube. Place the glass rod , paddle down, into the test tube.

4.3.2 Immersion. Lower the test tube into the holder so that the rim of the test tube is 1.5 ± .25 inches above the surface of the oil. Tighten the holder to prevent slippage of the test tube. The stopwatch is started immediately upon immersion.

4.3.3 Stirring sample. Stir the sample by twisting the glass rod. Do not lift the rod up and down. Continue turning the paddle for three minutes.

4.3.4 End point. The end point is defined as the time when the glass rod can be turned 90° and will spring back considerably upon releasing.

5. CALCULATIONS AND REPORTING

5.1 Unless otherwise specified, report the average of two tests in minutes and seconds to the nearest second. Test results should be considered suspect if the range for duplicate determinations exceeds 10 seconds for gel times less than 6 minutes or 20 seconds for gel times greater than 6 minutes.

M-R-7 Gel Time Determination of Liquid Resins at 132°C

1. SCOPE

1.1 This test method is applicable to liquid resins which undergo gelation at 132° ± 1°C. (Option A) or any other temperature (Option B).

2. EQUIPMENT

2.1 G.E. gel time meter
(Sunshine Scientific Instrument Co.)

2.2 Test tubes 18 x 150 mm.

2.3 Glass stirring rods 7-1/8" x 1/4"

2.4 Thermometer accurate to 1°C. for checking bath temperature

2.4.1 Option A

2.4.1.1 Voltage varied for heating bath

2.4.1.2 Monochlorobenzene, boiling point 132°C.

2.4.1.3 Water condenser

2.4.2 Option B

2.4.2.1 Thermostatically controlled oil bath

3. PROCEDURE

3.1 Place solvent or oil in the large beaker to maintain a 2" immersion depth of the test tube.

3.2 Turn on cooling water to condense the monochlorobenzene.

3.3 Heat the bath by either boiling the monochlorobenzene or circulating oil through a heating coil in the bath. Wait for an equilibrium temperature to be established.

3.4 Place 5 to 6 cc. of resin in a clean test tube, add a clean stirring rod with its attached holder and insert into heated bath. Couple the stirring rod to the torsion motor.

3.5 Turn on the test meter.

3.6 Gelation occurs when the buzzer rings and turns off the timer.

3.7 Notes (Cautions):

3.7.1 Monochlorobenzene is a toxic solvent, use with adequate ventilation. Handle with rubber gloves.

3.7.2 Keep torsion wire free from kinks and maintain a gap of 1.027 inches between vise jaws.

3.7.3 Adjust actuating switch to approximately 1/4".

3.7.4 Maintain stirring rod in the center of the test tube.

3.7.5 Keep a test tube in the bath when not in use.

M-R-8 RDS-Viscosity of Various Temperatures With Rheomatic Dynamics Spectrophotometer

1. SCOPE

1.1 This method describes a procedure for the determination of resin viscosity at various temperatures using the Rheometric Dynamics Spectrometer (RDS).

2. EQUIPMENT AND MATERIALS

2.1 Rheometrics Dynamic Spectrometer with 50 mm. parallel plates, and operators manual

2.2 Oven, forced air

2.3 Balance, 0.1 gm. accuracy

2.4 Stopwatch

2.5 Silicone Release paper

2.6 Tongue depressors

2.7 RDS systems manual

3. SAMPLE

3.1 Raw resins require one (1) oz. minimum

3.2 Powders require three (3) oz. minimum

3.3 Mixed resins require two (2) oz. minimum

4. PROCEDURE

4.1 General Test Parameter Capabilities

4.1.1 Temp. range - 200 ° to 395 °C. linear stepwise heating profile

4.1.2 Strain range is 0 to 100%

4.1.3 Frequency range is 0.01 to 100 radians/sec.

4.1.4 Physical separation of plates (gap) is 0.01 to 5.0 mm

4.2 Instrument Set Up (Method I)

4.2.1 Power up instrument and adjust air pressure to 45 psi.

4.2.2 Select the following starting front panel parameters:

4.2.2.1 Starting temperature - 30°C.

4.2.2.2 % Strain - 50 (per BMS 8-256)

4.2.2.3 Frequency rate - 10 (per BMS 8-256)

4.2.2.4 Mode - cure sweep

4.2.2.5 Test - Parallel plate

4.2.3 Select operational parameters by typed responses as follows:

4.2.3.1 Total time - 140 min.

4.2.3.2 Sweep rate - 2.0°C./min.

4.2.3.1 2 min./test point

4.2.4 Press: "Geometry Test"

4.2.4.1 Gap - 0.50 mm.

4.2.4.2 Radius - 25.00 mm.

4.2.5 Press "Plotter"

4.2.5.1 X-axis min. - 0

4.2.5.2 X-axis max. - 14O

4.2.5.3 Y-axis min. - 1EO

4.2.5.4 Y-axis max. - 1E5

4.2.5.5 *-yes, no for all other quantities

4.2.6 Press "Print"

4.2.6.1 *, ', " - Torque - yes*

4.2.6.2 all others - no

4.2.6.3 * Time and temperature are automatically selected.

4.2.7 Plotter and Paper

4.2.7.1 Insert K and E paper type 46-6210 (5 cy. x 7 in.)

4.2.7.2 Zero lower left and upper right corners of each chart by adjustment of the zero and venier controls respectively.

4.2.8 Sample Insertion

4.2.8.1 Parallel plates are brought into contact (>10% force normal) and the micrometer set to zero.

4.2.8.2 Sample material should be placed on plates preheated to 70°C. and allowed to flow as the gap is set. The plates are then allowed to return to approx. 30°C.

4.2.8.3 Using the single sweep mode, the resin viscosity is allowed to equilibrate for 2-3 minutes at 30°C. (% torque is monitored to prevent input overload).

4.2.9 Material Testing Using "Cure Mode" is initiated by the following Sequence:

4.2.9.1 Temperature = 30°C.

4.2.9.2 Mode - Cure

4.2.9.3 Test - Parallel Plate

4.2.9.4 % Strain = 50.00

4.2.9.5 Rate - 10.00 rad./sec.

4.2.9.6 START

4.2.9.7 Temperature = 240°C.

4.2.10 Viscosity of the resin sample over the temperature range will be plotted and printed. At the desired end of the test, shut down is as follows:

4.2.10.1 Temperature - 30°C

4.2.10.2 Reset

4.2.11 Plates may be cleaned by soaking in acetone

5. CALCULATION AND REPORTING

5.1 Report RDS viscosity at various temperatures required by the applicable specification.

5.2 Attach completed graph with all pertinent information (see para. 4.2.9)

M-R-9 Liquid and Gel Permeation Chromatography Determining Mol Weights

1. SCOPE

1.1 This procedure describes methods for conducting liquid chromatogaphy and gel permeation chromatography of phenolic resins. It analyzes the molweight of contituents.

2. EQUIPMENT AND MATERIALS

2.1 HPLC Spectra-Physics SP8000 with UV detector and autosampler, and operators manual.

2.2 M-Styragel chromatography columns, 2 X 100 A, 2 x 500 A <u>(USE NO WATER)</u>

2.3 Syringe, 10 cc.

2.4 Gas tight syringe, 5 cc.

2.5 Needles

2.6 Scissors

2.7 Centrifuge tubes, 50 ml. disposable

2.8 Centrifuge

2.9 Graduated cylinder, 10 ml.

2.10 Balance, Analytical, Sensitivity 0.001 gm.

2.11 Beaker

2.12 Millex-SR filter units, 0.5

2.13 Membrane TFE filter paper, 0.45

2.14 B & J tetrahydrofuran, UV grade, THF

2.15 Helium (zero grade) gas

2.16 Nitrogen gas

3. PREPARATION OF SAMPLE SOLUTION

3.1 <u>Resin Samples</u>

3.1.1 Weigh out 0.25 ± 0.02 gm. of resin into a glass vial (labeled)

3.1.2 Add 10 ml. 100% THF into the vial

3.2 Resin Mixes

3.2.1 Weigh out 3.0 ± 0.02 gm. of sample into a centrifuge tube

3.2.2 Add 25.0 ml. of 100% THF into the centrifuge tube

3.2.3 Agitate for 15-50 minutes

3.2.4 Centrifuge for 2-5 minutes at appropriate speed

3.3 Prepregs

3.3.1 Cut sample into approximate 1/2" x 1/2" squares

3.3.2 Weigh out 6.0 ± 0.5 gm. of sample into centrifuge tube

3.3.3 Add 25 ml. 100% THF into the centrifuge tube

3.3.4 Agitate for 15-50 minutes

3.3.5 Centrifuge for 2-5 minutes at appropriate speed

3.4 Solvent Preparation

3.4.1 Filter solvent (1-2 liters) by using 0.45 um. TFE filter

3.4.2 Degas with He 1/4 to 1/2 hour (high flow)

3.4.3 Decrease the flow rate before running

4. INSTRUMENT PROGRAMMING

4.1 Date Assignment

```
D: 00:00:00         00:00:00
month day year    hour min. sec.
D - Display
```

4.2 Report File Assignment

```
R:1 - report file
REPORT FILE 1
RENAME:
SAMPLE:-------------
SAMPLE VOLUME: 10 L
SAMPLE CONC.: 20 MG-ML
OPERATOR NAME: (your name)
DETECTOR 1: UV. 254NM 0.1 AUFS
DETECTOR 2: ;
```

COLUMN TYPE; SIZE: 2x100A, 2X500A, -STYRAGEL
MOBILE PHASE: Isocratic

A: ;
B: ;
C: THF
END OF DIALOG

4.3 Parameter File Assignment

S:1 parameter file 1

PARAMETER SET: 1

RENAME: ;

1. LINK: ;
2. INITIAL DELAY: 1
3. RUN TIME: 30
4. NO. OF CYCLES: 6
5. FLOW RATE: 2.00
6. FLOW MODE: QI
7. TEMPERATURE: 35
8. MOBILE PHASE FILE: 1
9. REPORT FILE: 1
10. GRAPH FILE: 1
11. DATA SYSTEM FILES: 1
12. CHAN 1:1
13. CHAN 2: ;
14. FULL TIME: 1
15. FLUSH TIME: 1
16. REPETITIONS PER VIAL: 1
17. TIMED EVENTS: XX

END OF DIALOG

4.4 Graph File Assignment

G = 1 graph file 1

G11
G1E5
G1I5
GVO.5
XX

EXIT GRAPH EDIT

4.5 Mobile Phase File Assignment

M = 2 mobile phase file 2

MOBILE PHASE FILE 2

```
WHICH SOLVENTS? C

END OF DIALOG

MI2

M;1

MOBILE PHASE FILE 1

WHICH SOLVENTS? C

END OF DIALOG

MI1
```

4.6 Data File Manipulation

```
FI1  Data system file (file center)

nm gpc-QC

CH 001
OD 002
IX 001
RN 001
NR 001
PW 015
PT 0150
MA 5000
FD
VE
FILE 001

RN 001    NR 001   PW 015      PT 0105
FS 000    MS 0     MA 5000     IM 00
PP 004    TI 000   RO 000
NM GPC-QC
CH 001    OD 002   IX 001      SN 000

FF  function file
APO
FD
VE
FILE 001

AR 0  IR 0   DC 0   NZ 0    FX 0   EC 0
TP 1  NP 0   NT 0   AA 0    XD 0   HD 0


XX

METHOD o
```

4.7 Review File Values

```
S-1

PARAMETER SET: 1

INITIAL DELAY: 1 RUN TIME: 30 NO. OF CYCLES: 6
FLOW RATE: 2.00  FLOW MODE: Q1  TEMPERATURE: 35
MOBILE PHASE FILE: 1 REPORT FILE: 1 GRAPH FILE: 1
CHAN 1:1
FILL TIME: 1  FLUSH TIME: 1  REPETITIONS PER VIAL: 1

R-1

-------------VOL: 10 UL  CONC:  20 mG-ML   NO:  1
(your name)
    FLOW RATE:  2.0     PRESSURE:  0000
COLUMN:  2x100A, 2X500A,  --STYRAGEL
   CHAN 1:  U.V.  254NM     0.1  A FS
   MOBILE PHASE: ISOCRATIC
  MOBILE PHASE FILE 1
    100.0% C
    G-1
    GRAPH FILE: 1
    Channel 1:  Plot Var.:  DET. 1  Chart Spd.: 0.5
    Polar: L ATTN.: 5   Zero: 0%  Inten.: 5
    Channel 2:  Inoperative
    M-1
    Mobile Phase File: 1
    100.0% C
```

5. PROCEDURE

5.1 HPLC: Start-up

5.1.1 Turn on He and N_2

5.1.2 Adjust the He pressure to 10 psi and N_2 pressure to 75 psi.

5.1.3 Turn on both switches, Wavelength Drive at "on" position. Set wavelength at 254 nm.

5.1.4 Turn on the Spectrophotometric Detector, make sure it is ".4 amp." Set range at 0.1 AUF

5.2 Automatic Operation

5.2.1 Be sure both detector and auto sampler are on

5.2.2 Keep He purge, the solvent with low flow rate

5.2.3 Type AT1
DID
FM1
MI1
SB1

5.2.4 Use the inner knob on the detector range selector to adjust the read out to -1200 (detector signal) or type GB and adjust the baseline 1 inch to the left margin, then type GX to start plotting.

5.2.5 Fill one gas tight syringe with sample solution and the other one with THF; attach each syringe to one Miller-SR filler unit.

5.2.6 Place the injector handle in the "LOAD" position and type SO to open the injector sample solenoid.

5.2.7 Inject 1 cc. of THF to flush the injector loop, then inject 1 cc. of sample solution and type SK to close the sample solenoid.

5.2.8 Wash the syringe with the THF twice; fill it with the next sample.

5.2.9 After LC, inject automatically, replace the injector handle in the "LOAD" position and repeat the injecting operation.

5.2.10 Check the condition by typing:

E- elapsed time since injection
T- present oven temperature
P- pressure
C- current % C at ternary valve
DID- dector signal
F- flow rate

5.3 Calibration (before running sample solutions)

5.3.1 Run a THF blank in order to plot the baseline

5.3.2 Run molecular weight standards and plot molecular weight vs. retention time on graph paper

Mole Weight Standards:

M.W.	Compound
93	Toluene
570	Polystyrene
955	"
3600	"

5.4 Termination

5.4.1 Type EX to end run

5.4.2 Type SX to stop parameter set

5.4.3 Shut down all gas flows

M-R-10 Purity of Solvents by Chromatography

1. SCOPE

1.1 This method describes a procedure for routinely checking purity of solvents through the use of gas chromatography. It is similar to ASTM-D3792.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

2.1.1 Gas Chromatograph with Thermal Conductivity Detector- Hewlett-Packard Model 810 or any other with similar capability and performance and operators manual (preferably equipped with an integrator).

2.1.2 Two 6 feet by 1/8 inch stainless steel columns with packing of Porapak Q. 80/100 mesh. (Water Associates)

2.1.3 Hamilton syringe 701N or equivalent (10 microliter capacity)

2.1.4 Pipettes, 10.0 ml, 1.0 ml, 0.1 ml

2.1.5 Air tight sample bottles

2.2 Materials

2.2.1 High Purity Solvents - Pure samples of each type of solvent to be checked as well as any suspected impurity solvents should be obtained. Nanograde purity is good. For acetone, Fisher A-20 is recommended.

2.2.2 Distilled Water

3. SAMPLING

3.1 Representative Sample The sample of solvent to be injected into the G.C. should be representative. Exercise due care in certain cases where the test solvent has a constantly changing make-up.

3.2 Number of Determinations. When quantifying solvent impurities, make at least 2 runs with the test samples to check accuracy.

4. PREPARATION

4.1 Column Conditioning

4.1.1 If columns are new they should be conditioned prior to use according to manufacturer's instructions.

4.1.2 Prior to sample injection, the column should be clear of any contaminates from any previous injections. An isothermal run of about 225°C. for 1/2 hour will clear the column of most contaminates. Care should be exercised that the column temperature does not exceed the maximum rated temperature (250° C.) since the column packing Porapak Q will start to degrade. This purging of the column should be done either with the columns disconnected from the thermal conductivity detectors, (Bridge current, off) or with the T.C. temperature well above the column temperature if the columns are connected. This will insure that material coming out of the column will not condense on a cooler T.C. filament. All other recommended precautions and procedures concerning column care contained in the G.C. operating manual should be followed.

4.2 Reference Sample Preparation. Various reference samples of pure solvents as well as samples containing known percentages of "impurity" solvents should be made up and stored in air tight bottles. These reference samples will be run later to produce Chromatograms for comparison to test solvents. The analysis at hand will dictate what "impurity" solvents to use in the reference sample and in what percentages. For example, if the quality of the recycled acetone obtained from distilling common pre-preg systems is to be checked, the main impurities might be methyl cellosolve, toluene, methanol, and maybe some water. Therefore, reference samples of pure acetone and 1%, 2%, 3%, 5%, and 10% by volume of these impurities in acetone would probably be desirable.

5.1 Run Conditions Although run conditions will vary with the particular analysis, the conditions listed below will be close to those used for common low boiling solvent analysis (acetone, etc.). If no idea of contaminate nature or percentages is known, a temperature program from 50°-195°C. is a good place to start.

5.1.1 Helium Flow - Pre-set, equal on "A" and "B" columns, 30-40 ml./min.

5.1.2 Main Power, oven power, recorder power, chart drive, oven control, bridge switch, detector temp. switch - all on.

5.1.3 Program Selector - Isothermal

5.1.4 Oven Cover - Automatic

5.1.5 Bridge Current - 200 milliamps

5.1.6 Detector (T.C.) Temperature - 200°C.

5.1.7 Injector Temperature - 195^{o}C.

5.1.8 Oven Temperature - 150^{o}C.

5.1.9 Chart Speed - 1.0 inch/min.

5.1.10 Attenuation - 16 (initially)

5.2 Sample Injection The integrator and recorder baselines will be quiet and without drift before each injection.

When both baselines are stable, 10.0 ml. of air is injected into column "A". The chart is immediately marked at the injection point. From this injection point to the maximum point of the air peak in minutes is Tm (retention time for unabsorbed gas).

After baseline has stabilized, a reference sample (2.0 ml) can then be injected. Again mark injection point and observe retention time for each component of reference sample. The adjusted retention time for each component is $(Tr'-Tm_1)$.

After again allowing the baseline to stabilize, the reference sample injections are followed by the test samples. Many different reference samples may then be needed to make an adequate qualitative and quantitative analysis.

6. COMPOUND IDENTIFICATION

6.1 Compound identification is based upon the adjusted retention time $(Tr'-Tm_1)$. The adjusted retention time is a function of the carrier gas, column temperature, column pressure drop, and other factors, thereby indicating the importance of maintaining constant conditions while comparing reference samples to test samples.

6.2 The recommended procedure for qualitative analysis is as follows: With the column at operating conditions, inject suitably sized samples of liquids, one compound at a time. Adjust the instrument attenuation so that the peak height is at least 50 percent of full scale. Inject the sample quickly into column "A". Again mark the injection point, and measure retention time (Tr') for each peak in minutes to at least 2 significant figures. Make duplicate determinations and use the average value for compounds relative to at least one injected standard reference material. All data should be recorded carefully with each run. For qualitative analysis, the retention times of the sample peaks must then be matched with those of known standards obtained under the same operating conditions. Occasionally, it is necessary to analyze a sample in 2 different columns under different conditions, measuring the retention times of the components and compare them with previously determined standards, in order to determine which of 2 (or more) compounds having the same retention time is present. With common solvents, one type of column like Porapk Q. is often all that is necessary.

7. QUANTITATIVE ANALYSIS

7.1 The areas under the peaks of the chromatogram are quantitative measures of the amounts of the corresponding compounds. The relative area is usually proportional to the concentration if the relative responses of the sample concentration components are equal. If this is not the case, the corrected area of the component is used. It is obtained by dividing the peak area by the relative response of that component. Therefore, best results require a calibration curve of peak area, (or height) versus known amounts of pure components for the given operating conditions.

7.2 Although the establishment of the true peak area by integration is the standard method for quantitative analysis, multiplying the peak by the width of the peak taken at the half-height gives proportional values especially for sharp, symmetrical, completely resolved peaks. With this method, a chart speed that will give at least a 5 mm. half-width should be used.

7.3 When the volumes injected are the same, the following Relationships apply:

$V_1 = (H_1/H_2) \times V_2$ and $W = V_1 \times (D_1/D_2)$

Where:

V_1 = Vol. percent of sample component
H_1 = Compound peak area or height in sample
H_2 = Compound peak area or height in standard
V_2 = Volume percent of sample component in standard
W = Weight percent of sample component
D_1 = Density of pure sample component
D_2 = Density of sample

7.4 An outline of the recommended procedure used for the quantitative calibration is as follows: With the column at equilibrium operating conditions, inject the liquid samples of pure components, one compound at a time. More than one at a time is acceptable if you're sure what peak corresponds to which compound. Vary the sample size (or concentration) to cover the desired range. Samples of a given size should be injected until at least three peaks have the same area, ideally ± 1 percent at the same attenuation. Adjust the attenuation in all cases to keep the peak on scale and with a height of at least 50 percent. Data should be converted to some attenuation previously chosen as a basis for calculations, for example, x 1, x 8, x 64, etc. Multiply the actual peak height by its corresponding attenuation and divide by the basis attenuation. Plot this peak height versus quantity of each compound and connect the data points with a smooth calibration curve. Label the axis on the plot and for each such plot show the following information: Data, column description, and operating conditions. Record also the purity of the compounds used. The syringe, pipette, or other device must be flushed at least 3 times with each new compound prior to injecting a sample of that component.

Actual sample injections will generate peaks which can then be easily quantitated using the calibration charts.

7.5 An example of a possible analysis follows:

All runs are made with identical conditions.

All samples injected are 2.0 ml.

Reference samples -Tr'

Acetone - 7.5 min.
Methanol - 2.7 min.
Ethanol - 5.0 min.

From Calibration Curves

Attenuation	Peak Area	Volume % of Ethanol
16	25	1
16	50	2
16	100	4
8	100	8

Sample peak of impurity in solution is Tr' = 5.0 min. and peak area of 75 at 16 attenuation. From previous calibration work, peak is ethanol. 3% by Volume.

8. CALIBRATION

8.1 Before each calibration and series of determinations (or daily) condition the column at 200°C. for 1 hr. with carrier gas flow.

8.2 Determination of Relative Response Factors - Anhydrous 2-propanol is used as an internal standard. The response factor to water relative to the standard is determined by means of the following procedure. See Fig. 1 for a typical chromatogram. It is good practice to determine the response factor daily or with each series of determinations.

8.2.1 Weigh about 0.2 gm. of water and 0.2 gm. of 2-propanol to the nearest 0.1 mg. into a septum sample vial. If it has been determined that a correction for the water content is necessary, weigh 2 ml. of dimethylformamide into the vial. If the dimethylformamide is anhydrous, simply add 2 ml.of it as weighing is not necessary.

8.2.2 Inject a 1 unit aliquot of the above solution onto the column and record the chromatogram. The retention order and approximate retention times after the air peak are 1. water, about 0.7 min; 2. 2-propanol, about 2.8 min.; and 3. DMF, about 7 min.

8.2.3 The preferred procedure to obtain the water content of the DMF is by Karl Fischer titration. If this has been done, calculate the response factor for water by means of the following equation:

$$R = \frac{W_i A_{H2O}}{(W_{H2O} + PW_S)A_i}$$

Where:

R = response factor
W_i = weight of 2-propanol, gm.
W_{H2O} = weight of water added, gm.
W_S = weight of Dimethyl-formamide, gm.
A_{H2O} = area of water added
A_i = area of 2-propanol peak
$P = \frac{\text{Weight \% water in DMF}}{100}$

8.2.4 If Karl Fischer titration is not available, the following procedure may be used to obtain a reasonable estimate of the response factor.

8.2.4.1 Inject the same size aliquot of DMF and 2-propanol mixture, but without added water, as a blank. Note the area of the water peak in the blank.

8.2.4.2 The response factor for water is calculated by means of the following equation:

$$R = \frac{W_i(A_{H2O} - B)}{W_{H2O}A_i}$$

Where:

R = response factor
W_i = weight of 2-propanol, gm.
W_{H2O} = weight of the water, gm.
A_i = area of 2-propanol peak
A_{H2O} = area of the water peak
B = area of the water peak in the blank

9. PROCEDURE

9.1 Weigh to the nearest 0.1 mg. 0.6 gm. of water-reducible sample and 0.2 gm. of 2-propanol into a septum vial. Add 2 ml. of dimethyl-formamide into the vial. Seal the vial. Prepare a blank containing the 2-propanol and dimethylformamide but no sample.

9.2 Shake the vials on a wrist action shaker or other suitable device for 15 min. To facilitate settling of solids allow the samples to stand for 5 min. just prior to injection into the chromatograph. A low speed centrifuge may also be used.

9.3 Inject a 1 ml.- sample of the supernatant from the prepared solutions onto the chromatographic column. Record the chromatograms using the conditions described in Table 1.

10. CALCULATIONS

10.1 Measure the area of the water peak and the 2-propanol internal standard peak and multiply each area by the appropriate attenuation factor to express the peak areas on a common basis. Use of an electronic integrator is recommended to obtain the best accuracy and precision. However, triangulation, planimeter, paper cut out, or ball and disk integrator may be used.

10.2 Determine the water concentration in the sample by means of the following equation:

$$H_2O, \% = \frac{A_{H2O} \ X \ W_i \ X \ 100}{A_i \ X \ W_p \ X \ R}$$

Where:

A_{H2O} = area of water peak
A_i = area of 2-propanol peak
W_i = weight of 2-propanol added, gm.
W_p = weight of sample, gm.
R = response factor determined in 8.2.3 or 8.2.4.2

10.3 Correction for Water Content of Solvent

10.3.1 If the blank indicates the presence of a detectable peak for water in the dimethylformamide used as solvent, make a correction in the calculation.

10.3.2 To make the correction, the water content of the dimethylformamide is determined either by chromatography (8.2.4) or, preferably, by Karl Fisher titration (8.2) Calculate the water content due to solvent by using the following equation:

$$H_2O(S)\% = \frac{(W_S)(P)100}{W_P}$$

where:

W_S = weight of dimethylformamide, gm.
W_P = weight of sample, gm.
$P = \frac{\text{weight \% water in DMF}}{100}$

10.3.3 The water content of the sample in this case is the difference between the total percent determined in 10.2 and the correction for the solvent water content as determined in 10.3.2. For isothermal operation set the column temperature at 140°C. After the 2-propanol has cleared the column adjust the temperature to 170°C. until DMF clears the column. Reset the temperature to 140°C for subsequent runs.

Table I Instrument Parameters (Typical Conditions)

Detector thermal conductivity 1.22m. inside Column diameter by 3.2mm. outside diameter packed with 60 to 80 mesh porous polymer packing

Temperatures:	
Sample inlet	200°C.
Detector	240°C.
Column	
Initial	80°C.
Final	170°C.
Program rate	30°C./min.
Carrier Gas	helium or nitrogen
Flow rate	50 ml/min.
Detector current	150 mA.
Specimen size	1 1

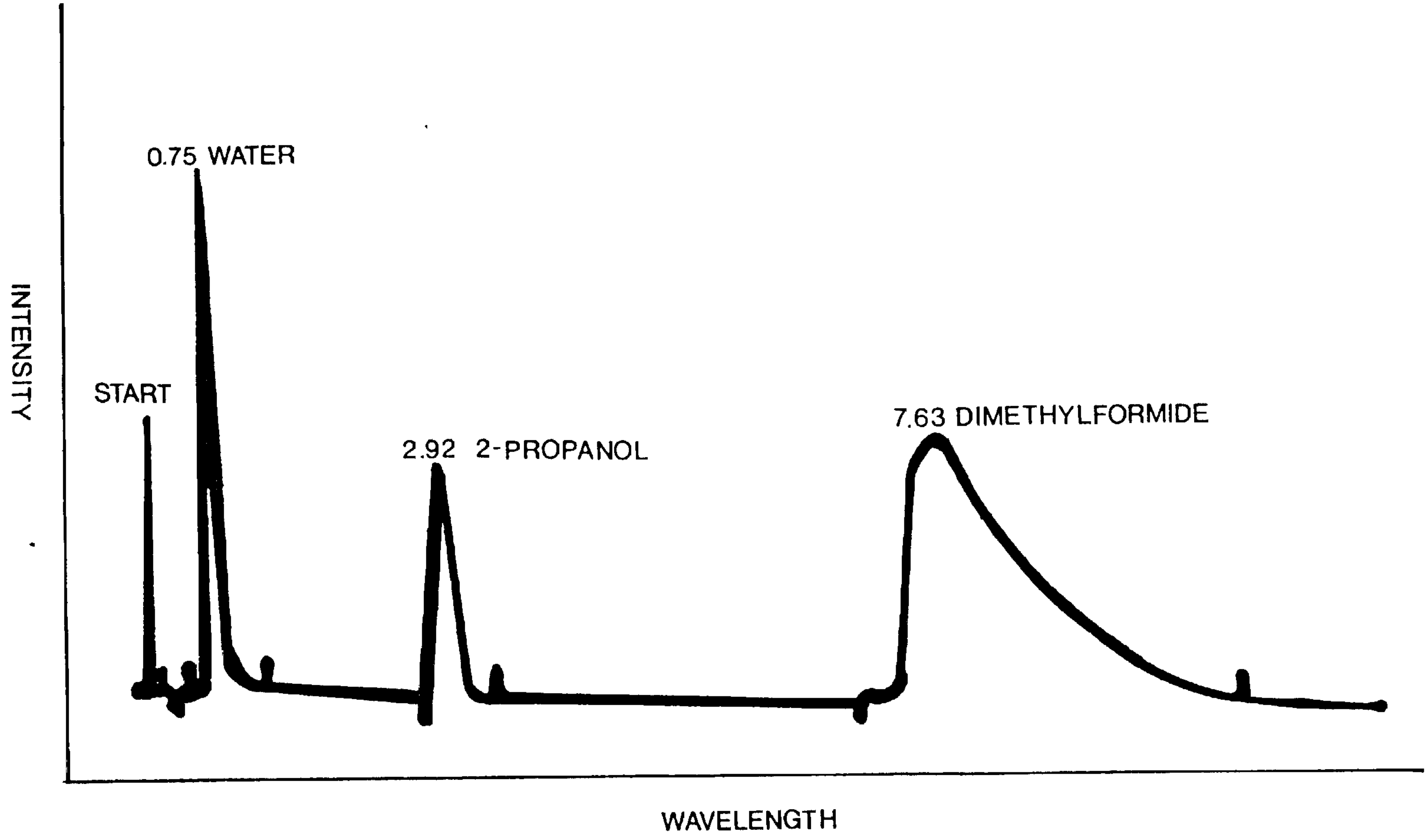


Fig. 1 - Typical Chromatograph

M-R-11 Infra-red Spectrophotometer Analysis of Resins

1. EQUIPMENT

1.1 Double beam infra-red spectrophotometer Perkin Elmer Model 21 or equivalent, and operators manual.

1.2 Matched sealed NaCl Cells 0.20 mm. path length.

2. EQUIPMENT OPERATION

2.1 Spectrophotometer must be warmed up for a period of at least 10 minutes before operation.

2.2 Before operation, spectrophotometer must be balanced in accordance with manufacturer's procedure. (See Operators manual.)

2.3 Sub-zero and noise checks must be run at one (1) week intervals to assure proper operation of instrument.

2.4 Cells used will be marked to assure the reference cell will always be used in the reference beam and the sample cell will always be used in the analytical beam.

2.5 A spectrum will be run at one (1) week intervals with acetone in both the sample cell and reference cell to assure that the cells are in proper operating condition. The spectrum obtained should not vary more than ± 3% @ 100% transmission from a straight line from 9.5 to 15.0 microns.

3. INSTRUMENT SETTINGS

3.1 Slit program..927

3.2 Response..1

3.3 Speed..0.8 microns/min

3.4 Auto suppression..0

4. PROCEDURE

4.1 Weigh out enough resin to produce a 4% solution of solids in spectrograde acetone. Note 1.

4.2 Filter mixture through S and S No. 589 Blue Ribbon filter paper. (Flute the paper for faster filtration).

4.3 Rinse reference and sample cells three time with spectrograde acetone. Flush each cell with dry nitrogen. Load reference cell with spectrograde acetone. Load sample cell with extract.

4.4 Balance spectrophotometer and set at 8.0 microns. Insert sample and reference cells. Absorbance at 8.0 microns shall be between 0.70 and 0.75. If absorbance is greater than 0.75 adjust concentration of remaining solution by addition of spectrograde acetone. If absorbance is less than 0.70 evaporate in a vacuum oven at room temperature.

4.5 Scan the spectrum from 2.0 to 15.0 microns.

4.6 Mark chart with material designation, lot or batch number, instrument settings, date, solvent, and operators initials.

4.7 Compare spectrum obtained with standard spectrum for presence of foreign absorption peaks. A standard spectrum will be furnished by the Fiberite Corp.

Note 1 - Double distilled CP Acetone may be used.

M-R-12 Moisture Content of Resins by Use of a Photo Volt Unit

1. APPARATUS

1.1 Aquatest IV or equivalent Photo Volt

2. EQUIPMENT

2.1 Glass sample vials with caps - a suitable vial volume is 24 ml.

2.2 Syringe - Tuberculin type 1 ml. size

2.3 Syringe needles - Need to be 5" to reach into the reagent.

2.4 Mechanical shaker - An ultrasonic bath can also be used if care is taken not to heat samples.

2.5 Vacuum oven

3. REAGENT

3.1 Methanol- Reagent grade, low in H_2O (must be less than .025%). Dry with molecular sieves.

4. SAMPLE PREPARATION

4.1 Dry the sample vials and caps by heating in a vacuum oven at 110°C. long enough to drive off any moisture (about 15 minutes).

4.2 Remove and place immediately into a desiccator to cool.

4.3 When cooled to room temperature, weigh approximately 3-5 grams of sample into each vial to the nearest 0.1 mg. (The desirable sample size contains 100-3000 micrograms of H_2O so adjust the sample size accordingly).

4.4 Add 20 ml. of Methanol using a volumetric pipet.

4.5 Prepare a solvent blank by pipeting 20 ml. of Methanol into dried vial and cap immediately.

4.6 Shake vials on a mechanical shaker for 30 minutes. The samples are now ready for analysis.

5. ANALYSIS

5.1 Determine the moisture content of the blank solvent first. Draw a 1 ml. sample into the syringe, being careful to remove all air bubbles. Inject sample into the Aquatest IV titration vessel. The sample must be discharged below the surface of the vessel solution.

5.2 Perform this step in triplicate and calculate the average micrograms of H_2O in the solvent blank. Perform triplicate runs on each of the sample vials and average the values.

6. CALCULATIONS

$$\frac{[(\text{mcgs } H_2O \text{ sample})-(\text{mcgs } H_2O \text{ in Blank})] \text{x } 20\text{ml. Meth x } 100}{\text{Sample wt. in g. x } 106}$$

= moisture content, % water

M-R-13 pH of Liquid Resins by Use of a pH Meter

1. SCOPE

1.1 This method covers a procedure for determining the pH value of liquid resins.

2. EQUIPMENT

2.1 pH meter. Accumet model 825 MP with glass body combination electrode and temperature compensator. Operators Manual would be of use.

2.2 pH buffers, 4, 7, 10.

2.3 150 ml. beakers

2.4 Distilled water.

3. PROCEDURE

3.1 <u>Calibration</u>

3.1.1 Perform a 1 point calibration using the pH buffer nearest to the expected pH measurement.

3.1.2 Remove the electrode from the storage solution, rinse with distilled water and place in fresh buffer solution of the necessary pH.

3.1.3 Press "stby/meas".

3.1.4 Press "1 pt cal".

3.1.5 Enter electrode efficiency from the most recent 2 point calibration. e.g. 91.4% is entered as ".9140" press "enter".

3.1.6 Enter pH buffer value used e.g. "7.00" press "enter".

3.1.7 Wait for millivolt reading to stabilize and press "enter".

3.1.8 Press "stby/meas".

3.2 <u>pH Measurement</u>

3.2.1 Remove the electrode from the buffer solution, rinse with distilled water and immerse in sample to be measured.

3.2.2 Press "stby/meas".

3.2.3 Press "mode" until pH 1 appears in the lower right corner (skip this step if pH 1 is already displayed).

3.2.4 Wait for reading to stabilize and read pH value to the nearest 0.1 pH unit.

3.2.5 Press "stby/meas", remove electrode from sample and rinse with acetone then distilled water.

3.2.6 Place the electrode back into the storage solution.

M-R-14 Resin Solids and Total Solids for Both Filled and Unfilled Systems

1. SCOPE

1.1 This method is used to determine the percent of non volatile material in solutions of formaldehyde-phenol resins or other resin solids.

2. EQUIPMENT

2.1 Analytical balance (0.001 gm. sensitivity)

2.2 Aluminum dry dish - Cenco No. 12720

2.3 Oven-gravity convection type-thermostatically controlled at 135 ± 1°C.

2.4 Desiccator

3. PROCEDURE

3.1 Preheat three drying dishes in the oven at 135 ± 1°C for 30 ± 2 minutes. Cool the dishes in the desiccator to room temperature and weigh to the nearest milligram (0.001 gm.).

3.2 Weigh 2.8 to 3.2 gm. of resin solution to the nearest milligram, into each of the tared, dried dishes.

3.3 Place the samples in the drying oven. Heat for 3 hours at 135 ± 1°C. and then transfer the samples to the desiccator and cool to room temperature.

3.4 Reweigh the samples to the nearest milligram.

4. CALCULATIONS

4.1 <u>For unfilled resin systems:</u>

$$\%\ \text{Resin Solids} = \frac{\text{Final total wgt. - wgt. of dish}}{\text{Original total wgt. - wgt. of dish}} \times 100$$

4.2 <u>For filled resin systems:</u>

$$\%\ \text{Total Solids} = \frac{\text{Final total wgt. - wgt. of dish}}{\text{Original total wgt. - wgt. of dish}} \times 100$$

M-R-15 Refractive Index on Resins

1. SCOPE

1.1 The refractive index is determined on all resins.

2. EQUIPMENT

2.1 Abbe refractometer, Bausch and Lomb Model 519A

2.2 Constant temperature bath (25.0° ± 0.5°C.)

2.3 Pumping system for water

3. PROCEDURE

3.1 Set the constant temperature bath at 25.0°C ± 0.5°C. Assemble pumping system to circulate water through the refractometer. Calibrate the refractometer with the standard sample.

3.2 Place a drop of resin on the glass prism and close. Turn on light and adjust instrument so that the line is at the intersection of the cross hairs.

4. CALCULATIONS

4.1 Report reading on the scale.

NOTE: Refractometer must be calibrated before each series of determinations. Calibration is accomplished using a sample of known refractive index supplied with the instrument. Proceed with the calibration as directed by the instrument manufacturer.

M-R-16 Specific Gravity by Hydrometers on Liquid Resins

1. SCOPE

1.1 This method is applicable for determining the specific gravity of liquid resins.

2. EQUIPMENT

2.1 Series of hydrometers

2.2 Ungraduated cylinder

2.3 Thermometer

3. PROCEDURE

3.1 Pour liquid resin into ungraduated cylinder and adjust temperature to 25° ± 0.5°C. Place into the liquid a hydrometer covering the range of specific gravity of the liquids being tested. Read the hydrometer at the level of the liquid and record.

M-R-17 Resin Reinforcement in Aldehyde Phenolic Prepregs

1. SCOPE

1.1 This method is used to determine the percent of non-volatile reinforcement in an aldehyde phenolic resin.

2. EQUIPMENT

2.1 Analytical Balance; accurate to 0.1 mg.

2.2 Crucibles

2.3 Oven - gravity convection type thermostatically - controlled at $135^{\circ} \pm 1^{\circ}C$.

2.4 Muffle furnace set at $788^{\circ} \pm 5^{\circ}C$.

3. PROCEDURE

3.1 Determinations are run in duplicate

3.2 Weigh approximately three grams of resin solution into tared crucibles which have been previously fired at $788^{\circ} \pm 5^{\circ}C$. for 1 hour.

3.3 Place crucibles in $135^{\circ} \pm 1^{\circ}C$. oven for 3 hours and transfer to a desiccator to cool.

3.4 Weigh to the nearest milligram and designate as W_1.

3.5 Place crucibles in $788^{\circ} \pm 5^{\circ}C$. muffle furnace for 5 hours ± 5.0 min.

3.6 Transfer crucibles to a desiccator to cool.

3.7 Reweigh samples to the nearest milligram and designate as W_2.

4. CALCULATIONS

$$\% \text{ Resin Reinforcement} = \frac{W_2 - \text{weight of crucible}}{W_1 - \text{weight of crucible}} \times 100$$

M-R-18 Degree of Advancement or Polymerization by Infra-Red Technique

1. SCOPE

1.1 This method describes a procedure for the determination of the degree of advancement or polymerization of aldehyde-phenolic resins. An infrared analytical technique is employed which differentiates molecular structural changes resulting from polymerization. This method applies to raw resin as well as preimpregnated fabric, to unmodified as well as modified resins, and to filled as well as unfilled prepregs.

