de Havilland Material Specification

TITLE:	CORROSION INHIBITING SEALANT
SPECIFICATION NUMBER:	DHMS S3.06
ISSUE:	С
AMENDMENT:	
DATE:	April 30, 2013
PAGE:	1 of 16

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	SIGNED ORIGINAL ON FILE	
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REVISION RECORD

Issue	Page	Description and Reason for Change
A		This is a complete revised issue. Detailed changes have not been noted.
Amd. 1	4	Removed Shore A Hardness value from Table 1
	14	QPL: Courtlads Aerospace has been changed to PRC DeSoto Int.
Amd. 2	5	Table 2 updated to include requirement for Hardness
	7	Para. 4.5 revised to include batch acceptance test.
В		This is a complete revised issue, Detailed changes have not been noted.
		Updated format
	3,7	Clarified curing time requirement
C		This is a complete revised issue.
	7	Added cure time requirement for Class E, Para. 4.5.2
	,	reduced cure time requirement for Class 2, rata. 1.3.2

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1 SCOPE

This specification covers the requirements for room temperature curing, two component, corrosion inhibiting synthetic rubber compounds for sealing.

1.1 Classification

The sealing compound shall be supplied in one of the following types and classes:

Types

Type I Chromated

Type II Non-Chromated

Classes

Class C For brush application on faying surface.
Class E For spray gun application as a coating.

The dash numbers following the Class C and Class E designation, indicate the minimum squeeze-out life (in hours) and minimum application life (in hours) respectively. For the purpose of this specification, C-80 and E-2 are covered.

2 APPLICABLE DOCUMENTS

The following document shall form the specification of the sealant defined herein.

2.1 Military Specifications

MIL-C-5541 Chemical Conversion Coatings on Aluminum and Aluminum Alloys

MIL-S-81733C Sealing and Coating Compound, Corrosion Inhibitive

2.2 U.S. Government Specifications

QQ-A-250/12 Aluminum Alloy 7075, Plate and Sheet TT-S-735 Standard Test Fluids, Hydrocarbon

2.3 American Society for Testing and Materials

ASTM D1002 Test Method for Strength Properties of Adhesives in Shear by Tension Loading

(Metal to Metal)

2.4 de Havilland Material Specifications and Standards

DHMS C4.01 Primer, Fluid Resistant, Epoxy

PPS 31.17 Manual Solvent Cleaning

QAMTR 021 Testing PR 1431G Type II & III Sealing Compounds

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3 REQUIREMENTS

The sealing compounds noted shall meet all the requirements specified in **Table 1**.

3.1 Materials

The basic ingredient used in the manufacture of this product shall be a synthetic rubber of the polysulfide type. The cure of the sealant shall result from the chemical reaction of the two components and shall not depend upon the evaporation of solvent.

3.2 Physical Properties

3.2.1 Colour

Unless otherwise specified, the colour of the sealing compound shall be as furnished by the manufacturer. The curing agent, if furnished separately, shall be of contrasting colour to facilitate mixing.

3.2.2 Appearance

The base compound and curing agent shall be of uniform blend and shall be free of skins, lumps, and jelled or coarse particles. There shall be no separation of ingredients which cannot be readily dispersed by mechanical agitation or mixing by hand.

3.2.3 Non-Volatile Content

The minimum percent non-volatile content of the freshly mixed compound, when tested as specified in **Para.4.1**. shall be as specified in **Table 1**.

3.2.4 Initial Viscosity

The initial viscosity of the base compound shall conform to <u>Table 1</u>, when tested as specified in <u>Para.4.2</u>.

3.2.5 Application Time

When tested per **Para.4.3** the application time shall be as specified in **Table 1**.

3.2.6 Squeeze Out Life (Class C only)

The residual sealant in each specimen shall be no thicker than 0.004" at the expiry of the times listed in **Table 1**, when tested per **Para.4.4**.

3.2.7 Curing Time

Standard Cure

When tested at $77 \pm 5^{\circ}$ F and $50 \pm 5\%$ relative humidity

- a) C-80 sealant is fully cured in 42 days.
- b) E-2 is fully cured in 7 days.

Accelerated Cure

For C-80, 24 ± 1 hour at standard condition, followed by 120 ± 2 hours at $158 \pm 5^{\circ}$ F.

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Table 1: Physical Properties

Type I	Requirements		
Properties	C-80	E-2	
Non-Volatile Content (%)	85 minimum	65 minimum	
Viscosity (poise)	1200-4000	50-150	
Application Life (hr)	24	2	
Squeeze Out Life (hr)	80	N/A	

3.3 Properties After Cure

3.3.1 <u>Soluble Chromate Content</u> (Type I only)

The cured sealing compound shall contain a minimum of 2.7% by weight when tested as specified in **Para.4.6**.