2. EQUIPMENT AND MATERIALS

2.1 <u>Equipment</u>

2.1.1 Infrared spectrophotometer, Beckman IR-8, or equivalent, and operators manual

2.1.2 Sodium chloride variable path cell

2.1.3 Sodium chloride liquid cell, 0.20 mm.

2.1.4 Funnel, 60°

2.1.5 Beakers, 100 ml.

2.1.6 Stirring rod, glass

2.1.7 Scissors

2.1.8 Graduated cylinder, 50 ml.

2.1.9 Syringe, 2 ml.

2.1.10 Centrifuge

2.1.11 Vacuum oven

2.1.12 Aluminum pans, 2 1/2" diam. 1/2" height

2.2 <u>Materials</u>

2.2.1 Acetone, water free, saturated with silica gel

2.2.2 Filter paper, S & S No. 589 Blue Ribbon, or equivalent

2.2.3 Infrared chart paper, 2.5 to 16 microns, absorbance

3. SAMPLING

3.1 Sufficient sample shall be taken to provide approximately 2 grams of resin solids.

4. PROCEDURE

4.1 Operation and Calibration

For operation and calibration of the spectrophotometer, see SCM-7-01 for the IR-8 instrument or other equivalent procedures.

4.2 Adjustment of variable cell

The variable path cell shall be adjusted to the exact thickness of the 0.20 mm. sodium chloride cell. In order to determine that the thicknesses are closely matched, fill both cells with acetone using a syringe. When properly matched, the resulting spectrum will not vary by more than ± 3.0% from 100.0% transmission when measured between $9.\bar{5}$ and 15.0 microns. Adjust the variable path cell as necessary in order to attain the above tolerance.

4.3 Solution Preparation

4.3.1 Raw Resins Raw resins known or suspected of having solvents other than acetone shall be devolatilized under vacuum without heat for a minimum of four hours or until disappearance of the absorption bands indicating their presence. This may require as long as 24 hours for some solvents such as isopropanol. (As an alternate, isopropanol can be removed by adding 30 ml. acetone to 2 gm. aldehyde-phenolic resin and periodically stirring at RT without vacuum over 0.5 hr. during which both acetone and isopropanol are largely volatilized). Add sufficient acetone to the resin to effect a 4 to 6 % solids solution. Stir with a glass stirring rod and allow to stand for ten minutes. If the solution is not clear, centrifuge, or filter using S & S No. 589. Seal filtrate in a vial if there is to be any time delay. Continue per instruction in Para. 4.4.

4.3.2 Preimpregnated Resins Cut the sample into approximately 1/2 inch squares or lengths. If the dry resin content of the prepreg is known, use the following formula to determine amount of prepreg and acetone to use:

$$\frac{191}{\text{DRC}} = \frac{\text{grams of prepreg in 36 ml. of acetone}}{\text{Dry Resin Content, gm.}}$$

If the dry resin content of the prepreg is not known, arbitrarily use 6 grams of prepreg in 36 ml. of acetone. Place the prepreg and acetone in a 100 ml.

beaker. Stir with a glass rod and allow to stand for a minimum of 20 minutes, with occasional stirring. Filter using S & S No. 589. Seal filtrate in a vial if there is to be any time delay. Extraction efficiency should be determined. Extraction efficiency must be at least 90% (see Para. 6.1) in order to achieve an accurate result.

4.4 Adjustment of Concentration Fill the 0.20 mm. cell with the resin-extract from Para. 4.2, using a syringe. Fill the variable path cell with water free of acetone. Position both cells in their respective holders in the spectrophotometer. Without placing the pen upon the chart paper, start machine operation at 11 microns by setting to "Scan" position. Determine absorbance at approximately 12.15 microns (12.1 to 12.2 microns). The concentration of the extract must be further adjusted (if necessary) to produce linear absorbance between 40% and 45% at this wavelength. This may be accomplished by removing the sample and adding acetone (if the absorbance is too great) or by partially volatilzing the acetone at room temperature (if the absorbance is too small).

4.5 Scan Place the pen to 5.0 microns and place on chart paper. Start machine operation by setting to scan position. Scan the spectrum from 5.0 to 16.0 microns at 0.54 microns per minute. The pen will automatically raise off the chart when the scan is complete. Remove the chart and label it in accordance with Para. 5.2. Turn function control to "STOP" and return pen carriage to 2.5 microns. Tilt pen backwards to prevent accidental contact with metallic chart bed.

4.6 Clean Up Remove cells from the instrument. Clean thoroughly with acetone followed by dry nitrogen or dry air. Place the cleaned and dried cells in a desicator.

5. CALCULATIONS AND REPORTING

5.1 Calculations Calculate the Advancement Index as follows:

5.1.1 Construction of tangential base line. Construct tangential base line over the absorbance occurring at approximately 9.8 microns, extending from approximately 9.45 to 10.2 microns. Also construct a tangential base line over the absorbance occurring at approximately 12.15 microns extending from approximately 11.5 to 12.5 microns.

5.1.2 Construction of perpendiculars. Construct a line perpendicular to the wavelength at the absorptions occurring at approximately 9.8 to 12.15 microns which passes through the maximum absorbance and the tangential base line.

5.1.3 Determination of absorbance. Determine the absorbance units (to three significant figures) between the maximum absorbance and the tangetial base line at approximate 9.8 and 12.15 microns absorptions. This is the base line absorbance.

5.1.4 Advancement Index. The Advancement Index, Infrared Polymerization Index (IRPI) and IRZB are as follows:

$$\text{Advance. Index (IRPI)} = \frac{\text{Base Line Absorb. @ 12.15 Micr.}}{\text{Base Line Absorb. @ 9.8 Microns}}$$

$$\text{Base Line (IRZB)} = \frac{\text{Log Base Line @ 12.15 Microns}}{\text{Log Base Line @ 9.8 Microns}}$$

5.2 Reporting. Record and report the following information:

5.2.1 Spectrum number

5.2.2 Sample identification

5.2.3 Lot or batch number

5.2.4 Date of test

5.2.5 Gain, balance and speed settings

5.2.6 Advancement Index (IRPI) or Baseline Index (IRZB) to the nearest 0.01 unit

5.2.7 Identity of the operator

5.2.8 Extraction efficiency if calculated

6. NOTES

6.1 Extraction Efficiency. It is desirable to determine extraction efficiency. This can be done by a variety of normal laboratory techniques.

M-R-19 Differential Scanning Calorimetry for Determining the Heat of Polymerization

1. SCOPE

1.1 This procedure describes a method for conducting differential scanning calorimetry.

2. EQUIPMENT AND MATERIALS

2.1 Perkin-Elmer DSC-1B or equivalent analyzer and operators manual

2.2 Sample pans, crimped

2.3 Nitrogen gas supply

3. SAMPLING

3.1 Obtain a small (1 oz.) representative sample.

4. PROCEDURE

4.1 Perform test with a Perkin-Elmer DSC-1B or equivalent analyzer. Sample pan must be crimped.

Temperature range:	Room temp. to 325°C. (598°K)
Rate of heating:	20 c./minute
Sensitivity range:	10 mcal./sec. (20 mvfs)
Reference:	Empty gold pan plus lid on reference side
Atmosphere:	Nitrogen (40 ml./min.)

4.2 The peak area is determined by extrapolating the baseline under the peak. Measure the peak area with a planimeter or equivalent. Record the temperature of onset, peak (s), maximum, and completion of reaction. The heat of polymerization shall be calculated as follows:

$$H_s = \frac{H_r \times W_r \times A_s \times R_s \times S_r}{W_s \times A_r \times R_r \times S_r}$$

where :

H = heat of polymerization, cal/g.
W = weight, mg.
A = peak area, in.^{2}
S = chart speed, in./sec.
R = range setting, mcal./sec., in.

Subscripts:

r = reference (calibration standard)
s = sample

4.3 Melting Point Determinations

4.3.1 Polymeric materials and organic solids melting points may be determined by the apex of the peak of melting endotherm.

4.3.2 Pure metal melting points may be determined as the onset of the melt endotherm.

M-R-20 pH of Resins and Aqueous Solutions with pH Meter

1. SCOPE

1.1 This method describes a procedure for the determination of pH of aqueous solutions and liquid resins.

1.2 This method is similar to ASTM-E70-52T and will yield equivalent results.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

2.1.1 pH meter with glass electrodes

2.1.2 Beakers, 200 ml., tall form

2.2 Materials

2.2.1 Buffer standards of pH 4, 7, 10

2.2.2 Distilled water

2.2.3 Denatured ethyl alcohol

3. SAMPLING

3.1 Sample Size. A minimum sample of 100 ml. is required to immerse the electrodes.

3.2 Number of determinations. Unless otherwise specified, duplicate determinations will be run on each sample.

4. PROCEDURE

4.1 Standardization of pH meter. The pH meter must be standardized daily prior to use as follows (see 6.1):

4.1.1 Allow sufficient time for complete warm-up of pH meter amplifier.

4.1.2 Immerse clean electrodes in standard buffer solution.

4.1.3 If necessary, set temperature control knob to temperature of buffer.

4.1.4 Read pH meter.

4.1.5 Using the calibrating control knob, set pH meter to indicate pH value of buffer.

4.1.6 Place pH meter in "Stand-by" condition.

4.1.7 Remove buffer and rinse electrodes with distilled water.

4.2 Determination of pH. The pH of the solution to be tested will be found as follows:

4.2.1 Immerse electrodes in sample.

4.2.2 If necessary, set temperature control knob to temperature of samples.

4.2.3 Read pH meter.

4.2.4 Remove sample and rinse electrodes thoroughly with proper solvent.

5. CALCULATIONS AND REPORTING

5.1 Reporting. If the range of determinations exceeds 0.1 pH units, the results should be considered suspect. Report the average of duplicate determinations to two significant figures.

6. NOTES

6.1 Standardization. The pH meter should be calibrated weekly in accordance with SCM-7-02. If for any reason the pH meter cannot be calibrated properly, consult SCM-7-02 for possible electrode failure and electrode servicing.

M-R-21 Karl Fisher Water Determination in Materials Chemicals

1. SCOPE

1.1 To determine the percentage of water in various materials and chemicals, titrate with stabilized Karl Fisher reagent.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

2.1.1 Aquatrator (Junior) - Precision Scientific Co., 3737 W. Cortland St., Chicago Illinois 60647, and operators manual.

2.1.2 Analytical Balance - Mettler, Model B5; Will Scientific, Cat. #2742 (et al).

2.1.3 Desiccator

2.1.4 Dropping Bottle

2.2 Materials

2.2.1 Karl Fisher Reagent - Fisher Scientific Co., Cat. #SO-K-3.

2.2.2 Methanol (Borden)

3. ASSEMBLY

3.1 Follow "Operating Instruction" booklet supplied with instrument. If assistance is required, contact Bainbridge Quality Control Office, of Precision Scientific, Co. Arrangements will be made for assembly assistance and operating know-how.

Note I: In assembly, do not set up the Water-Methanol burette and drying tubes for same. This is a modification by the laboratory in which we eliminate the Back Titration Method; using dry Methanol in the Reaction Vessel and Distilled Water for standardizing, etc.

Note II: In place of Drierite, use Silica Jel which we feel is a more effective desiccant and which also can be oven dried and reused, a property lacking with the Drierite.

4. DETERMINATION OF ENDPOINT

4.1 After assembly and plug-in, energize unit by turning the Mode Selector switch from the "OFF" position to "DIRECT ADJUST".

4.2 With DIRECT ADJUST knob, position indicating needle to full scale "20" for normal operations.

Note: Always turn the DIRECT ADJUST knob counter-clockwise before switching to the ADJUST position. This will prevent accidental damage to the meter needle if the DIRECT ADJUST knob is set too far clockwise.

4.3 Turn MODE SELECTOR switch to DIRECT TITRATE.

Note: In a direct titration the Micro-Ammeter needle will start at a low meter reading and increase as the endpoint is approached.

4.4 Add the dry Methanol into the reaction vessel so that it covers the electrodes.

Note: The Methanol must cover the electrodes, however, the electrodes must not be so low as to be damaged by the Teflon stirring bar.

4.5 Set the stirring at the desired slow rate.

4.6 Fill the Karl Fisher burettes by pumping the appropriate plunger. Be sure to place your finger over the hole below the plunger. When the reagent has filled the burette, release your finger and the plunger. The reagent will then syphon to the "0" ml. mark.

Caution: Do not allow titrant to enter the drying tube which is inserted in the stopper on the burette.

4.7 Dispense Karl Fisher solution until the black needle holds steadily between 17/19.

4.8 Remove Sample Filling Port Stopper and place 2 drops of distilled water so the current drops. Replace stopper and vacuum purge the vessel by depressing and holding the pump switch. (Amount of purging depends on the humidity present in the room. Normally, 15 seconds is adequate for humid ambient conditions.)

Note: See "Operating Instructions", Section IV, Subtitle A, "Vacuum Pump System".

4.9 Check burette to insure that reagent is at the "0" line, titrate until an endpoint is reached. Initial addition of titrant will cause current needle to swing upscale and then drop as titrant reacts with the water. Further addition swings the current needle in increasing upscale increments. As endpoint is approached, the increments become smaller. If a single drop maintains this endpoint for a specified time (will not fade), stop the titration and record the meter value so noted. <u>This is the Endpoint.</u>

<u>Note:</u> It is extremely important that whatever meter value is chosen for the endpoint, that this value be used for both calibration and actual sample titrations. It is also extremely important that the full-scale adjustment be left at the same value for calibrating and actual sample titrations, i.e., "20" for normal operations.

5. DETERMINATION OF KARL FISHER FACTOR (K. F. Solution only)

5.1 After determining Endpoint the system is operable.

5.2 To determine "Factor", again add a few drops of distilled water to the Methanol solution. Record sample weight.

5.3 Vacuum purge the system. Set the stirring at the desired rate.

5.4 Fill the K. F. burette. Titrate with K. F. Solution until the <u>Endpoint</u> is reached.

5.5 Record mls. of K. F. Solution used.

5.6 The milligrams of Water per milliter of Solution is called "Factor", <u>which must be determined at least once a day.</u>

6. DIRECT TITRATION METHOD

6.1 Energize unit by turning the MODE SELECTOR switch from the OFF position to DIRECT ADJUST.

6.2 With DIRECT ADJUST knob, position indicating needle to Full Scale "20" for normal operations.

6.3 Turn MODE SELECTOR switch to DIRECT TITRATE.

6.4 Determine "Factor" if necessary (at least once a day).

6.5 Add sample to Methanol Solution. Record sample weight.

6.6 Vacuum purge system. Set the stirring at the desired rate.

6.7 Fill the K.F. Burette. Titrate with K.F. Solution until the Endpoint is reached.

6.8 Record ml. of K.F. Solution used.

7. CALCULATION

$$\frac{\text{Milliters K.F. Soln. x "Factor"}}{\text{Milligrams of Sample used}} \times 100 = \%\ \text{water}$$

8. NOTES

8.1 ACCURACY: Several factors will influence accuracy; mainly moisture pick-up, dirty burettes, spent drying chemicals, insufficient volumes of titrant, chemical reaction with other chemical (Ref: "AQUAMETRY" by Mitchell and Smith, Vol. 5)

8.2 The moisture problem is greatly reduced by use of the vacuum pump whenever the sample stopper is removed. Drying tubes are clearly exposed and should be changed whenever the contents have absorbed moisture as indicated by the color change. All connections are positive and easily checked.

8.3 Droplets of reagent in the burette bore clearly indicate that the burette should be cleaned.

8.4 An endpoint setting (in direct titration) which is too high will be toward the insensitive portion of the meter scale. If the current needle never approaches this setting, obviously the endpoint setting is too high or the span is too low (high resistance solution with corresponding low current reading).

8.5 The platinum electrode tips can become coated and insensitive. They should be cleaned in warm sulphuric acid-dichromate solution. Rinse with distilled water and acetone, and dry thoroughly. When removing the vessel, wipe any liquid droplets from the underside of the gasket. When adding a powdered sample with a dry powder funnel, be sure the sample does not adhere to the vessel walls.

8.6 DRAINING REACTION VESSEL: Several sample determinations can be run before the liquid level in the vessel reaches the burette tips. It is suggested that a portion of this liquid be transferred to the new reaction vessel. However, rather than remove the vessel, an alternate choice is to stop the stirring, remove the sample inlet stopper, and syphon the liquid to a level just above the electrode tips. Replace the stopper, vacuum purge with dry air, and readjust the stirring. Syphoning is accomplished by squeezing the air out of a plastic wash bottle (provided), inserting its tube into the reaction vessel and withdrawing the unwanted solution.

9. TROUBLE SHOOTING GUIDE

9.1 Follow "Operating Instruction" booklet supplied with instrument. Please read. THIS IS IMPORTANT for maximum efficiency.

10. PARTS LIST

10.1 It is suggested the following parts be purchased and stocked. Note parts list operating instruction.

10.1.1 Electrode, platinum

10.1.2 Reagent, Karl Fisher (Refrigerate unused bottles)

10.1.3 Desiccant, Silica Jel*

10.1.4 Burette

*Tel-Tale Silica Gel from Davison Chemical Co., Baltimore, MD. 21203. This is Grade 42, Mesh Size 6/16, comes in 1 lb. cans.

NOTE: Save reagent bottle caps, drill for replacements for cap with stopper.

M-R-22 Free Formaldehyde in Aldehyde Phenolic and Furan Type Resins

1. SCOPE

1.1 When Hydroxylamine Hydrochloride reacts with dissociated Free Formaldehyde, Formaldoxime is formed with a quantitative liberation of Hydrochloric Acid.

1.2 The liberated acid is titrated with base. This method is used since it is accurate in the presence of alkali sensitive materials as well as in the presence of aldehyde phenolic groups.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

2.1.1 pH Meter (Beckman or equivalent)

2.1.2 Ground glass dropping bottle with rubber bulb, 15 ml.

2.1.3 Beakers, 150 ml., 250 ml.

2.1.4 Burettes, 25 ml. (2)

2.1.5 Graduate Cylinders, 25 and 50 ml.

2.1.6 Analytical balance, accurate to 0.0001 gm.

2.2 Materials

2.2.1 Sodium Hydroxide, 0.5N, 0.1N

2.2.2 Sulfuric Acid, 0.5N, 0.1N

2.2.3 Hydroxylamine Hydrochloride - prepared by dissolving 70 gm. of amine salt in 1 liter distilled water. This solution is adjusted to pH 4.0 with 0.5N Sodium Hydroxide before use.

2.2.4 Methanol, CP (formaldehyde free)

2.2.5 Distilled water

3. PROCEDURE

3.1 METHOD A - For aldehyde-phenolic resins containing less than 5% free formaldehyde:

3.1.1 Follow in duplicate:

3.1.1.1 Fill the medicine dropping bottle with the resin.

3.1.1.2 By difference, weigh accurately 3.00 gm. of sample into a 250 ml. beaker.

3.1.1.3 Add 20 ml. of methanol and 20 ml. of distilled water from graduate cylinders.

Note: More methanol may be added; enough to completely disperse the sample. In such a case record ml. of methanol used and carry out a blank with the same amount of methanol plus 20 ml. water. For novolacs, use methanol only.

3.1.1.4 Adjust pH to 4.00 using 0.1N sulfuric acid or 0.1N sodium hydroxide. Employ the electrodes and stir vigorously with a glass stirring rod after each addition of titrant. Do not touch the electrodes with the stirring rod.

3.1.1.5 Add 50 ml. of the hydroxylamine hydrochloride which has a pH of 4.00 from a graduate cylinder.

3.1.1.6 Stir and let stand 5 minutes.

3.1.1.7 Titrate the sample with 0.5N sodium hydroxide, using the electrodes and stirring as above, to pH 4.00. Record ml. sodium hydroxide used.

3.1.1.8 Run blank on all reagents at least once a day.

3.2 METHOD B - For aldehyde-phenolic resin containing 5% or more free formaldehyde

3.2.1 As above, except under (3.1.1.6) - Stir and let stand 40 minutes.

3.3 METHOD C - For phenolic resins containing urea:

3.3.1 As in Method A, except under (3.1.1.7) - Add cracked ice, adjust pH meter to 10°C temperature setting and titrate the sample with 0.5N sodium hydroxide using the electrodes and stirring as above to pH 4.00. Record ml. sodium hydroxide used.

3.4 METHOD D - For furan resins containing both urea and phenol and less than 5% free formaldehyde:

3.4.1 As in Method A, except under (3.1.1.3) - Add 20 ml. methanol; under (3.1.1.5) - Add 15.0 ml. of hydroxylamine hydrochloride reagent which has a pH of 4.00.

4.0 CALCULATION

4.1 $\frac{\text{mls. 0.5N NaOH}}{2}$ = Free CH_2O (using N/2 NaOH & exactly 3 gm. sample).

4.2 If 1N sodium hydroxide is used:

mls. 1N NaOH = Free CH_2O (using 1N NaOH and exactly 3 gm. sample)

-OR-

$$\frac{\text{mls.} \times N \times 3.002}{\text{Wt. Sample}} = \%\ \text{Free } CH_2O$$

M-R-23 Chang's Index - A Measure of Degree of Cure

1. SCOPE

1.1 This method describes a procedure for an arbitrary measure of the relative degree of cure, advancement, or polymerization of liquid aldehyde-phenolic resins or aldehyde-phenolic prepregs.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

2.1.1 Buret, 50 ml.

2.1.2 Balance, analytical, sensitivity of 0.0001 gm.

2.1.3 Triple beam balance

2.1.4 Pipet, 25 ml.

2.1.5 Aluminum cups, 1.5 gm., diameter 2 - 3/8 inches, height 5/8 inch

2.1.6 Hypodermic syringes, 2 ml., 10 ml., 30 ml. with delivery needles

2.1.7 Oven, forced air

2.1.8 Desiccator

2.1.9 Filtering crucibles, fritted glass, medium or fine

2.2 Materials

2.2.1 Distilled water

2.2.2 Acetone

2.2.3 Filter paper, Whatman #41, #42, or equivalent

3. SAMPLING

3.1 Liquid resins.

3.1.1 10 grams for resins between 50% and 100% solids

3.1.2 20 grams for resins between 30% and 50% solids

3.1.3 For resins near 20% solids, measure solids per 4.2 and proceed to 4.3.

3.2 Prepreg materials

3.2.1 15 grams for materials of between 40% and 50% dry resin content

3.2.2 20 grams for materials of between 30% and 40% dry resin content

4. PROCEDURE

4.1 Preparation of intermediate solutions: Prepare duplicate solutions as follows:

4.1.1 Liquid resin. Mix in a flask the appropriate sample size weighed on the triple beam balance, with about 50 ml. of acetone. Determine the resin solids of the mixture according to the method of 4.2.

4.1.2 Prepreg materials. Soak the appropriate sample size, chopped to about 1/2" x 1/2" and weighed in a flask on a triple beam balance, with 50 ml. of acetone for 15 minutes minimum. Swirl occasionally. Filter the extracted material through Whatman #41 or #42 filter paper. If the material contains fine filler, thorough filtration may require the use of a medium or fine fritted glass filtration crucible. Determine the resin solids of the solution per 4.2.

4.2 Determination of resin solids of intermediate solutions. Determine resin solids of intermediate solutions as follows:

4.2.1 Weighing and devolatilization. Weigh on the analytical balance by difference from 2 ml. syringe, 2 ml of intermediate solution or liquid resin of 3.1.3. Discharge the solution into a tared aluminum solids dish. Volatilize in a forced air oven at $275 \pm 5^{\circ}F$. for 30 minutes. Cool in a desiccator and reweigh.

4.2.2 Calculation of resin solids. Calculate resin solids as follows:

$$\text{Resin solids, wt.\%} = \frac{\text{Wt. of sample after volatil.}}{\text{Wt. of sample before volatil.}} \times 100$$

4.3 Preparation of 4.0% resin solids solution. Prepare the final solution as follows:

4.3.1 Calculations for dilution. Calculate W_1, the weight in grams of intermediate solution to be used as follows:

$$W_1 = \frac{160}{\text{\% resin solids from 4.2.2}}.$$

Calculate W_2, the weight of acetone to be used, as follows:

$$W_2 = 40 - W_1$$

4.3.2 Dilution. On the analytical balance, introduce W_1 into a 125 ml. flask from a large syringe as quickly as possible to the nearest 0.001 gm. Immediately introduce W_2 to the nearest 0.001 gm. into the flask from a large syringe as quickly as possible. Long, steel delivery needles simplify these operations. Stopper the flask and swirl lightly to mix.

4.3.3 Verification of 4.0% resin solids. For adequate precision, solids must be between 3.95% and 4.0%. Occasionally verify resin solids by the procedure of 4.2.2 except use an 8 ml. sample size. This change in sample size keeps the total weight of resin used roughly equal for both determinations.

4.4 Titration. Pipet 25 ml. of the final solution of 4.3 into a 125 ml. erlenmeryer flask. Titrate with distilled water to the first perceptible, persistent end point. Recognition of the end point is made easier if a strong compact light beam is shined through the flask during titration. Colloidal particles scatter the beam at the end point.

5. CALCULATIONS AND REPORTING

5.1 Reporting. Report the average number of ml. of water used as Chang's Index. Duplicate determinations should be considered suspect if they differ by more than 0.3 ml.

M-C-1 Linear Density of Yarn and Cord Expressed in Denier

1. SCOPE

1.1 This method covers the determination of linear density of tire or industrial yarn or cord from a high traverse package; such as, a pin, tube, or bobbin. Linear density is corrected to commercial moisture regain and expressed in denier units.

1.2 This method is consistent with ASTM Methods D 885-75, Section II and D 1907-75, Option 3.

2. APPLICABLE DOCUMENTS

2.1 ASTM Standards

D 123-75 Definitions of Terms Relating to Textile Materials

D 885-75 Testing Tire Cords, Tire Cord Fabrics, and Industrial Filament Yarns made from Man-made Organic-Base Fibers.

D 1907-75 Test for Yarn Number by the Skein Method

D 1909-68 Table for Commercial Moisture Regains for Textile Fibers.

3. DEFINITIONS

3.1 Density, linear, - mass per unit length

3.2 Denier, - a measure of linear density in gm. per 9000 m.

3.3 Commercial moisture regain, - an arbitrary value formally adopted as the regain to be used with weight of the moisture-free material when calculating (1) the yarn number, (2) the commercial or legal weight of a shipment of delivery or any specific textile material, or (3) the weight of a specific component in the analysis of fiber blends.

4. SUMMARY OF METHOD

4.1 A skein of yarn is wound on a reel, oven-dried, and weighed. The linear density is calculated from the weight and length of the skein.

5. USES AND SIGNIFICANCE

5.1 The determination of linear density consists of measuring a length of yarn or cord, drying and weighing it. Since yarns and cords are extensible to varying degrees, it is essential to measure length by reeling under controlled tension.

5.2 Predried air is used in drying. If predried air is not used, then small amounts of moisture will be retained by the fiber in the oven, the actual amount varying with the type of fiber, the temperature, and the relative humidity of the air entering the oven.

6. APPARATUS

6.1 Reel., 1 m. ± 0.4%, equipped with a yarn or cord tensioning device and a revolution counter.

6.2 Brabender Moisture Tester, Semi-automatic, Type FD or FDA, or equivalent, supplied with a current of air at a rate sufficient to change the air in the oven at least once every 4 min. the current of air shall be predried (less than 0.01 grams of water per 1000 L.) by circulating through dehydrating units.

6.3 Rack for holding supply package, over-end for yarn tube or cone, side unwinding for bobbins of cord.

7. SAMPLING AND SPECIMENS

7.1 One skein specimen is taken from each sample

8. PROCEDURE

8.1 Adjust yarn or cord tension device on reel to 4 oz.

8.2 Position sample package on holder and lace through guides and tension device and attach to an arm of the reel. Let-off is over-end for yarn tube or cone and from side for bobbin or cord.

8.3 Set counter to zero

8.4 Wind to exact revolutions the following length skeins:

Nominal Yarn or Cord Denier	Revolutions
1100/1	81
1650/1	55
2200/1	41

Nominal Yarn or Cord Denier	Revolutions
1100/2	37
1650/2	24
1650/3	15
2200/3	11

8.5 Turn reel so that arm to which the end was attached is in the same position as at the start. With scissors cut and remove the skein from the reel. Fold the skein to fit into the Brabender drying cup.

8.6 Dry to constant weight in the Brabender Moisture Tester at 105 ± 2° C. In practice it has been found that the equipment described and starting with a preheated Brabender, constant weight is reached in 1 hr.

8.7 With the test specimen cup in position over the built-in balance, lift the cup with the control lever and turn "On" the illuminated dial. Add gram weights to the weight cup to bring the scale reading in the illuminated dial between "7.6" and "8.6". Record the weight added to the weight cup to the nearest 1 mg and the dial reading to the nearest 0.1 percent.

9. CALCULATIONS

9.1 Calculate the linear density to the nearest whole denier using the equation below.

Linear Density,

$$\text{denier} = \frac{(21.0 - W - 0.1\ B)\ 9000\ (1 + R)}{L}$$

where:

W = weight added to the Brabender weight cup, gm.
B = reading on the Brabender illuminated dial, %.
R = commercial moisture regain, for rayon 11.0 percent, expressed as a decimal.
L = length of yarn or cord in skein specimen, m.

Note: The expression (21.0 - W - 0.1 B) is the weight in grams of the specimen as determined by the built-in balance in the Brabender Moisture Tester.

10. REPORT

10.1 State that the specimen was tested as directed in AVTEX STM 003-75.

10.2 Report the following information:

10.2.1 Linear density to nearest whole denier

10.2.2 Identity of analyst.

M-C-2 Tensile Properties of Yarn, Cord, and Fabrics of Twisted Materials

1. SCOPE

1.1 This method covers the determination of tensile properties of tire or industrial yarns made of rayon, cords twisted from such yarn and fabrics woven from such cord, products that are made specifically for use in the manufacture of pneumatic tires or reinforcing other rubber goods and for other industrial applications.

1.2 Options are included for the testing of (1)conditioned and (2) oven-dried specimens.

1.3 This method is consistent with ASTM D 885-72 for sections applicable except as noted.

2. APPLICABLE DOCUMENTS

2.1 ASTM Standards

D 76-67, Specifications for Tensile Testing Machine for Textile Materials

D 123-75, Definitions of Terms Relating to Textile Materials

D 885-75, Testing Tire Cords, Tire Cord Fabrics and Industrial Filament Yarns Made From Man-Made Organic Base Fibers

3. DEFINITIONS

3.1 Atmosphere for testing tire cords and industrial yarns, air maintained at a relative humidity of 55 ± 2 percent and at a temperature of 24 ± 1° C. (75 ± 2°F).

3.2 LASE, load at specified elongation as interpreted from the load-elongation curve.

3.3 EASL, elongation at specified load as interpreted from the load-elongation curve.

3.4 For definitions of tensile test and other textile terms used in this method refer to ASTM Definitions D 123-75 and Method D 885-74.

4. SUMMARY OF METHOD

4.1 A specimen of yarn or cord is clamped in a tensile testing machine and then stretched until broken. Breaking load, breaking elongation, load at a specified elongation (LASE) and elongation at a specified load (EASL) are obtained directly.

4.2 This method covers two options with respect to moisture content of the specimens at the time of testing:

4.2.1 Option 1, conditioned (in moisture equilibrium for testing with the standard atmosphere for testing).

4.2.2 Option 2, oven-dried.

4.3 This method covers taking specimens from tubes, cones or bobbins and cord specimens from woven fabric tabbies.

4.4 Testing of yarn or cords other than rayon usually require different conditioning prior to testing and different values of reading LASE, Refer to ASTM D 885-75.

5. USES AND SIGNIFICANCE

5.1 The levels of tensile properties obtained when testing yarns and cords are dependent on the history of the specimen and on the specific conditions used during the tests. Among these conditions are rate of loading, type of clamps, gage length of the specimen, temperature and humidity of the atmosphere, rate of airflow across the specimen and temperature and moisture content of the specimen. The relative importance of these factors varies with each type of fiber. Testing conditions accordingly must be specified precisely in order that test results on a specific sample will be reproducible.

5.2 For further statement on uses and significance see ASTM D 885-75, Section 13.

6. APPARATUS

6.1 Tensile Testing Machine - Instron with C or CT Load cell. The instron is a single-strand testing machine of the constant-rate-of-extension type (CRE). The specifications and methods of calibration and verification shall conform to ASTM D 76-67.

6.2 Clamps - Pneumatic capstan type, automatically operated

For yarn -Instron 4C, Cat. 2714-004

For cord -Instron 4D, Cat. 2714-006

6.3 Pretensioning Device - Instron 4C or 4D (Cat. A58-29 or A2-93.

6.4 Oven - A ventilated drying oven controlled in a temperature range of 105 ± 3°C. The oven shall be supplied with a current of air sufficient to change the air once every 4 min. Exit port for air must be open. The specimens shall be protected from direct radiation of the heating units. The oven shall be supplied with air from the testing room, see 3.1. Ovens meeting these requirements are Precision Scientific Cat. 1236 or Freas Model 835A.

6.5 Drying Rack - A removable rack to fit into the oven, 6.3 See Figure 1.

6.6 Clips - 1 in. Bulldog or Hunt No. 0, two required for each group of specimens.

6.7 Denier Reel - hand operated.

6.8 Twist Counting Instrument - Alfred Suter, 20 in., hand operated.

6.9 Pressure Sensitive Tape.

7. CONDITIONING

7.1 Option 1, Conditioned. Samples shall be at equilibrium with the standard atmosphere for testing, 3.1. Equilibrium shall be reached by adsorption. Exposure overnight has been found to be sufficient. Note 1.

Note 1 - When inter-laboratory comparisons were made, percent moisture regain in the yarn cord at the time of testing shall be reported. A 1 percent change in regain corresponds to about 2 percent change in tensile strength, inverse relationship.

7.2 Option 2, Oven-dried. Cord samples shall be conditioned overnight in the standard atmosphere for testing to allow setting and equalizing of twist.

8. PREPARATION AND HANDLING OF SPECIMENS

8.1 All yarn specimens shall be tested for twist. Box process yarns and pre-twisted continuous process yarns specimens shall have 3 tpi, Z twist, inserted with the twist counting instrument prior to mounting in the tensile testing machine for conditioned specimens and prior to mounting in the dryer racks for oven-dried specimens. See ASTM D 885-75.

8.2.1 Yarns with twist and cords. Specimens are taken directly from the tube, cone, bobbin or tabby for mounting in the tensile testing instrument.

8.2.1.1 For samples on tubes, cones or bobbins, specimens shall be taken from the center portion of the traverse since yarn or cord in the transverse reversal at either end of the package can contain abnormal twist that may not equalize along the length of the yarn or cord in handling the specimen.

8.2.1.2 Strip at least two yards of yarn or cord to eliminate yarn or cord at the end of the transverse and to eliminate yarn or cord that may have lost twist by running out to a free end.

8.2.1.3 Hold the end of the yarn or cord firmly in one hand. Unwind approximately 20 in. and with the other hand grasp the yarn or cord firmly and then cut with scissors.

8.2.1.4 The specimen shall not be allowed to kink or bend into a radius less than 1 in. to prevent migration of twist. If it does discard it and take another specimen.

8.2.1.5 The specimen shall be held firmly until after mounting in the clamps of the Instron instrument to prevent loss of twist.

8.2.1.2 Yarns without twist. Select specimens as in 8.2.1.1

8.2.2.1 Attach end in right-hand clamp of the twist counting instrument. With left-hand clamp set at 20 in. lace yarn through left-hand clamp and the pre-tensioning device. Pull to pre-tension the yarn specimen and close the left-hand clamp. Insert 60 turns Z twist (3 tpi).

NOTE - tpi is threads per inch.

8.2.2.2 Remove the specimen from the twist counting instrument and handle as in 8.2.1.4.

8.3 Option 2. Oven-dried.

8.3.1 Yarns with twist and cord on cones or bobbins.

8.3.1.1 Strip sample and hold specimens as in 8.2.1.1 & 8.2.1.2.

8.3.1.2 Attach the end of the yarn or cord to an arm of the denier reel and unwind onto the reel sufficient revolutions of yarn or cord to provide the number of specimens required from a package. Bobbins shall be unwound from the side. Tubes or cones shall be unwound over-end.

8.3.1.3 Prior to cutting the yarn or cord from the supply package, wrap a piece of pressure sensitive tape around the band of yarns or cords from a package and press firmly to hold the band. Approximately 20 in. around the reel apply a second piece of pressure sensitive tape.

8.3.1.4 Mark on the tape identification of the sample.

8.3.1.5 Cut the band at the ends on the outside of the 20 in. section, apply a clip to either end and hang in the dryer rack. As in 8.2.1.4 the band of specimens shall not be allowed to kink or be slack when hung between hooks across from each other in the rack. If band is not slack or if it sags excessively to allow twisting or kinking, discard the band and wind another adjusting the distance between the two pieces of pressure sensitive tape.

8.3.2 Yarns without twist. Select specimens as in 8.2.1.1.

8.3.2.1 Insert twist and handle specimens as in 8.2.2.1 & 8.2.2.2.

8.3.2.2 Transfer twisted specimen to two pieces of pressure sensitive tape, mounted adhesive side up 20 in. apart on table. When sufficient specimens have been so prepared from the tube or cone, apply a second strip of pressure sensitive tape on top of the specimens and the tapes on the table.

8.3.2.3 Mark on the tape on one end the identification of the sample.

8.3.2.4 Remove the tapes from the table and mount each end in clips. Hang clips in the dryer rack as in 8.3.1.5.

Note 2 - Special dryer racks may be used where the twisted specimens are mounted in spring clips of the racks when the specimen is removed from the twist counting instrument.

8.3.3 Cords in fabric tabbies.

8.3.3.1 Test tabbies of tire cord fabric shall be mounted on a stiff medium; i.e., paperboard, to prevent kinking of the cords. If not so mounted or if cords show evidence of kinking, and replacement tabby is not available, it shall be noted on the test report.

8.3.3.2 With scissors cut the filling yarns and remove from an area about 3/4 in. wide to provide sufficient cord specimens for testing. Cut into the heavy tabby area about 1 in. parallel to the band of cords selected and then cut across the cords selected and then cut across the cords to remove the band of cords. Mount in a clip. Cut into the other heavy tabby area in like manner and mount the second end of the band of cord specimens in a clip. Hold clips to prevent kinking of the cords at any time as in 8.2.1.4.

8.3.3.3 Identify the band of cord specimens with marking on a small piece of paper under one of the clips.

8.3.3.4 Hang the band of cord specimens by the clips in the dryer rack as in 8.3.1.5.

8.3.4 Place rack in the oven and dry for a minimum of an hour for yarn and three hours for cord or a maximum of seven hours.

9. PROCEDURE

9.1 Adjustments to Instron testing instrument.

9.1.1 Select a load scale such that the estimated breaking load of the specimen is below 90 percent of the full-scale load of the Instron and the area of interest of the initial part of the load-elongation curve; i.e., LASE or EASL, is above 10 percent of the full-scale load of the Instron.

9.1.2 Rate of operation of the Instron cross-head shall be 100 percent/min of the nominal gage length of the specimen; i.e., 10 in./min.

9.1.3 Rate of operation of the Instron chart paper shall be 200 percent/min of the nominal gage length of the specimen; i.e., 20 in./min. Note 3.

Note 3 - This combination of rates of cross-head and chart paper is readily obtainable with standard gearing supplied by Instron. One inch of chart paper on the abscissa (time) axis is equal to 5 percent extension of the specimen which facilitates interpreting initial areas of the load elongation curve, LASE and EASL, for a high modulus material as oven-dried rayon. This rate of cross-head movememnt is at variance with that specified in ASTM D 885-75, Section 15.2 and Table 2 i.e., 120 percent/min. This difference is insequential as to its effect on tensile properties of yarn or cord either from the rate of extension or the cooling effects on oven-dried specimens.

9.1.4 Adjust the Instron so that, in the starting position, the distance from nip to nip of the clamps along the axis of the specimen (including the portion in contact with the snubbing surfaces)
is 10 ± 0.1 in.

9.1.5 Adjust the pre-tension device to 4 oz (113 g) tension on the specimen with the clamps closed prior to start of cross-head movement.

Note 4 - the amount of tension developed in the specimen varies significantly with the rate of pull on the specimen to actuate the pneumatic clamp. A very slow pull produces a low tension; a hard jerk a high tension.

9.1.6 Adjust air pressure for pneumatic clamps to 30 psi for yarn and 2 ply cord and 40 psi for 3 ply cord.

9.2 Option 1 - Test the conditioned specimens in the standard atmosphere for testing.

9.3 Option 2 - If more than 25 seconds elapse between taking the oven-dried specimen from the oven and breaking it in the testing machine, discard the specimen and take another.

Note 5 - This method covers testing oven dried specimens directly from the oven; as such the time from the oven to the instant of break of the specimen is important since it affects the temperature of the specimen, 5.1. This is at variance with ASTM D 885-75, Section 22.2.3, where provisions are made for cooling the dried specimen to room temperture before testing. The latter is a more precise and sophisticated provision of the method.

9.4 Remove the specimen of yarn or cord from the sample package or tabby for conditioned testing Option 1, or from the oven for oven-dried testing, Option 2, and handle in such a manner that no change in twist can occur prior to closing the jaws of the clamps on the specimen. Do not touch with the bare hand that portion of the specimen which will be between the nips of the clamps. Lace the specimen over the horn and down between the jaws of the upper clamp and close the clamp by pressing the cord in the left hand against the pneumatic switch on the upper clamp. Lace the specimen around the horn and up through the jaws of the lower clamp and over the pulley of the pre-tension device.

9.5 Close the lower clamp with a sharp steady pull on the end of the specimen in the right hand. Note 4 above.

9.6 With the Instron clamp set on "Auto", the cross-head automatically starts moving to separate the clamps when the lower clamp is closed.

9.7 Discard the test on a specimen that breaks within 1/8 in. of the nip of a jaw.

9.8 Repeat 9.2 through 9.6 for the required number of specimens.

9.9 Read from the chart the individual specimen breaking load in pounds and elongation in percent, and the elongation at specified load (EASL) or load at specified elongation (LASE) as required by the specification.

9.10 To facilitate reading the chart do not superimpose more than five breaks on the same starting point on the chart.

10. CALCULATIONS

10.1 Calculate the average breaking load to the nearest 0.1 lbs.

10.2 Calculate the average breaking elongation to the nearest 0.1 percent.

10.3 Calculate the average EASL or LASE where and as required by the specification.

10.4 For yarns calculate the average breaking tenacity in grams per denier to the nearest 0.01 gf/d.

$$\text{Breaking Tenacity, gf/d} = \frac{S(453.6)}{D}$$

where: S = average breaking load in pounds
D = linear density in denier as determined by ASTM Method D 885-72, section 11, or M-C-1
gf/d = grams force per denier
1 oz. = 453.6 grams

11. REPORT

11.1 Report the following information.

11.1.1 Option used. (Conditioned or oven-dried).

11.1.2 Average breaking load.

11.1.3 Average breaking elongation.

11.1.4 Average LASE or EASL, where specified.

11.1.5 Average breaking tenacity, for yarn only.

11.1.6 Full scale load setting of Instron in pounds.

11.1.7 Number of specimens for each test.

11.1.8 Identification of technician who carried out the tests.

11.1.9 Identification of sample.

11.1.10 Any abnormal condition of specimen, Ref. 8.3.3.1.

Note 6. Designation is a sequential number for ASTM Standard Test Methods and Practices followed by a two digit number indicating the year of issue or revision. A small case letter indicates subsequent revision in one year, as 102-75A.

Note 7. Revision editorial except for 8.3.4.

Note 8. tpi is turns (or twists) per inch in this test.

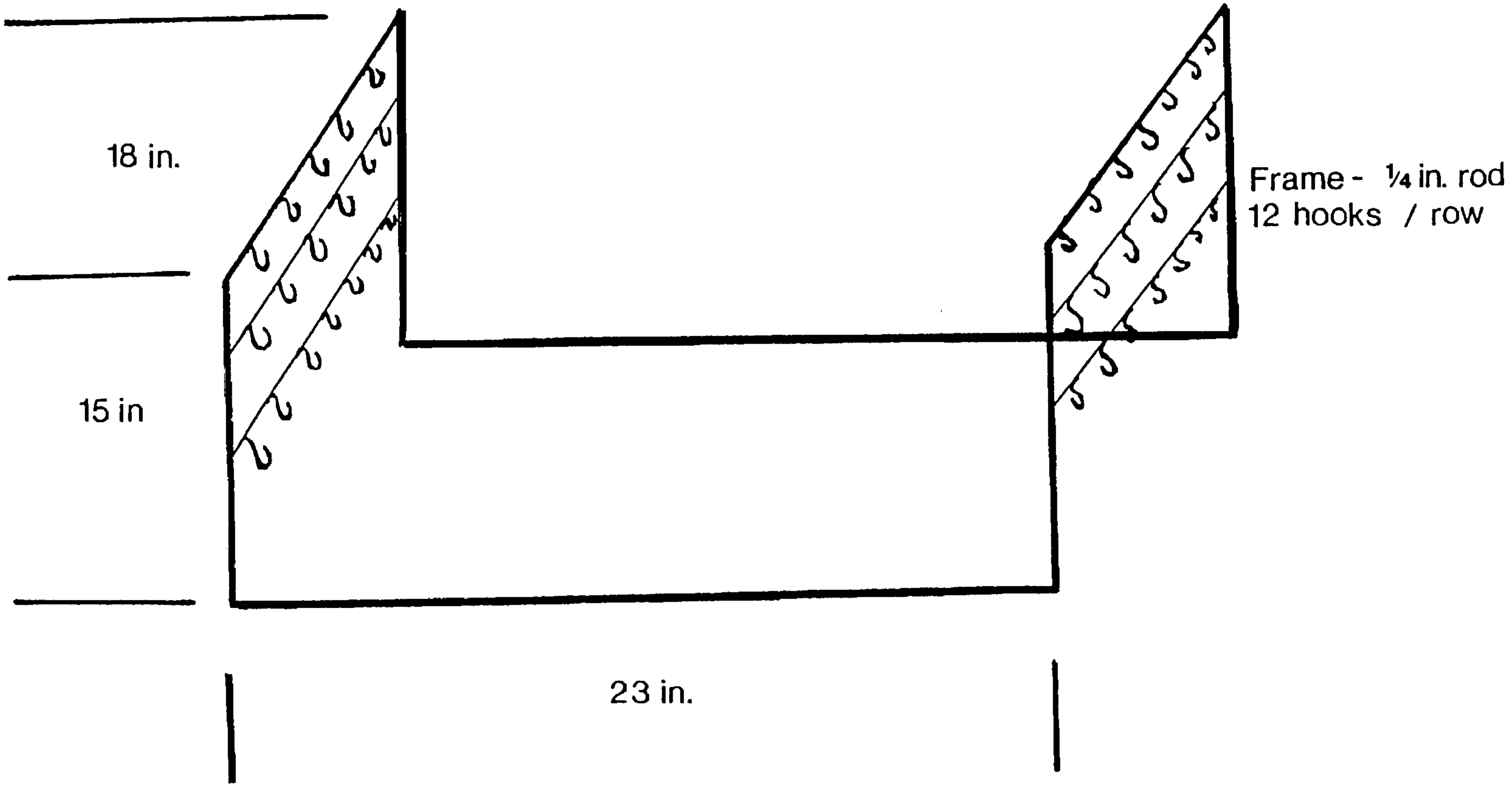


Fig. 1

C-2

M-C-3 Twist, Determination in Yarn, Cord

1. SCOPE

1.1 This method covers the determination of amount and direction of twist in single filament yarn or cord.

1.2 For cords, this method covers the determination of the twist of the cords and the twist in the single yarn before cording.

1.3 This method is consistent with ASTM Methods D 885- 75, Section 32 and D 1423-71, Section 9.3 through 9.5.

2. APPLICABLE DOCUMENTS

2.1 ASTM Standards

D 123 - 75 Definitions of Terms Relating to Textile Materials.

D 885 - 75 Testing Tire Cords, Tire Cord Fabrics and Industrial Filament Yarns made from Man-Made Organic Base Fibers.

D 1423 - 71 Twist in Yarns by the Direct-Counting Method.

3. DEFINITIONS

3.1 Twist, the number of turns about its axis per unit of length observed in a yarn or other textile strand expressed in turns per inch.

3.2 Direction of twist, the direction of twist in yarns and other textile strands is indicated by the capital letters "S" and "Z". Yarn has S twist if, when the yarn is held in a vertical position, the visible spirals or helices around its central axis conform in direction of slope to the middle portion of the letter "S" and Z twist if the visible spirals or helices conform in direction of slope to the central portion of the letter "Z." When two or more yarns, either single or plied, are twisted together, the letters "S" and "Z" are used in a similar manner to indicate the direction of the last twist inserted.

3.3 Tire Cord, a twisted or formed structure composed of two or more single or plied industrial yarns. Each individual single or plied yarn element in a cord has the same nominal twist, direction of twist, length, and tension. The direction of twist is used to combine the single or plied yarn elements into a cord structure in a direction opposite to that used in the yarns.

3.4 Cord twist, the amount of twist in a cord made from two or more single or plied yarns. Cord twist is based on the initial length of a cord specimen.

3.5 Single twist, the amount of twist in each single yarn element in a tire cord structure based on the length of the element after twist has been removed from the cord.

3.6 Original twist, the twist in a single or plied or cabled yarn as the component was before incorporation into the more complex structure.

3.7 Twist take-up, the change in length of a yarn or other textile strand caused by twisting expressed as a percent of the original untwisted length.

3.8 For definitions of other terms used in this method refer to ASTM Definitions D 123-75.

4. SUMMARY OF METHOD

4.1 The turns of twist in a known length of yarn are counted as they are being removed by rotating one end of the specimen while the other end remains fixed until the elements of the yarn being tested are parallel and free from twist. The number of turns required to remove the twist is reported per unit length.

4.2 The amount of twist in the component elements of a plied or cabled yarn is determined as follows: one end of the yarn is fixed while the other end is rotated until the structural components are parallel. Any one or all of these components may then be used as test specimens.

5. USES AND SIGNIFICANCE

5.1 The determination of twist in a straight section of a yarn is not the simple straight-forward operation it appears to be, for the test results may be greatly influenced by variations in test procedures and techniques. In all manipulations, extreme care is necessary to prevent rotation of either end of the specimen, which would alter the twist.

5.2 The twist in a yarn before it is packaged may be different from that of the yarn after it has been withdrawn from the package because of changes in tension and the effect of the method of withdrawal. If the yarn is withdrawn over-end, a slight increase or decrease in twist will take place, depending upon the direction of the twist in the yarn, the direction of winding on the package, and the length of the turn (or wrap) on the package.

5.3 The optimum amount of twist depends upon the use for which the yarn is intended. The amount of twist affects both the strength and elongation properties of the yarn with increased twist being associated with increased elongation. The relationship between twist and strength is more complex.

5.3.1 In filament yarns, some twist (up to 7 tpi) (280 tpm) or a suitable sizing is desirable to facilitate textile operations. A small increase results in a loss in strength. However, higher twist in such yarns may be used to subdue luster or increase elongation, or to secure other special effects, as in crepe fabrics.

5.4 For further information on Uses and Significance of twist, refer to ASTM Method D 1423-71, Section 5.

6. APPARATUS

6.1 Twist tester, Alfred Suter, Precision Scientific Co., or equivalent consisting of a pair of clamps, one of which is rotatable in either direction and positively connected to a revolution connector. The tester may be hand- or power-driven. The position of one clamp (or both clamps) shall be adjustable to accommodate specimens of the lengths of 10 and 20 in. and to permit measuring the change in length during untwisting. Means shall be provided for applying the specified tension of the specimen and for determining the specimen length with an accuracy of ± 0.002 in. (0.5 mm). The movable but nonrotatable clamp shall be capable of being traversed with substantially no friction to permit determining the untwisted length of the specimen under the specified tension. The counting device shall be re-setable to zero count and shall indicate the total number of turns to the nearest whole turn.

6.2 Tensioning Weights, for Twist Tester, 1 oz and 4 oz.

6.3 Pick needle

7. SAMPLING

7.1 Select yarn or cord samples to represent the test lot.

7.2 When sampling tubes, cones, or bobbins, take specimens from the side, not over end, of the package, and from the center of the traverse, not at the ends of the package. This is extremely important in taking specimens for twist and tensile tests since the yarn or cord structure is disturbed and twist migrates at a traverse reversal and when taking a specimen for test purposes the twist is not as likely to return to its original location as when the yarn or cord is removed from the package in production.

7.3 When sampling fabric tabbies, leave the tabby mounted on its mounting board. Cut the filling yarns and remove them from areas of the fabric to be sampled.

8. PROCEDURE

8.1 Set the clamps to secure a nominal gage length of 20 ± 0.02 in. for yarn and cord from bobbin of 10 ± 0.02 in. for cord from fabric tabby. For the Alfred Suter Precision instrument this is accomplished by setting the assembly holding the left side clamp fixture to a setting of 17 ± 0.02 in. for bobbin specimens and 7 ± 0.02 in. for tabby specimens. Set the counter to zero. Set the tension in the left side clamp for 4 oz. Lock the left side floating clamp with the floating pointer at 3 ± in.

8.2 With the yarn or cord package to be tested located near and behind the twister testing instrument, strip yarn or cord from the package by unwinding from the side for about 3 yd. to a side yarn or cord that may have lost twist due to an unrestrained end. Continue stripping until yarn or cord has passed a traverse reversal so that the specimen to be tested will have come from the center portion of the package, 7.2.

8.3 Without releasing the yarn or cord lace it through the clamp on the right side of the instrument. Tighten the clamp. Guide the specimen through the clamp at the left and around the hook of the tensioning device. Pull on the end of the specimen until the pointer on the floating left side clamp reads exactly 3 in. Tighten the left clamp on the specimen. Release the left side clamp device.

8.3.1 When mounting specimens of cord from a fabric tabby, grasp one cord near the heavy end of the tabby, cut the cord between the point grasped and the heavy end of the tabby. Without releasing the cord lace it through the right clamp and tighten. Release the cord. Grasp the other end of this cord near the other heavy end of the tabby. Cut the cord between this point grasped and the heavy end of the tabby. Hold taut to prevent the cord specimen from going slack and kinking. Proceed as in 8.3 to lace the left side clamp.

8.4 Set the gearing to the right side clamp to untwist the specimen with the counter moving in an increasing direction. Remove the twist completely by turning the right side rotation clamp until the cord elements are parallel as determined by passing a pick needle between the untwisted elements from one clamp to the other. For a yarn the right side side clamp is rotated until the filaments are essentially parallel as determined by passing a pick needle within the filaments from one clamp to the other. Record the direction of twist and the number of turns in the specimen, to the nearest whole number.

8.5 For a cord specimen the twist in the single element is determined by removing by cutting all but one of the single elements (yarns) between the clamps. The yarns removed are cut near, within 0.25 in, of both clamps. Without changing the original tensioning weight record the length of the one single yarn remaining by reading the scale on the floating left clamp. This value is added to the setting for the left assembly, 8.1.

9. CALCULATIONS

9.1 Calculate to the nearest 0.1 tpi the amount of twist in each specimen in turns per inch.

Twist, tpi = N/L

where:

N = number of turns to the nearest whole number observed in the specimen of cord or single element.
L = length of the specimen to the nearest 0.05 in. before untwisting, 8.1 for an original yarn or cord, 8.5 for an element of a cord.

9.2 Calculate the average twist to the nearest 0.1 tpi for the specimens tested.

10. REPORT

10.1 State that the specimens were tested as directed in AVTEX STM 006-76.

10.2 Report the following information:

10.3 Average turns per inch for yarn; single and ply for cord.

10.4 Direction of twist for yarn; single and ply for cord.

10.5 Identity of analyst.

M-C-4 Finish on Rayon Yarn Using Freon and Isopropanol Extraction

1. SCOPE

1.1 This method covers the determination of finish on rayon yarn using Freon and isopropanol solvents with Soxhlet extraction apparatus.

1.2 This is a general method for finish and follows closely ASTM Method D 2257-75, except the alcohol specified is isopropanol instead of methanol.

2. APPLICABLE DOCUMENTS

2.1 ASTM Standards

D 123-75, Definitions of Terms Relating to Textiles

D 2257-75, Extractable Matter in Yarn

3. DEFINITIONS

3.1 Finish - a lubricant added to a fiber or yarn in the manufacturing process that is necessary for handling and processing.

3.2 For definitions of other textile terms used in this method, see ASTM Definitions D 123-75.

4. SUMMARY OF METHOD

4.1 A specimen is extracted in a Soxhlet apparatus, first with Freon 113 and then with isopropanol. The solvents are evaporated, combined, and the residue dried and weighed. The amount of extracted matter is reported as a percentage of the original weight of the specimen.

5. USES AND SIGNIFICANCE

5.1 This method is used for the determination of the amount of finishing material applied to the yarn during manufacture.

5.2 Finishing materials are added to a yarn to enable it to be wound and processed.

5.3 Besides the quantitative result, examination of the extracted residue for odor, physical state etc, for deviations from normal frequently may be revealing as to the yarn manufacturing procedure including finishing application.

5.4 The residues of the two may be separated and weighed to determine their ratios for deviations from normal and also for analysis for identification of components.

6. APPARATUS AND REAGENTS

6.1 Soxhlet Extraction Apparatus, Pyrex glass, equipped with ground glass joints. Extractor body 50 mm. internal diameter, 250 mm. length, capacity to top of siphon 200 ml., flask size 250 or 300 ml. capacity.

6.2 Stainless or monel discs 1 3/4 in. diameter perforated with four 1/4 in. holes, for insertion in the Soxhlet body to prevent specimen plugging siphon outlet.

6.3 Electric heaters, for heating flasks, equipped with controllers for continuous variation of heater temperature; such as, Precision Type H heaters with Superior Electric Co. Type 116 Powerstats.

6.4 Drying oven, ventilated, capable of maintaining a temperature of 105 ± 3°C.

6.5 Steam bath, for predrying residue.

6.6 Analytical balance, sensitivity of 0.01 gm. for weighing specimen.

6.7 Analytical balance, sensitivity of 0.0001 gm. for, weighing residue.

6.8 Freon 113, available from E. I. Dupont DeNemours and Co. a halogenated hydrocarbon solvent - 1,1,2 trichloro-1,3,3 trifluoroethane.

6.9 Isopropyl alcohol, industrial grade.

6.10 Tongs.

6.11 Evaporation dish, aluminum, 50 ml. usable capacity.

7. SAFETY PRECAUTION

7.1 Normal safety precautions must be exercised with flammable solvents.

8. SAMPLING AND SPECIMEN SIZE

8.1 From each package to be tested, take a specimen weighing 25 ± 0.1 gm.

9. CONDITIONING

9.1 Neither preconditioning nor conditioning is necessary.

10. PROCEDURE

10.1 Place the disc level in the bottom of the Soxhlet extractor apparatus. Place each specimen in a separate Soxhlet body such that the top of the specimen is below the top of the siphon. Insert the extractor body into a flask.

10.2 Pour sufficient Freon 113 over the specimen to fill the extractor body to the siphoning level and furnish sufficient excess to prevent the flask from going dry during the extraction. A total of about 225 ml. is required.

10.3 Attach the condenser and adjust the heater such that the boiling rate produces a siphoning about every 20 minutes. Carry out the extraction for 1 1/2 hours.

10.4 At the end of a siphoning disassemble the condenser, squeeze the specimen against the inside of the Soxhlet body, with a stirring rod. Transfer specimen to an oven for minimal drying. Reassemble the Soxhlet and boil to fill the body with the Freon 113 solvent from the flask. Disassemble and set aside the Freon 113 in the body. Solvent remaining in the flask should be 20-30 ml.

10.5 Pour the contents of the boiling flask into a tared evaporating dish. Rinse the flask twice with about 5 ml. each of Freon 113 and pour into the evaporating dish.

10.6 Place the evaporating dish on the steam bath and evaporate the solvent with as little boiling as possible to prevent losses.

10.7 Remove the specimen from the oven and place it again into the Soxhlet body. Reassemble the Soxhlet apparatus to a second flask.

10.8 Repeat 10.2 using isopropanol.

10.9 Repeat 10.3.

10.10 At the end of a siphoning disassemble the condenser, squeeze the specimen against the inside of the Soxhlet body and discard the specimen. Reassemble the Soxhlet and boil to fill the body with the isopropanol solvent from the flask. Disassemble and set aside the isopropanol in the body. Solvent remaining in the flask should be 20-30ml.

10.11 Pour the contents from this second boiling flask into the evaporating dish on the steam bath. Rinse the second flask twice with 5 to 10 ml. of isopropanol into the evaporating dish.

10.12 Evaporate to dryness on the steam bath, see 10.6.

10.13 Transfer the aluminum dish with the tongs to the drying oven and dry at $105^{\circ}C \pm 3^{\circ}C$ oven temperature to constant weight; that is, until consecutive 15-minute dryings, cooling in a desiccator and weighing show a change in weight of less than 0.0001 gm.

10.14 Record the weight to the nearest 0.0001 gm.

11. CALCULATIONS

11.1 Calculate the percent of combined extracts for each specimen to the nearest 0.01 percent.

$$F, \text{ percent} = 100\ L/W \qquad (1)$$

where:

F = combined extract percent, as is or constant basis
L = weight of combined extract, gm.
W = weight of specimen, origin, gm.

Footnote:

This method is similar to FMC Method FP 1 of 1 June 1966.

M-C-5 pH of Fiber and Fabrics in Distilled Water and Saline Solutions

1. SCOPE

1.1 This method covers the determination of the pH of fiber in distilled water.

1.2 Other AVTEX Methods for pH are STM 127, pH of fiber by Indicator and STM 129, pH of fiber in saline solution by pH meter.

2. APPLICABLE DOCUMENTS

2.1 ASTM Standards

D 123-75 Definitions of Terms Relating to Textile Materials.

3. DEFINITIONS

3.1 pH - a measure of acidity or alkalinity represented by the logarithm of the reciprocal (negative logarithm) of the concentration of hydrogen ions.

3.2 For other textile terms in this method, refer to ASTM Definitions D 123-75.

4. SUMMARY OF METHOD

4.1 A specimen of fiber is soaked in distilled water.The solution is measured for pH using a pH meter.

5. USES AND SIGNIFICANCE

5.1 The acidity or alkalinity of fiber is important for certain end uses; such as, nonsensitivity of product to human skin.

5.2 This is an empirical method since it is the pH of a specific sample weight and solution volume.

6. APPARATUS

6.1 pH meter with electrodes, combination probe or probes.

6.2 Beakers, 100 ml, 250 ml.

6.3 Graduated Cylinder, 100 ml.

6.4 Analytical Balance, sensitivity of 0.01 gm.

6.5 Glass stirring rod.

6.6 Magnetic stirrer.

6.7 pH buffer solutions, 5 pH, 10 pH.

6.8 Distilled water.

6.8.1 Neutralize stock solution of distilled water to 7.0 ± 0.2 pH with meter using 0.1 N HCL or 0.1 N NaOH, using Magnetic stirrer.

7. SAMPLES AND SPECIMENS

7.1 Take representative samples of fiber or yarn of the manufacturing operation.

7.2 From each sample weigh one specimen of 10 ± 0.1 gm.

8. PROCEDURE

8.1 Calibrate the pH meter using the buffer solutions.

8.1.1 Wash probe with distilled water from a wash bottle. Immerse usable part of probe into distilled water in a beaker. Lift probe from water and rinse with distilled water from a wash bottle. Drain. Do no wipe.

8.1.2 Pour about 50 ml. of one of the two buffer solutions into a 100 ml. beaker. Immerse the probe into the buffer solution. Allow meter indicating needle to come to rest. Adjust the meter to indicate the pH of the buffer solution.

8.1.3 Remove probe and wash as directed in 8.1.1.

8.1.4 Pour about 50 ml. of the second buffer solution into a second 100 ml. beaker. Immerse the probe into the buffer solution. Allow meter indicating element to come to rest. Meter should check pH of second buffer within ± 0.05 pH. If meter reading is outside this tolerance a problem exists with either the meter or the buffer solutions which must be resolved.

8.2 Measure 100 ml. of distilled water, pH 6.8, into 250 ml. beaker. Place beaker on magnetic stirrer.

8.3 Using meter adjust pH of water to pH 7.0 ± 0.05, mixing with magnetic stirrer, using 0.01 N HCl or 0.01 N NaOH.

8.4 Place a 10 ± 0.1 gm. specimen of fiber into the beaker. Immerse with stirring rod and agitate fiber in solution for 1 min. ± 5 sec.

8.5 At end of 1 min. pour out water into a 100 ml. beaker, squeezing the fiber with the glass rod. Place pH probe (or probes) into 100 ml. beaker and measure pH of solution. Swirl or otherwise agitate solution.

9. REPORT

9.1 State that specimen was tested as directed in AVTEX STM 057-76b.

9.2 Report pH of solution.

9.3 Report identity of analyst.

M-C-6 Shrinkage of Yarn and Cord in Water and Ambient Temperature

1. SCOPE

1.1 This method covers the shrinkage of rayon yarn or cord in meter lengths when exposed to water at room temperature.

2. APPLICABLE DOCUMENTS

2.1 ASTM Standards

D 123-75 Definitions of Terms Relating to Textiles

3. DEFINITIONS

3.1 Shrinkage - the change in length of a meter length of yarn or cord expressed as a percent of the length prior to exposure to the shrinkage medium.

3.2 For definitions of other textile terms used in this method, refer to ASTM Definitions D 123-75.

4. SUMMARY OF METHOD

4.1 A meter of conditioned yarn or cord is measured under a tensioning weight sufficient to straighten but not to stretch the yarn or cord before and after exposure to water at room temperature followed by drying and conditioning. The change in length is expressed as a percentage of the length before exposure.

5. USES AND SIGNIFICANCE

5.1 Shrinkage in yarn or cord is a measure of the residual strain in the yarn following manufacture.

5.2 The amount of shrinkage in yarn or cord affects wet processing characteristics; e.g. wet treatment.

6. APPARATUS

6.1 Measuring scale - a table with marks 1 m apart, mm scale for at least 20 mm. interior to the 1 meter marks at one end and a pulley at the end of the table.

6.2 Weight - 4 oz., with clip.

6.3 Marking pen.

6.4 Tray

6.5 Water

6.6 Oven, drying, capable of maintaining 105 ± 5°C.

7. SAMPLING

7.1 Test 2 samples per package.

8. CONDITIONING

8.1 Condition sample prior to testing.

9. PROCEDURE

9.1 Measure a specimen by extending the yarn or cord over the measuring scale. Hang a 4 oz. weight on the end of the sample over the pulley, holding onto the other end.

9.2 Mark a specimen by placing marks on the yarn or cord exactly in line with the one meter marks on the table. Disconnect the weight, cut the sample from the package approximately 2 m. long with the marked specimen in the center.

9.3 Coil without tangling on a tray. Immerse the tray in water at room temperature. Sample must not be allowed to float.

9.4 At the end of 1/2 min. remove the tray with the sample from the water, drain to remove excess water, place in oven at 105 ± 5°C. for 1 hr. to dry.

9.5 Remove tray from oven and condition sample for 2 hours in the laboratory.

9.6 Place the sample on the measuring scale as at the time of marking with one end held and the other end extended over the pulley with the 4 oz. weight attached. Align one mark on sample with a mark on the scale. Measure and record the length between the two marks on the sample to the nearest mm.

10. CALCULATION

10.1 Calculate the shrinkage in the specimen to the nearest 0.1 percent using Equation 1.

$$S, \text{ percent} = 100\,(M-L)/M \qquad (1)$$

where:

S = Shrinkage, percent
M = Original length of specimen, 1 m.
L = Length of specimen after exposure, m.

M-C-7 Moisture Regain Content of Fiber

1. SCOPE

1.1 This method covers the determination of the moisture content of yarn.

1.2 This method is consistent with ASTM Method D 2654, Procedure 2.

2. APPLICABLE DOCUMENTS

2.1 ASTM Standards

D 123-75 Definitions of Terms Related to Textile Materials

D 2654-71 Moisture Content and Moisture Regain of Textile Material.

3. DEFINITIONS

3.1 Moisture content - the amount of moisture in a material determined under prescribed conditions and expressed as a percentage of the weight of the moist material, that is, the original weight comprising the dry substance plus any moisture present.

3.1.1 Moisture content is also referred to as moisture on an "as is", "as received", or "wet" basis.

3.1.2 Moisture content differs from moisture regain only on the basis used for calculations.

3.1.3 Moisture calculations are commonly based on the weight of a specimen which has been dried by heating in an oven. If the air in the oven contains moisture, the oven-dried specimen will contain some moisture even when it no longer shows a significant change in weight. In order to ensure that the specimen is moisture-free, it must be exposed to desiccated air until it shows no further significant change in weight.

3.2 For definitions of other textile terms in this method refer to ASTM Definitions D 123-75

4. SUMMARY OF METHOD

4.1 A 10 gm. specimen of yarn is weighed and dried to constant reweight in a Brabender oven supplied with desiccated air. The moisture content is read.

4.2 Since the original weight is 10 gm., the read-out of the Brabender in-oven balance is in moisture content.

5. USES AND SIGNIFICANCE

5.1 This method utilizes a 10 gm. sample. It is an excellent test to measure the variations in moisture content of specimens of yarn. Because of the specimen size it is not recommended for commercial weight determination.

6. APPARATUS

6.1 Brabender oven, operated at 105 ± $3^{o}C.$, supplied with a current of air at a rate sufficient to change the air in the oven at least once every 4 min., the air to the oven shall be predried by circulating through Electro Dryer units.

6.2 Brabender predrying balance

6.3 Reel, 1 m.

7. SAMPLES AND SPECIMENS

7.1 Take care to not allow the yarn to change moisture, either before or after drying.

7.2 Strip sufficient yarn from the package to remove the outside layer of yarn. In practice this is accomplished by taking samples for other test less sensitive to moisture; such as, finish, linear density, etc. from the outer layers. Unwind a skein of about 11-12 gm. Cut from reel. Weigh to exactly 10 grams using the Brabender preweight balance.

7.3 Take 1 specimen per sample.

8. PROCEDURE

8.1 Daily check the calibration of the Brabender balances.

8.1.1 Check the predrying balance by placing a 10 gm. weight in the specimen pan and adjusting balance to indicate 10 gm.

8.1.2 Check the in-oven balance by placing a 10 gm. weight on the balance pan. Read-out scale should read zero. Place a 9 gm. weight on the balance pan. Read-out scale should read 10.0.

8.2 Place the 10 gm. specimen in a cup in the oven at 105 ± 3 °C. Dry to constant weight as follows: Dry for 1 hr. Stop the fan and weigh specimen in the oven. Record percent moisture content to the nearest 0.1 percent. Start the fan and dry for an additional 1/2 hr. Stop the fan and reweigh the specimen. If the change in reading is greater than 0.1 percent moisture content, continue drying and checking the weight every 15 min. until the specimen has reached constant weight.

9. REPORT

9.1 Report the following:

9.2 Average moisture regain for specimens tested.

9.3 Report identity of analyst.

M-C-8 Ash Content of Fiber at Increasing Temperatures

1. SCOPE

1.1 This method covers the determination of the ash content of rayon fiber by exposure at increasing temperature in a muffle furnace.

2. APPLICABLE DOCUMENT

2.1 ASTM Standards

D 123 - 75 Definitions of Terms Related to Textile Material.

3. DEFINITIONS

3.1 Ash, dry method - the residue after combustion for 3 hours in muffle furnace, 1 hour at 200°C, 1 hour at 400°C and 1 hour at 800°C.

3.2 For definitions and textile terms in this method refer to ASTM definitions D 123-75.

4. SUMMARY OF METHOD

4.1 A specimen of rayon fiber or yarn is ignited in a muffle furnace for 3 hours, 1 hour at 200°C ± 25°C, 1 hour at 400°C ± 25°C and 1 hour at 800°C ±25°C. The residue remaining is expressed as a percentage of the "as is" weight of the original specimen.

5. USES AND SIGNIFICANCE

5.1 The ash content of rayon fiber or yarn is indicative of the method of manufacture.

6. APPARATUS

6.1 Porcelain crucible, Coors 12, approx. 250 ml. capacity for staple fiber and Coors 09, approximately 50 ml. capacity for yarn.

6.2 Muffle furnace, electric, HEVI Duty Electric Company, with controller, Thermolyne Temcometer, Sybron Corporation, or equivalent, capable of maintaining 200 ± 25° C., 400 ± 25° C. and 800 ± 25° C.

6.3 Crucible tongs.

6.4 Analytical balance, sensitivity of 0.0001 gm.

6.5 Desiccator with desiccant.

7. USES AND SIGNIFICANCE

7.1 The test specimen shall be selected to represent the test unit.

8. PROCEDURE

8.1 Determine the crucible tare weight by heating in the muffle furnace at 800°C ± 25°C for 10 minutes, cool slightly and place in the desiccator to cool to room temperature. Weigh to the nearest 0.0001 gm.

8.2 Weigh in the tared crucible a specimen of fiber or yarn of about 5 gm. to the nearest 0.0001 gm.

8.3 Place the crucible with the specimen in the muffle furnace at 200 ± 25°C. for 1 hour. Increase the temperature to 400 ± 25°C. for one hour. Increase the temperature to 800 ± 25°C. for 1 hour.

8.4 Cool the crucible and residue slightly and place in a desiccator to cool to room temperature. Weigh to nearest 0.0001 gm.

9. CALCULATIONS

9.1 Calculate the percentage of ash to the nearest 0.01 percent.

$$\text{Ash, percent} = \frac{R - T}{W - T}(100)$$

where:

R = weight of crucible and residue after combustion, gm.
W = weight of crucible and original specimen, gm.
T = tare weight of crucible, gm.

10. REPORT

10.1 State that the specimens were tested as directed in AVTEX STM 092-76.

10.2 Report the following.

10.2.1 Ash content to nearest 0.01 percent.

10.2.2 Identity of analyst.

M-C-9 Zinc Extraction by Use of HCl and Measurement by Atomic Absorption

1. SCOPE

1.1 This method covers the determination of extractable zinc in rayon fiber or yarn by atomic absorption method. The zinc is extracted by bringing a specific weight of specimen to a boil in a specific volume of HCl.

2. APPLICABLE DOCUMENTS

2.1 ASTM Standards

D 123-75 Definitions of Terms Relating to Textile Materials

3. DEFINITIONS

3.1 For definitions of textile terms used in this method refer to ASTM Definitions 123-75.

4. SUMMARY OF METHOD

4.1 A specimen of fiber or yarn is boiled in 1 N HCl. The solution is diluted and evaluated by Atomic Absorption Spectrophotometer against a reference standard.

5. USES AND SIGNIFICANCE

5.1 The amount of zinc in rayon fiber or yarn is indicative of the manufacture of the product.

5.2 The amount of zinc in rayon fiber or yarn may affect end use performance.

6. APPARATUS AND REAGENTS

6.1 Analytical balance, sensitivity of ± 0.001 gm.

6.2 Beaker, 250 ml.

6.3 Hot plate in hood, electric.

6.4 Pipettes, 2 ml., 5 ml., 20 ml.

6.5 Volumetric flasks, 100 ml., 1000 ml.

6.6 Graduated cylinder, 100 ml.

6.7 Distilled water.

6.8 HCl, 1 N, reagent grade.

6.9 Zinc, standard solution, 1 ppm.

6.9.1 Zinc, Standard Solution 1000 ppm, Harleco, Phila.

6.9.2 Pipette 20 ml. of the 1000 ppm standard zinc solution into a 1000 ml. volumetric flask and make to volume with distilled water. Mix. Identify as Solution A. Pipette 5 ml. of solution A into a 100 ml. volumetric flask and make to volume with distilled water. Mix. This solution contains 1 ppm zinc. Identify as Sol. B.

6.10 Atomic Absorption Spectrophotometer, Perkins-Elmer Model 360 and operators manual.

6.10.1 Light source-zinc hollow cathode lamp.

6.10.2 Flame type - air-acetylene, oxidizing, lean, blue.

6.10.3 Wave length setting - 214 nm.

6.10.4 Slit setting - 0.7 nm.

6.11 Hengar granules (boiling chips)

7. SAMPLING

7.1 Representative sample to provide duplicate specimens 3-4 gm.

8. CONDITIONING

8.1 Neither preconditioning or conditioning is necessary.

9. PROCEDURE

9.1 Weigh a 3.5 ± 0.1 gm. specimen to the nearest 0.001 gm.

9.2 Place specimen in 250 ml. beaker containing a few Hengar chips. Add 100 ml. of 1 N HCl. Mark level of solvent with fiber completely immersed by placing on the outside of the beaker the bottom edge of a gum paper label even with the top of the solvent level. Bring to boil on hot plate under a hood. Remove and cool to room temperature. Add 1 N HCl to bring solvent volume back to bottom edge of the label. Mix thoroughly with glass rod.

9.3 Pipette 5 ml. aliquot into a 100 ml. volumetric flask. Dilute to volume with distilled water. Mix.

9.4 Calibrate the Atomic Absorption Spectrophotometer.

9.4.1 Set zero transmission with distilled water blank.

9.4.2 Set 100 percent transmission with 1 ppm zinc standard.

9.5 Read specimen solution, 9.3, on Atomic Absorption Spectrophotometer.

9.6 Repeat for duplicate specimen whenever first specimen falls out of specifications.

10. CALCULATION

10.1 Calculate amount of zinc to nearest 0.005 percent.

zinc, percent = $(R \times D) / S\ (10^4)$

where:

R = reading of AA, direct in ppm for 1 ppm full scale calibration.
D = Dilution factor, (100) (100) / 5
S = specimen weight, gm.

11. REPORT

11.1 State the specimens were tested as directed in AVTEX Method STM 118-76.

11.2 Report the following information:

11.2.1 Average amount of zinc to nearest 0.005 percent

11.2.2 Range of duplicate specimens to nearest 0.005 percent if two were run.

11.2.3 Identification of analyst.

M-C-10 Sulfur Content of Yarn, Both Sulfate and Elemental Sulfur

1. SCOPE

1.1 This method covers the determination of sulfur, in sulfate and free sulfur form, in rayon fiber.

2. APPLICABLE DOCUMENTS

2.1 ASTM Standards

D 123-75 Definitions of Terms Related to Textile Materials.

3. DEFINITIONS

3.1 For definition of textile terms in this method refer to ASTM Definition D 123-75.

4. SUMMARY OF METHOD

4.1 A specimen of yarn is dissolved in conc. nitric acid. Conc. hydrochloric acid is added with potassium chlorate and the solution boiled down to release the nitric acid and nitrates. The alkalinity is adjusted with ammonium hydroxide and the sulfate and sulfur converted to barium sulfate with the addition of barium chloride. The barium sulfate precipitate is filtered, washed, dried and weighed. The weight is converted to free sulfur and reported as a percentage of the original specimen.

5. USES AND SIGNIFICANCE

5.1 The amount of sulfur present in a rayon fiber is indicative of method of manufacture of the fiber.

5.2 Sulfur content is of importance to some uses of rayon fiber and is covered in grading standards.

6. APPARATUS

6.1 Beaker, 250 ml., Phillips conical

6.2 Analytical balance, sensitivity ± 0.0001 gm.

6.3 Muffle furnace, capable of 800°C.

6.4 Gooch crucible, Coors 7 M.

6.5 Filtering flask, with adapter for Gooch crucible.

6.6 Asbestos, purified acid washed long fiber, J. T. Baker Chem. Co.

6.7 Graduated cylinders, 25 ml., 200 ml.

6.8 Hot plate

6.9 Oven, capable of $105^{\circ} \pm 3^{\circ}$ C.

6.10 Pipette

6.11 Desiccator with desiccant

7. REAGENTS

7.1 Nitric acid, conc., reagent grade

7.2 Hydrochloric acid, conc., reagent grade

7.2.1 1:1 solution of HCl on volume basis

7.3 Potassium chlorate, reagent grade

7.4 Methyl orange indicator

7.5 Ammonium hydroxide, conc., reagent grade

7.6 Barium chloride, reagent grade

7.6.1 Barium chloride, 5 percent solution, weight-volume basis prepared with distilled water

7.7 Silver Nitrate, reagent grade

7.7.1 Silver nitrate, 10 percent solution, weight-volume basis prepared with distilled water

8. SAMPLES AND SPECIMENS

8.1 Weigh a specimen of 1 ± 0.001 gm. fiber.

9. PROCEDURES

9.1 Place the 1 gm. specimen of fiber in a 250 ml. Phillips beaker. Add 15 ml. of concentrated nitric acid and warm slightly. Agitate the mixture until the fiber is dissolved. When the yarn is practically free from sulfate and free sulfur, the solution will be clear.

9.2 Place the beaker containing the solution on a hot plate in a hood and boil down to about 10 ml. Cool to room temperature. Add 20 ml. of conc. hydrochloric acid and a few crystals of potassium chlorate. Continue boiling until the volume is reduced to about 10 ml. Cool slightly, add another 20 ml. of hydrochloric acid and boil until brown fumes are no longer visible. At this point the solution is free of nitric acid or nitrates.

9.3 Remove the beaker from the hood. Add 3-5 drops of methyl orange. Neutralize the solution with concentrated ammonium hydroxide to yellow color. Dilute with 20 ml. distilled water. Make solution acidic with 1:1 hydrochloric acid (pink), add 1 ml. excess. Bring to a boil and add 15 ml. of 5 percent barium chloride solution with stirring, adding the first 4 ml. very slowly dropwise. Again bring to boil and boil for 2 min. Allow to stand at least 2 hrs. or overnight.

9.4 Preparation of Gooch crucible.

9.4.1 Prepare a slurry of asbestos in distilled water in a beaker.

9.4.2 Place the Gooch crucible in the adapter on the filtering flask. Pour the slurry of asbestos slowly into Gooch crucible with suction applied to the flask until a matt develops that completely covers the bottom of the crucible about 2 mm. thick. Wash with 200 ml. of distilled water to remove any fine fibers: Dry in an oven at 105 ± 3°C. for 30 min. Transfer to muffle furnace 800°C. and ignite to constant weight. Thirty min. is generally adequate followed by cooling to room temperature in a desiccator, and weighing to 0.0001 gm. for tare weight of prepared Gooch crucible.

9.5 Filter solution from 9.3 through tared prepared Gooch crucible in adapter on filtering flask with suction applied. Scrub Phillips beaker with a rubber policeman to completely transfer all the contents to the crucible.

9.6 Wash precipitate with hot distilled water until free of chlorides as determined by absence of turbidity in the filtrate with the addition of a few drops of 10 percent silver nitrate solution. 200 ml. of hot distilled water has been found to be adequate generally.

9.7 Dry prepared Gooch crucible with precipitate in an oven at 105 ± 3°C. for 30 min.

9.8 Transfer to the muffle furnace at 800°C. and ignite to constant weight, that is, until it loses no more than 0.1% of its weight at 30 min. intervals of drying followed by cooling in the desiccator.

9.9 The precipitate is barium sulfate.

10. CALCULATIONS

10.1 Calculate the amount of sulfate and free sulfur as elemental sulfur in the specimen to the nearest 0.001 percent.

Sulfur, percent = 100 (K x P) / W

where:

K = ratio of atomic weight of sulfur to molecular weight barium sulfate, 0.1373
P = weight of barium sulfate precipitate, gm.
W = weight of fiber specimen, gm.