3.3.2 Specific Gravity

The specific gravity of the cured sealing compound shall be as specified in **Table 2** when tested per **Para.4.7**.

3.3.3 Resistance to Hydrocarbon Fluid (Weight loss and flexibility):

Following exposure to the hydrocarbon fluid per <u>Para.4.8</u>, the cured sealant shall exhibit a reduction in weight of no more than 10% and shall be free from cracks when bent 180 degrees over a 1/8" radius mandrel. Cracks which develop at avoid or other preparation defects, shall be discounted.

3.3.4 Resistance to Heat

When tested as per <u>Para.4.9</u>, the cured compound shall not blister, crack, or show evidence of "blowing" at rest or when wrapped 180 degrees on a mandrel.

3.3.5 Shear Strength

The shear strength of the compound shall be as specified in <u>Table 2</u>, when tested in accordance with <u>Para.4.10</u>, as received and after immersion. All specimens shall exhibit a minimum 95% cohesive failure.

3.3.6 <u>Storage Life</u>

3.3.7 The base compound and curing agent, after 6 months storage at temperature between 35° to 80° F, shall show no hardening, separation, or settling of the material when tested as specified in Para.4.11. After the storage period, the mixed sealing compound shall meet the requirements for application life, squeeze out life and curing time.

3.3.8 Workmanship

The workmanship shall be in accordance with high grade manufacturing practice for this type of material. It shall be suitable for its intended purpose and free of defects which may affect its performance.

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Table 2: Mechanical Properties

Properties After Full Cure	Requirements
Soluble Chromate Content	2.7% by Weight
Specific Gravity	1.65 Max.
Shear Strength	C-80 : 200 psi Min.
	E-2: 100 psi Min.
Shore A Hardness (class C)	30 Min.

4 TEST METHODS

4.1 Non-Volatile Content

Five to ten grams of the mixed sealing compound shall be transferred as soon as possible to a dish about 8 centimeters in diameter. A tight fitting cover shall immediately be placed over the dish and the weight determined to the nearest milligram. The cover shall then be removed and the sealing compound heated for 72 1 hours at 70° 1° C (158° 2° F), and the compound transferred to a desiccator and cooled to room temperature, the cover replaced, and the weight determined to the nearest milligram. The percent non-volatile content shall be calculated as follows:

% Non-Volatile =
$$\frac{W2}{W1} \times 100$$
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W1 - Initial Weight

W2 - Final Weight

4.2 Initial Viscosity

The viscosity shall be determined with the sample of base compound in a 1-quart can. The can shall be filled with base compound to within 1/2 inch of the top, covered, and stored at standard conditions for a minimum of 8 hours. The base compound shall be thoroughly mixed by stirring for 3 minutes. The container shall be closed and the material allowed to stand for 1 hour. The Broolfield Model RVF Viscometer or equivalent shall be used with the reading being reported as poise. Spindle identification and speed shall be as follows:

C-80-No. 6 spindle at 2 rpm

E-2-No. 5 spindle at 10 rpm

4.3 Application Time

4.3.1 Class C

The compound prepared in accordance with the manufacturer's instructions shall have an application

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time of not less than the time in hours specified in <u>Table 1</u>. At the end of the application time, 50 grams of sealant per minute (minimum)shall be extruded when tested as follows:

The sample shall be mixed and prepared in accordance with the manufacturer's instructions. 2 to 3 inches of sealing compound shall be extruded initially to clear trapped air. At the end of the rated application time, measured from the beginning of the mixing period, the sealing compound shall be extruded through a Semco 440 nozzle (0.125" orifice at 90 5 psig) or equivalent into a preweighed cup for 1 minute and the weight in grams of sealing compound determined.

4.3.2 <u>Class E</u>

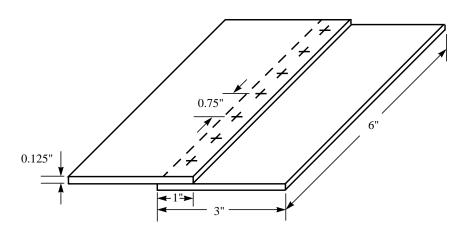
The viscosity of the mix shall be 300 poise maximum when tested at the expiry of the application time specified in <u>Table 1</u>.