M-C-11 Rayon Yarn Requirements for Carbon Textiles

1. SCOPE

1.1 This specification covers the requirements for rayon yarn to be used in the manufacture of carbon textiles.

2. APPLICABLE DOCUMENTS

2.1 Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue specified below shall apply:

2.1.1 **FMC Fiber Division**

	Measurement of:	Similar Methods in this manual
FMC STM 003:	Yarn Denier	M-C-1
FMC STM 005:	Yarn Strength	M-C-14, 2
FMC STM 006:	Yarn Twist	M-C-3
FMC STM 010:	Yarn Finish Content	M-C-4
FMC STM 057:	Yarn pH	M-C-5
FMC STM 069:	Yarn Shrinkage	M-C-6
FMC STM 092:	Ash Content	M-C-8
FMC STM 114:	Moisture Content	M-C-43
FMC STM 140:	Sulfur Content	M-C-10
FMC STM 118:	Zinc Content	M-C-9

3. REQUIREMENTS

3.1 Qualification - The material finished under this specification shall be a product which has been tested and has passed the qualification tests specified herein.

3.2 Material - Unless otherwise specified, the material purchased to this specification shall be rayon code 2175, made by the FMC Fiber Division. The yarn shall not be treated with a finish of any kind other than the standard water-dispersable finish originally applied.

3.3 Chemical Requirements - The chemical requirements of the yarn shall be as follows:

Ash Content, Wt. %	0.7 Maximum
Sulfur Content, Wt.%	0.25 Maximum
Zinc Content, Wt.%	0.07 Maximum
pH	5.0 - 8.0
Finish Extractables, Wt.%	0.2 - 1.0

3.4 Physical Requirements - The physical requirements of the yarn shall be as follows:

	Min.	Max.
Twist, turns, per inch, (tpi)	1.6	2.4
Conditioned, Strength, Lbs.	10.3	13.5
Conditioned Elongation, %	5.5	9.5
Moisture Content, wt.%		13.0
Denier, gms./9000 meters	1590.0	1710.0
Shrinkage, %	2.9	4.7

4. QUALITY ASSURANCE PROVISIONS

4.1 Quality Control System Requirements - FMC Fiber Division specifcations apply in all particulars relevant to material supplied.

4.2 Lot. Unless otherwise specified, all Rayon yarn shipped in one truckload shall be considered a lot for purposes of inspection and tests. The invoice number for a particular truckload is the lot number identification.

4.3 HITCO Inspection - The HITCO inspector shall make such inspections as are necessary to determine that the material is in accordance with the requirements of this specification with regards to purchase order, packaging, marking, packing, and acceptance or qualification tests.

4.4 Supplier Inspection - Unless otherwise specified, the supplier is responsible for the performance of inspections necessary to determine that the material is in accordance with the requirements of this specification with regards to purchase order, packaging, marking, packing, and acceptance or qualification tests. These inspections shall be made prior to submission for customer inspection and acceptance. Except as otherwise specified, the supplier may utilize his own facilites or any commercial facility acceptable to the customer. Inspection records of the examinations and tests shall be kept complete and available to the customer.

4.5 Customer Rejection - Any lot inspected at one time may be rejected in its entirety and be subject to return for failure to meet purchase order requirements.

4.6 Classification of Tests - The inspection and testing of the material shall be classified as follows:
Qualification Tests
Acceptance Tests

4.7 Qualification Tests - The qualification tests for this material shall consist of all tests listed in Table I. At the discretion of HITCO these tests may be repeated subsequent to the intial qualification.

4.7.1 Sampling for Qualification Tests - Unless otherwise specified, ten (10) representative samples of a sufficient size to perform the qualification tests shall be selected at random from the lot presented for inspection.

4.7.2 Qualification Test Report - The material supplier shall furnish to customer three copies of a report of tests verifying that the material conforms to the qualification requirements of this specification.

TABLE I

Characteristic		Test Method in this manual
Ash Content	FMC STM 092	M-C-8
Sulfur Content	FMC STM 140	M-C-10
Zinc Content	FMC STM 118	M-C-9
pH	FMC STM 057	M-C-5
Finish Content	FMC STM 010	M-C-4
Twist	FMC STM 006	M-C-3
Conditioned Strength	FMC STM 005	M-C-14, 2
Conditioned Elongation	FMC STM 005	M-C-14, 2
Moisture Content	FMC STM 037	M-C-7
Denier	FMC STM 003	M-C-1
Shrinkage	FMC STM 069	M-C-6

4.8 Acceptance Tests - The acceptance tests for this material shall consist of all listed in Table II.

TABLE II

Characteristic		Test Method in this manual
Ash Content	FMC STM 092	M-C-8
pH	FMC STM 057	M-C-5
Finish Content	FMC STM 010	M-C-4
Twist	FMC STM 006	M-C-3
Denier	FMC STM 003	M-C-1
Conditioned Strength	FMC STM 005	M-C-14, 2
Conditioned Elongation	FMC STM 005	M-C-14, 2
Moisture Content	FMC STM 657	M-C-7
Shrinkage	FMC STM 069	M-C-6

4.8.1 Sampling Plan for Acceptance Tests - Unless otherwise specified, all shipments shall be acceptance tested for conformance to this specification. One sample shall be randomly selected from each 3300 pounds of yarn produced and tested to the requirements of Table II.

4.8.2 Rejection for Acceptance Tests - If any sample fails any one or more of the acceptance tests described in Table II, another package shall be randomly sampled from the same 3300 pound batch. The retest data shall be averaged with the original data obtained on the first sample. If the average test data passes the minimum requirements, the 3300 pound batch is accepted. If the average test data is below the minimum requirements, the 3300 pound batch being tested shall not be shipped to the weaver or to HITCO. In the event that subsequent failures are repetitive to a significant degree as determined by the supplier, the supplier may request a specification amendment subject to the review and approval of HITCO.

4.8.3 Acceptance Test Report - The material supplier shall furnish to customer, with each shipment, three copies of a report of tests verifying that the lot of material conforms to the acceptance requirements of this specification.

4.9 Test Procedure - The test procedure shall be in accordance with the applicable procedures listed in Tables I and II.

		Test Methods This Manual
Ash Content	FMC STM 092	M-C-8
Sulfur Content	FMC STM 140	M-C-10
Zinc Content	FMC STM 118	M-C-9
pH	FMC STM 057	M-C-5
Finish Content	FMC STM 010	M-C-4
Twist	FMC STM 006	M-C-3
Conditioned Strength	FMC STM 005	M-C-14, 2
Conditioned Elongation	FMC STM 005	M-C-14, 2
Denier	FMC STM 003	M-C-1
Shrinkage	FMC STM 069	M-C-6

5. PREPARATION FOR DELIVERY

5.1 Packaging - The yarn shall be packaged on tubes. Each layer of tubes in the case shall be separated by a divider. There shall be 8-10 pounds of yarn per tube, 4 to 10 pound tubes may be supplied as ordered by the customer.

5.2 Packing - Unless otherwise specified, the rayon yarn shall be packed in a clean, dry case of the standard size and type normally used by FMC Fiber Division. The case shall be so constructed as to insure acceptance by common or other carrier for safe transportation at the lowest rate, to the point of delivery specified by the purchase order.

5.3 Marking - Cases shall be marked in accordance with FMC Fiber Division standard practices. Marking shall include, but not be limited to the following information:

1650/720/2.O-Z, Rayon Yarn, MDS 2015, Code 2175.
Manufacturer's names.
Manufacturer's invoice number.

6. NOTES

6.1 Ordering Data - Procurement documents should specify, but not be limited to the following information:

1650/720/2.O-Z, Rayon Yarn, MDS 2015, Code 2175.
Place of inspection.
Place of Delivery.
Weight of Material.

AVTEX FIBER SPECIFICATIONS FOR BURLINGTON

Product	Carbonizing Yarn
Date of Issue	11/30/76
Code	2175
Description	1650/720 Filament Yarn

Test Method	Min.	Max.	Test Method	Test Methods This Manual
Cond. Strength, lbs.	10.3	13.5	STM 005	M-C-2,14
Cond. Elongation, %	5.5	9.5	STM 005	M-C-2,14
Finish, %	0.2	1.0	STM 010	M-C-4
Shrinkage, %	2.9	4.7	STM 069	M-C-6
Twist, T.P.I.	1.6	2.4	STM 006	M-C-3
Denier	1590	1710.0	STM 003	M-C-1
pH	5.0	8.0	STM 057	M-C-5
Moisture, %	----	13.0	STM 087	M-C-7
Ash Content, %	----	0.7	STM 092	M-C-8
Sulfur Content, %	----	0.25	STM 140	M-C-10
Zinc Content, %	----	0.07	STM 118	M-C-9

E. J. Epp, Manager
Technical Services

This specification is effective through December, 1977. If it is to be reissued, Avtex Manager of Technical Services must be advised in writing during October, 1977.

These specifications relate to Avtex analytical results. Other laboratories may get different levels.

M-C-12 Carbon Fabric Specifications

1. SCOPE

1.1 This specification covers one type of carbon fabric used in solid rocket motor nozzle components.

1.2 The carbon fabric covered by this specification is intended for use in the manufacture of solid rocket motor nozzle components.

2. REQUIREMENTS

2.1 Material. The material furnished under this specification shall be a pan base carbon fiber (T-300) woven into a satin harness 8 fabric and supplied with properties as defined in 3.1.

2.2 Yarn. Three thousand filament yarn shall be used to weave the fabric.

2.3 Tracer Yarns and Fabric Finishes. Tracer yarns and fabric finishes within the fabric shall be acceptable.

3. CLASSIFICATION OF INSPECTIONS

3.1 Inspection requirements specified herein are classified as quality conformance tests.

	Test Methods This Manual
Visual examination	M-C-31
Fiber ultimate tensile strength	M-C-14
Fiber density	M-C-15
Weight	M-C-16
Thread Count	M-C-17
Thickness	M-C-18

4. QUALITY ASSURANCE PROVISIONS

4.1 Sampling. Each lot of material shall be sampled for inspection as follows:

4.2 Lot. A lot shall consist of that quantity of material which is manufactured at one time, using the same process, and under the same conditions of manufacture. Any change in materials, formulation, processes, tools or conditions from lot to lot shall require approval by Thiokol. A lot shall not consist of more than 250 pounds.

4.3 Samples. A material sample of sufficient quantity to perform all tests specified herein shall be selected from the outside end of each master roll.

4.4 Test Specimens. A minimum of three specimens shall be tested from each sample for each of the properties requiring test specimens. All test results shall be reported to the procuring organization. The average test results of three test specimens shall be reported in addition to the reporting of the individual test specimen value. The average results of each test shall meet the requirements.

5. DEFINITIONS

5.1 Master Roll. A master roll is the wide broadgoods fabric roll shipped to the procuring activity.

5.2 Sample. A sample is a cut-out section of a cloth master roll.

5.3 Specimen. A specimen is a cut-out section of a sample used for testing.

5.4 Typical Materials. The following materials are typical of the material covered by this specification:

5.4.1 Fabric Style ES-1252 manufactured by Woven Structures

5.4.2 Fabric Style W-133 manufactured by Fiberite

M-C-13 Visual Examination of Broadgood Rolls

1. SCOPE

1.1 Each roll of raw material shall be visually examined.

2. MATERIAL

2.1 Broadgoods. The material shall have no holes, tears, non-uniform weave, resin build-up, contaminants, or other defects which would render more than 5 percent of any roll unsuitable for the intended use.

2.2 Tape. Tape material shall be free from dirt, grease, oil, or any other contaminates. Tape shall be uniform in appearance and free from holes, tears, nonuniform weave and any other defects which may be detrimental to the use of the material for the purpose intended.

M-C-14 Fiber Ultimate Tensile Strength Specification Limits of Carbon Fiber

1. SCOPE

1.1 A certificate of compliance from the carbon fiber supplier shall be submitted and will be acceptable for verification of requirement.

1.2 Limits (psi)

Minimum	312,000
Maximum	--

M-C-15 Fiber Density Specification Limits of Carbon Fiber

1. SCOPE

1.1 A certificate of compliance from the carbon fiber supplier shall be submitted and will be acceptable for verification of requirement.

1.2 Limits (g/cc)

Minimum	1.7
Maximum	--

M-C-16 Fiber Weight Specification Limits of Broadgoods

1. SCOPE

1.1 The weight of the material on all master rolls shall be determined in accordance with ASTM D 1910-64.

1.2 Limits (ounces / sq.yd.)

Minimum	10.0
Maximum	12.0

M-C-17 Thread Count Specification Limits on Master Rolls of Broadgoods

1. SCOPE

1.1 The thread count of the material on all master rolls shall be determined in accordance with ASTM D 1910-64.

1.2 Limits (Yarns per inch)

	Warp Direction	Fill Direction
Minimum	22.0	21.0
Maximum	26.0	25.0

M-C-18 Thickness Specification Limits of Master Rolls of Broadgoods

1. SCOPE

1.1 The thickness at 5 ± 3 psi tensioning on all master rolls shall be determined in accordance with ASTM D 1777-64.

1.2 Limits (inch)

Minimum	0.016
Maximum	0.021

M-C-19 Carbon Reinforcement, 1200 ppm sodium, Specifications for Rocket Nozzle Components

1. SCOPE

1.1 This specification covers one type of carbon reinforcement for use in solid rocket motor nozzle components.

1.2 The carbon reinforcement covered by this specification is intended for use in the manufacture of carbon cloth aldehyde-phenolic for reinforced plastic nozzle components.

2. REQUIREMENTS

2.1 Qualification. The material furnished under this specification shall be a product which has met the qualification requirements.

2.2 Material. The material furnished under this specification shall be a rayon-based carbon eight harness satin fabric finally processed at a temperature range of 2000 to 2850 degrees Fahrenheit (F).

3. CLASSIFICATION OF INSPECTIONS

3.1 Inspection and testing of the carbon reinforcement shall be classified as follows:

3.1.1 Qualification and Conformance Inspections

	Methods in This Manual
Visual Examination	M-C-13
Carbon Assay and Moisture Content	M-C-20
Ash Content	M-C-8, 21
Weight	M-C-16
Breaking Strength	M-C-22, 2
Specific Gravity	M-C-23
Thread Count	M-C-17
Thickness	M-C-18
Sodium Content	M-C-24

4. SAMPLING

4.1 Qualification. Two rolls of material shall be processed and tested. Sodium, ash and carbon specimens shall be removed from the beginning end and at 5 other equally spaced axial locations along the length of each roll. One of each specimen shall be removed at left, center, and right locations at each axial station; 21 total test sites. Three each specimens for all other properties shall be removed from the exposed end of each roll. Samples of material (approximately 2 x 2 inches) shall be removed from each test site and sent to Morton Thiokol. These samples shall be tested for weight loss in air and for alkali and alkaline earth metals by spectrographic emission, for information only. One additional roll of material shall be randomly selected from the first production lot and subjected to the same tests as specified for the two initial rolls.

4.2 Quality conformance. Each roll in the lot shall be tested for carbon, sodium and ash content. Six specimens, three from each end of the rolls, shall be tested. Three specimens shall be removed from the exposed end of sampled rolls and tested for moisture, weight, breaking strength, specific gravity, thickness, and thread count. The number of rolls to be selected for test shall be in accordance with Table I. In addition, one roll shall be selected at random from each of the first 10 production lots and tested for sodium and ash content as specified.

Table I: Sampling

Number of rolls in lot	Number of sample rolls to be selected for testing
1 to 3	All
4 to 10	3
11 to 45	5

4.3 Lot. A lot shall consist of that quantity of material which is manufactured at one time, using the same process, and under the same conditions of manufacture. A lot shall not consist of more than 2500 pounds. Individual rolls shall not exceed 125 pounds.

4.4 Accept-reject criteria. The lot of material (qualification or quality conformance) shall be acceptable provided all individual values for sodium, ash and carbon content, and the average values for all other properties for each roll selected meet the requirements. In the event that a single specimen fails to meet sodium or ash content, two repeat tests may be conducted. The roll shall be acceptable with respect to sodium and ash content if both repeat tests meet the applicable requirements. Failure to meet sodium, ash, or carbon content requirements within one roll shall reject that roll. Report all individual and average values and test data.

M-C-20 Ash Content of Filler Material

1. SCOPE

1.1 The ash content determination shall be performed as follows:

NOTE: The ashed samples shall be used to measure sodium content. Care should be exercised not to contaminate the sample with sodium. Samples should not be touched with the hands.

2. PROCEDURE

2.1 Take an approximate 4 gram representative sample. Place test specimen in a beaker and dry in an air circulating oven at 225 ± 5 °F. for a minimum of one hour.

2.2 Remove the specimen from the oven and cool in a desiccator.

2.3 Weigh approximately 2 grams of the cooled specimen to the nearest 0.1 mg. into separate tared ceramic or platinum crucibles.

2.4 Heat the crucible in a muffle furnace at 600 ± 15°C for 16 to 18 hours or until constant weight is achieved.

2.6 Remove the crucible containing the ashed specimen, place in a desiccator to cool and weigh to the nearest 0.1 mg.

2.7 Obtain the post-heat weight and calculate the percent ash as follows:

$$\text{Percent ash} = \frac{(A-B) \times 100}{W}$$

Where:

A =weight of crucible plus ash, gm.
B = weight of crucible, gm.
W = weight of original specimen, gm.

2.8 Report ash content to the nearest 0.10 percent.

2.9 Retain specimen for measurement of sodium content.

2.10 Limits (percent ash)

Minimum	0.0
Maximum	0.5

M-C-21 Breaking Strength Specifications for Broadgood Rolls

1. SCOPE

1.1 The breaking strength test shall be conducted as specified in ASTM D 1682.

1.2 Limits (lb/inch of width)

	Warp direction	Fill direction
Minimum	20	15
Maximum	--	--

M-C-22 Specific Gravity Specification of Broadgoods

1. SCOPE

1.1 The test for specific gravity shall be conducted in accordance with ASTM C 135 or C 604 may be used as an alternate method.

1.2 Limits (at 25°C)

Minimum	1.8
Maximum	1.9

M-C-23 Sodium Content of Filler Material by Flame Emission Method

1. SCOPE

1.1 The sodium content determination shall be in accordance with the following:

2. PROCEDURE

2.1 Obtain ash sample as in M-C-20.

2.2 Wash down the walls of each crucible with distilled or laboratory grade deionized water and add approximately 5 milliliters (ml.) concentrated hydrochloric acid to each.

2.3 Bring the acid mixture to a boil, cool the crucibles transfer the contents of each to a one liter volumetric flask and dilute to volume with distilled water or deionized water.

2.4 Determine the sodium content of each flask by flame emission at 589 nanometers (nm) using the instrument manufacturers operating instructions and a calibration curve for sodium which is linear between 0.1 and 2.5 micrograms per milliliter.

2.5 Report sodium content to the nearest 10 parts per million (ppm).

M-C-24 Raw Graphite Fabric Visual and Physical Inspection

1. SCOPE

1.1 Scope. This instruction describes a detailed procedure for the inspection and grouping of raw graphite fabric.

1.2 Classification. Raw graphite fabric shall be classified into the three following groups. The classification depends on visual and physical characteristics.

Group A
Group B
Group C

2. APPLICABLE DOCUMENTS

2.1 The following documents, the issue in effect at the time, form a part of this instruction:

2.1.1 U. S. Polymeric, Inc.:
(a) QCi-9-1, Material Identification, Storage and Control
(b) QCi-10-1, Indication of Inspection by Stamps

3. PROCEDURE

3.1 Machine set-up. Set up the inspection machine as follows:

3.1.1 Vacuum. Turn on the vacuum and check the vacuum bag for leaks and dirt. Change the vacuum bag if necessary.

3.1.2 Threading the leader. Starting from the unwind side of the inspection machine, thread a leader under the first roller, up over the top roller, down through the vacuum head, under the next roller and then to the rewind roller. See Figure 1.

3.1.3 Unpacking. Carefully unpack the fabric saving the polyethylene wrapping for repackaging. Use gloves, do not touch with hands, because of sodium contamination of product.

3.1.4 Mounting the fabric. Place the roll of fabric on the unwind shaft and secure the roll with chucks. Using two-sided tape, attach the leader to the fabric as close to the leading edge as possible.

3.1.5 Alignment of fabric. Run the fabric through the machine at low speed to the windup end with the brake off. Center the fabric on the rollers if necessary. Remove the leader and tape the fabric to a 3-inch ID core on the wind-up shaft providing good alignment and uniform tension on the fabric between the windup shaft and the first roller.

3.1.6 Maintaining alignment. Maintain good alignment on the windup shaft by adjusting the position of the unwind shaft.

3.1.7 Inspection speed and tension. Start the machine and maintain slow speed with the brake on. Adjust the brake to provide optimum tension.

3.2 Inspection and classification. General inspection shall be conducted from the back of the machine, viewing the fabric in the vertical position and over the top roller; see areas A and B of Figure 1. Inspect for bagginess in area C. Sampling shall be as designated.

3.2.1 Group A. Group A rolls shall conform to the following:

3.2.1.1 Defects. The roll shall be free from visual defects including bags, puckers, brittleness or stiffness, weave defects, holes and tears. All workmanship must be of the highest quality.

3.2.1.2 Color. Color shall be uniform throughout the roll.

3.2.1.3 Pieces. There shall be no more than three pieces in the roll.

3.2.1.4 Length. The minimum length of any piece in a roll shall be 20 yards.

3.2.2 Group B. Group B rolls shall conform to the following:

3.2.2.1 Color. Color shall be uniform throughout the roll.

3.2.2.2 Brittleness and stiffness. There shall be no brittle or stiff areas in the roll.

3.2.2.3 Creases. There shall be no more than two (2) creases per width of fabric. Creases shall be a minimum of two (2) yards apart. No crease shall fold over or be deeper than three eights (3/8) inch nor longer than six inches.

3.2.2.4 Weak material. The material shall not be weak, permitting stretching or distortion during inspection.

3.2.2.5 Weave defects. There shall be no excessive weave distortion or pulled threads.

3.2.2.6 Holes and tears. Holes and tears greater than three eights (3/8) inch in any one dimension shall be a minimum of three (3) yards apart in the warp direction. There shall not be more than two (2) per sixty (60) yards. Holes shall not be greater than one (1) inch.

3.2.2.7 Width. The width of the fabric shall not vary more than two (2) inches throughout the roll. There shall not be ragged uneven edges.

3.2.2.8 Length. The minimum length of any piece in a roll shall be nine (9) yards.

3.2.2.9 Bag. Bag shall not exceed the following limits when measured in a 30 inch horizontal span:

3.2.2.9.1 The bag shall be no greater than one and one-half (1-1/2) inches in depth by three-fourths (3/4) the width of the fabric or

3.2.2.9.2 The bag shall be no greater than two and one-half (2-1/2) inches in depth by one-half (1/2) the width of the fabric or

3.2.2.9.3 The bag shall be no greater than four (4) inches in depth by one-third (1/3) the width of the fabric.

3.2.2.9.4 In no case shall the bag exceed four (4) inches in depth.

3.2.3 Group C. Group C shall be rolls that contain one or more defects in excess of those for groups A or B.

3.2.4 Borderline material. Borderline material shall be placed in the lower of the two groups in question.

3.2.5 Unlisted defects. Any defects which are not listed above found within a roll of material shall be brought to the attention of the shift supervisor or leadman, when found, in order that an immediate evaluation and grading can be made on the material.

3.3 Packaging. Each roll of fabric shall be repackaged in the original shipping container with the polyethylene wrapping as originally received. Rolls originally supported shall be repackaged in the same manner.

3.4 Marking. Each roll shall be marked in accordance with QCi-9-1 and QCi-10-1.

3.5 Segregation. Non-conforming items shall be segregated in accordance with QCi-9-1.

4. REPORTING

4.1 Duplicate inspection reports shall be kept for each roll inspected. The inspection reports for Group A and B shall be stamped "visually accepted" and reports for Group C shall be stamped "Hold for material review" in accordance with QCi-10-1. One copy of the report shall be forwarded to Incoming Quality Control and the other shall be retained in inspection.

5. NOTES

5.1 Definitions.

5.1.1 Bag. The deviation of the fabric from the horizontal plane established by two (2) parallel bars over which the fabric is spanned.

5.1.2 Creases. Creases are small bags or wrinkles appearing in the fabric.

5.2 Handling of high purity fabrics. When handling high purity fabrics, gloves must be worn to avoid contamination.

VISUAL INSPECTION OF RAW GRAPHITE FABRIC

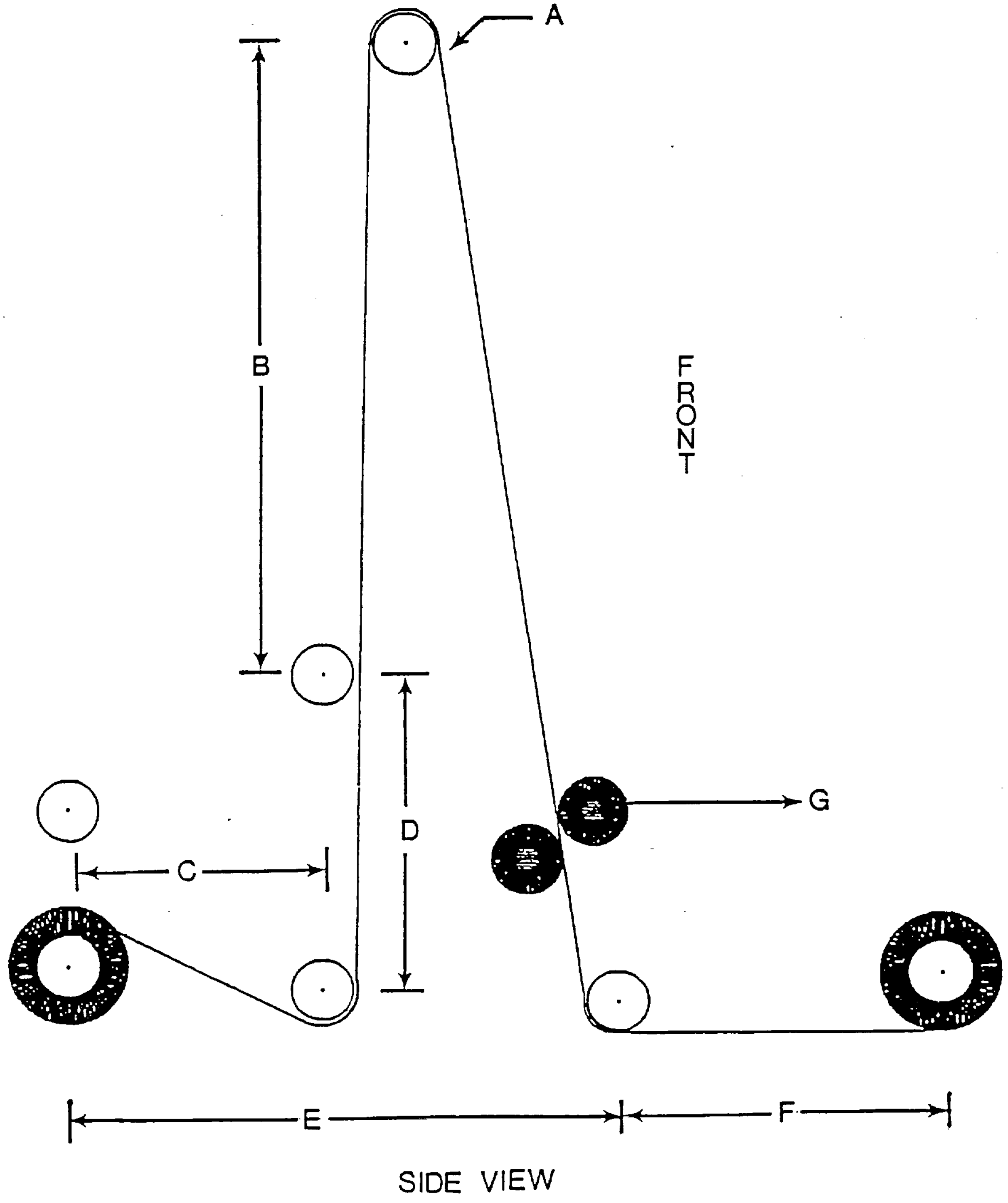


Fig. 1

M-C-25 pH of Fabric by Both Cold and Hot Water Extraction

1. SCOPE

1.1 This procedure describes two methods for the determination of pH of raw fabrics. Method I describes a procedure for the determination of pH using cold water extraction. Method II describes a procedure for the determination of pH using hot water extraction.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

2.1.1 Pan balance, minimum sensitivity 0.1 gm.

2.1.2 Beakers, 150 ml. tall form

2.1.3 pH meter, Corning model 7 or equivalent

2.1.4 Watch glass, 65 mm. (2-1/2")

2.1.5 Hot plate

2.2 Materials

2.2.1 Distilled water, freshly boiled, pH 7.00 ± 0.05

3. SAMPLING

3.1 Sample size. The test requires a representative sample of at least 8 gms.

3.2 Number of determinations. Unless otherwise specified, conduct duplicate determinations on each sample.

4. PROCEDURE

4.1 Method I. Cold water extraction of fabrics for determinations of pH.

4.1.1 Extraction. Weigh 4.0 ± 0.2 gms. of fabric, cut to approximately 1/2" x 1/2" pieces, into a 150 ml. beaker. Add 100 ± 2ml. of room temperature freshly boiled distilled water with a pH of 7.0 ± 0.05. Stir the fabric-water mixture and ascertin that the fabric has been thoroughly wetted. Cover with a watch glass and allow to stand at room temperature for 60 ± 5 minutes.

4.1.2 Determination of pH. If necessary, calibrate the pH meter in accordance with SCM-7-02. Rinse the electordes with distilled water and immerse the electrodes about one inch in the solution to be tested.

Turn the instrument to the "read" position and allow 15 seconds to pass before taking a reading. Record the reading to the nearest 0.02 pH unit.

4.2 Method II. Hot water extraction of fabrics for the determination of pH.

4.2.1 Extraction. Weigh 4.0 ± 0.2 grams. of fabric, cut to approximately 1/2" x 1/2" pieces, and transfer to a 200 ml. beaker. Add 100 ± 2 ml. of freshly boiled, distilled water with a pH of 7.0 ± 0.05. Cover with a watch glass and boil on a hot plate for 20 to 30 minutes. Remove from the hot plate and allow the solution to reach room temperature. Add freshly boiled, distilled water with a pH of 7.0 ± 0.05 to bring the volume to 100 ± 2 ml. and determine the pH as in 4.1.2.

5. CALCULATIONS AND REPORTING

5.1 Report the average of two tests to 0.01 pH unit. Duplicate determinations should be considered suspect if the range exceeds 0.5 pH unit.

M-C-26 Fabric Thread Count for Both Warp and Fill Direction

1. SCOPE

1.1 Scope. This method describes a procedure for the determination of the number of warp and fill yarns per inch of woven cloth.

1.2 Equivalent method. This method is similar to CCC-T-191 b, Method 5050.1.

2. EQUIPMENT AND MATERIALS

2.1 Equipment. Thread counter apparatus with magnifier

3. SAMPLING

3.1 Sample size. Sufficient sample shall be obtained so as to permit specified number of determinations to be made in each the warp and fill directions.

3.2 Determinations. Unless otherwise specified, five determinations will be made in each the warp and fill directions.

4. PROCEDURE

4.1 Preparation of sample. The cloth shall be laid out smoothly and without tension on a horizontal surface.

4.2 Determination of thread count.

4.2.1 Area of determination. The actual count shall be determined from representative sections of the cloth along the warp and fill directions. No count shall be made nearer than 3 inches to the selvage edge, or either end, and no two determinations should include the same threads.

4.2.2 Inches for each count. If there are more than 25 threads per inch, count one inch for each determination; if there are fewer than 25 threads per inch, count three inches per each determination made.

5. CALCULATIONS AND REPORTING

5.1 Average the specified number of determinations for each warp and fill direction and report these values, rounded off to the nearest whole number.

M-C-27 Fabric Weight Using Small Specimens of Unconditioned, Conditioned, and Devolatilized Methods

1. SCOPE

1.1 This method describes a procedure for determination of fabric weight using small specimens. Unconditioned, conditioned, and devolatilized methods are included.

1.2 The procedure is equivalent to CCC-T-191, Method 5041.

2. EQUIPMENT

2.1 Scissors

2.2 Analytical balance, sensitivity 0.0001 gm.

2.3 Steel template, 6.76 inches square

2.4 Stanley knife or equivalent

2.5 Desiccator with conditioning chemicals giving a relative humidity of 65 ± 2%

2.6 Oven capable of maintaining $70^{\circ} \pm 3^{\circ}F$.

2.7 Desiccator with silica gel or equivalent

2.8 Timer

2.9 Oven, forced air

3. SAMPLE SIZE

3.1 One linear yard shall be taken for test.

4. PROCEDURE

4.1 From the sample, cut at random five (5) specimens each 6.76 inches square. No specimen shall be closer than three inches to the selvage. Weigh individual specimens to 0.01 gm.

4.2 If the conditioned method is used, precondition specimens for 24 ± 2 hours at 65 ± 2% relative humidity and $70^{\circ} \pm 3^{\circ}$ F. Weigh individual specimens to 0.01 gm.

4.3 If devolatilized method is used, omit paragraph 4.2 and suspend specimens in a forced air oven at $320^{\circ} \pm 5^{\circ}F$. for 15 ± 0.25 minutes. Weigh individual specimens to 0.01 gm.

5. CALCULATIONS AND REPORTING

5.1 The individual specimen weight in grams is equivalent to its weight in ounces per square yard.

5.2 To calculate unconditioned fabric weight (as received), obtain the average of the five values of paragraph 4.1. Designate as Unconditioned Fabric Weight, W_1. The range of the five fabric weights should not exceed 3.0%.

5.3 To calculate conditioned fabric weight, obtain the average of the five values of paragraph 4.2. Designate as Conditioned Fabric Weight, W_2. The range of the five fabric weights should not exceed 2.0%.

5.4 To calculate devolatilized fabric weight, obtain the average of the five values of paragraph 4.3. Designate as Devolatilized Fabric Weight, W_3. The range of the five fabric weights should not exceed 2.0%.

5.5 To calculate volatile content, use the following formula:

$$\%\ \text{Volatile Content} = \frac{W_1 - W_3 \times 100}{W_1}$$

M-C-28 Apparent Volume and Density of Raw Fabric, Prepregs, and Cured Material

1. SCOPE

1.1 This procedure describes a method for determinations of apparent volume of raw fabrics, prepregs, cured parts and other related materials by use of an air comparison pycnometer. After apparent volume has been measured, apparent density can be calculated. This method is suitable for the analysis of non-surface active, non-compressible materials.

NOTE: Instrument limitations. Materials such as carbon fabric will not yield reproducible values, however graphite fabric will yield reasonably accurate results even though it is surface active.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

2.1.1 Pycnometer, Model 930, Beckman, or equivalent and operators manual.

2.1.2 Oven, vacuum

2.1.3 Balance, analytical, minimum sensitivity 0.001 gm.

2.1.4 Gas bottle with regulator

NOTE: Usable gases. Any inert gas may be used. Unless otherwise specified use helium.

2.1.5 Vacuum source

2.1.6 Laboratory knife

3. SAMPLING

3.1 Sample Size. Obtain a representative size sample of approximately five (5) grams for bulky materials (raw fabrics should be about 1" by 36"). For solid materials (cured parts), a sample size up to 50 c.c. can be measured. A greater degree of accuracy can be achieved by using larger sample sizes, but the sample size will be dependent upon the ability of the sample to fit into the sample cup (maximum volume 50 c.c.).

3.2 Number of determinations. Unless otherwise specified, one determination will suffice but, multiple measurements must be made, see paragraph 4.3.1.

4. PROCEDURE

4.1 Sample conditioning. Unless otherwise specified, condition sample by inserting in a vacuum oven heated to $275^{\circ} \pm 5^{\circ}F$. After exposure under full vacuum for 20 to 30 minutes, remove sample from oven and immediately weigh to nearest 0.001 gm.

4.2 Instrument calibration.

4.2.1 Initial warm-up. At the start of a series of measurements the instrument should be loosened and warmed-up. This is done by turning both the measuring and reference handwheels in and out several times with the purge and coupling valves open.

4.2.2 Volume check. Accuracy of the unit should be checked for volume by inserting either the 1" or 1-1/2" steel test ball in the sample cup, and measuring volume of the ball per paragraph 4.3. The volume readings obtained from the test balls should be a consistent 8.58 ml. for the 1" steel ball and 28.96 ml. for the 1-1/2" steel ball. If the ball measurement exceeds a volume tolerance of ± 0.15 ml. for either ball, refer to the Instruction Manual for adjustment instructions.

NOTE: Ball checks. When checking several samples during one series of tests, periodic checks of the ball should be made to make sure the instrument is still reading accurately. This is best done by checking the ball in between every third or fourth sample that is being tested.

4.2.3 Zero check. The zero check procedure is identical to paragraph 4.3, but is made with the sample cup clean and empty. If the final counter reading is other than zero, refer to the Instruction Manual for adjustment instructions.

4.3 Specimen volume measurements. Unless otherwise designated, the following procedure shall be utilized in determination of apparent volume:

4.3.1 Adjust gas pressure regulator on the helium tank to a pressure no greater than 2 psi. Turn vacuum supply on.

4.3.2 With purge and coupling valves open, rotate both handwheels in a counter-clockwise direction to the extreme position. Turn the measuring wheel until starting number is reached (located on a plate affixed to the side of the case above the measuring handwheel).

4.3.3 Insert previously conditioned and weighed specimen into the sample cup and insert sample cup into position and secure it firmly.

4.3.4 Open purge and coupling valves.

4.3.5 Open vacuum valve and allow 10 seconds for system to evacuate, then close valve.

4.3.6 Open gas (helium) valve and allow 5 seconds for pressure to equilibrate, then close gas valve.

4.3.7 Open vent valve for 5 seconds for pressure equilibrium, then close vent and purge valves.

4.3.8 Wait for 10 seconds then loosen the coupling valve (this should be rotated a couple of times)

4.3.9 Wait for 10 seconds then, turn both handwheels clockwise simultaneously until the reference wheel stops. Apply minimum amount of pressure after the wheel initially stops. Keep the pointer on the scale during this process.

4.3.10 Wait for 10 seconds and adjust pointer by image alignment to the zero mark with the measuring handwheel (See Instruction Manual).

4.3.11 Open coupling valve. Read specimen volume on counter directly in ml.

4.3.12 Open purge valve and vent valve, then close vent valve and repeat steps 4.3.2 through 4.3.11 until consistent readings are obtained on each specimen.

NOTE: When opening coupling valve, observe null (differential pressure) indicator. If it shifts position, true null was not obtained and the run should be repeated.

5. CALCULATIONS AND REPORTING

5.1 Calculations. To calculate apparent density from apparent volume use the following formula:

NOTE: Specific gravity. Density and specific gravity may be considered identical for all practical purposes in the measurements of materials whose density does not vary appreciably with temperature.

$$\text{Density} = \frac{\text{Weight of dried specimen, gms.}}{\text{Volume reading obtained, c.c.}} = \text{gm./cc.}$$

5.2 Reporting. Report specific gravity to four significant figures. In addition to reporting specific gravity, report specimen weight and apparent volume.

$$\text{Specific Gravity} = \frac{\text{Density}}{\text{Density of } H_2O} = \frac{\text{Density}}{0.9975} = \text{Sp.G.}$$

M-C-29 Raw Carbon Fabric Inspection Into Both Minor and Major Defect Categories

1. SCOPE

1.1 Raw Carbon Fabric Inspection

2. MATERIAL

2.1 Run material as follows or as instructed by Quality Control and Production management.

2.2 CSA(1105,1183) Fill side up VCK-VCL(1073,1113) Warp side up

3. DEFECTS

Defects are classified as either minor or major. Large minor defects, i.e. those exceeding the allowables shall be considered major.

3.1 Table of Minor Defects and Allowables

	Minor Defects	Allowables
3.1.1	Oil free finger prints causing localized weave distortion	3 per linear yard
3.1.2	Frayed fill bands across the width, e.g. rack bands	6" long by width of roll
3.1.3	Puckers	≤ 9 square in.area
3.1.4	Mispicks, floats, skips, end outs, missing picks	4 per linear yd.
3.1.5	Kinks	
3.1.5.1	Clustered- 24 or less per 6" x 6" area	2 clusters/linear yd.
3.1.5.2	Continuous - not closer than 3 inches	42 or less/linear yd. (2% of end count)
3.1.6	Bagginess	
3.1.6.1	One edge only (20 yds. maximum)	Loss of tension ≤ 6" in. from the edge
3.1.6.2	Both edges (20 yds. maximum)	Loss of tension ≤ 4" in. from the edge

3.2 Table of Major Defects and Allowables

	Major Defect	Min. Defect/yd.
3.2.1	Any minor in excess of the allowables	0.1
3.2.2	Spot - any noticeable caused by oil, dirt or foreign material	0.1
3.2.3	Folds or wrinkles - any occurance greater than 6 inches or less than 2 inches apart	0.5
3.2.4	Holes- any occurance greater than 1/4 inch dia.	0.1
3.2.5	Splice - bad splice(not flat) good splice	0.5 0.2
3.2.6	Cut or tear- any occurance which cannot be considered a hole	0.5
3.2.7	Change in warp and fill side at splice	Reject Roll

4. INSPECTION

4.1 Inspect and fill out an inspection sheet recording only those defects greater than the allowables.

4.2 Defective yardage equals the amount of affected yardage but not less than minimum of 0.1 or 0.5 as above.

4.3 Material with 3.0% or less defective material shall be approved. Material with 3.0% or more defective material shall be rejected.

5. CALCULATIONS

$$\text{Percent Defective} = \frac{\text{Total number of def. yds}}{\text{Total ydg.}} \times (100\%)$$

M-C-30 Broadgoods Visual Inspection and Criteria For Both Minor and Major Defects

1. WRAPPING REQUIREMENTS

1.1 3" diameter core 2-6" longer than material width.

1.2 Fabric shall be taped to the full width of the core.

1.3 Use a minimum of 1 unit desiccant bags per Mil-D-3464 and 6 mil *poly bags and tape excess at material edges to core to create a good seal.

1.4 Wrap material such that the material will unwrap per below.

1.5 Width - 36-48 inches

1.6 Roll size - No minimum, 14" diameter maximum

2. Defects are classified as either minor or major. Large minor defects, in those exceeding the allowable shall be considered major.

2.1	Minor defects (do not remove)	Allowable
2.1.1	Oil free finger prints causing localized wave distortion	3 per linear yard
2.1.2	Frayed fill bands across	6" long by width of roll
2.1.3	Puckers	≤ 9 square inch area
2.1.4	Mispicks, floats, skips, end outs, missing picks	4 per linear yard
2.1.5	Kinks	
2.1.5.1	Clustered - 24 or per 6" x 6" area	2 clusters per linear yard (2% of end count)
2.1.5.2	Continuous - Not closer than 3"	42 or less per linear yard (2% of end count)
2.1.6	Abnormal slot or spring marks less than 2" wide	2 per linear yard
2.1.7	Contamination that can be brushed away	

2.2 Major defects (record but do not remove)

		Min. Allowances Per Yard
2.2.1	Holes less than 1" diameter	0.1
2.2.2	Tears or cracks less than 4" long	0.1
2.2.3	Wrinkle foldovers less than 4" long	0.1
2.2.4	Heavy resin spots up to 1" diameter	0.1

2.3 Major defects (remove and splice roll)

2.3.1 Spots caused by oil, grease, or foreign contamination. Spots caused by treater fuzz are acceptable, however, record occurances and report to your supervisor.

2.3.2 Treater folds and wrinkle foldovers can not be removed.

2.3.3 Contamination, that which can not be removed.

2.3.4 Holes larger than 1" diameter.

2.3.5 Weak or brittle areas.

2.3.6 Heavy resin spots in excess of 1" diameter.

2.3.7 Tears or cracks greater than 4" long.

2.3.8 Selvage (trim and remove)

2.4 Splicing

2.4.1 Open splice (do not bond together with tape).

2.4.2 Splice must allow the material to roll up smoothly.

2.4.3 Splices can be no less than 5 yards apart nor closer than 5 yards from the end of the roll.

2.4.4 Flag all splices and record on inspection sheet.

3. INSPECTION

3.1 Inspect and fill out an inspection sheet recording only those major defects greater that the allowable.

3.2 Defective yardage equals the amount of affected yardage but not less than minimum of 0.1 linear yard.

3.3 Material with 5.0% or less defective material shall be approved. Material with 5.0% or more defective material shall be rejected.

4. CALCULATIONS

$$\% \text{ Defective} = \frac{\text{Total No. of defective yards}}{\text{Total yardage}} \times 100$$

M-F-1 Ash Content of Fillers, Pigments and Related Materials

1. SCOPE

1.1 Scope. This method describes a procedure for the determination of ash content of ignitable fillers, pigments and related materials which will ash at 1750°F.

1.2 Equivalent procedures. This procedure is equivalent to ASTM-D1506-59.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

2.1.1 Muffle furnace

2.1.2 Oven, forced air

2.1.3 Analytical balance, minimum sensitivity, 0.1 mg.

2.1.4 Crucibles, porcelain, wide form with covers

3. SAMPLING

3.1 Sample size. Obtain a representative sample of at least 5 gm.

3.2 Number of determinations. Unless otherwise specified, run duplicate determinations.

4. PROCEDURE

4.1 Drying sample. Condition about 5 gm. of sample in a porcelain crucible in a forced air oven for 60 ± 5 minutes at 225 ± 5°F.

4.2 Weighing. Weigh about 2 gm. to the nearest 0.0001 gram into a prefired and weighed porcelain crucible.

4.3 Ignition. Place sample into muffle furnace at 1750 ± 50° F. Ignite for about 4 hours. A longer time period may be necessary. Keep the furnace door open about 1/4 inch to admit air. Check for completeness of ashing by cooling the sample in a desiccator, weighing, reigniting for 1 hour, then cooling in a desiccator and re-weighing, as often as necessary, to contant weight.

4.4 Final weight. When ashing is determined to be complete, cool the sample in a desiccator, then reweigh crucible and residue to 0.0001 gm.

5. CALCULATIONS AND REPORTING

5.1 Calculations. Calculate ash content as follows:

$$\text{Ash Content, Wt. \%} = \frac{W_3-W_1}{W_2-W_1} \times 100$$

where:

W_1 = weight of prefired crucible, gms.
W_2 = weight of crucible plus sample, gms.
W_3 = weight of crucible plus residue, gms.

5.2 Reporting. Unless otherwise specified, round off and report ash content to 0.01%. Duplicate determinations should be considered suspect if they differ by more than 0.1% ash content.

M-F-2 Particle Size and Distribution Using a Sub-Siever Sizer

1. SCOPE

1.1 This procedure describes the method for determining the particle size of a powdered material using the Fisher Sub-Siever Sizer.

2. EQUIPMENT AND MATERIALS

2.1 Fisher Sub-Siever Sizer, and operators manual

2.2 Brass Sample Tube 14-313-5

2.3 Brass Porous Plugs

2.4 BX Filter Paper Discs 3/4" Dia.

2.5 Small Powder Funnel

2.6 Sample Thief

3. SAMPLING

3.1 A representative sample (approximately 1.0 ounce) is required.

3.2 When drums or other large containers are to be sampled, the use of a sample thief is required to insure a representative sampling.

3.3 An average of at least three (3) determinations are required.

4. OPERATING PROCEDURE (See Operators Manual)

4.1 Before using the Sub-Sieve Sizer, make certain that the drying tube is filled with the indicating drying agent, 10-20 mesh Drierite. The Drierite has been treated with a substance that indicates the state of dryness of the agent, the condition of which can be determined by observing the drying tube through the small window in the left side of the cabinet. A blue color indicates that the agent is satisfactory. A pink color indicates that the agent must be replaced with fresh stock.

4.2 The Sample Packing Assembly, consisting of the rack and pinion, (1) compact the powder sample after the sample has been weighed into the sample tube, and (2) also measure the actual height of the packed powder sample. Before these can be done, however, the assembly must be adjusted to compensate for the thickness of the two porous plugs and filter papers which serve to confine the sample in the sample tube.

4.3 The sample packing assembly can be adjusted by the following:

4.3.1 Set two porous plugs with two filter papers between them on the brass post (J), making certain that the lower plug touches the upper end of the post.

4.3.2 Using the Rack and Pinion Control (E), guide the rack down onto the top porous plug. The tip of the pointer should now coincide exactly with the baseline on the Calculator Chart. If not, loosen the locknut that holds the lower brass post in its mount and adjust the height of the post by screwing it up or down.

4.3.3 When the tip of the pointer coincides exactly with the baseline on the Calculator Chart, the initial level of the liquid meniscus in the manometer must be made to exactly coincide with the upper edge of the curved portion of the metal cross bar attached to the rack. Use the Manometer Level Control but only when the sample tube is not attached to the instrument. Once the adjustment has been made for a particular determination, the Manometer Level Control, must not be turned until that determination has been completed. Check the manometer level before each successive determination and correct as necessary. All water level measurements are taken with reference to the upper edge of the curved portion of the cross bar, and all chart readings with reference to the extreme tip of the pointer mounted on this same cross bar.

4.4 Porosity Scale

4.4.1 The porosity of a packed sample, or the ratio of the air space in the sample bed to the total volume occupied by the packed sample, is read directly from the Porosity Scale located along the bottom edge of the Calculator Chart. A given sample may be packed to many different porosities. Therefore, an Optimum Porosity Point, or the porosity setting at which a particular powder can be measured on the Sub-Sieve Sizer with the minimum possibility of error, must be found. For micropulverized dicyandiamide (dicy) this Optimum Porosity Point has been determined to be 0.625.

4.5 Measuring Particle Size

4.5.1 Plug the line cord into an applicable receptacle, flip the power switch (L) to ON, and allow the instrument to warm-up for 20 minutes to give it a chance to stabilize. While the unit is warming up, perform steps 4.5.2 through 4.5.8.

4.5.2 Lay a paper disc over one end of the sample tube. Then, with the perforated surface of the plug against the surface of the paper disc, push one of the two porous plugs about one-half inch into the tube, forcing the paper to crimp around the edges.

4.5.3 Place the tube vertically in the rubber support with the paper side of the plug up.

4.5.4 Weight (to within 0.01 gm.) a sample of dry powder equal in grams to the true density of the sample. (For each new lot of dicy, its density must be calculated with the use of the Helium Pycnometer.)

4.5.5 Using a powder funnel, completely transfer the weighed sample into the sample tube. Tap the side of the tube to settle the powder.

4.5.6 Lay a second paper disc over the top of the sample tube, and force the other porous plug and paper disc down into the tube.

4.5.7 Place the sample tube on the brass post (J) with the lower plug touching the upper end of the post.

4.5.8 Using the Rack and Pinion Control (E), lower the rack until the flat bottom end touches the upper plug.

4.5.9 Move the Calculator Chart so that the pointer is set at the Optimum Porosity Point * for the sample to be tested. (For Dicy this is 0.625).

4.5.10 Using the Rack and Pinion Control, lower the rack until the tip of the pointer on the rack is set on the Sample Height Curve on the chart.

4.5.11 Without moving the chart, raise the rack and remove the sample tube, being careful not to disturb the sample.

4.5.12 Mount the sample tube between the rubber cushioned supports just to the right of the brass post (J) and twist the Clamp Assembly Control (H) to the right until the tube is locked into place, making an air-tight seal at both ends of the tube.

4.5.13 Adjust the Pressure Control (G) until bubbles rise in the Pressure Regualtor Standpipe at the rate of two to three per second. The liquid level in the manometer tube will rise slowly and reach a maximum height within 30 seconds to several minutes, depending upon particle size.

4.5.14 Once the liquid in the manometer reaches its maximum level, and without disturbing the position of the Calculator Chart, turn up the rack until the upper edge of the cross bar coincides with the liquid meniscus in the manometer.

4.5.15 Read the particle size off the chart and record the reading.

4.5.16 Repeat steps 4.5.2 - 4.5.15 two or more times until three (3) reproducible readings are obtained, and report the average particle size value.

4.5.17 In order to determine the Optimum Porosity Point for a given sample, perform steps 4.5.9 - 4.5.15 and repeat, each time moving the Calculator Chart to the left 0.05 Porosity Units, beginning with 0.80. Discontinue the analysis when the chart runs off scale at 0.40 or when the sample is packed as tight as possible.

4.5.18 Plot all readings on a sheet of graph paper and connect the points. The mid-point of the plateau of the curve is the optimum porosity point.

M-F-3 Percent Inorganic Filler Reinforcement In a Resin

1. SCOPE

1.1 This method is used to determine the percentage of inorganic filler used as reinforcement in a resin.

2. EQUIPMENT

2.1 The apparatus shall consist of the following:

2.2 Analytical balance capable of weighing to the nearest milligram (0.001 gm.).

2.3 Centrifuge (International Model V-2) equipped with suitable head, trunions and shields to handle 50 ml. centrifuge tubes. Capable of 3,000 RPM.

2.4 Drying oven thermostatically controlled and capable of maintaining $135^{\circ} \pm 1.0^{\circ}C$.

2.5 Centrifuge tubes 50 ml. capacity.

2.6 Aluminum weighing dish approximately 57 mm dia. x 11 mm. high.

3. SAMPLES

3.1 Resin solid samples shall consist of three 2.8-3.2 gm. samples.

3.2 Filler samples shall consist of three 7-10 gm. samples of resin taken from the same container as the resin solids sample.

3.3 It is important that samples be at room temperature and be thoroughly stirred before weighing out resin solids and filler samples to prevent settling of the filler material.

4. PROCEDURE

4.1 Determine the resin solids of the sample by weighing three 2.8-3.2 gm. samples of thoroughly stirred resin into three separate tared aluminum dishes. Weigh each sample to the nearest milligram and designate this weight as W_1.

4.2 Place the samples in the aluminum dishes in the thermostatically controlled drying oven which is at $135^{\circ} \pm 1.0^{\circ}C$ for a period of 3 hours ± 5 min.

4.3 After the samples have been in the oven for three hours, remove them from the oven and place them in a desiccator to cool before weighing. After the samples have cooled, weigh the samples and designate the weight as W_3.

5. FILLER CONTENT

5.1 Determine the filler content of the resin by weighing three 7-10 gm. samples of thoroughly stirred resin into three separately tared 50 ml. centrifuge tubes. Weigh the samples to the nearest milligram. Designate this weight as W_3.

5.2 Dilute the resin in the centrifuge tubes with 35 ml. of acetone. Stir the contents of the centrifuge tube with a glass stirring rod and wash the rod clean of all resin with a minimum of acetone from a wash bottle. Place the samples in the centrifuge and counterbalance with identical centrifuge tubes filled with acetone to the same level as the resin samples. (It is important to use the same solvent as in the samples due to weight loss of the acetone by evaporation in the centrifuge.)

5.3 Centrifuge the samples at 3,000 RPM for 20 minutes. Remove the samples. Pour off the supernatant liquid making certain no filler is removed. Fill the sample tubes with 35 ml. of acetone and stir with a glass stirring rod. Wash the stirring rod clean with a minimum amount of acetone from a wash bottle. Return the samples to the centrifuge and spin at 3,000 RPM for 20 min. a second time. Pour off the supernatant liquid and repeat the procedure a third time. After the third spinning, pour off the supernatant liquid and wash down the outside of the centrifuge tubes to remove any resin which may have spilled over the side of the tubes. Place the tubes in the drying oven at $135^\circ \pm 1.0^\circ$C. for three hours. Remove the tubes at the end of this period and place them in a desiccator to cool. When the specimens have cooled, re-weigh to the nearest milligram. Designate the weight as W_4.

6. CALCULATIONS

6.1 Calculate the resin solids as follows:

$$W_2/W_1 = \text{Resin Solids} = A$$

$$\text{Average} = (A_1+A_2+A_3)/3 = A_{\text{Avg.}}$$

6.2 Calculate the percent filler as follows:

$W_4/A_{\text{avg.}} \times 100$ = % filler in dry resin

Where:

W_1 = Original weight of resin, filler and solvent, gms.
W_2 = dry weight of resin and filler, gms.
W_3 = original weight of resin filler and solvent in centrifuge tube, gms.
W_4 = weight of filler remaining in centrifuge tube, gms.

M-F-4 Filler Content in Filled Resin Systems

1. SCOPE

1.1 This method is used for the determination of filler content in filled resin systems.

2. EQUIPMENT

2.1 Analytical balance, sensitivity 0.0001 gm.

2.2 50 ml. tri-pour beakers

2.3 Disposable syringes (3cc.)

2.4 Membrane filtration funnel

2.5 Whatman 4.25 cm. GF/F filters

2.6 Whatman 4.7 cm. EPM 2000 filters

2.7 Aluminum weighing pans

2.8 Vacuum system

2.9 Air circulating oven $163^\circ \pm 3^\circ$C.

2.10 Desiccator

2.11 Stirring rod with rubber policeman

2.12 Teflon gasket 47 mm.

3. SAMPLING

3.1 It is important that samples obtained from resin mixes are thoroughly mixed. Lab sub-samples also must be thoroughly mixed and at room temperature prior to use. These precautions must be taken to prevent settling of the filler material. This analysis is to be performed in triplicate. Mix samples with air driven stirrer for 15 minutes minimum.

4. PROCEDURE

4.1 Determine the total solids content of the material.

4.2 Precondition several pairs of filters (one Whatman EPM 2000 on the bottom and one Whatman GF/F on the top) by filtering approximately 25 ml. of acetone through the pair of filters. Place the filters in an aluminum weigh pan and dry at $163^\circ \pm 3^\circ$C. for 20 minutes or until dry. Preconditioned filters are to be stored in a desiccator.

4.3 Weigh a preconditioned pair of filters and pan (from 4.2) to the nearest 0.0001 gram and place the pair of filters, with the smaller filter on top, in the membrane filtration apparatus (W_2). A teflon gasket is to be placed on top of the stainless steel screen prior to placing the filters in the apparatus.

4.4 Transfer 2-3 grams of resin to a 50 ml. tri-pour beaker using a disposable syringe. Record this weight to the nearest 0.0001 gram and designate as W_1.

4.5 Add 35-45 ml. of acetone to the 50 ml. beaker and stir thoroughly. Transfer the contents to the assembled filtering funnel (4.3). Repeat twice, adding all rinses to the filtering funnel.

4.6 Filter the contents of the filtering funnel through the pair of filters. Stir the solution frequently to avoid settling of the filler onto the shoulder of the filtering funnel. Rinse the sides of the filtering funnel with acetone to insure that all the filler is entrapped in the filters.

4.7 Remove the filters from the filtering funnel and place back into the original weighing pan (the top filter should be placed into the pan first and covered by the larger, bottom filter). Transfer any filler remaining on the bottom rim of the filtering funnel to the weighing pan with a small metal spatula. Close loosely around the edges and dry. Cool to room temperature in desiccator and reweigh to the nearest 0.0001 gm. (W_3).

5. CALCULATIONS

$$W_1 \times \frac{\text{Avg. \% Total Solids}}{100} = W_5$$

$$\frac{W_4}{W_5} \times 100 = \text{\% Filler}$$

Where:

W_1 = Original weight of resin, filler, solvent, gm.
W_4 = Dry filler weight ($W_3 - W_2$), gm.
W_5 = Total solids weight of initial sample, gm.

M-F-5 Particle Size by the Horiba Centrifugal Automatic Analyzer

1. SCOPE

1.1 This procedure describes the method for determining particle size of powders and liquids using the Horiba CAPA-500 Centrifugal Automatic Partical Analyzer.

<u>Note:</u> This particle size method is in no way an alternative to the manual method. The manual method should be used in conjunction with this method.

2. EQUIPMENT AND MATERIALS

2.1 Horiba CAPA-500 Centrifugal Automatic Particle Analyzer and Operators Manual.

2.2 Cuvettes, pyrex with teflon lids, 14 (W) x 14 (D) x 45 (H) mm.

2.3 Glass Vials, 25 ml. with caps

2.4 Pipettes, disposable, 10 ml.

2.5 Pasteur Pipettes

2.6 Pipette Bulb

2.7 Ultrasound Bath

2.8 Beakers, 500 ml.

2.9 Spatula

2.10 Analytical Balance, ± 0.0001 gms. sensitivity

2.11 Kimwipes

2.12 Ethylene Glycol, 99%+ purity

2.13 Glass Microspheres, 1 - 5 um.

2.14 Glass Microspheres, 34.8 um.

2.15 Glass Microspheres, 110.6 um.

2.16 Acetone, industrial

2.17 Vacuum

3. PREPARATION OF STANDARDS AND SOLUTIONS

3.1 Standards

3.1.1 Depending on the particle size range desired, there are three glass microspheres standards available:

3.1.1.1 1 - 5 um.

3.1.1.2 34.8 um.

3.1.1.3 110.6 um.

3.1.2 Weigh 10.0 mg. of standard into a 25 ml. glass vial. Add 20 ml. of ethylene glycol. Mix well by shaking, and place vial in ultrasound bath for thirty minutes.

3.2 Samples

3.2.1 Depending on the samples to be run, each method for sample preparation will be different, namely the solvent used and the concentration of the sample desired; therefore, various addendums have been added at the end of the method to take care of this situation.

3.2.2 For carbon black powders and resins, please refer to Addendum 1.

3.2.3 For E-03 and E-24 finishes, please refer to Addendum 2.

4. PROCEDURE

4.1 Blank

4.1.1 The blank must be run first, before running any standards or samples.

4.1.2 Use the same solvent used for the samples to run the blank; e.g. ethylene glycol.

4.1.3 Fill both cuvettes with solvent. The liquid level in the cuvette should be between the two engraved lines. Cap the cuvette, and make sure there are no air bubbles.

4.1.4 Program the following parameters into the Horiba analyzer:

4.1.4.1 Solvent viscosity

4.1.4.2 Solvent density

4.1.4.3 Sample density

4.1.4.4 Diameter (max.)

4.1.4.5 Diameter (min.)

4.1.4.6 Diameter (div.)

4.1.4.7 Centrifugal/Sedimentation Mode

4.1.4.8 Speed if centrifugal mode is selected

4.1.5 Depending on the samples to be run, each method for particle size analysis will have different values for the parameters described in Section 4.1.3.

4.1.6 For carbon black powders and resin, please refer to Addendum 1.

4.1.7 For E-02 and E-24 finishes, please refer to Addendum 2.

4.1.8 Place one cuvette in reference cell holder, and turn the cell holding bar until the cuvette is fixed.

4.1.9 Place the other cuvette in sample cell holder, and turn the cell holding bar until the cuvette is fixed.

4.1.10 Close the sample chamber and press the "BLANK" button on the control panel. Measurement is made for thirty-two seconds, and the mean value is stored.

4.1.11 Samples are now ready to be run.

4.2 Standards

4.2.1 Fill cuvette with standard, as prepared in section 3.1. The liquid level in the cell should be between the two engraved lines. Cap the cuvette carefully, making sure no air bubbles are introduced.

4.2.2 Place cuvette in sample cell holder, and turn the cell holding bar until the cuvette is fixed.

4.2.3 The cuvette in the reference cell holder should contain only ethylene glycol. Secure the reference cell with the cell holding bar.

4.2.4 The Horiba Analyzer should be programmed in the following manner:

4.2.4.1 Solvent viscosity is 19.90 cps

4.2.4.2 Solvent density is 1.11 gm./cc

4.2.4.3 Sample density is 2.30 gm./cc

4.2.4.4 Diameter (max.) is 10.0 microns

4.2.4.5 Diameter (min.) is 0.01 microns

4.2.4.6 Diameter (div.) is 1.0 microns

4.2.4.7 Speed is 2,000 rpm

4.2.4.8 Sample concentration is 0.5 mg/ml

4.2.4.9 Running time is 12 minutes, 1 second

The above parameters are for glass microspheres standard, 1 - 5 minutes.

4.2.5 Close the sample chamber. Press the "SET" button. The running time should appear, in this case, 12 minutes, 1 second.

4.2.6 Press the "START" button. Sample is now running. After run stops, data will start printing out.

4.2.7 Make sure that the blank is run before running the standard, see Section 4.1.

4.2.8 Each standard must be run twice.

4.3 Samples

4.3.1 Fill cuvette with sample. The liquid in the cell should be between the two engraved lines. Cap the cuvette carefully making sure no air bubbles are introduced.

4.3.2 Place cuvette in sample cell holder, and secure the cell by turning the cell holder bar.

4.3.3 The cuvette in the reference cell holder should contain only solvent, the same solvent used for the sample. Secure the cell by turning the cell holding bar.

4.3.4 The Horiba Analyzer should be programmed as follows:

4.3.4.1 For carbon black powders and resins, refer to Addendum 1.

4.3.4.2 For E-02 and E-24 finishes, refer to Addendum 2.

4.3.5 Close the sample chamber. Press the "SET" button. The running time should appear.

4.3.6 Press the "START" button. Sample is now running. After run stops, data will start printing out.

4.3.7 Make sure that the blank is run before running any samples. See Section 4.1.

4.3.8 Each sample must be run twice.

5. RESULTS

5.1 It is crucial that the absorbance, shown in the absorbance vs. time graph, does not exceed 1.0.

5.1 The average diameter, D(avg.), is based on 50% of the cumulative distribution.

5.1 The average diameter must not vary by more than 10% from sample to sample.

5.1 The average diameter for the glass microspheres standard, 1-5 microns, should be about 4.3 ± 0.1 microns. The value should be entered in the particle size calibration log for future reference.

PARTICLE SIZE

ADDENDUM 1

CARBON BLACK POWDERS AND RESINS SAMPLE PREPARATION

1. SAMPLE PREPARATION

1.1 Weigh 2 mg. of sample in a 25 ml. vial. Add 20 ml. of ethylene glycol; concentration is 0.1 mg./ml. Mix well in ultrasound bath for thirty minutes.

1.2 Take 2 ml. of 0.1 mg./ml. solution and add 20 ml. of ethylene glycol in a 25 ml. vial; concentration is 0.01 mg./ml. Mix well in ultrasound bath for thirty minutes.

2. HORIBA ANALYZER DATA

2.1 Solvent is ethylene glycol

2.2 Solvent viscosity is 19.90 cps.

2.3 Solvent density is 1.11 gm./cc.

2.4 Sample density is 1.90 gm./cc.

2.5 Diameter (max.) is 5.0 microns

2.6 Diameter (min.) is 0.01 microns

2.7 Diameter (div.) is 0.50 microns

2.8 Speed is 4,000 rpm

2.9 Sample concentration is 0.01 mg./ml.

2.10 Running time is 11 minutes, 31 seconds

PARTICLE SIZE

ADDENDUM 2

E-03 AND E-24 FINISHES

1. SAMPLE PREPARATION

1.1 Weigh 120 mg. of sample in a 25 ml. vial if the percent solids is 20%. Add 20 ml. of deionized water; concentration is 6.0 mg./ml. Mix well in ultrasound bath for thirty minutes.

1.2 Weigh 60 mg. of sample in a 25 ml. vial if the percent solids is 40%. Add 20 ml. of deionized water; concentration is 3.0 mg./ml. Mix well in ultrasound batch for thirty minutes

2. HORIBA ANALYZER DATA

2.1 Solvent is deionized water

2.2 Solvent viscosity is 0.94 cps.

2.3 Solvent density is 0.99 gm./cc.

2.4 Sample density is 1.25 gm./cc.

2.5 Diameter (max.) is 2.0 microns

2.6 Diameter (min.) is 0.01 microns

2.7 Diameter (div.) is 0.25 microns

2.8 Speed is 5,000 rpm

2.9 Sample concentration is 6.0 mg./ml. (20% solids), or 3.0 mg./ml. (40% solids)

2.10 Running time is 6 minutes, 34 seconds

M-F-6 Carbon Assay and Moisture Content of Carbon Filler

1. SCOPE

1.1 Carbon assay and moisture content. The carbon assay and moisture content determination shall be in accordance with the following:

2. PROCEDURE

2.1 Dry a 300 milligram specimen to a constant weight at 225 + 5 °F. before combustion and base the percent carbon (to the nearest 0.1 percent) on the weight of the dried sample.

2.2 Conduct the carbon assay in accordance with ASTM D 3176 or by the alternative method specified.

2.3 Calculate the moisture content in accordance with the following:

$$\text{Percent moisture} = \frac{W - W_1}{W} \quad (100)$$

Where:

W = specimen weight prior to drying , gram (gm.)

W_1 = specimen weight after drying, gm.

2.4 Report the results to the nearest 0.10 percent.

2.5 Alternative carbon assay determination. The alternative carbon assay determination shall be accomplished using a LECO CHN-600 Carbon, Hydrogen, Nitrogen Analyzer, or equivalent, have an operators manual available, as follows:

2.5.1 Insert the dried sample into the top of a tared tin capsule.

2.5.2 Crimp the top of the capsule with forceps to prevent escape of the sample material.

2.5.3 Place the capsule, with sample back on the LB-20 balance to determine net sample weight. When the weight stabilizes, push the ENTER key.

2.5.4 Place capsule in the analyzer sample chamber.

2.5.5 Push the analyzer key to begin analysis.

2.5.6 When the analysis is complete, the results will be displayed and a print-out will occur.

2.5.7 Report percent carbon test results to the nearest 0.10 percent

M-F-7 Particle Size Test for Fillers by SEM

1. SAMPLING

1.1 Representative samples are to be obtained by selecting a number of containers and sampling each container with a thief. Mixing and selecting the appropriate size sample and storing in air tight containers until ready for analysis.

2. TEST METHODS

2.1 Scanning Electron Microscope (SEM) Method was applied to a visual sampling and 20,000x magnification. The minimum and maximum particle size is determined, and a representative group of particles spanning the full range is selected. From this selected group each particle is measured and an arithmetic average size is calculated.

NOTE: The two methods, SEM and the HORIBA method do not give equivalent results, and hence should be used in conjunction with the manual method.

P-P-1 Volatile Content of Uncured Prepregs

1. SCOPE

1.1 The volatile content of each sample of the uncured material shall be determined in accordance with the following:

2. PROCEDURE

2.1 Cut a 16 ± 2 square inch specimen with a minimum width of 1.5 inches across the width of the prepreg fabric.

2.2 Weigh the specimen to the nearest 0.01 gram (W_1).

2.3 Place specimen in a recirculating oven preheated and stabilized to 325 ± 10° F. for 10 ± 1 minutes. Specimens should be placed to get adequate air circulation.

2.4 Remove specimen and place in a desiccator and cool to room temperature.

2.5 Remove from the desiccator and weigh the specimen to the nearest 0.01 gram (W_2).

2.6 Calculate percent volatiles as follows:

$$\text{Percent Volatiles} = \frac{W_1 - W_2}{W_1} \times 100$$

Where:

W_1 = uncured weight of specimen, gm.
W_2 = final weight of specimen, gm.

2.7 Report volatile content of specimen to the nearest 0.1 percent.

2.8 Limits (wt percent)

	Normal	Retest Properties
Minimum	3.5	3.5
Maximum	6.00	6.5

P-P-2 Resin Solids, Filler and Cloth Content of Uncured Material

1. SCOPE

1.1 These properties of the uncured material shall be determined for each sample in accordance with the following.

2. PROCEDURE

2.1 Place a 1.5 ± 0.2 gram specimen weighed to the nearest 0.01 gram (specimen weight) in a 250 ml. beaker.

2.2 Calculate devolatilized specimen weight (W) as follows:

$$W = \text{specimen wt} - \frac{(\%\ \text{volatiles}) \times (\text{specimen wt.})}{100}$$

Where: Percent volatiles is the value determined from the volatiles content.

2.3 Add 50 ml. of N, N dimethylformamide (DMF) and bring to a boil. Gently stir the boiling DMF and prepreg cloth for a minimum of 5 minutes. Remove beaker from heat source and cool for a minimum of 5 minutes while stirring gently.

2.4 Decant and transfer the liquid solution to a second 250 ml. beaker.

2.5 Rinse the remaining fabric with DMF several times to ensure complete extraction of resin and filler from fabric and add the rinses to a second 250 ml. beaker.

2.6 Finally rinse the fabric with acetone to remove all traces of DMF.

2.7 Add all rinses to the second 250 ml. beaker.

2.8 Dry a filter and a crucible or filtering crucible at 350 ± 10 °F. for a minimum of 30 minutes. Weigh filter and crucible or filtering crucible to the nearest 0.01 gm. (W_2).

2.9 Dry the fabric in an oven for 15 ± 1 minutes at 325 ± 10 °F., cool in a desiccator to room temperature and reweigh to the nearest 0.01 gm. (W_1).

2.10 Using an aspirator or vacuum pump, filter the contents of the second 250 ml. beaker through a filter and crucible or filtering crucible into a vacuum flask.

2.11 Rinse the crucible and filter or filtering crucible several times with acetone to remove all resin.

2.12 Dry the crucible and contents, at 350 ± 10 °F. for 20 ± 1 minutes, cool in a desiccator to room temperature and reweigh to the nearest 0.01 gm. (W_3).

2.13 Calculate the resin solids content, filler content, and cloth content as follows:

$$\text{Resin solids content \%} = \frac{W - (W_1 + W_4)}{W} (100)$$

$$\text{Filler content \%} = W_4/W\ (100)$$

$$\text{Cloth content \%} = W_1/W\ (100)$$

Where:

W = calculated devolatilized weight of prepreg specimen, gm.
W_1 = weight of fabric after resin and filler removal, gm.
W_2 = crucible and filter or filtering crucible weight, gm.
W_3 = filler + crucible or filler + filtering crucible weight, gm.
W_4 = W3-W2 = filler weight, gm.

2.14 Report the resin solids, filler content, and cloth content to the nearest 0.10 percent.

2.15 Limits (wt percent)

	Dry Resin Solids	Carbon Filler	Cloth
Minimum	32.0	8.0	47.0
Maximum	37.0	16.0	60.0

P-P-3 Resin Flow of Uncured Material

1. SCOPE

1.2 Resin flow for each sample of the uncured material shall be determined in accordance with the following.

2. PROCEDURE

2.1 Using a 4-inch by 4-inch ± 1/8-inch template, cut four squares across the width of the fabric. All squares shall be bias cut to eliminate fiber loss in testing. Stack the squares uniformly on each other to make a specimen. Where the width of the tape is less than 4 inches wide, cut the tape in 4-inch lengths and place side by side to fabricate a 4-inch by 4-inch specimen ply. Stack four plies alternately placed 90 degrees to the previous ply direction. Place each ply uniformly on each other.

2.2 Weigh the specimen of 4 plies to the nearest 0.01 gram and record as W_1.

2.3 Place the specimen between release film. Preheat the press to 325 ± 10 °F., position the specimen in the middle of the press plate and apply the press load of 150 ± 10 psig. immediately. Press load the specimen for a minimum of 10 minutes at 325 ± 10 °F.

2.4 Remove the specimen from the press and cool to ambient temperature.

2.5 Using a knife, scrape off the resin flash to the original size of the specimen. Do not remove any reinforcement from the original dimensions.

2.6 Reweigh the specimen to the nearest 0.01 gram and record as W_2.

2.7 Calculate the percent resin flow as follows:

$$\text{Percent flow} = \frac{W_1 - W_2}{W_1} \times 100$$

Where:

W_1 = uncured weight of specimen, gm.

W_2 = final weight of specimen, gm.

2.8 Report resin flow to the nearest 0.1 percent.

2.9 Limits (wt. percent)

	Normal	Retest
Minimum	10.0	8.0
Maximum	20.0	23.0

P-P-4 Carbon Cloth, Phenolic, Preimpregnated, Medium Sodium and Uncured Content

1. SCOPE

1.1 This specification covers medium sodium carbon cloth reinforcement preimpregnated with aldehyde-phenolic resin for rocket motor nozzle components.

2. MATERIAL

The material shall be a carbon fabric reinforcement impregnated with a carbon filled phenolic resin and either slit into straight or bias tape for wrapping or supplied as broadgoods. The cut tape ends shall be joined by splicing and stitching with nylon thread. Bias tape and straight tape cut ends shall be joined by butt splicing. Broadgoods roll widths shall be 36-48 inches and shall be rolled onto the paper core with the warp side of the impregnated fabric on the outside face of the rolls selvage shall be removed to provide two parallel roll edges.

2.1 Resin. The resin system shall conform to the requirements of MIL-R-9299, Grade A.

2.2 Reinforcement. The reinforcement shall be a carbon fabric conforming to STW4-3184.

2.3 Resin filler. The resin filler shall be carbon powder with a minimum carbon assay of 90 percent.

2.4 Splicing thread. The splicing thread used for the stitching specified in 3.2 shall be in accordance with STW4-2919.

2.5 Age of material. The material shall be shipped within 3 months of manufacturing data and shall be tested and certified within 60 days of shipment.

2.6 Process of manufacture. Any change in process or material after initial qualification testing as required by this specification shall be cause to require requalification.

3. STORAGE LIFE

The material shall have a shelf life of 6 months after the date of manufacture when stored at 50 degrees Fahrenheit (F), maximum, in sealed plastic bags with a 1-unit (minimum) desiccant bag conforming to MIL-D-3464. After date of receipt of the broadgoods, the maximum cumulative out-of-storage time shall be 30 days. Storage life requirements shall apply until the material has been completely tape wrapped onto a mandrel to form a rough component shape.

3.1 Shelf life extension. The shelf life of broadgoods and tape rolls may be extended for three additional 6 month periods provided the material, upon retest, conforms to the retest requirement for resin flow, volatile content, and residual volatile content. The maximum allowable cumulative shelf life of broadgoods and tape rolls slit from broadgoods shall not exceed 24 months from the date of manufacture of the original broadgoods lot. The number of broadgoods and tape rolls to be tested for shelf life extension shall be as follows:

No. of broadgoods or tape rolls in lot	No. of broadgoods or tape rolls to be tested
1-3	ALL
4-40	4
41-65	5
66-100	6

A lot of material shall be acceptable if the average of the three tests for each selected roll meets the shelf life extension requirements. Failure of any of the selected rolls to meet these requirements shall require all rolls within the lot to be tested for only those material properties which failed to meet specification requirements.

3.2 Conditioning for shelf life extension retest. Material which fails to meet the maximum allowable volatile and resin flow requirements may be conditioned to extend the shelf life by subjecting all the rolls in the lot which failed to meet the requirements of 3.1 to the following:

3.2.1 Place material, broadgoods and/or slit tape, in vacuum chamber supported through core(s) to prevent material damage.

3.2.2 Expose material to vacuum of less than 1 inch of Mercury (Hg) and a temperature of 60 to 90 degrees F for a period of 7 to 9 days.

3.2.3 After completion of drying process, randomly select rolls to be tested using the following sampling plan. Obtain samples from outside and inside each sampled roll and retest for volatile content and resin flow.

No. of broadgoods rolls or tape rolls remaining in the original lot	No. of broadgoods rolls or tape rolls to be selected for retest sampling
1 - 2	All
3 - 40	3
41 - 65	4
66 - 100	5

3.2.4 Failure of any of the selected rolls to meet the requirements shall require all conditioned rolls to be tested.

3.3 Out-of-Storage time extension. The 30-day out-of-storage time may be extended for one additional 30-day period provided that each roll has met the shelf life extension requirements and the average of three tests has met the retest requirements.

3.4 Preparation and storage of samples selected for retest. Each lot of broadgoods or tape roll samples shall be promptly put into an individual plastic bag, sealed, properly labeled, and stored at 50 degrees F., maximum. Prior to testing and removal from the plastic bag, the samples shall be conditioned at 60 to 80 degrees F. for 2 hours, minimum. The samples shall be tested within 72 hours after removal from the broadgoods or tape rolls.

4. CLASSIFICATION OF INSPECTIONS

The inspection requirements specified herein are classified as qualification inspections and quality conformance tests.

4.1 Uncured Material

4.1.1 Visual examination M-C-31

4.1.2 Volatile content P-P-26

4.1.3 Dry resin solids F-14

4.1.4 Carbon filler Content M-F-55

4.1.5 Cloth Content P-P-2

4.1.6 Resin flow P-P-3

4.1.7 Sodium Content M-C-24

4.2 Cured Material

4.2.1 Density F-1

4.2.2	Residual volatiles	F-16
4.2.3	Resin Content	F-14
4.2.4	Compressive Strength	F-4
4.2.5	Interlaminar Shear	F-5
4.2.6	Thermal Conductivity	F-6
4.2.7	Coefficient of thermal expansion	F-7
4.2.8	Flexural strength	F-8
4.2.9	Tensile strength	F-9

5. SAMPLING

Each lot of carbon cloth aldehyde-phenolic shall be sampled for inspection as follows:

5.1 Lot. A manufacturing lot of preimpregnated material shall consist of material which is impregnated in one continuous production run, using one batch of the impregnator's resin and additives mixed in one vessel at one time.

5.2 Lot size. A lot of carbon cloth aldehyde-phenolic shall not consist of more than 8000 pounds.

5.3 Lot acceptance test samples.

5.3.1 Sampling for uncured material properties. Each sample shall be of sufficient size to perform all of the uncured material inspections and tests. Each sample shall be marked with the roll number, lot number, and the location in the roll from which the sample was taken. Each broadgoods roll in the lot shall be sampled at the beginning of the roll, and at the end of the roll.

5.3.2 Sampling for cured material properties. A sample of sufficient size to perform all the cured material inspections and test specified herein shall be taken from the outside end of each broadgoods roll selected at random from each lot. The number of rolls to be selected shall be determined as follows:

Number of rolls in lot	Number of rolls to be selected for sampling
1 to 2	All
3 to 40	3
41 to 65	4
66 to 110	5

Failure of a roll sample to conform to the requirements of this specification shall be cause to withdraw that roll and shall require that all other rolls in that lot be tested for only those material properties which failed to meet specification requirements.

5.3.3 Sampling for sodium content. Samples of sufficient size to perform sodium test shall be removed from the exposed end of each broadgoods roll selected at random from each lot. Failure of a roll to conform to the requirements of this specification shall be cause to withdraw that roll and shall require all other rolls in that lot to be tested for sodium. The number of rolls to be selected shall be as follows:

Number of rolls in lot	No. of rolls to be selected for sampling
1 to 2	all
3 to 40	3
41 to 65	4
66 to 110	5

5.4 Test requirements. Three tests shall be performed for each of the material properties specified herein on the samples taken from each broadgoods roll sampled. All individual test results shall be reported. The average values of the test results and at least two of the three individual test values shall be used to determine conformance to the requirements of this specification. Where applicable, testing of individual specimens for more than one property shall be permissible.

5.4.1 Test conditions. Unless otherwise specified, all tests shall be performed under ambient conditions. Where the temperature or time is critical to the results of the test, these controls shall be specified in the applicable test procedure.

5.4.2 Supplier retest. When the average test value of any lot fails to conform and the cause of the failure can be attributed to improper preparation of the specimen or testing machine anomaly, a new test shall be conducted on a sample from the same broadgoods roll from which the failed specimens were removed.

6. DEFINITIONS

6.1 Stage. A method of stabilizing and retaining the aldehyde-phenolic polymer in a solid state on the carbon cloth by temperature cycling.

6.2 Coefficient of thermal expansion. The coefficient of thermal expansion is the slope of the secant line between the 75 degrees F. and 400 degrees F. line intersection of the linear expansion curve.

6.3 Tape roll. A tape roll is a narrow width of impregnated cloth tape from a broadgoods roll.

6.4 Sample. A sample is a cut-out section of the impregnated broadgoods roll or tape roll.

6.5 Specimen. A specimen is a cut-out section of the sample used for testing.

6.6 Broadgoods. Broadgoods material is a wide roll of carbon cloth fabric impregnated with resin.

P-P-5 Moleweight of Resins and Prepregs by Chromatography

1. SCOPE

1.1 This procedure describes methods for conducting liquid Chromatography and gel permeation chromatography of aldehyde-phenolic resins. It determines the moleweight of the constituents.

2. EQUIPMENT AND MATERIALS

2.1 HPLC Spectra-Physics SP8000 with UV detector and autosampler, with appropriate operators manuals.

2.2 Styragel chromatography columns, 2 X 100 A, 2 x 500 A <u>(USE NO WATER)</u>

2.3 Syringe, 10 cc.

2.4 Gas tight syringe, 5 cc.

2.5 Needles

2.6 Scissors

2.7 Centrifuge tubes, 50 ml. disposable

2.8 Centrifuge

2.9 Graduated cylinder, 10 ml.

2.10 Balance

2.11 Beaker

2.12 Millex-SR filter units, 0.5

2.13 Membrane TFE (Tetrafluroethane) filter paper, 0.45

2.14 B & J tetrahydrofuran, UV grade, THF

2.15 Helium (zero grade) gas

2.16 Nitrogen gas

3. PREPARATION OF SAMPLE SOLUTION

3.1 <u>Resin Samples</u>

3.1.1 Weigh out 0.25 ± 0.02 gm. of resin into a glass vial (labeled)

3.1.2 Add 10 ml. 100% THF into the vial

3.2 Resin Mixes

3.2.1 Weigh out 3.0 ± 0.02 gm. of sample into a centrifuge tube

3.2.2 Add 25.0 ml. of 100% THF into the centrifuge tube

3.2.3 Agitate for 15-50 minutes

3.2.4 Centrifuge for 2-5 minutes at appropriate speed

3.3 Prepregs

3.3.1 Cut sample into approximate 1/2" x 1/2" squares

3.3.2 Weigh out 6.0 ± 0.5 gm. of sample into a centrifuge tube

3.3.3 Add 25 ml. 100% THF into the centrifuge tube

3.3.4 Agitate for 15-50 minutes

3.3.5 Centrifuge for 2-5 minutes (Speed-7)

3.4 Solvent Preparation

3.4.1 Filter solvent (1-2 liters) by using 0.45 um. TFE filter (Tetrafluoroethene)

3.4.2 Degas with He 1/4 to 1/2 hour (high flow)

3.4.3 Decrease the flow rate before running

4. INSTRUMENT PROGRAMMING

4.1 Date Assignment

D: 00:00:00 00:00:00
month day year hour min. sec.
D - Display

4.2 Report File Assignment

R:1 - report file
REPORT FILE 1
RENAME:
SAMPLE:-------------
SAMPLE VOLUME: 10 L
SAMPLE CONC.: 20 MG-ML
OPERATOR NAME: (your name)
DETECTOR 1: UV. 254NM 0.1 AUFS
DETECTOR 2: ;
COLUMN TYPE; SIZE: 2x100A, 2X500A, -STYRAGEL
MOBILE PHASE: Isocratic

A: ;
B: ;
C: THF
END OF DIALOG

4.3 Parameter File Assignment

S:1 parameter file 1

PARAMETER SET: 1

RENAME: ;

1. LINK: ;
2. INITIAL DELAY: 1
3. RUN TIME: 30
4. NO. OF CYCLES: 6
5. FLOW RATE: 2.00
6. FLOW MODE: QI
7. TEMPERATURE: 35
8. MOBILE PHASE FILE: 1
9. REPORT FILE: 1
10. GRAPH FILE: 1
11. DATA SYSTEM FILES: 1
12. CHAN 1:1
13. CHAN 2: ;
14. FULL TIME: 1
15. FLUSH TIME: 1
16. REPETITIONS PER VIAL: 1
17. TIMED EVENTS: XX

END OF DIALOG

4.4 Graph File Assignment

G = 1 graph file 1

G11
G1E5
G1I5
GVO.5
XX

EXIT GRAPH EDIT

4.5 Mobile Phase File Assignment

M = 2 mobile phase file 2

MOBILE PHASE FILE 2

WHICH SOLVENTS? 2

```
END OF DIALOG

MI2

M;1

MOBILE PHASE FILE 1

WHICH SOLVENTS? C

END OF DIALOG

MI1
```

4.6 Data File Manipulation

```
FI1  Data system file (file center)

nm gpc-QC

CH 001
OD 002
IX 001
RN 001
NR 001
PW 015
PT 0150
MA 5000
FD
VE
FILE 001

RN 001     NR 001    PW 015       PT 0105
FS 000     MS 0      MA 5000      IM 00
PP 004     TI 000    RO 000
NM GPC-QC
CH 001     OD 002    IX 001       SN 000

FF  function file
APO
FD
VE
FILE 001

AR 0   IR 0   DC 0   NZ 0   FX 0   EC 0
TP 1   NP 0   NT 0   AA 0   XD 0   HD 0

XX

METHOD
```

4.7 Review File Values

```
S-1
```

```
PARAMETER SET: 1

INITIAL DELAY: 1 RUN TIME: 30 NO. OF CYCLES: 6
FLOW RATE: 2.00  FLOW MODE: Q1  TEMPERATURE: 35
MOBILE PHASE FILE: 1 REPORT FILE: 1 GRAPH FILE: 1
CHAN 1:1
FILL TIME: 1  FLUSH TIME: 1  REPETITIONS PER VIAL: 1

R-1

-------------VOL: 10 UL  CONC:  20 mG-ML   NO:  1
(your name)
    FLOW RATE:  2.0     PRESSURE:  0000
COLUMN:  2x100A, 2X500A,  --STYRAGEL
    CHAN 1:  U.V.  254NM     0.1  A-FS
    MOBILE PHASE FILE 1
    100.0% C
    G-1
    GRAPH FILE: 1
    Channel 1:  Plot Var.:  DET. 1  Chart Spd.: 0.5
    Polar: L ATTN.: 5   Zero: 0%  Inten.: 5
    Channel 2:  Inoperative
    M-1
    Mobile Phase File: 1
    100.0% C
```

5. PROCEDURE

5.1 HPLC: Start-up:

5.1.1 Turn on He and N_2

5.1.2 Adjust the He pressure to 10 psi. and N_2 pressure to 75 psi.

5.1.3 Turn on both switches, Wavelength Drive at "on" position. Set wavelength at 254 nm.

5.1.4 Turn on the Spectrophotometric Detector, make sure it is ".4 m amp" Set range at 0.1 AUFf

5.2 Automatic Operation

5.2.1 Be sure both detector and auto sampler are on

5.2.2 Keep He purge, the solvent with low flow rate

5.2.3 Type AT1
DID
FM1
MI1
SB1

5.2.4 Use the inner knob on the detector range selector to adjust the read out to -1200 (detector signal) or type GB and adjust the baseline 1 inch to the left margin, then type GX to stop plotting.

5.2.5 Fill one gas tight syringe with sample solution and the other one with THF; attach each syringe to one Miller-SR filler unit.

5.2.6 Place the injector handle in the "LOAD" position and type SO to open the injector sample solenoid.

5.2.7 Inject 1 cc. of THF to flush the injector loop, then inject 1 cc. of sample solution and type SK to close the sample solenoid.

5.2.8 Wash the syringe with THF twice; fill it with the next sample.

5.2.9 After LC, inject automatically, replace the injector handle in the "LOAD" position and repeat the injecting operation.

5.2.10 Check the condition by typing:

E- elapsed time since injection
T- present oven temperature
P- pressure
C- current % C at ternary valve
DID- detector signal
F- flow rate

5.3 Calibration (before running sample solutions)

5.3.1 Run a THF blank in order to plot the baseline

5.3.2 Run molecular weight standards and plot molecular weight vs. retention time on graph paper

Mole Weight Standards:

M.W.	Compound
93	Toluene
570	Polystyrene
955	"
3600	"

5.4 Termination

5.4.1 Type EX to end run

5.4.2 Type SX to stop parameter set

5.4.3 Shut down all gas flows

P-P-6 Infra-red Spectrum of Resins and Prepregs

1. SCOPE

1.1 This method is used for determination of infrared spectrum of liquid and solid resins or prepreg.

2. APPARATUS

2.1 Beckman 4250, Beckman Acculab 8 or equivalent, and operators manual.

2.2 Sodium chloride disk.

2.3 Solvent: Spectrograde acetone, THF or methylene chloride.

3. PROCEDURE

3.1 Place a small amount of material to be tested in a 50-ml. beaker. Add about 15 ml. of spectroquality acetone and stir with a glass stirring rod to dissolve. If the material doesn't dissolve in acetone, spectroquality THF or methylene chloride may be used.

3.2 Place several drops of the solution on the sodium chloride crystal and allow to dry at room temperature.

3.3 Zero the I.R> beam as the instrument manual describes.

3.4 Make a quick scan of the region from 2000 cm^{-1} to 1000 cm^{-1}. When the transmittance is between 15 and 25 percent at most intense peak the right concentration is on the crystal. Twenty percent is the ideal transmittance.

3.5 Start the scan at 90% transmittance. Scan the sample in the infrared from 2.5 to 16 microns.

3.6 Do any necessary calculations.

P-P-7 Resin Content of Prepregs by Soxhlet Extraction

1. SCOPE

1.1 This method describes a procedure for the determination of the resin content on carbon, graphite, fortisan and nylon prepregs by soxhlet extraction. Special provisions are included to determine the resin content of filled systems.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

2.1.1 Balance, analytical, sensitivity 0.0001 gm.

2.1.2 Bottle, wash

2.1.3 Crucible, filtering, fritted glass, medium grade, 30 ml.

2.1.4 Crucible holder

2.1.5 Desiccator

2.1.6 Extraction thimbles, pyrex, extra coarse, 25 x 85 mm.

2.1.7 Flask, filtering, 500 ml.

2.1.8 Furnace, muffle, at least 1000 ± 25°F

2.1.9 Oven, forced air (325° ± 5°F.)

2.1.10 Soxhlet extraction assembly, 125 ml. capacity with hot plate

2.1.11 Vacuum system

2.2 Materials

2.2.1 Extraction thimbles, paper, fat-extracted, 25 x 85 mm

2.2.2 Solvent, technical grade (see 4.5)

2.2.3 Filter, glass fiber, 5.5 cm., Reeve Angel grade 934AH or equivalent.

3. SAMPLING

3.1.1 Broadgoods. The sample shall include the entire width of the unit being tested, less the first two inches in from each selvage edge. A sufficient number of strips, 1 x 2 inch from the usable width, shall be cut into 1/2 inch by 1/2 inch squares, to provide enough samples for the required number of soxhlet extractions per sample. These squares shall then be well mixed, using extreme care not to knock off any resin, before the actual samples are taken.

3.1.2 Chopped Stock. Obtain a representative bulk sample of 10-15 grams and mix thoroughly, using extreme care not to knock off any resin.

3.2 Number of determinations. Unless otherwise specified, run duplicate determinations on each sample.

4. PROCEDURE

4.1 Volatile content. Determine volatile content of the prepreg per PTM-17, PTM-32, or as specified.

4.2 Thimble type. Unless otherwise specified, the following thimble types shall be used:

MATERIAL CARRIER	THIMBLE TYPE
Carbon and graphite	Paper
Fortisan	Glass
Nylon	Glass

4.3 Thimble conditioning. Unless otherwise specified, condition as follows:

4.3.1 Paper. Identify with a soft lead pencil and dry for 2 hours at 325° ± 5°F. in a forced air oven. Remove in sets of 3 and weigh immediately to 0.001 gm. (W_1). Store in a desiccator together with recorded weight.

4.3.2 Glass. Dry for 30 minutes at 950° ± 25°F. in a muffle furnace. Cool in a desiccator and weigh to 0.001 gm. (W_1).

4.4 Sample Weighing. Unless otherwise specified, transfer a 2.5 ± 0.5 gm. sample to the weighed thimble and weigh to 0.001 gm. (W_3). When paper thimbles are used, they must be reweighed to 0.001 gm. (W_2) before addition of the sample. See note 6.1 for precautions regarding paper thimbles.

4.5 Extraction. Position the thimble within the Soxhlet extraction assembly. Add 90 ml. of solvent, adding a portion of the 90 ml. to the extraction tube so that about 2/3 of the thimble is covered, the remainder is added to the boiling flask. The following solvents are suggested for the listed resins. However, experience shall dictate solvent to be used (see note 6.2).

RESIN	SUGGESTED SOLVENT
Phenolic	Ethyl alcohol or dimethylformamide (DMF)
Epoxy	Acetone or methyl ethyl ketone (MEK)
Silicone	Piperidine or toluene
Polyester	Acetone or MEK

Turn on the cooling water for the condenser. Turn on the hot plate to effect heating of the solvent. When condensation of the solvent occurs, adjust hot plate temperature to effect 3 to 10 reflux changes per hour. For high boiling solvents, such as DMF, it may be necessary to wrap the extraction tube with aluminum foil. Ensure that the solvent is condensing and dropping into the extraction tube. Continue to reflux for a minimum of four hours.

4.6 Determination of filler when present. If filler has appeared in the extract, the filler weight must be determined as follows:

4.6.1 Conditioning of crucibles. Place a glass fiber filter in a fritted glass crucible (see note 6.3). Condition in a forced air oven for 15 to 20 minutes at 325° ± 5°F. Allow the crucibles to cool in a desiccator and weigh to 0.001 gm.

4.6.2 Filtering. Set up the vacuum system and filter the resin extract. Using a wash bottle of acetone or appropriate solvent wash out the extraction tube and flask, putting all washings in the filtering crucible. Wash the residue clean of resin. About 50 ml. of acetone or appropriate solvent will usually suffice. Do not stir the solution on the filter.

4.6.3 Unremovable filler. If after washing the extraction flask with solvent there is some filler which is caked on the flask and cannot be removed, the following procedure can be used for carbonaceous filled aldehyde-phenolic resins: Place the flask in a muffle furnace at 750° ± 25°F. for a minimum of one hour. Cool in a desicattor and weigh to 0.001 gm. If the flask has been prepared, subtract the tared weight and add this filler weight to that of 4.6.4 If the flask has not been prepared, place in a muffle furnace and burn off the carboneceous filler at 1000 ± 50°F. for a minimum of one hour. Cool in a desiccator and weigh to 0.001 gm.

4.6.4 Drying filter crucible. Dry the filter crucible in a forced air oven for a minimum of one hour at 325° ± 5°F. Cool in a desiccator and weigh to 0.001 gm. Add this filler weight to the carrier weight of 4.7.

4.7 Drying thimbles. Remove thimbles from the extraction assembly. Drain off the solvent and dry to essentially constant weight at 325° ± 5°F.in a forced air oven (see note 6.4). If paper thimbles are used, remove from the oven in sets of three or less and weigh immediately to 0.001 gm. (W_4). Glass thimbles may be cooled in a desiccator before weighing.

5. CALCULATIONS AND REPORTING

5.1 Calculations where paper thimbles are used.

5.1.1 Dry resin content (DRC). Calculate dry resin content (resin solids) as follows:

$$\text{DRC, wt. \%} = 100\ [\ 1 - [(W_4-W_1)/(W_3-W_2)\ \times\ (1-V/100)]]$$

where:

W_1 = weight of dried thimble, gms.
W_2 = weight of thimble after storage in desicator,gms
W_3 = weight of thimble plus sample, gms.
W_4 = weight of thimble plus extracted sample, gms.
V = volatile content, wt.%

5.1.2 Wet resin content (WRC). Calculate wet resin content (total pick-up) as follows:

$$\text{WRC, Wt. \%} = 100\ [\ 1\ -\ [(W_4\ -\ W_1)/(W_3-W_2)]\ ]$$

5.1.3 Wet resin solids (WRS). Calculate wet resin solids as follows:

$$\text{WRS, wt.\%}=100[1-[(W_4-W_1/W_3-W_2)]-(V/100)]=\text{WRC}-V$$

5.2 Calculations where glass thimbles are used.

5.2.1 Dry resin content (DRC). Calculate dry resin content (resin solids) as follows:

$$\text{DRC, wt.\%} = 100\ [\ 1-[(W_4-W_1)/(W_3-W_1)(1-(V/100))]]$$

where symbols are specified in 5.1.1

5.2.2 Wet resin content (WRC). Calculate wet resin solids as follows:

$$WRC, wt.\% = 100 [1-(W_4-W_1)/(W_3-W_1)]$$

5.2.3 Wet resin solids (WRS). Calculate wet resin solids as follows:

$$WRS, wt.\% = 100 [1-[(W_4-W_1)/(W_3-W_1)] - (V/100)]=WRC-V$$

5.3 Reporting. Unless otherwise designated, report resin content to the nearest 0.1%. Duplicate determinations should be considered suspect if they differ by more than 1.5%.

6. NOTES

6.1 Paper thimbles. When paper thimbles are used, it is good laboratory practice to periodically determine a blank on the procedure. This is especially true when conducting the extraction on a new product for the first time. Under normal conditions, single thimbles shall be used. However, particle size of a filler may make it necessary to use double thimbles.

6.2 Selection of solvent. For carbonaceous filled aldehyde-phenolics with carbon or graphite reinforcement, the following rule shall apply: DMF must be used when resin content is less than 31% and/or volatile content is less than 3.8%.

6.3 Use of glass fiber filter. A glass fiber filter is used to prevent the fritted glass filter from becoming clogged with the filler.

6.4 Thimble drying time. The drying time for the thimble is normally two hours minimum.

6.5 Calculation of resin content. This method has used carrier weight for the calculation of resin content. There are occasions where it may be desirable to calculate resin content by using the resin extract which has been dried down by the appropriate means and weighed. Calculating resin content using resin extract typically gives higher results and more variability than when thimble weight is used.

P-P-8 Volatile Content of Prepregs

1. SCOPE

1.1 The purpose of this method is to describe a procedure for the determination of volatile content of preimpregnated materials.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

2.1.1 Steel templates, 4 inches by 4 inches and 2 inches by 2 inches

2.1.2 Hooks, "S" shaped

2.1.3 Oven, forced air

2.1.4 Oven, dead air

2.1.5 Mettler balance, or equivalent, capable of weighing to 0.001 gram

2.1.6 Stanley knife or equivalent

2.1.7 Wide mouth ceramic crucibles, Coors No. 1 or equivalent

2.1.8 Desiccator

3. SAMPLING

3.1 Sample Size

3.1.1 Prepregs other than unidirectional tapes. Three specimens 4 inches by 4 inches are cut randomly from a representative sample one foot from edge of prepreg.

3.1.2 Unidirectional tapes. Three specimens 2 inches by 2 inches (or equivalent of 4 square inches per specimen) shall be randomly cut from a sample representative of the material.

4. PROCEDURE

4.1 Prepregs other than unidirectional tapes.

4.1.1 Preparing samples. Cut three 4 inch by 4 inch specimens using steel template and weigh collectively to the nearest 0.001 gm.

4.1.2 Testing samples. Attach "S" shaped hook to one corner of each specimen and place the specimens in the designated type of oven for required time and temperature as per the chart in 4.2.2. Suspend the specimens individually, allowing room for free air circulation between each. Do not allow the door to remain open longer than 10 seconds while loading the specimens. If dripping of resin occurs repeat, hanging each specimen by diagonal corners. Remove the specimens from the oven, desiccate for 5 minutes and reweigh collectively to the nearest 0.001 gm. Desiccation may be waived unless specifically required by customer specification or test method, provided the weighing is done immediately upon removal of the specimens from the oven.

4.2 Unidirectional tapes.

4.2.1 Preparing samples. Cut three 2 inch by 2 inch (or equivalent) specimens and weigh each into a previously tared crucible, to the nearest 0.001 gm.

4.2.2 Testing samples. Place the crucibles in the designated type of oven for the required time and temperature as per the chart. Do not allow the oven door to remain open longer than 10 seconds while loading oven.

PREPREG TYPE	* TEM., ^{O}F.	*TIME, MIN.
Phenolics or Phenylsilanes	320	9
Polyesters	220	8, Dead Air
Standard Epoxies	320	9
Special Epoxies	275	15
Silicones	320	5
Paper, Melamines	320	8, Dead Air
Paper, Polyesters	220	10, Dead Air
Polyimides	450	10
Miscellaneous	**	**

* All temperature tolerances are ± 5^{O}F; all time tolerances are ± 0.25 minutes. All ovens are forced air except as noted.

** For miscellaneous prepreg types, temperatures and time are as per customer specification.

Remove the crucibles from the oven, desiccate for 5 minutes and reweigh each to the nearest 0.001 gm.

5. CALCULATIONS AND REPORTING

5.1 Prepregs other than unidirectional tape.

5.1.1 Calculate volatile content as follows:

$$\% \text{ Volatile} = \frac{W_1 - W_2}{W_1} \text{ X } 100$$

where:

W_1 = Original weight, gms.
W_2 = Final weight, gms.

5.2 <u>Unidirectional tapes.</u>

5.2.1 Calculate volatile content as follows:

$$\% \text{ Volatile} = \frac{(W_1 - W_c) - (W_2 - W_c)}{W_1 - W_c} \text{ X } 100$$

where:

W_1 = Weight of crucible and original sample, gms.
W_c = Weight of crucible, gms.
W_2 = Weight of crucible and sample from oven, gms.

5.3 <u>Repeated tests should agree within 0.2%.</u>

P-P-9 Gel Time for Thermosetting or Thermoplastic Resins

1. SCOPE

1.1 This procedure describes a method for determining gel time of thermosetting or catalyzed thermoplastic resins using a cure plate.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

2.1.1 Stop watch, 0.1 sec. divisions

2.1.2 Cure plate

2.1.3 Thermometer, 20 to 600°F. , accurate to ± 2°F.

2.1.4 Tongue depressor slivers, about 1/8 inch wide

2.1.5 Bent medicine droppers, with useful capacity of approx. 1 ml.

2.1.6 Cleaning Blade

2.1.7 Oven, to at least 500 ± 2°F

3. SAMPLING

3.1 Sample size. A representative sample of about 10 gms. is required.

3.2 Number of determinations. Unless otherwise specified, make three determinations.

4. PROCEDURE

4.1 Preconditioning cure plate. Unless otherwise specified, precondition the cure plate to one of the following temperatures, consistent with resin type:

RESIN TYPE	TEMPERATURE	
Silicones (uncatalyzed)	480 ± 3°F.	
Silicones (catalyzed)	350 ± 2°F	
Polyesters	275 ± 2°F.	
Phenolics, Phenyl Silanes	325 ± 2°F.	
Epoxies, (General)	325 ± 2°F.	(unless otherwise specified)
Polimides	400 ± 3°F.	
Epoxies (Special)	350 ± 3°F.	

4.2 Preparation of cure plate and resin. Clean the cure plate thoroughly. The sample must have a solids content specific to that resin system. Adjust if necessary.

4.3 Starting gel. Using an eye dropper, draw in as much sample as it will hold. This will be 1.0 ± 0.2 grams. Transfer completely to the spot on the cure plate directly above the thermometer. Commence timing with a stop watch.

4.4 Detection of end point. Do not disturb the resin blob until a few seconds prior to minimum gel time expected. At this time, the gel time is close. With the clean tip of the sliver, poke at the resin with an up and down movement (see Note 6.1). As gel time approaches, the sliver will carry strings of resin. When the resin is gelled, the sliver will not carry any resin and the resin will have rubbery appearance. Note the time in seconds required for gelation. Repeat the test for a total of three results.

5. CALCULATIONS AND REPORTING

5.1 Average the three results and report the value in seconds or minutes and seconds. The results should be considered suspect if the range of the three results exceeds 10% of the average gel time.

6. NOTES

6.1 When poking at the resin, do not attract resin to a certain point, but merely poke and move lightly across in order to pick up a small drop which will string when lifted upward. If the resin is gelled when it is disturbed, repeat the operation but probe earlier.

6.2 For E-715 and E-735, use constant stroking of the resin until gelation occurs.

P-P-10 Volatile, Fiber, and Filler Content of Prepregs

1. SCOPE

1.1 This procedure describes methods of determining the volatile content, fiber content and filler content of EPDM prepregs such as R2066P.

2. EQUIPMENT AND MATERIALS

2.1 Aluminum weighing dishes, approx. 1.5 gm.

2.2 Oven, air circulating, at least 300 ± 5°F

2.3 Timer

2.4 Desiccator with desiccant

2.5 Porcelain crucible, 25 ml. capacity, with cover

2.6 Muffle furnace, at least 1500 ± 25°F

3. SAMPLING

3.1 A representative sample of 4 inches by the width of the material (normally 40 inches) is required.

3.2 The number of determinations shall be as specified in each individual test below.

4. PROCEDURE

4.1 Volatile Content

4.1.1 Volatile content shall be determined in accordance with the following:

4.1.1.1 Cut 3 specimens each 2" x 2" from each sample to be tested

4.1.1.2 (W_1) Weigh each 2" x 2" to nearest mg. individually and place into an aluminum dish

4.1.1.3 Condition sample and aluminum dish in circulating air oven @ 285 ± 10°F for 55 ± 2 min.

4.1.1.4 Remove, cool in desicattor, and weigh (2 x 2 sample only) to the nearest mg. (W_2)

4.1.2 Calculation

$$\text{Volatile Content, \%} = \frac{(W_1 - W_2)}{W_1} \times 100$$

where:

W_1 = Original sample weight, grams

W_2 = Sample weight after 55 minutes @ 285°F., gm.

Report individual and average values.

4.2 Total Filler Content

4.2.1 Determine the filler content as follows:

4.2.1.1 Cut the 2" x 2" (W_2) sample into 1/2" x 1/2" and place into a previously fired (1450 ± 10°F.) and tared crucible.

4.2.1.2 (W_3) weigh to the nearest mg. sample and crucible, then cover crucible, and place in a muffle furnace for 1 hr. ± 5 min. at 800° ± 10°F.

4.2.1.3 Remove crucible and place into a desiccator to cool to touch (do not remove cover from the crucible).

4.2.1.4 (W_4) Weigh crucible and sample without cover, gm.

4.2.2. Calculation

Total Filler Content,

$$\text{Total Filler Content,} = 100 - \frac{W_3 - W_4}{W_2} \times 100$$

where:

W_2 = Sample weight after 55 ± 5 min. @ 280° ± 5°F, gm.

W_3 = Crucible and sample (without cover) before burn off, gm., to nearest mg.

W_4 = Crucible and sample (without cover) after burn off, gm., to nearest mg.

Report the individual and average values for these results.

4.3 Fiber Content

4.3.1 Determine fiber content by calculation as follows:

4.3.2 Calculation

$\frac{\text{Total Filler}}{29.8} \times 26.7$ (these constants apply only to R2066P)

P-P-11 Extraction of Prepregs for Determination of Acetone Soluble Material

1. SCOPE

1.1 Scope. This method describes a procedure for determining the amount of acetone-soluble material in prepreg, molded or laminated phenolic products.

1.2 Equivalent methods. This method is equivalent to Federal Test Method Standard No. 406, Method 7021 and ASTM-D494-46.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

2.1.1 Specimen fragmentator - a suitable drill press, bench lathe, planer, milling machine, grinder, or file will be needed to breakup specimen into fragments.

2.1.2 Sieves, U.S. Standard, 40 and 140 mesh

2.1.3 Weighing bottle, 2 oz. glass stoppered

2.1.4 Soxhlet extraction assemble, 125 ml. capacity with hot plate

2.1.5 Balance, analytical, sensitivity 0.001 gm.

2.1.6 Drying chamber capable of maintaining 122 ± 4^{o}F

2.2 Materials.

2.2.1 Extraction thimble, paper, 25 mm. D X 80 mm. L.

2.2.2 Aluminum dishes, diameter 2-1/2 inches, height 1-1/16 inches

2.2.3 Acetone, reagent grade

3. SAMPLING

3.1 Sample size. Each sample shall weigh a minimum of 15 grams. The sample shall be a composite, representative of the prepreg, molded or laminated part from which it was taken.

3.2 Number of determinations. Unless otherwise specified, make three determinations on each sample.

4. PROCEDURE

4.1 Prepreg. Prepreg materials shall be cut in squares no larger than 1/2 inch.

4.1.2 Molded or laminated parts. Molded or laminated parts shall be prepared as follows:

4.1.2.1 Grinding. The method of grinding up the material for sieving should be such that no undue heating of the particles will occur which might tend to further cure the material. Drilling is the preferred method where the size and shape of the part permits. In any case, the particles should be of the smallest size practicable, so that they will pass through a 40-mesh screen with a minimum of reworking or grinding.

4.1.3 Screening. Screen the material through a 40-mesh sieve. Regrind the part which does not pass through the 40-mesh sieve until it does, resieve, and blend with the original part which passed. Place the sample on a 140-mesh sieve and screen by using either a standard screen shaker (such as the Rotap) or by hand screening. In the latter case, the screen should be rotated with a slight tapping for a period of one minute or more until there are virtually no further screenings.

4.1.4 Bottling sample. The sample for analysis should be removed from the 140-mesh screen and placed in the two-ounce bottle. Care should be taken that the sample is immediately placed in this bottle and closed up so as to prevent absorption of moisture by undue exposure.

4.2 Extraction. From the material in the bottle, accurately weigh out 1.0 ± 0.05 gm. into a tared, open texture, quantitative filter paper 12 to 15 cm. in diameter. Place the filter paper containing the test specimen in a standard, single thickness, paper extraction thimble, 80 mm. by 25 mm. Close the thimble so that none of the sample can float out. Place the thimble in an extraction tube and add 50 ml. of acetone. Start the water in the condenser and adjust the heat so there are between 15 and 20 dumps per hour. Carefully maintain this rate for four hours minimum. After the siphon empties, remove the flask and empty the contents into a weighed aluminum dish. Rinse the flask with acetone three times and add the washings to the extract in the dish.

4.3 Drying. Place the dish in a well ventilated drying chamber maintained at 122 ± 4°F. and dry the sample to constant weight. Keep the dish in a desiccator between dryings to prevent moisture pickup.

5. CALCULATIONS AND REPORTING

5.1 Calculations.

5.1.1 Acetone extractables. Calculate the percent of acetone extractable materials as follows:

$$E = \frac{W_3 - W_2}{W_1} \times 100$$

where:

E = acetone extractables, wt. %
W_1 = sample weight, gm.
W_2 = tare weight of aluminum pan, gm.
W_3 = final weight of pan and extract, gm.

5.1.2 Percent uncured resin. Calculate percent uncured resin as follows:

$$U = E/T$$

where:

U = uncured resin, wt.%
T = resin solids, wt. % (see Note 6.1)

5.1.3 Degree of polymerization. Calculate degree of polymerization as follows:

$$\text{Degree of polymerization} = 100 - U$$

5.2 Reporting. Unless otherwise specified, report the following information:

5.2.1 Percent acetone extractable material

5.2.2 Degree of polymerization

5.2.3 Cure cycle used in preparation of sample.

6. NOTES

6.1 The resin solids figure must be representative of the resin solids of the sample extracted.

P-P-12 Method of Conducting Thermogravimetric Analysis of Resins and Prepregs.

1.0 SCOPE

1.1 This procedure describes the method for conducting thermogravimetric analysis of resins and prepregs.

2.0 EQUIPMENT AND MATERIALS

2.1 Perkin-Elmer Model TGS-2 Thermoanalyzer or equivalent

2.2 Perkin-Elmer DSC-2 Programer, or equivalent

2.3 Appropriate operators manual

3.0 SAMPLING

3.1 Obtain a small (1 oz.) representative sample

4.0 PROCEDURE

4.1 Balance Controls

4.1.1 Power - on

4.1.2 Filter - lo

4.1.3 Recorder Mode - short (See recorder direction manual)

4.1.4 Recorder Range - 10 mg.

4.1.5 Weight Suppression switch - 100 mg.

4.1.6 Weight Suppression thumb sheen dial - 00.000

4.2 Use DSC-2 as the TG Programmer

4.2.1 Sample holders empty

4.2.2 Temperature RANGE Calibration - as calibrated

4.2.3 Temperature ZERO Calibration - as calibrated

4.2.4 Lower LIMIT - 350oK (or as required)

4.2.5 Cooling RATE - 160o/min.

4.3 Heater Control Unit

4.3.1 ZERO and RANGE Setting - as determined from calibration procedure (or as indicated on calibration card 993-9235)

4.3.2 Power - HEAT

4.3.3 oC - oK switch (on back panel) oK

4.4 Recorder

4.4.1 CHART SPEED -slow (e.g., 5 mm/min.) change to faster chart speed when temperature is started

4.4.2 Weight Pen: RANGE - 20 mv.; right-hand zero

4.4.3 Second Pen: Thermocouple: RANGE - 1 volt, right-hand zero; or FDC: RANGE - 10 mv, left-hand zero

P-P-13 Percent Volatiles Content of Prepregs

1. SCOPE

1.1 This procedure is applicable for determining the percent volatiles content for impregnated material.

2. EQUIPMENT

2.1 Cutting Board or Template

2.2 Analytical Balance, sensitivity 0.001 gm.

2.3 Air-Circulating Oven, $325^\circ \pm 10^\circ F$.

3. PROCEDURE

3.1 This test is to be run in triplicate. Cut three 4" x 4" ± 1/8" straight cut from left, center, right, across the width of broadgoods.

3.1.1 Weigh the specimen to the nearest 0.01 gram (W_1).

3.1.2 Place specimen in a circulating oven, preheat and stabilize to 325 ± 10 degrees F. for 10 ± 1 minutes. Specimens should be placed to get adequate air circulation.

3.1.3 Remove specimen and place in a desiccator and cool to room temperature.

3.1.4 Remove from the desiccator and weigh the specimen to the nearsest 0.01 gram (W_2).

3.1.5 Calculate percent volatiles as follows:

$$\text{Percent Volatiles} = \frac{W_1 - W_2}{W_1} \times 100$$

Where:

W_1 = uncured weight of specimen, gm.
W_2 = final weight of specimen, gm.

3.1.6 Report volatile content of specimen to the nearest 0.1 percent.

P-P-14 Flow Character of "B" Staged Broadgoods

1. SCOPE

1.1 This test is applicable for the determination of the flow characteristics of "B" staged broadgoods material.

2. EQUIPMENT

2.1 Cutting Board or Template

2.2 Analytical Balance, sensitivity 0.001 gm.

2.3 Laminar press capable of meeting temperature and pressure requirments of this method, $325^{\circ} \pm 10^{\circ}$F, 150 ± 10 psig.

3. PROCEDURE

3.1 This test is run in triplicate. Cut one specimen each from left, center, and right across a piece of broadgoods.

3.1.1 Cut four 4-inch by 4-inch ± 1/8 inch squares from each sample location. All squares shall be bias cut to eliminate fiber loss in testing. Stack the squares uniformaly on each other to make a specimen.

3.1.2 Weigh the specimen of four plies to the nearest 0.1 gm. and record.

3.1.3 Place the specimen between release film. Preheat the press to $325^{\circ} \pm 10\ ^{\circ}$F., position the specimen in the middle of the press plate and apply the press load of 150 ± 10 psig. immediatly. Press load the specimen for a minimum of 10 minutes at 325 ± 10 degrees F.

3.1.4 Remove the specimen from the press and cool to ambient temperature.

3.1.5 Using a knife, scrape off the resin flash to the original size of the specimen. Do not remove any reinforcement from the original dimensions.

3.1.6 Reweigh the specimen to the nearest 0.01 gm. and record as W_2.

3.1.7 Calculate the percent resin flow as follows:

$$\text{Percent Flow} = \frac{W_1 - W_2}{W_1} \times 100$$

Where:

W_1 = uncured weight of specimen, gm.
W_2 = final weight of specimen, gm.

3.1.8 Report resin flow to the nearest 0.1 percent.

P-P-15 Resin Solids, Filler, and Cloth Content of Prepregs

1. SCOPE

1.1 This method is applicable for determining the resin solids, filler, and cloth content of prepregs having an epoxy or aldehyde-phenolic resin matrix.

2. PROCEDURE

2.1 Resin solids, filler content and cloth content. These components of the uncured material shall be determined for each sample in accordance with the following:

2.1.1 Place a 2 1/4 ± 1/4" square specimen weighed to the nearest 0.01 gm. (R) (specimen weight) in a 250 ml. beaker.

2.1.2 Calculate devolatilized specimen weight (W) as follows:

$$W = \text{specimen wt.} - \frac{(\text{percent volatiles})(\text{specimen wt})}{100}$$

Where: percent volatiles is the value determined from the volatile content.

2.1.3 Add 50 ml. of N, N dimethylformamide (DMF) and bring to a boil. Occasionally gently stir the boiling DMF and prepreg cloth for a minimum of 5 minutes.

2.1.4 Decant and transfer the liquid solution to the second 250 ml. beaker.

2.1.5 Rinse the remaining fabric with DMF several times to ensure complete extraction of resin and filler from fabric and add the rinses to a second 250 ml. beaker.

2.1.6 Finally rinse the fabric with acetone to remove all traces of DMF.

2.1.7 Add all rinses to the second 250 ml. beaker.

2.1.8 Dry a filter and a crucible or filtering crucible at 350 ± 5°F. for a minimum of 30 minutes. Weigh filter and crucible or filtering crucible to the nearest 0.01 gm. (W_2).

2.1.9 Dry the fabric in an oven for 20 minutes minimum at 325 ± 10°F. minimum. Cool in a desiccator to room temperature and reweigh to the nearest 0.01 gm. (W_1).

2.1.10 Using an aspirator or vacuum pump, filter the contents of the second 250 ml. beaker through a filter and crucible or filtering crucible into a vacuum flask.

2.1.11 Rinse the crucible and filter or filtering crucible several times with acetone to remove all resin.

2.1.12 Dry the crucible and contents at 350 ± 10 degrees F. for 20 minutes minimum. Cool in a desiccator to room temperature and reweigh to the nearest 0.01 gm. (W_3).

2.1.13 Report the resin solids, filler content, and cloth content to the nearest 0.10 percent.

3. CALCULATIONS

3.1 Calculate the resin solids content, filler content and cloth content as follows:

3.1.1 Resin Solids content (percent) = $\frac{W-(W_1+W_4)}{W}$ (100)

3.1.2 Filler content (percent) = W_4/W (100)

3.1.3 Cloth content (percent) = W_1/W (100)

3.1.4 Where:

W = calculated devolatilized weight of prepreg specimen, gm.
W_1 = weight of fabric after resin and filler removal, gm.
W_2 = crucible and filter or filtering crucible weight, gm.
W_3 = filler + crucible and filter or filtering crucible weight, gm.
W_4 = $W_3 - W_2$ = filler weight, gm.

P-P-16 Resin Solids, Filler and Cloth Content of Prepregs

1. SCOPE

1.1 This method is applicable for determining the resin solids, filler and cloth content of prepregs having an epoxy or aldehyde phenolic resin matrix.

2. EQUIPMENT

2.1 Cutting board

2.2 250 ml. beakers

2.3 Membrane filtration funnel

2.4 Whatman 4.25 cm. GF/F filters

2.5 Whatman 4.7 cm. EPM 2000 filters

2.6 Analytical balance, sensitivity 0.0001 gm.

2.7 Aluminum weighing pans

2.8 Vacuum pump or aspirator

2.9 Circulating air oven, $163^{\circ} \pm 3^{\circ}C$

2.10 200-230 mesh filtering sieve

2.11 Acetone or DMF technical grade

2.12 Desiccator

2.13 Ultrasonic cleaner or hot plate

2.14 Teflon gasket 47 mm. (optional)

3. PROCEDURE

3.1 Precondition several pairs of filters (one Whatman EPM 2000 on the bottom, one Whatman GF/F on the top) by filtering approximately 25 ml. of acetone through the pair of filters. Place the filters in an aluminum weighing pan and dry @ $163^{\circ} + 3^{\circ}$ C.for 20 minutes or until dry. Preconditioned filters are to be stored in a desiccator.

3.2 Weigh a 2 1/4 ± 1/4" square specimen to the nearest 0.0001 gm. and place in a clean 250 ml. beaker. Alternative sample sizes (Specimen Weight) may be specified.

3.3 Ultrasonically extract the sample twice, for three minutes each time, in 50-75 ml. of acetone. Fresh solvent is to be used for each three minute extraction. Ultrasonic extraction must be performed under a fume hood. Stir the samples several times during extraction. Alternatively, the samples may be extracted for five minutes with 50-75 ml. of boiling DMF. (For prepreg weights over 6 gm. use 150 ml. of solvent.)

3.4 Weigh a preconditioned pair of filters and pan (from 3.1) to the nearest 0.0001 gm. and place the pair of filters, with the smaller filter on top, in the membrane filtration apparatus (W_2).

Note: If the filter sticks to the funnel base, a teflon gasket may be placed between the bottom filter and the stainless steel screen, and repeat.

3.5 Hold the specimen in the beaker with a glass stirring rod while decanting the liquid solution through a 200-230 mesh filtering sieve placed on top of the filtering funnel.

3.6 Rinse the remaining fabric twice with acetone to ensure complete extraction of the resin and filler. Complete rinsing is indicated by a clear acetone solution.

3.7 Add all rinses to the filtering funnel per Step 3.5.

3.8 Remove the fabric and place into a preweighed aluminum drying dish. Any fiber remaining on the filtering sieve is to be transferred to the aluminium weighing pan using a rubber policeman. Dry the fabric in an air circulating oven @ $163^{\circ} \pm 3^{\circ}$C. for 20 minutes or until dry. Cool in a desiccator to room temperature and weigh to the nearest 0.0001 gm. (W_1).

3.9 Filter the contents of the filteing funnel through the pair of filters. Rinse the sides of the filtering funnel with acetone to insure that all the filler is entrapped in the filters.

3.10 Remove the filters from the filtering funnel and place back into the original weighing pan (the top filter should be placed into the pan first and covered by the larger, bottom filter). Transfer any filler remaining on the bottom rim of the filtering funnel to the weighing pan with a small metal spatula. Close loosely around the edges and dry in an air circulating oven @ $163^{\circ} \pm 3^{\circ}$C. for 20 minutes or until dry. Cool to room temperature in a desciccator and reweigh to the nearest 0.0001 gm. (W_3).

4. CALCULATIONS

Devolatilized specimen wt.

$$W = \text{Specimen wt.} - \frac{(\%\ \text{Volatiles})(\text{Specimen Wt.})}{100}$$

$$\text{Resin Solids}\ \% = \frac{W - (W_1 + W_4)}{W} \times 100$$

$$\text{Filer Content}\ \% = W_4/W \times 100$$

$$\text{Cloth Content}\ \% = W_1/W \times 100$$

Where:

W = Calculated devolatilized specimen wt., gm.
W_1 = Weight of fabric after resin and filler removal, gm.
W_2 = Preconditioned filter weight, gm.
W_3 = Filter and filler weight, gm.
$W_4 = W_3 - W_2$ = filler weight, gm.

P-P-17 Sodium and Ash Content by Flame Emission for Prepregs

1. SCOPE

1.1 The procedure determines sodium and ash content of materials as applicable.

NOTE: Care should be taken not to contaminate the samples with sodium by touching with hands.

2. EQUIPMENT

2.1 Beakers

2.2 Analytical balance, sensitivity 0.0001 gm.

2.3 Muffle furnace, above 600 ± 15°C

2.4 Desiccator

2.5 Circulating air oven

2.6 Cermamic or platinum crucibles

2.7 250 ml. volumetric flasks

2.8 ASTM Type I 16.6 ohm resistivity

2.9 Concentrated hydrochloric acid

2.10 Hot plate

2.11 Perkins-Elmer atomic absorption instrument or equivalent and operators manual

3. PROCEDURE

3.1 Take specimens which weigh approximately 3 grams. Place test specimens in individual beakers and dry in an air circulating oven at 225° ± 5 degrees F. for a minimum of one hour.

3.2 Remove the specimens from the oven and cool in a desiccator.

3.3 Weigh approximately 2 grams of each cooled specimen to the nearest 0.1 mg. into separate tared ceramic or platinum crucibles (W_1).

3.4 Heat the crucibles in a muffle furnace at 600 ± 15 degrees C. for 16 to 18 hours or until constant weight is achieved.

3.5 Remove the crucibles containing the ashed specimens, place in a desiccator and cool.

3.6 Wash down the walls of each crucible with distilled water and add approximately 5 milliliters (ml.) concentrated hydrochloric acid to each.

3.7 Bring the acid mixture to a boil. Cool the crucibles. Transfer the contents of each to a 250 ml. volumetric flask and dilute to volume with distilled water.

3.8 Determine the sodium of each flask by flame emission at 589 nanometers (nm.) using the instrument manufactures operating instructions and a calibration curve for sodium which is linear between 0.1 and 2.5 micrograms per milliliter.

3.9 The calibration curve is made by measuring the absorbance on a minimum of 3 standard solutions, generally in the 1 ppm to 10 ppm liquid range. Either plot the absorbance vs. ppm liquid on graph paper or run a linear regression analysis. The correlation coefficient should be >.99 to assure linearity. Using the graph or linear fraction obtain the ppm of the liquid sample (Part 3.8) by inputting the absorbance of the sample. (Note: The absorbance of the sample should be bracketed by the standards).

4. CALCULATIONS

$$\text{ppm of Sol. Sample} = \frac{\text{ppm of liq. sample x dilution ratio}}{W_1}$$

$$\text{\% Ash} = W_2/W_1 \text{ x } 100\%$$

4.1 Report the results to 3 significant figures.

P-P-18 Compression, Residual Volatile, Dowel Shear, and Resin Content of Prepregs

1. MOLDING PROCEDURE

1.1 Material Designation Mx -4926

1.2 Specification STW5-3140

1.3 I. P. Card Number

2. SPECIMEN REQUIREMENTS - COMPRESSION

2.1 Mold one laminate 10" warp marked x 10" x 0.125" ± .025".

2.2 Pre-cut 10" warp direction and 10" fill direction.

2.3 Staging: 25 minutes @ 250°F ± 5°F.

2.4 Cure temperature: 325°F ± 10°F.

2.5 Cure Pressure: 1,000 ± 50 psi

2.6 Cure time: 120 ± 15 minutes.

2.7 Cool under pressure before removing from press.

3. INSPECTION REQUIREMENTS

3.1 Absence of blisters, voids, cracks, porosity, uneven surface delamination, or contamination.

4. SPECIMEN REQUIREMENTS - RESIDUAL VOLATILE

4.1 Mold 1 laminate 4 ± 1/4" x 4 ± 1/4" x 0.250 ± 0.050" thick.

4.2 Mark warp direction

4.3 Cure temperature 325°F ± 10°F.

4.4 Apply contact pressure for 30 ± 5 seconds, and dump pressure for two cycles before slowly applying maximum pressure. Do not prestage.

4.5 Maximum pressure, 1,000 ± 50 psi.

4.6 Cure time: 120 ± 15 minutes

4.7 Cool under pressure before removing.

5. SPECIMEN REQUIREMENTS - DOWEL SHEAR

5.1 Mold one 2" diameter by 2 1/2" thick billet.

5.2 Use approximately 180 plies.

5.3 Have all plies in same direction.

5.4 Staging: 25 minutes at 250°F ± 5°F.

5.5 Cure temperature: 325°F ± 10°F.

5.6 Hold at 325°F., cure temperature (325°F ± 10°F.)

5.7 Cure pressure: 1000 ± 50 psi.

5.8 Cure time: 120 ± 15 minutes.

5.9 Cool under pressure before removing from press.

6. SPECIMEN REQUIREMENTS - RESIN CONTENT

6.1 Cut 32 rectangles 1/2" x 5" per specimen (Pyrolysis bars).

6.2 Staging: 255 ± 5°F. for approximately 2-6 minutes.

6.3 Cure temperature: 325°F. ± 10°F.

6.4 Cure pressure: 1,000 ± 50 psi.

6.5 Cure time: 10 ± 1 minute

6.6 Pull Hot.

6.7 There should be no resin flash. If there is resin flash, repeat with a longer staging time.

P-P-19 Dowel Shear Strength of Fiber Reinforced Laminates

1. SCOPE

1.1 This method measures the shear strength of fiber reinforced plastic laminates based on Fed. Test Method 406, Method 1041a.

2. EQUIPMENT

2.1 Universal test machine - 10,000 lbs. capacity.

2.2 Shear jig per Fed. Test Method 406, Method 1041

3. PROCEDURE

3.1 Machine the specimen to 0.375 + 0.000/ -0.002 inch diameter by 1.125 inch minimum length.

3.2 Measure the diameter, d., of the test specimen, in.

3.3 Place the specimen in the 0.375 inch diameter three plate double shear jig, per above reference. Fig. 1.

3.4 Load the machine at 0.015 to 0.025 inch per minute.

3.5 Record the maximum load carried by the specimen, L.

4. CALCULATIONS

$$\text{Shear strength} = \frac{2L}{\frac{\text{Pi } d^2}{4}} \quad \text{or} \quad \frac{L}{2A} = \text{psi}$$

Where:

L= maximum load carried, lbs.
d = diameter, in.
A = cross sectional area, $\frac{\text{Pi } d^2}{4}$, inches^2

4.1 Report to 3 significant digits.

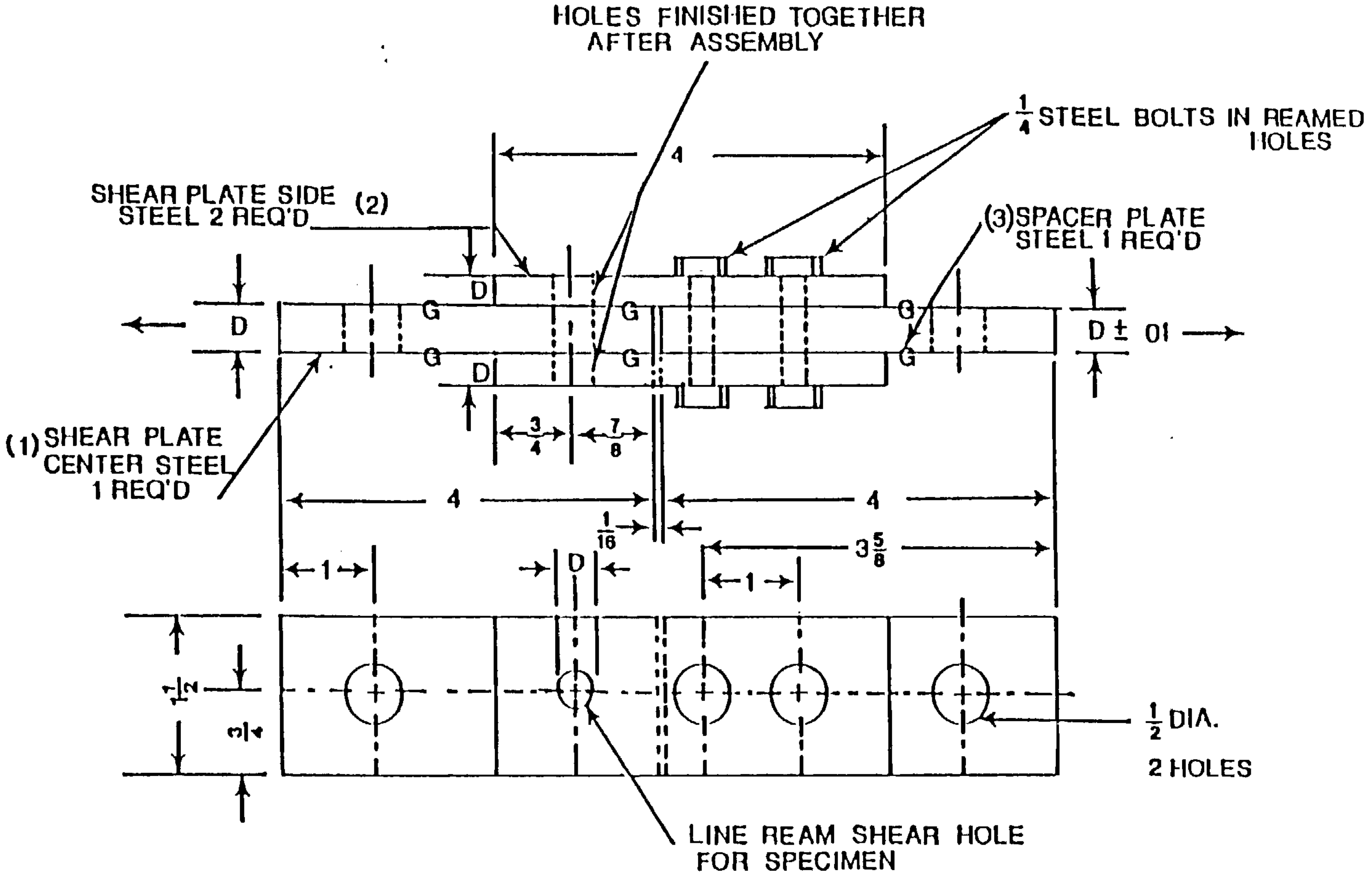

HOLES FINISHED TOGETHER AFTER ASSEMBLY
1/4 STEEL BOLTS IN REAMED HOLES
4
SHEAR PLATE SIDE STEEL 2 REQ'D (2)
(3)SPACER PLATE STEEL 1 REQ'D
D
G
G
D
G
G
D
G
G
D ± 01
(1)SHEAR PLATE CENTER STEEL 1 REQ'D
3/4
7/8
4
4
1/16
3 5/8
1
D
1
1 1/2
3/4
1/2 DIA.
2 HOLES
LINE REAM SHEAR HOLE FOR SPECIMEN

Figure 1

P-P-20 Compression Strength of Fiber Reinforced Laminates

1. SCOPE

1.1 This method measures the compression strength and modulus of fiber reinforced plastic laminations.

2. EQUIPMENT

2.1 Universal test machine of 10,000 lb. capacity, and operators manual.

2.2 Double cross specimen holders per ASTM D 695 mounted on machine L-shaped ASTM D 695. The holder(s) will allow access to strain gage(s) when modulus is taken.

2.3 Strain gages, such as Micro-Measures CEA-00-062UW-120 or equivalent, with amplification to the load chart.

3. PROCEDURE

$t = 0.125 \pm 0.025$ inches

$w_1 = 0.75 \pm 0.020$ inches

$w_2 = 0.50 \pm 0.015$ inches

$L = 3.13 \pm 0.030$ inches

$s = 1.5 \pm 0.030$ inches

$r = 1.5$ inches

3.1 t and w_2 are measured with 0.157 inches radius ball-flat micrometer.

3.2 The loading nose, platen and specimen ends (4 Planes) are all parallel to better than 0.0010 in. per 1.0 in. in any direction.

3.3 The sample is held between two I shaped pieces, Figure 1. These are supported with finger tight nuts and bolts on the inside through the sample consistent with number 2 above.

3.4 Environmentally conditioned specimens are held within ± 5°F for 10 + 1 min prior to dry tests and 2 ± 1 minute prior to wet tests.

3.5 The precision of the test is better than 1%. The strength is calculated as pounds/in.^{2}

$$\text{Strength} = C = \frac{P}{A}$$

$$\frac{C}{1000} = \text{ksi}$$

and is usually reported in ksi to three significant digits. The modulus

$$E = \frac{m}{A}$$

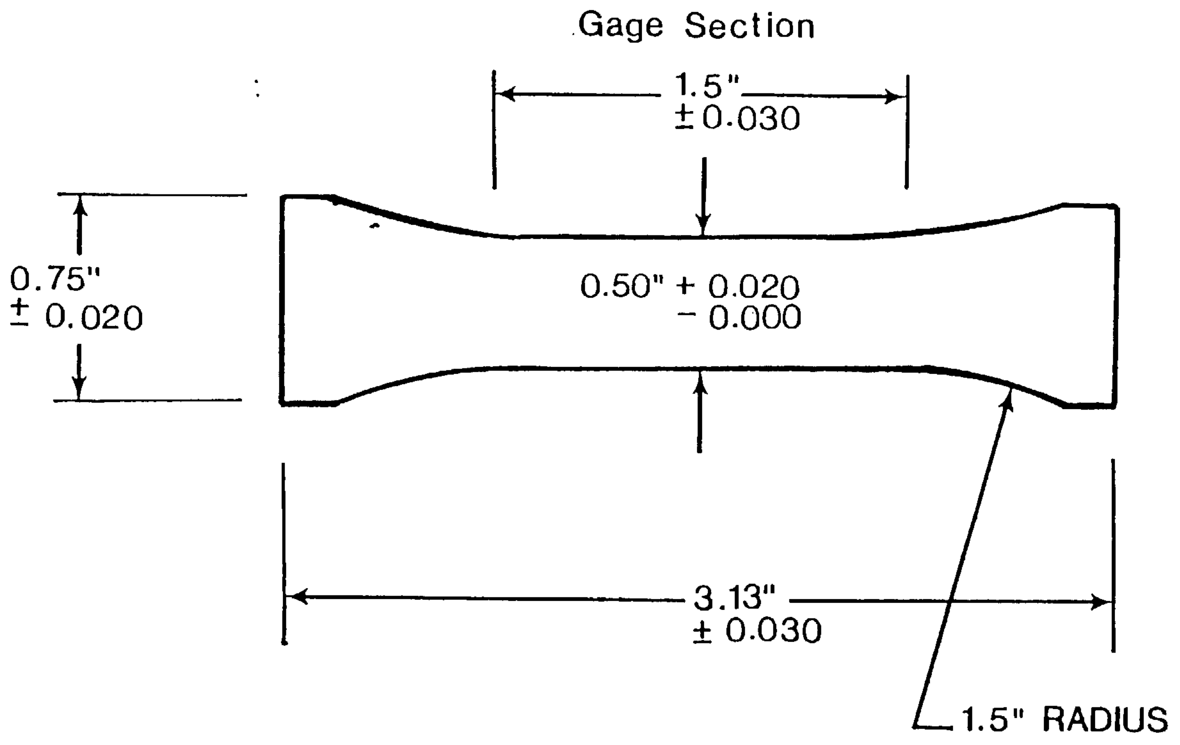
$$\frac{E}{1{,}000{,}000} = \text{Msi}$$

and is usually reported in Msi to three significant figures.

P = Maximum load (lb) carried by the specimen.

m = Load taken from the stress-strain curve from 20 to 40% of a typical P value.

A = Original minimum cross sectional area, in^2.



Test Specimen Thickness – 0.125 ± 0.025"

Figure 1

P-P-21 Resin Content of Prepregs Making Use of Factor "K"

1. SCOPE

1.1 This method is applicable for all carbon-reinforced aldehyde-phenolic compounds. The Fixed Carbon Content Factor, K, is determined for each individual compound and is a function of the amount of carbonaceous char formed under these controlled conditions. K is calculated from data compiled from the pyrolysis of samples of the molding material in which the resin solids have been determined gravimeterically through pyrolysis then they can be correlated with the known resin solids of the specimens.

Example: Gravimetric Determination 34.0%
Wt. Loss by Pyrolysis 16.504

$$K = \frac{34.0}{16.504} = 2.060$$

2. APPARATUS

2.1 Balance, analytical, precision not more than 0.0001 gm.

2.2 Lindberg Model 550035A (or equivalent) Tube Furnace at least 1500 ± 25°F.

2.3 Vacuum Pump

2.4 Clamps

2.5 Vacuum Tubing, Rubber, Water

2.6 Manometer

2.7 Vacuum Trap, Dewar Type

2.8 Type Drain Lines

2.9 Vycor Tube

2.10 Heavy Wall Vacuum Flasks

3. PROCEDURE

3.1 Wipe specimens clean using acetone and allow to dry in air 30 minutes minimum before testing.

3.2 Record each specimen weight to nearest 0.0001 gm. (W_1).

3.3 Place test specimen in Vycor tube, place tube into the tube furnace and tightly attach tube to test apparatus.

3.4 Start vacuum pump and evacuate system to 3 inches of mercury absolute maximum. Check for air leaks by clamping off hose to vacuum pump and noting manometer, leak rate must be less than 0.20 inches of mercury per minute. Recheck joints until this level is achieved.

3.5 With vacuum on and pressure stabilized at 3 inches of mercury maximum, place the Vycor Tube in the preheated tube furnace at 1500 ± 25°F.

3.6 Continue pyrolysis for approximately 30 minutes or until manometer or gauge reading is same reading as it was before pyrolysis, whichever is longer.

3.7 When pyrolysis is complete, turn off furnace, remove tube from furnace, and cool under vacuum until specimen is at room temperature.

3.8 When specimen is cool, turn off vacuum and carefully let air into system. When pressure equilibrium is reached, remove specimen from test tube and weigh to nearest 0.0001 gm. (W_2).

4. CALCULATE

Calculate resin content as follows:

$$\text{Resin content, percent} = \frac{W_1 - W_2}{W_1} \times 100 \times K$$

Where:

W_2 = Weight of specimen after pyrolysis, gm.
W_1 = Weight of specimen before pyrolysis, gm.
K = Fixed carbon content factor, to be supplied by prepreg manufacturer.

P-P-22 Specific Gravity and Density of Prepregs

1. SCOPE

1.1 This method covers the determination of the specific gravity and density of solid plastics by displacement of liquid and determination of the change in weight. Based on ASTM D 792, Method A.

2. EQUIPMENT

2.1 Analytical balance of 0.1 mg. accuracy

2.2 Sample suspension device

2.3 Alcohol, wetting agent, or equivalent

2.4 Distilled water

2.5 Copper wire

3. PROCEDURE

3.1 Each sample of 1/2 to 1 1/2 gm. is weighed. It is called W_1. The precision here and in the following measurement is 0.1 mg. The temperature is 23 ± 2°C.

3.2 A piece of thin, approximately 0.01 in., copper wire is weighed with as much of it immersed in the water as will be in 3.4. If the specific gravity of the sample is less than 1 and, if it floats, a sinker is weighed with the wire. This weight is W_3.

3.3 Pre-wet the sample suspended by the wire in lab. alcohol and then in an intermediate distilled water bath.

3.4 W_2 is the weight of the sample completely immersed in a third bath of distilled water.

4. CALCULATIONS

$$\text{Specific gravity} = W_1/\ (W_1 - (W_2 - W_3))$$

$$\text{Density} = 0.9975 \times \text{specific gravity above } (\text{gm.}/\text{cm}^3.)$$

4.1 The density is the specific gravity as measured above times the density of water at this temperature.

P-P-23 Residual Volatile Content of Molded Articles

1. SCOPE

1.1 This method is used to determine the volatile content remaining trapped in a molded article. This volatile content may consist of entrapped water of condensation, unreacted phenol, residual solvent, plasticizers, or volatile mold release agents.

2. EQUIPMENT

2.1 Balance accurate to 0.001 gm.

2.2 Desiccator

2.3 Air circulating oven, 163 ± 5°C at least

3. PROCEDURE

3.1 Cut a specimen 1.000 ± 0.050" by 1.000 ± 0.050" from the center 2" x 2" section of the test panel. Wipe the specimen clean with MEK and air dry 20 minutes minimum.

3.2 Place the specimen in a desiccator for 18 hours minimum.

3.3 Weigh the specimen, (W_1).

3.4 Place the specimen in the oven for 24 hours minimum, at 163 ± 5°C.

3.5 Remove the specimen from the oven and cool in a desiccator for approximately 30 minutes or until room temperature is reached.

3.6 Reweigh the specimen, (W_2).

4. CALCULATIONS

$$\text{percent volatile} = \frac{W_1 - W_2}{W_1} \times 100\%$$

Where:

W_1 = initial weight, gm.
W_2 = final weight, gm.

5. REPORT

5.1 Report the individual and average results to the nearest 0.1%, as percent residual volatile.

P-P-24 Infra-Red Spectrum of Liquid and Solid Resins and Prepregs

1. SCOPE

1.1 This method is used for determination of infrared spectrum of liquid and solid resins or prepreg.

2. APPARATUS

2.1 Beckman 4250, Beckman Acculab 8 or equivalent and operators manual.

2.2 Sodium chloride disk.

2.3 Solvent: Spectrograde acetone, THF (Tetrahydrofuran) or methylene chloride.

3. PROCEDURE

3.1 Place a small amount of material to be tested in a 50-ml. beaker. Add about 15 ml. of spectroquality acetone and stir with a glass stirring rod to dissolve. If the material doesn't dissolve in acetone, spectroquality THF or methylene chloride may be used.

3.2 Place several drops of the solution on the sodium chloride crystal and allow to dry at room temperature.

3.3 Zero the I.R. beam as per the instrument manual.

3.4 Make a quick scan of the region from 2000 cm^{-1}. to 1000 cm^{-1}. When the transmittance is between 15 and 25 percent at most intense peak the right concentration is on the crystal. Twenty percent is the ideal transmittance.

3.5 Start the scan at 90% transmittance. Scan the sample in the infrared from 2.5 to 16 microns.

3.6 Do any necessary calculations.

P-P-25 Residual Volatile Content in Molded Articles

1. SCOPE

1.1 This method is used to determine the volatile content remaining trapped in a molded article. This volatile may consist of entrapped water of condensation, unreacted phenol, residual solvent, plasticizers, volatile mold release agents or a combination thereof.

2. TEST SPECIMENS

2.1 The test specimen shall consist of a 1.000 + 0.050" x 1.000 ± .050" x 0.125 + 0.025". Three specimens shall be tested for each determination. Direct comparison of values between samples shall not be made unless all samples so compared do not vary by more than ± 10% from a nominal given thickness.

3. APPARATUS

3.1 <u>Balance</u>. An accurate analytical balance capable of weighing to the nearest milligram shall be used.

3.2 <u>Oven</u>. A thermostatically controlled air circulating oven capable of maintaining the test temperature within ± 2° C. shall be used.

4. PROCEDURE

4.1 <u>Conditioning specimens</u>. Specimens shall be conditioned in a desiccator over silica gel for a period of 18 hours prior to weighing.

4.2 <u>Initial Weight</u>. After the specimens have been conditioned they shall be individually weighed on an analytical balance to the nearest milligram. This weight is to be designated as W_1.

4.3 <u>Devolatilization</u>. After the specimens have been weighed they shall be placed in an air circulating oven maintained at 163 + 2° C. for a period of 4 hours + 5 min. After this time the specimens shall be removed and placed in a desiccator over silica gel and allowed to cool to ambient temperature. After reaching ambient temperature the specimens shall be re-weighed to the nearest milligram and this weight designated as W_2.

5. CALCULATIONS

5.1 Percent Weight Loss

The volatile shall be expressed as a percentage weight loss as compared to the original specimen weight (W_1).

$$\text{Percent weight loss} = \frac{W_1 - W_2}{W_1} \times 100$$

Where:

W_1 = the initial weight of the test specimen, gm.
W_2 = the final weight of the test specimen, gm.

5.2 The values obtained for three specimens shall be averaged and reported as weight loss for the sample tested.

P-P-26 Thermogravimetric Analysis (a) Chemical Test Methods

1. SCOPE

Chemical Test Method by:
Thermogravimetric Analysis

2. **MATERIAL** CCA-3 Fabric (512°C. Test)

3. **EQUIPMENT** Setting

3.1 Heater Control: Load
Zero - + 20
Range - + 10
TGS - 2

3.2 Auto Balance:
Power - on
Filter - Lo
Tare - Pointer at the middle of green line
Recorder Range - 100
Recorder Mode - Short

3.3 Recorder:
Pen 2 - (Zero pen by holding zero switch down; adjusting knob until pen is "0" on the right hand side of the chart)
Range - 1 volt
Power - Servo
Pen 1 (Zero by following instruction of Pen 2)
Range - 1 mv.
Polarity - (-)
Chart Speed - 5 mm./min.

3.4 Differential Scanning Calorimeter:
Power - On
Lower - Unit - 100°C
Upper Limit - 512 ± 10°C
Cooling Range - 160°C/min.
Heating Rate - 160°C/min.

3.5 Air flow rate - 80 ml./min. (Zero gas air)

3.6 Operators manuals.

4. TEST METHOD

4.1 Cut three samples 1/4" x 1/4" of fabric

4.2 Disengage spring latch of balance cover to release gas pressure

4.3 Loosen screw, then lower down furnace

4.4 Swing loading platform underneath platinum pan

4.5 Place samples on pan

4.6 Raise furnace carefully, tighten screw, and engage spring latch (Note: Bubbles should appear on the outlet-tubing that is immersed in water; if not, turn balance cover until bubbles come out)

4.7 Turn heater control to heat and recorder mode to Mg

4.8 Make mark on chart by lowering Pen-1 and advancing chart manually and raising pen again (reading will be 1 mg per small division, this will give the weight of sample).

4.9 Turn recorder mode to % sample

4.10 Adjust Pen-1 until it reads 100 on the left hand of chart by turning the "% adjust knob"

4.11 Lower both pens, power knob - chart, push "heat" button on DSC

4.12 Pen-2 should level off at 512-514°C. mark, otherwise adjust upper limit and push heat until 512-514°C. mark is attained on chart

4.13 When Pen-2 reaches 512-514°C, start timer for 20 minutes

4.14 Push "cool" button after 20 minutes, raise both pens, power knob-servo, and recorder mode-short

5. CALCULATIONS

5.1 Read % loss directly from chart (1% loss/small division on chart) using Pen-1

Note: Lower furnace only when temperature drops to 150°C. or less.

P-P-27 Thermogravimetric Analysis (b)

1. SCOPE

Chemical Test Method By:
Thermogravimetric Analysis

2. MATERIAL CCA-3 Fabric (1000°C. Test)

3. EQUIPMENT-Setting

3.1 Heater Control: Load
Zero - + 20
Range - + 10
TGS - 2

3.2 Auto Balance:
Power - On
Filter - Lo
Tare - Pointer at the middle of green line
Recorder Range - 100
Recorder Mode - Short

3.3 Recorder:
Pen 2 (Zero pen by holding zero switch down; adjusting knob until pen is "0" on right hand side of chart)

Range 1 volt
Polarity (-)
Pen 1 (Zero by following instruction of Pen 2)
Range No mv.
Polarity (-)
Chart Speed 5mm./min.

3.4 Differential Scanning Calorimeter:
Power On
Lower Unit 1000°C.
Upper Limit 1000°C.
Cooling Range 40°C./min.
Heating Range 20°C./min.

3.5 Air flow rate 80 ml./min. (zero gas air)

3.6 Operators manuals.

4. TEST METHOD

4.1 Cut three samples of fabric 1/4" x 1/4".

4.2 Disengage spring latch of balance cover to release gas pressure.

4.3 Loosen screw, then lower down furnace.

4.4 Swing loading platform underneath platinum pan.

4.5 Place samples on pan.

4.6 Raise furnace carefully, tighten screw, and engage spring latch.

NOTE: Bubbles should appear on the outlet-tubing that is immersed in water if not, turn balance cover until bubbles come out.

4.7 Turn heater control to heat and recorder mode to Mg.

4.8 Make mark on chart by lowering Pen-1 and advancing chart manually and raising pen again (reading will be 1 mg per small division, this will give the weight of sample).

4.9 Turn recorder mode to % sample.

4.10 Adjust Pen-1 until it reads 100 on the left hand of the chart by turning the "% adjust knob".

4.11 Lower both pens, power knob- chart, push "heat" button on DSC.

4.12 Pen-2 should level off at 1000°C. mark, otherwise adjust upper limit and push heat until 1000°C. mark is attained on chart.

4.13 Push "cool" button after 1000°C. is reached, raise both pens, power knob servo, and recorder mode-short.

5 CALCULATIONS

5.1 Read % loss directly from chart (1% loss/small division on chart using Pen-1

NOTE: Lower furnace only when temperature drops to 150°C. or less.

P-P-28 Thermogravimetric Analysis

1. SCOPE

1.1 This procedure describes the method for conducting thermogravimetric analysis of resins and prepregs.

2. EQUIPMENT AND MATERIALS

2.1 Perkin-Elmer Model TGS-2 Thermoanalyzer or equivalent

2.2 Perkin-Elmer DSC-2 Programer, or equivalent

2.3 Appropriate operators manuals.

3. SAMPLING

3.1 Obtain a small (1 oz.) representative sample

4. PROCEDURE

4.1 <u>Balance Controls</u>

4.1.1 Power - on

4.1.2 Filter - lo

4.1.3 Recorder Mode - short (see sec. 7D1,6 and 7D2,1 in operators manual)

4.1.4 Recorder Range - 10 mg.

4.1.5 Weight Suppression switch - 100 mg.

4.1.6 Weight Suppression thumb steel dial - 00.000

4.2 <u>Use DSC-2 as the TG Programmer</u>

4.2.1 Sample holders empty

4.2.2 Temperature RANGE Calibration - as calibrated

4.2.3 Temperature ZERO Calibration - as calibrated

4.2.4 Lower LIMIT - 350^{o}K. (or as required)

4.2.5 Cooling RATE - 160^{o}/min.

4.3 <u>Heater Control Unit</u>

4.3.1 ZERO and RANGE Setting - as determined from calibration procedure (or as indicated on calibration card 993-9235)

4.3.2 Power - HEAT

4.3.3 ^{o}C. - ^{o}K. switch (on back panel) ^{o}K.

4.4 Recorder

4.4.1 CHART SPEED slow (e.g., 5 mm/min.) change to faster chart speed when temperature is started

4.4.2 Weight Pen: RANGE - 20 mv.; right-hand zero

4.4.3 Second Pen: Thermocouple: RANGE - 1 volt, right-hand zero; or FDC: RANGE - 10 mv., left-hand zero

P-P-29 Resin Content by Means of Pyrolysis of Prepregs

1. SCOPE

1.1 Resin content shall be determined in accordance with the following.

2. PROCEDURE

2.1 Prepare test specimens as follows:

2.1.1 Cut 32 rectangles (1/2 inch by 5 inches nominal) from a prepreg sample.

2.1.2 Stage the 32 rectangles at 255 ± 5°F. to reduce flow.

Note: Staging time is dependent on the resin system and flow and must be determined from experience.

2.1.3 Using the 32 rectangles, mold a bar 1/2 inch by 1/2 inch by 5 inches.

2.1.4 Cure the specimen at 325 ± 10°F.and 1000 ± 10 psi. for 10 ± 1 minutes. If any resin flashes, repeat and use longer staging time.

2.1.5 Cut the bar into approximately three equal lengths to form three test specimens.

2.2 Wipe the specimen clean using methyl ethyl ketone (MEK) and allow to air dry for 20 minutes minimum before testing.

2.3 Record specimen weight to the nearest 0.001 gram (W).

2.4 Place the test specimen in the Vycor test tube and tightly attach the tube to the test apparatus.

2.5 Start the vacuum pump and evacuate the system to 10 ± 5 mm. of mercury (Hg) absolute. Check for air leaks by clamping off the hose to the vacuum pump and noting the manometer. The leak rate must be less than five millimeters (mm.) (0.02 in.) per minute. Recheck joints until this level is achieved.

2.6 With the vacuum on and the pressure stabilized at 10 ± 5 mm. Hg, place the Fisher burner under the specimen. Adjust the flame area to cover the sample completely, as in Fig. 1.

NOTE: A tube furnace maintained at 1500 ± 25°F.may be used in place of the Fisher burners.

2.7 Continue pyrolysis for approximately 30 minutes or until the manometer or gauge reading is the same reading as it was prior to pyrolysis, whichever is longer.

NOTE: If residue collects in the Vycor test tube, place the Fisher burners under those areas until the burnout is complete.

2.8 When pyrolysis is complete, turn off burners and cool under vacuum until specimen is at room temperature (touch Vycor test tube just under test specimen very carefully).

2.9 When the specimen is cool, turn off vacuum and carefully let air into the system. When pressure equilibrium is reached, remove specimen from test tube and weigh to the nearest 0.001 gram (W_1).

2.10 Calculate dry resin content as follows:

Dry resin content, % = $(1.00 - W_1/W)100(K)$

Where:

W_1 = weight of specimen after pyrolysis, gm.
W = weight of the specimen prior to pyrolysis,gm.
K = a constant as shown below

K Factors

U. S. Polymeric FM 5055	Fiberite MX 4926
2.008	2.060

Note: These constants are unique for each material.

2.11 Report the dry resin content to the nearest 0.1 percent.

2.12 Limits (percent dry resin content by weight)

Minimum	30.0
Maximum	38.5

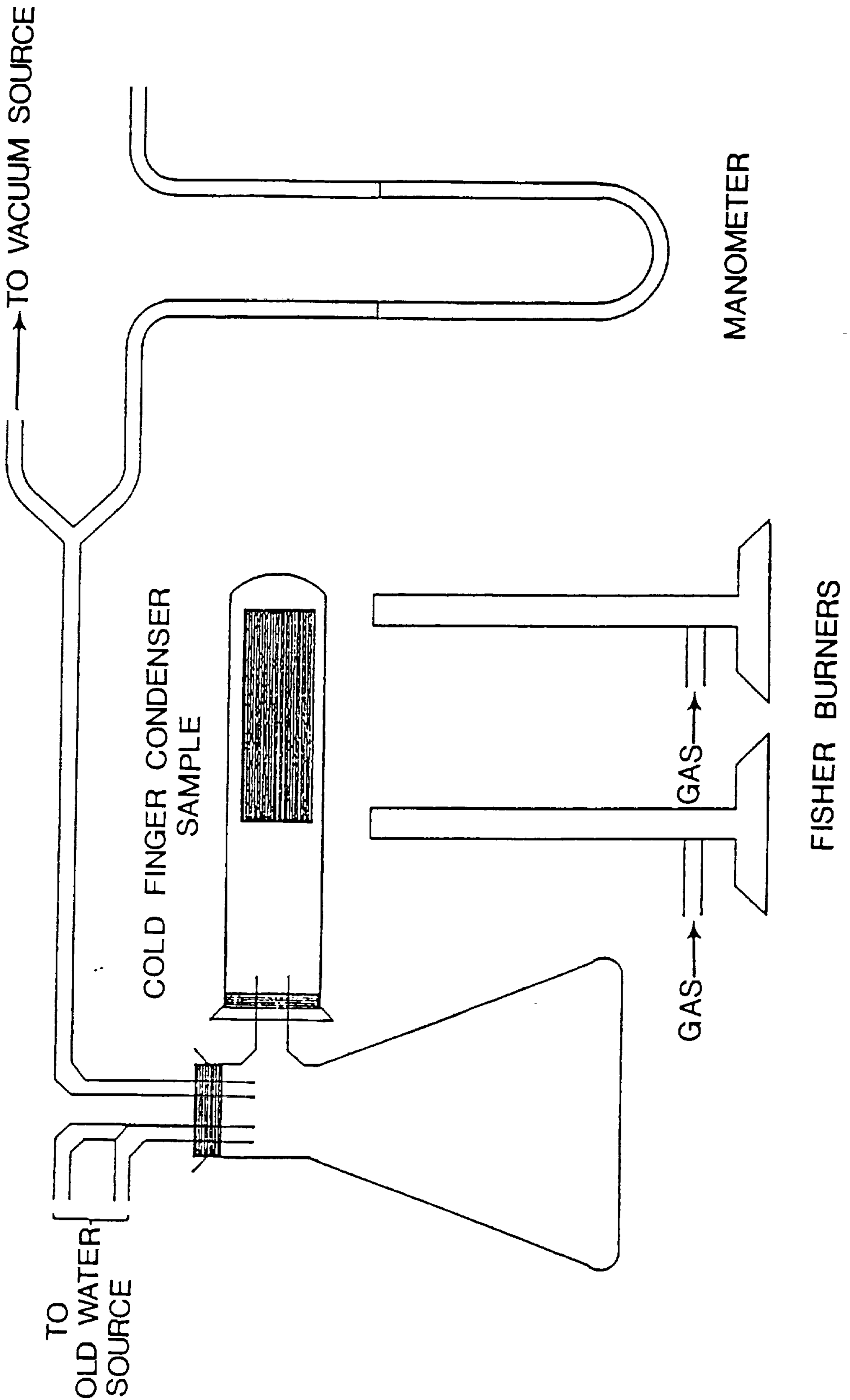

TO VACUUM SOURCE
MANOMETER
COLD FINGER CONDENSER
SAMPLE
GAS
GAS
FISHER BURNERS
TO
COLD WATER
SOURCE

Fig. 1

P-P-30 Tack Temperature of Prepregs by Lap Shear

1. SCOPE

1.1 Scope. This method describes a procedure for establishing the minimum tack temperature of prepreg materials by lap shear where the specimen overlap is under a specified pressure while heating. This method does not give a shear strength.

1.2 Purpose. The purpose of this method is to confirm blocking temperature of pattern grade material.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

2.1.1 Asbestos laminates, 3" x 3"

2.1.2 Hot plate, variable temperature control

2.1.3 Metal block, 2" x 2" x 1/4", weight 125 ± 5 grams.

2.1.4 Press device, capable of applying 50 ± 2 lbs. of force

2.1.5 Stop watch and/or electric timer

2.1.6 Weight, 15 lbs. (or other specified weight)

2.2 Materials.

2.2.1 Cellophane

3. SAMPLING

3.1 The sample should be large enough to provide a sufficient number of 1" x 3" specimens to determine minimum tack temperature . Unless otherwise specified, specimens shall be cut so as to optimize sample size and shall be in either the warp or fill direction.

4. PROCEDURE

4.1 Preparation of test specimen.

4.1.1 Preconditioning hot plate. Unless otherwise specified, preheat the hot plate to the specified temperature (±3°F.). Place the 2" x 2" x 1/4" block and the 15-pound (or other specified) weight on the hot plate during this preheat time.

4.1.2 Heating test specimen. Take two 1" x 3" specimens and form a one-inch overlap, placing warp side to fill side. Place between a fold of cellophane. Place the cellophane-wrapped specimen on the hot plate, place the 2" x 2" x 1/4" metal block on the overlap, and place the 15 pound (or other specified) weight on the 2" x 2" x 1/4" metal block. Allow the specimen to heat for 30 ± 2 seconds. Remove the weight and metal block and take specimen from the hot plate.

4.1.3 Specimen does not bond. If the specimen has not bonded, confirm this condition with an additional specimen before changing temperature.

4.1.4 Specimen is bonded. As quickly and delicately as possible, (5 seconds maximum), place the overlapped area between the platens of a room temperature press. The platens of the press shall be covered with asbestos laminates which are covered with cellophane. Unless otherwise specified, apply 50 ± 2 lbs. until the specimen is cool (about 1 to 2 minutes).

4.2 Testing specimen. Clamp one end of the specimen in jaws of spring load dial scale. Load other end of the specimen at a constant rate thus forming a lap shear test. Continue loading until shear or break occurs.

4.3 Minimum tack temperature.

4.3.1 Specimen supports weight. If the specimen supports a measurable load (see 6.1), decrease temperature (usually at 10°F. intervals) until specimen will not support a measurable load.

4.3.2 Specimen does not support weight. If the specimen does not support a measurable load, continue testing, increasing the hot plate temperature (usually at 10°F. intervals) until the specimen will support a measurable load. Report load in pounds. Test two additional specimens at this temperature.

5. CALCULATIONS AND REPORTING

5.1 Reporting.

5.1.1 Report the average load in pounds of three specimens at minimum tack temperature.

5.1.2 Report the minimum tack temperature at which a measurable load is supported. The minimum tack temperature must be established by a minimum of three specimens.

NOTES: A measurable load for the purpose of this test shall not be less than three pounds. The measurable load shall be three pounds.

P-P-31 Wet and Dry Flow by Flash Removal or Punched Disc

1. SCOPE

1.1 This is a method for the determination of flow by either flash removal or punched disc. Both wet (prepreg tested as is) and dry (devolatilized) flows are incorporated.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

2.1.1 Laboratory press, hydraulic, 500 ± 5 psi, 350 ± 5°F

2.1.2 Analytical balance, 0.0001 gm. sensitivity

2.1.3 Caul plates, stainless steel, 1/16" thick, 6" x 6" (optional)

2.1.4 Steel templates, 2" x 2" and 4" x 4"

2.1.5 Laboratory knife

2.1.6 Punch press with cutting die, 2.500" diameter

2.1.7 Calipers, graduated in 0.01 inch

2.2 Materials.

2.2.1 Release films, cellophane, mylar, aluminum foil, teflon, coated fabric.

2.2.2 TX - 1040 light weight teflon coated glass fabric, or equivalent

2.2.3 Mochburg Fabric, Grade CW-1850 (West Coast Paper Co., Seattle, Wash.) or equivalent.

3. SAMPLING

3.1 Sample size. The sample shall be large enough so that the required number of 4" x 4" plies may be cut. Unless otherwise specified, cut specimens on the bias. The total specimen weight should be 25 to 30 grams, except in the case of the high modulus graphite tape. The following table will serve as a guide in determining the number of plies per specimen:

MATERIAL GLASS (TYPE)	NUMBER OF PLIES
108	16
120	10
128	8
143	6
341	6
181	6
182	4
183	3
184	3

MATERIAL	NUMBER OF PLIES
Asbestos	10
Carbon	4
Graphite	4
High Modulus Graphite (tape)	3

3.2 Number of determinations. Conduct one determination, on each sample, unless otherwise specified.

4. PROCEDURE

4.1 Temperature and pressure for resin type. Each resin type has unique flow properties which must be tested at temperatures and pressures peculiar to that type. Unless otherwise specified, use the following standard pressures and temperatures:

RESIN TYPE	PRESSURE, psi	TEMP, °F.
Phenolics		
Press Grade	500	325
Vacuum Grade	15	325
All Other	150	325
Epoxies		
Unidirectional Tape	15	325
Vacuum Grade	15	275
All Other	50	275
Polyesters		
Press Grade	50	275
Vacuum Grade	15	275
Silicones	50	350
Other Resins	Must be specifically designated	
All resin system being utilized with the high modulus graphite tapes	200	300

NOTE: Pressure for epoxy systems. Use caution in selecting pressure and temperature for epoxy systems as a wide variety of applications require varied conditions.

4.1.1 Tolerances:

4.1.1 Pressure shall have a tolerance of ± 5% but with a maximum of ± 5 psi.

4.1.2 Temperature shall have a tolerance of ± 5°F.

4.2 Lay-up for tapes. The following lay-up procedure shall be used for tapes:

4.2.1 Lay-up for tapes.

4.2.1.1 Using strips about 4" long, lay up four (4) plies at least 4" square. Lay up the plies so that the strips of each successive ply are orientated 90° to the strips in the preceding ply. Cut flow specimens using 4" x 4" template. Do not cut specimens on the bias unless so designated.

4.2.2 Unidirectional tapes other than the high modulus graphite.

4.2.2.1 Cut enough strips to make up four (4) plies 10" x 10". Lay up the plies so that the fibers of each successive ply are orientated 90° to the preceding ply.

4.2.2.2 Using a 4" x 4" template, cut specimen on the bias. Weigh each specimen to the nearest 0.01 gm.

4.2.2.3 Sandwich the 4-ply specimen with two (2) 6" x 6"plies of 181 glass fabric on each side and place between cellophane release film. Proceed to 4.3.2.

4.2.3 High modulus graphite unidirectional tapes.

4.2.3.1 Cut three plies, 4" x 4" evenly spaced across the width of the material and orient them 0o, 90o, 0o. Weigh the layup to the nearest 0.01 gm.

4.2.3.2 Sandwich the specimen with one 6" x 6" ply of TX-1040 release fabric, followed by two (2) 6" x 6" plies of Mochburg bleeder fabric (each side). Place the entire layup between cellophane or teflon release film. Proceed to 4.3.2.

4.3 Pressing Specimens

4.3.1 If wet flow is to be determined, weigh the proper number of specimens to 0.01 gm. or as specified. This weight is not necessary if dry flow is all that is desired.

NOTE: Weighing. If greater accuracy is desired, weigh to 0.001 gm. In general, if the flow is 5% or less, weigh to nearest 0.0001 gm.

4.3.2 Properly align the specimens between release film. Position in the center of a press, reconditioned to the specified temperature, and close to the specified pressure as rapidly as possible.

NOTE: Caul plates. Caul plates will not normally be used unless specifically designated.

4.3.3 The time in the press shall be 10 minutes minimum but otherwise 3±1 minutes greater than the gel time.

4.3.4 Release pressure at end of time interval and immediately remove the release films if used. If dry flow is to be determined, use care in removing the release films so that none of the flash is lost.

4.4 Weighing the flash removal.

4.4.1 If dry flow is to be determined, weigh the laminate with flash to 0.01 gm. or as specified.

4.4.2 Scrape off the flash down to the original size with a dull knife, being careful to avoid removing any reinforcement from original dimensions.

4.4.3 Reweigh the laminate to 0.01 gm. or as specified.

4.5 Punched disc. If punched disc flow is to be determined, proceed as follows:

4.5.1 Use a punch press to remove a 2 1/2 inch diameter disc from the center of the laminate.

4.5.2 Weigh the disc to 0.001 gm. and measure the diameter to 0.01 inch.

5. CALCULATIONS AND REPORTING

5.1 Calculations. Calculate flow as follows:

5.1.1 Flow by flash removal.

5.1.1.1 Wet flow. Calculate wet flow as follows:

$$\text{Flow, Wt. \%} = \frac{W_1 - W_3}{W_1} \times 100$$

where:

W_1 = Original weight, gm.
W_2 = Weight of laminate after pressing but before flash removal, gm.
W_3 = Weight after flash removal, gm.

5.1.1.2 Dry Flow. Calculate dry flow as follows:

$$\text{Flow, Wt.\%} = \frac{W_2 - W_3}{W_2} \times 100$$

where symbols are as specified in 5.1.1.1

5.1.2 Flow by punched disc.

5.1.2.1 Wet flow. Calculate wet flow as follows:

$$\text{Flow, Wt. \%} = \frac{W_1 - RW_4}{W_1} \times 100$$

where:

W_1 = Original weight, gm.
W_4 = Weight of 2.50 inch disc, gm.
R = Ratio of area of 4" x 4" laminate to area of 2.50 inch disc

NOTE: Punched disc flow. Some specifications require flow ito be calculated using dry weight in the denominators. This is not a standard calculation and cannot be defined as a wet or dry flow; however, the calculation is given below for reference:

$$\text{Flow} = \frac{W_1 - RW_4}{RW_4} \times 100$$

NOTE: Place 6.4 here, but without the number.

5.2 Reporting. Unless otherwise specified, report flow to three significant figures. Test results should be considered suspect if the range for triplicate determinations exceeds 2.5% for flows greater than 10%. For lower flows, tested at 150 psi or greater, the range of triplicate flows should not exceed 1.5%.

P-P-32 Viscosity - RDS for Prepregs, by Parallel Disc

1. SCOPE

1.1 Prepreg Viscosity by Parallel Plate

1.1.2 Run Parameters (Operators manual)

1.1.2.1 Temperature to be as specified in Spec. of QPL

1.1.2.2 Rate = 10 rad./sec

1.1.2.3 % Strain = 10% strain for starting temperature 30°C. to 70°C. 50% strain for starting temperature in excess of 70°C.

1.1.2.4 Mode: Cure Sweep

Total Time	140 minutes
°C/min.	2.0°C minutes
Time/test	2.0 minutes/Test

1.1.2.5 Test: Parallel Plate: 25mm. for aldehyde-phenolic, 50mm. for epoxy/polyimides/phenolic

1.1.2.6 Geometry: Gap- see below
Radius - see 1.1.2.5

Gap setting must be made after thermal equilibration of material and plates. After equilibration, set force normal to 50% full scale and read gap setting. Use 50% force nominal gap setting for all calculations.

1.1.2.7

Plot		Epoxy/Polyimide	Phenolic
X = axis	min.	0	0
	max.	140	280
Y = axis	min.	1EO	1E2
	max.	1E5	1E7

Print: n^*, n^1, n, time, temperature, rate
Paper: Rheometerics 5 cycle x 7 inch Semi-log #4662-10 K&E Paper

1.2 Sample Insertion

1.2.1 Set desired temperature, place prepreg specimen on lower parallel plate and return assembly to test machine. Set oven temperature to about 70°C. in order to allow the prepreg sample to become pliable. Set gap as in 1.1.2.6.

1.3 Testing Initiate the test by the following sequence:

Temperature: See 1.1.2.3
Mode: Cure Mode
Test: Parallel Plate
% Strain: See 1.1.2.3
Rate: 10 rad./sec.
Start

1.4 Shut down after test:

Press reset
Remove plates and clean in acetone
Shut down power to heat and torque motor

F-1 Density of Cured Material

1. SCOPE

1.1 The density of the cured material shall be determined in accordance with the following.

2. PROCEDURE

2.1 Cut sufficient plies to produce a test panel 0.125 ± 0.025 by 4 inches (minimum) by 5 inches (minimum), mark the warp direction and cure panel as specified. When testing tape widths which are 4.0 inches or less prepare a test panel whose width is equal to that of the tape. The outer 0.100 inch of the panel shall not be used for preparation of the specimens.

2.2 Cut a specimen 1-inch by 1-inch by panel thickness at random from the test panel. Wipe the specimen clean using methyl ethyl ketone (MEK) and allow to air dry 20 minutes, minimum, before testing.

2.3 Weigh and record the weight of the specimen in air to the nearest 0.1 mg. (W_1).

2.4 Weigh the specimen in water at 73 ± 3 °F. and record the weight to the nearest 0.1 mg. using a wire and sinker if required (W_2).

NOTE: Weigh specimen in a suitable liquid other than water if it is affected by water or is lighter than water.

2.5 Calculate density using water as follows:

$$\text{Density (gm./cc.)} = \frac{0.9975\, W_1}{(W_1 + W_3 - W_2)}$$

Where:

W_1 = weight of specimen in air, gm.
W_2 = weight of specimen, wire and sinker suspended in water, gm.
W_3 = weight of wire and sinker suspended in water to the same depth as in the W2 determination to the nearest 0.1 mg.

2.6 Calculate density using other liquids as follows:

$$\text{Density (gm./cc.)} = \frac{0.9975\, W_1\, S}{(W_1 + W_3 - W_2)}$$

Where:

W_1 = weight of specimen in air, gm.
W_2 = weight of specimen and wire suspended in the liquid, gm.
W_3 = weight of wire suspended in the liquid to the same depth as in the W_2 determination, gm.
S = predetermined specific gravity of the liquid at 73 ± 3 °F.

2.7 Report the density to the nearest 0.01 gm./cc.

2.8 Limits (gm./cc)

Minimum	1.40
Maximum	1.52

F-2 Residual Volatile Content of Cured Material

1. SCOPE

1.1 The residual volatile content of the cured material shall be determined in accordance with the following:

2. PROCEDURE

2.1 Cut sufficient plies to produce a test panel 0.250 ± 0.050 thickness by 4 ± 0.125 by 4 ± 0.125 inches. Cure the panel as specified with the following exceptions.

2.2 Apply contact pressure for 30 ± 5 seconds and dump pressure for 2 cycles before slowly applying maximum pressure. (The material shall not be prestaged.)

2.3 When testing tape widths which are 4.0 inches or less, prepare a test panel whose width is equal to that of the tape. The outer 0.100 inch of the panel shall not be used for preparation of the specimens.

2.4 Cut a specimen 1.000 ± 0.050 inch by 1.000 ± 0.050 inch by panel thickness from the center 2 inch by 2 inch section of the test panel. Top and bottom molded surfaces shall not be machined. Wipe the specimen clean using MEK and allow to air dry 20 minutes minimum before testing.

2.5 Place the specimen in a desiccator and desiccate for 18 hours minimum.

2.6 Weigh the specimen to the nearest 0.01 gram and record as W_1.

2.7 Place the specimen in an air circulating oven, preheated and stabilized at 325 ± 10 °F. for approximately 30 minutes; condition the specimen at a temperature of 325 ± 10 °F. for 24 hours minimum.

2.8 Remove the specimen from the oven and cool in the desiccator for approximately 30 minutes or until the specimen reaches room temperature.

2.9 Reweigh the specimen to the nearest 0.01 gram. Record as W_2.

2.10 Calculate the percent residual volatiles as follows:

$$\text{Percent volatiles} = \frac{W_1 - W_2}{W_1} (100)$$

Where:

W_1 = original weight of specimen in grams
W_2 = devolatilized weight of specimen in grams

2.11 Report residual voaltiles to the nearest 0.01 percent.

2.12 Limits (percent volatiles by weight)

Minimum	-
Maximum	2.5

F-3 Compressive Strength of Cured Material Made from Prepregs

1. SCOPE

1.1 The compressive strength shall be determined in accordance with the following:

2. PROCEDURE

2.1 Cut sufficient plies to produce a test panel 0.125 ± 0.025 by 4 inches (minimum) by 5 inches (minimum) and mark the warp direction. (All pieces aligned in same direction.)

2.2 Cure the plies into a panel between separator film on smooth caul plates as follows:

2.2.1 Orient plies such that warp fibers of each ply are aligned in the same direction. Mark warp direction.

2.2.2 Place the stack of plies into a press preset at 325 ± 10 degrees F.

2.2.3 Increase the pressure slowly to 1000 ± 50 psi., allowing the resin to stage to minimize flash at ply edges.

2.2.4 Hold the pressure and temperature for 120 ± 15 minutes.

2.2.5 Decrease the temperature and pressure to ambient.

2.2.6 The outer 1/2 inch of the edges of the test panel shall not be used for preparation of specimens.

2.3 Machine a specimen to the desired dimensions ~~shown in Fig. 1~~.

2.4 Measure the width and thickness of the specimen to the nearest 0.001 inch in the gauge section area. Calculate and record the cross-section area.

2.5 Place the specimen in the support jig (Figure 1), centered so the ends project an even amount beyond both ends of the jig. Tighten finger tight only.

2.6 Place the specimen and the support jig in the testing machine and align so that the specimen ends are parallel with the surface of the compression tool. Adjust the crosshead of the testing machine until it just contacts the top of the specimen.

2.7 Set the speed control at a cross head indicated rate of 0.050 in./minute and start the machine.

2.8 Record the maximum load carried by the specimen during the test.

2.9 Calculate the compressive strength by dividing the maximum load by the original cross-section area.

2.10 Report compressive strength to the nearest 10 psi.

2.11 Limits (psi.)

	Warp Direction	Fill Direction
Minimum	25,000	20,000
Maximum	65,000	55,000

2.12 **NOTES FOR FIG. 2**

Note 1 - Cold rolled steel

Note 2 - Furnished four steel machine screws and nuts, round bead, slotted, length 1-1/4 in.

Note 3 - Grind surface denoted "Gr"

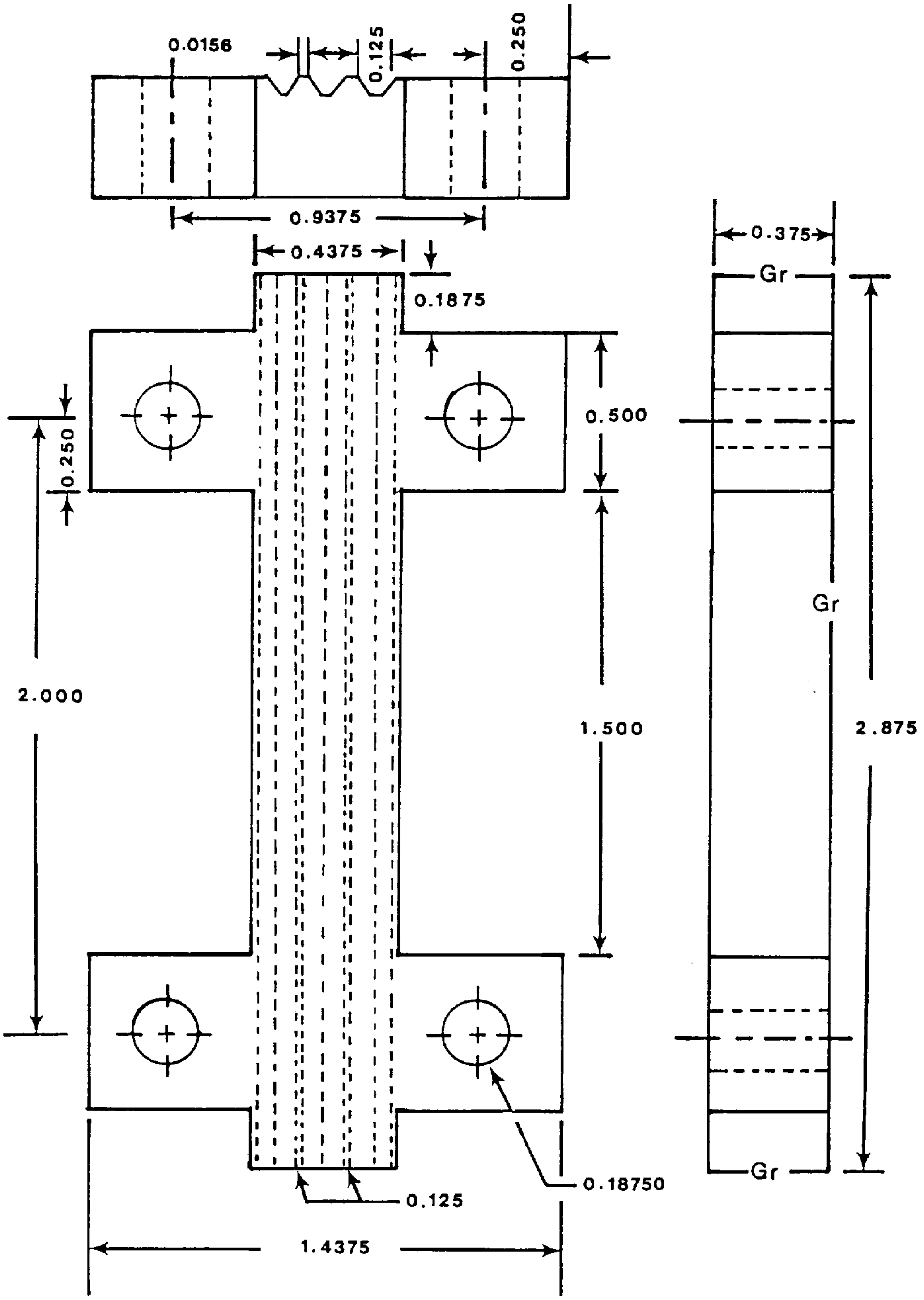

0.0156
0.125
0.250
0.9375
0.4375
0.1875
0.375
Gr
0.250
0.500
2.000
1.500
Gr
2.875
0.18750
0.125
Gr
1.4375

Fig. 1

F-4 Interlaminar Shear of Cured Material

1. SCOPE

1.1 The interlaminar shear strength test shall be in accordance with the following:

2. PROCEDURE

2.1 Cut sufficient plies to produce a test panel 1.5 ± 0.25 inch thick across the square plies of minimum dimensions of 1.5 ± 0.25 in.

2.2 Spray the mold with release agent and layup plies into mold.

2.3 Cure at 1000 ± 50 psi. and 325 ± 10 degrees F. for 2 hours ± 15 minutes.

2.4 After cure, remove the mold from the press, remove the test panel from the mold and allow the panel to cool to room temperature.

2.5 Cut a specimen of sufficient size to produce a 0.375 inch diameter by 1.125 inch minimum length finished specimen with the length being perpendicular to the plies from the test panel.

2.6 Center the specimen to get the length of the cylindrical specimen perpendicular to the plies and machine the specimens to a diameter of 0.375 + 0.000/-0.002 inch diameter.

2.7 Measure the diameter of the test specimen and calculate the cross-sectional area (A).

2.8 Place the specimen in the 0.375-inch diameter three plate double-shear jig shown in Figure 1.

2.9 Place the shear fixture in the testing machine, align, and pin the ends of the fixture.

2.10 Set the machine at an indicated crosshead speed of 0.015 to 0.025 inch-per-minute and test.

2.11 Record the maximum load carried by the specimen during the test and record as L, lb.

2.12 Calculations:

Interlaminar shear (psi.) = L/2A

Where:

L = the maximum load carried by the specimen during testing in pounds
A = The original cross-sectional area of the specimen in square inches.

2.13 Report the interlaminar shear to the nearest 10 psi.

2.14 Limits (psi.)

minimum	2,400
maximum	6,500

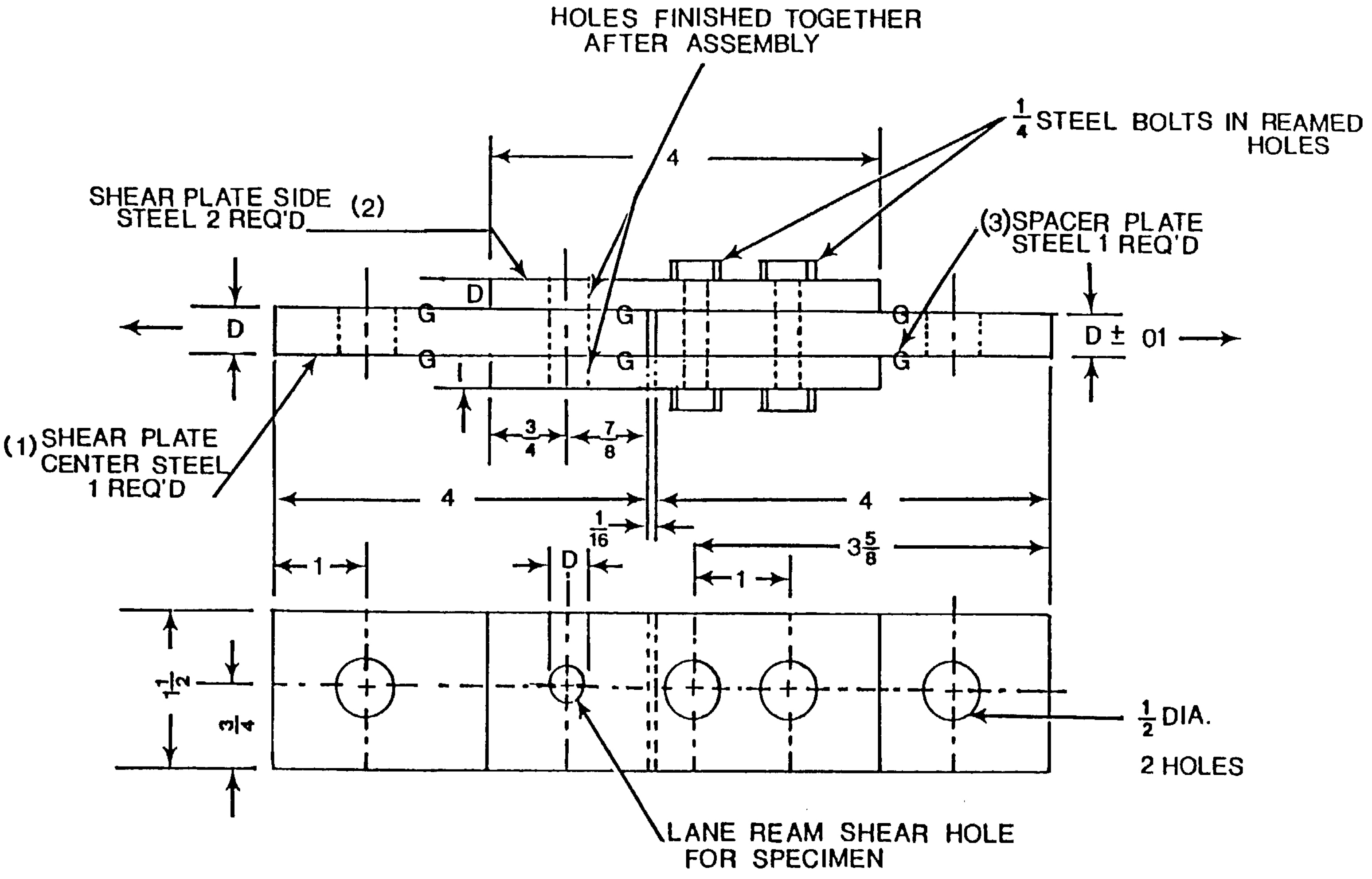

HOLES FINISHED TOGETHER AFTER ASSEMBLY
$\frac{1}{4}$ STEEL BOLTS IN REAMED HOLES
SHEAR PLATE SIDE STEEL 2 REQ'D (2)
(3) SPACER PLATE STEEL 1 REQ'D
(1) SHEAR PLATE CENTER STEEL 1 REQ'D
4
D
G
D ± 01
$\frac{3}{4}$
$\frac{7}{8}$
$\frac{1}{16}$
$3\frac{5}{8}$
1
$1\frac{1}{2}$
$\frac{3}{4}$
$\frac{1}{2}$ DIA.
2 HOLES
LANE REAM SHEAR HOLE FOR SPECIMEN

Figure 1

F-5 Thermal Conductivity of Cured Material

1. SCOPE

1.1 Thermal conductivity shall be determined in accordance with ASTM C 177 and as follows:

2. PROCEDURE

2.1 Prepare a test panel of required size and cure as follows:

2.1.1 Place the stack of plies into a press preset at 325 ± 10 °F.

2.1.2 Increase the pressure slowly to 1000 ± 50 psi., allowing the resin to stage to minimize flash at ply edges.

2.1.3 Hold the pressure and temperature for 120 ± 15 minutes, or 1 hour per inch of thickness, which ever is greater.

2.1.4 Decrease the temperature and pressure to ambient.

2.1.5 The outer 1/2 inch of the test panel shall not be used for preparation of specimens.

2.2 Data for thermal conductivity shall be reported for a temperature of 250 ± 20°F.

2.3 Maximum test specimen thickness shall be 9.00 inches.

2.4 Report thermal conductivity to the nearest 0.01 BTU./ft. hr. °F.

2.5 Limits (BTU./ft. hr. °F)

	With Ply	Across Ply
Minimum	0.10	0.10
Maximum	1.1	1.1

F-6 Coefficient of Thermal Expansion of Cured Material

1. SCOPE

1.1 The coefficient of thermal expansion shall be determined in accordance with ASTM E 228 and as follows:

2. PROCEDURE

2.1 Prepare a test panel of required size and cure as follows:

2.1.1 Place the stack of plies into a press preset at 325 ± 1 °F.

2.1.2 Increase the pressure slowly to 1000 ± 50 psi., allowing the resin to stage to minimize flash at ply edges.

2.1.3 Hold the pressure and temperature for 120 ± 15 minutes, or 1 hour per inch of thickness, whichever is greater.

2.1.4 Decrease the temperature and pressure to ambient.

2.1.5 The outer 1/2 inch of the edges of the test panel shall not be used for preparation of specimens.

2.2 Data for coefficient of expansion shall be reported for a temperature range from 75 to 400 °F.

2.3 Maximum test specimen thickness shall be 4.0 inches.

2.4 Report coefficient of thermal expansion to the nearest 0.01 in./in. °F x 10^{-6}.

2.5 Limits (in./in. °F x 10^{-6})

	Across Ply	With Ply
Minimum	5.0	2.0
Maximum	20.0	9.0

F-7 Flexural Strength of Cured Material

1. SCOPE

1.1 Flexural strength shall be determined in accordance with ASTM D 790, Method I, and as follows:

2. PROCEDURE

2.1 Cut sufficient plies to prepare required test panel and mark the warp direction.

2.2 Cure the plies into a panel as follows:

2.2.1 Orient plies such that warp fibers of each ply are aligned in the same direction. Mark warp direction.

2.2.2 Place the stack of plies into a press preset at 325 ± 10 °F.

2.2.3 Increase the pressure slowly to 1000 ± 50 psi., allowing the resin to stage to minimize flash at ply edges.

2.2.4 Hold the pressure and temperature for 120 ± 15 minutes, or 1 hour per inch of thickness, whichever is greater.

2.2.5 Decrease the temperature and pressure to ambient. The outer 1/2 inch of the edges of the test panel shall not be used for preparation of specimens.

2.3 Maximum test specimen thickness shall be 0.25 inch.

2.4 Report flexural strength to the nearest 10 psi.

2.5 Limits (psi.)

	Warp Direction	Fill Direction
Minimum	25,000	20,000
Maximum	55,000	55,000

F-8 Tensile Strength of Cured Material

1. SCOPE

1.1 Tensile strength shall be determined in accordance with ASTM D 638 and as follows:

2. PROCEDURE

2.1 Prepare a test panel of the required size and cure as follows:

2.1.1 Orient plies such that warp fibers of each ply are aligned in the same direction. Mark warp direction.

2.1.2 Place the stack of plies into a press preset at 325 ± 10 °F.

2.1.3 Increase the pressure slowly to 1000 ± 50 psi., allowing the resin to stage to minimize flash at ply edges.

2.1.4 Hold the pressure and temperature for 120 ± 15 minutes, or 1 hour per inch of thickness, whichever is greater.

2.1.5 Decrease the temperature and pressure to ambient. The outer 1/2 inch of the edges of the test panel shall not be used for preparation of specimens.

2.2 A thickness of up to 0.25 inch can be used for test specimens. Report each determination and the average of three specimens to the nearest 10 psi.

2.3 Limits (psi.)

	Warp Direction	Fill Direction
Minimum	15,000	10,000
Maximum	40,000	35,000

F-9 Dry Resin Content of Cured Material

1. SCOPE

1.1 This method describes a procedure for the determination of the dry resin content in molded carbon reinforced aldehyde phenolic plastics.

2. EQUIPMENT AND MATERIALS

2.1 Burners, Fisher

2.2 Test tubes, vycor, 25 X 200 mm.

2.3 Vacuum source

2.4 Manometer or vacuum gauge

2.5 Condenser, cold finger type

2.6 Clamps

2.7 Rubber stoppers

2.8 Vacuum tubing

2.9 Press for molding carbon bars 1/2" x 1/2" x 5"; at least 310 ± 10°F and 1000 ± 100 psi.

2.10 Desiccator

2.11 Cutting assembly for cutting carbon bars

2.12 Balance, analytical, 0.0001 gm. sensitivity

2.13 Timer

3. SAMPLING

3.1 Sample size. A broadgoods sample 6 inches by 20 inches is required.

3.2 Number of determinations. Unless otherwise specified, run duplicate determinations on each sample.

3.3 Preparation of test specimens.

3.3.1 Cutting. Cut 32 rectangles from the prepreg measuring 1/2 inch by 5 inches.

3.3.2 Staging. Stage the 32 rectangles at 255° ± 5°F. to reduce flow. Staging time is dependent upon resin system and flow and must be determined from experience.

3.3.3 <u>Molding.</u> Using the 32 rectangles, mold a bar 1/2" x 1/2" x 5" ± 1/32". Cure the specimen at 310° ± 10°F. and 1900 ± 100 psi. for 15 ± 1 minutes. If there is any resin flash, begin again and use a longer staging time. Cut the bar into approximately three equal lengths. Tag and store in a desiccator.

4. PROCEDURE

4.1 <u>Setting up apparatus.</u> Set up the apparatus as shown in Figure 1. Weigh the test specimen to 0.01 g. Place the test specimen in the vycor test tube and attach the tube to the side arm of the vacuum flask. All joints must be air tight. Start the vacuum pump, evacuate the system, and check for air leaks by shutting the valve leading to the vacuum pump. The leak rate should be less than five millimeters of Hg per minute (0.2 inch) as measured by a manometer or vacuum gauge.

4.2 <u>Pyrolysis.</u> With the vacuum on and the pressure stabilized, place the Fisher burners under the specimen so that the flame area covers it completely. Continue pyrolysis for 30 minutes, or until the manometer or vacuum gauge reading is the same as it was before pyrolysis, whichever is longest.

4.3 <u>Cooling Sample.</u> When pyrolysis is complete, turn off the burners and allow the specimen to cool under vacuum to room temperature. When the specimen is cool, turn the vacuum off, and carefully let air into the system to restore pressure equilibrium.

4.4 <u>Reweighing specimen.</u> Remove the vycor test tube from the vacuum flask. Weigh the specimen to 0.01 gm. when cooled to ambient, or transfer to a desiccator and weigh when convenient.

5. CALCULATIONS AND REPORTING

5.1 <u>Calculations.</u> Calculate the dry resin content (DRC) of the sample as follows:

$$\% \text{ DRC} = (1.00 - W_2/W_1)\ 100 \times K$$

where:

W_1 = weight of sample before pyrolysis, g.
W_2 = weight of sample after pyrolysis, g.
K = constant peculiar to material being tested, which must come from prepreg manufacturer.

5.2 Reporting. Unless otherwise specified, round off and report dry resin content to the nearest 0.01% on worksheets and to the nearest unit in the designated place for figures on final reports. Duplicate determinations should be considered suspect if they differ by more than 0.2%.

6.0 NOTES: Number of specimens per tube. Two or more specimens may be put in a tube so long as all specimens are completely covered by the flame from the burners.

F-10 Residual Volatiles of Cured Material

1. SCOPE

1.1 Method for determining residual volatile in cured laminates.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

2.1.1 Laboratory forced air oven, 325°F.± 5°F. capability

2.1.2 Laboratory press, hydraulic, 16,000 lbs. ± 50 lbs., 325°F. ± 5°F. capability

2.1.3 Analytical balance, 0.0001 gm. sensitivity

2.1.4 4" x 4" steel templates

2.1.5 Laboratory knife

2.1.6 Carbide bladed table saw or hack saw

2.2 Materials

2.2.1 Teflon coated glass cloth release fabric

3. SAMPLING

3.1 Sample size: The sample shall be large enough so that the required number of 4" x 4" plies, cut on the bias, may be obtained to make a laminate 0.250" thick.

4. PROCEDURE

4.1 Laminate: Using 4" x 4" template, cut required number of plies on the bias. Stack and load at 325°F ± 5°F, press between Teflon coated glass cloth release material. Apply contact pressure for 30 seconds ± 2 seconds, dump press; repeat twice and apply 1,000 ± 50 psi for four hours ± 10 minutes at 325°F. ± 15°F. Remove laminate from press, remove flash with knife, and cut three 1" x 1" pieces from laminate as follows: (Refer to Figure 1)

4.1.1 Cut 1" x 4" center strip parallel to two outer edges.

4.1.2 Cut three 1" x 1" pieces from this strip, starting at one end without trimming outside edge.

4.1.3 Desiccate pieces.

4.2 Test for Residual Volatiles: Each sample described above, after desiccation for a minimum of 18 hours, is weighed. This is sample weight. Samples are placed in forced air oven for four hours ± 10 minutes at 325°F. ± 15°F., removed and weighed hot. Four-hour weight loss is calculated:

Weight Loss = Initial Sample Wt. - Hot Wt. after 4 hrs.

Residual Volatile of each sample = $\frac{\text{Weight Loss}}{\text{Sample Weight}} \times 100\%$

Residual Volatile of mat. = Arithmetic Average of above three samples

4.3 Tolerances:

0.250" Laminate Thickness, ± 5%
325°F Press, ± 15°F.
1,000 psi. Press ± 5%
325°F. oven, ± 10° F.

5. REPORTING

5.1 Report Residual Volatile to 2 significant figures.

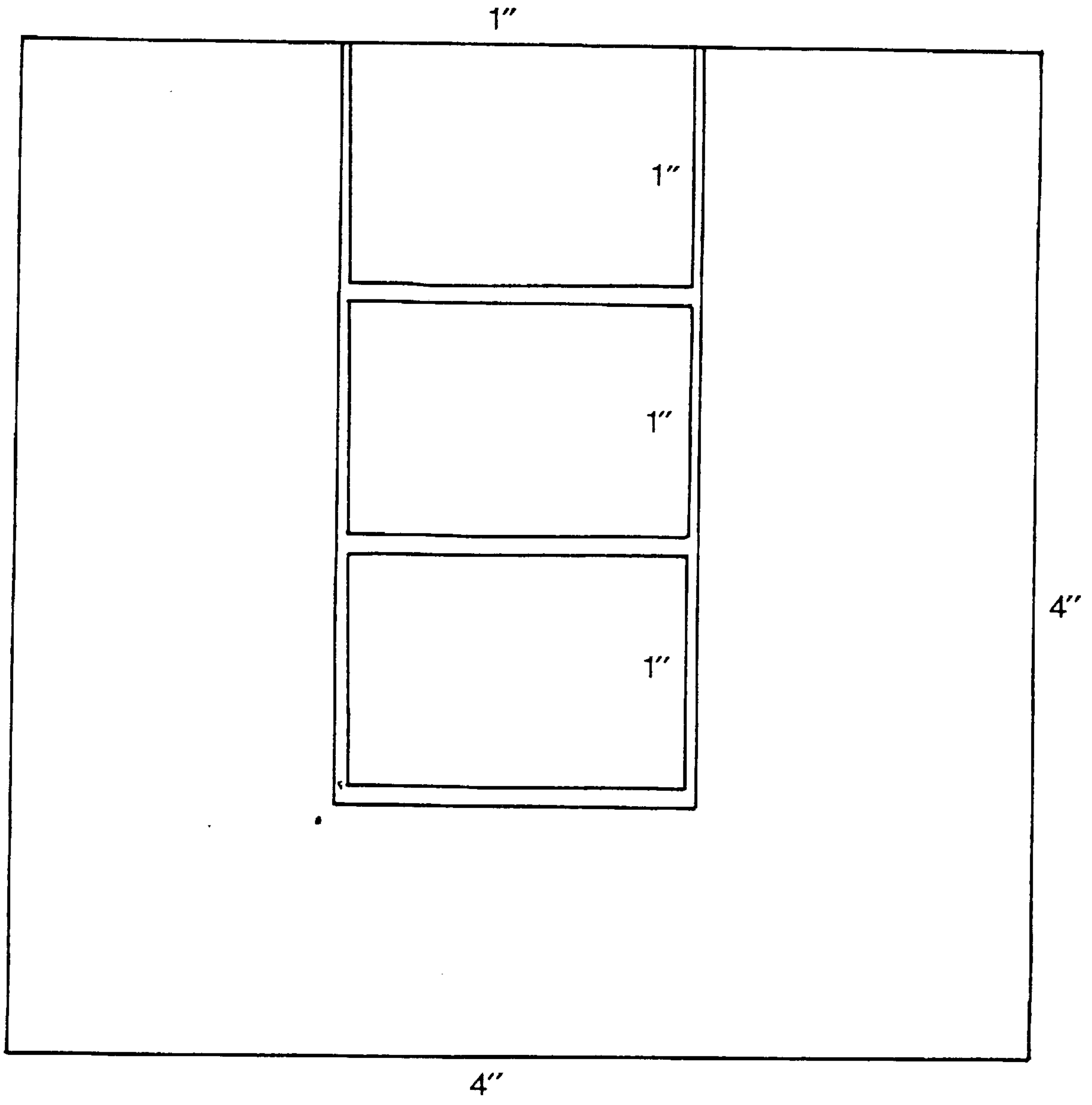


Fig. 1

F-11 Linear Thermal Expansion of Cured Material Using DSC

1. SCOPE

1.1 Scope. This is a method for the determination of linear thermal expansion using the differential scanning calorimeter. The thermal expansion of plastic is reversible. Upon it are superimposed changes in length due to changes in moisture content, degree of polymerization, loss of plasticizer or solvents, release of stresses or other factors which are mainly irreversible. This method of test is intended to yield the linear thermal expansion excluding these incidental factors. In general, it will not be possible to exclude all of these spurious factors and thus the method can be expected to give only approximate values. This method is an adaptation of ASTM E228-71.

2. EQUIPMENT AND MATERIALS

2.1 Equipment.

2.1.1 Differential Scanning Calorimeter and an operators manual

2.1.2 DSC User's Manual

2.1.3 Helium Gas source

3. SAMPLING

3.1 Preparation. All samples shall be cut or shaped from moldings, castings, or laminates by methods under conditions that give a minimum of strains or molecular anisotropy.

3.2 Dimensions. Samples shall be 1/4 by 1/4 by 1/4 inches with the ends machined flat and parallel with 0.005 inch tolerances.

4. PROCEDURE

4.1 Calibrate the DSC as per user's manual

4.2 Turn on the helium atmosphere or other gas as specified

4.3 Set approximate upper and lower temperatures

4.4 Measure sample height, width, and thickness to nearest 0.0001 inch

4.5 Place sample into the TMS (DSC) apparatus, noting the ply direction

4.6 Zero out the chart

4.7 Raise temperature to upper limit at a rate of 40°F.± 2°F.per minute. Hold upper limit for 5 minutes ± 0.5°F.

4.8 Cool sample at 40°F.± 5°F. per minute until lower limit is reached. Hold at lower limit for 5 minutes ± 0.5 minute.

4.9 Repeat steps 4.7 and 4.8 three (3) times. Disregard the first cycle of heating and cooling. This was done to condition the sample.

4.10 Measure the temperature difference at the end of each heating or cooling period.

4.11 Measure the shrinkage difference at the end of the heating and cooling periods with each cycle.

5. CALCULATIONS AND REPORTING

5.1 Calculations: Calculate the coefficient of linear thermal expansion as follows:

$$E = \frac{L}{LxT}$$

where:

E = coefficient of thermal expansion, in./in./°F. x 10^{-6}
L = test length of specimen at room temp., inch
T = temperature difference, °F., over which the change in length was measured
delta L = Average of change in length due to heating and cooling.