4.4 Squeeze Out Life

Three faying surface seal samples (can also be used for cure time test) shall be prepared as follows:

- 4.4.1 Clean the panels surface using MEK, DS 108 or Diestone DLS and measure/record the thickness of each panel between the holes, as shown in **Figure 1**.
- 4.4.2 Mix the sealant in accordance with the manufacturer's instructions and apply 0.02 inch thickness of sealant to three of the panels as shown in **Figure 1** (care should be taken to avoid allowing excess sealant in the holes).
- 4.4.3 Assemble by inserting seven NAS 1303-2 bolts through the holes in each of the panels with sealant, then place the uncoated panel over the bolts onto the sealant coated panel.
- 4.4.4 Insert flat washers and MS 21042-3 nuts over the threaded portion of the bolts and slowly torque (as evenly as possible) to produce a sealant thickness of 0.010 0.015 inch (check with a micrometer).
- 4.4.5 Allow the test panels to stand at standard conditions for 80 hours (specified squeeze out time).
- 4.4.6 Torque each nut (only once) to 30 in/lb and hold the torque wrench at 30 in/lb for 4-6 seconds.
- 4.4.7 Remeasure panel thickness at 30-35 minutes after the torquing operation and calculate the sealant thickness. The maximum allowable residual sealant thickness is 0.004 inch.

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FIGURE 1. Test Panel Configuration for Squeeze Out Life and Cure Time

4.5 Curing Time

4.5.1 Class C

Allow the torqued test specimens (from Para.4.4) to cure for 28 days at $77\pm5^{\circ}$ F and $50\pm5\%$ relative humidity, then disassemble one of the samples and break apart the faying surface seal. Visually examine the faying surfaces to ensure the absence of a gasket seal or for evidence of incomplete cure. If sealant cure is incomplete, allow the remaining two specimens to cure for and additional 14 days and subsequently re-examine. Presence of a gasket seal is the criterion for rejection.

4.5.2 Class E

Prepare test panels (shear strength) as per <u>Para.4.10</u>. Apply approximately 0.010 inch thick sealant by spraying to each mating surface . Allow the solvent to flash off for approximately one hour before bonding the mating surfaces. Cure panels for 48 hours at $77 \pm 5^{\circ}$ F and $50 \pm 5\%$ relative humidity. Machine 5x1 inch wide test pieces (normal to the overlap) as per_Figure 2. Perform shear test as per ASTMD1002, using a jaw separation of 2 inches per minute. Measure the dimensions of the bond area of each specimen and calculate the shear strength in Psi. Record the mode of failure as a percentage of the bond area. The shear strength shall meet the requirement listed in <u>Table 2</u>.

4.5.3 Hardness Class C

Prepare a 0.25 ± 0.05 inch thick by 2.5 ± 0.125 inch diameter sealant specimen with a flat surface. Cure as specified in <u>Para.3.2.7</u>. The hardness of the specimen shall be determined in accordance with ASTM D2240 and shall met the requirement listed in <u>Table 2</u>.

4.6 Soluble Chromate Content

The soluble chromate content in the compound shall be determined by the following procedure. (Three

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samples are required for determination):

4.6.1 Sample Preparation

A minimum of 25 grams of sample cured in accordance with <u>Table 1</u> shall be filed off such that all of the sealant will pass through an ASTM No. 40 sieve. A 5 gram sample of filed material shall be place into a 250 ml. Erlenmeyer flask and 100 ml. of distilled water added. The sample shall then be covered with a watchglass, placed on a hot plate, and boiled for 1 hour.

After boiling for the time specified, the flask shall be removed from the heat and the liquid decanted into a 500 ml flask. The extraction procedure shall be repeated three times.

4.6.2 Titration

After the filtrate collected above has cooled to ambient temperature, 10 ml. of concentrated hydrochloric acid and 2 grams of potassium iodide shall be added and the mixed solution titrated with 0.1 N sodium thiosulfate until the brown colour of iodide is almost gone. Two ml. of freshly prepared starch solution shall then be added and the titration continued to a clear greenish end-point. The volume in ml. of sodium thiosulfate required for titration shall be recorded.

4.6.3 Calculation

The percentage of soluble chromate, for purposes of this specification, shall be calculated as magnesium chromate pentahydrate as follows:

$$\frac{\text{Volume of Na}_2\text{S}_2\text{O}_3 \text{ (ml) } \text{ x Normality of Na}_2\text{S}_2\text{O}_3 \text{ x } 0.0768 \text{ x } 117.6}{\text{Weight of Sample (g)}} = \% \text{ MgCrO}_4 ● 5H_2\text{O}$$

(Results shall be reported to the nearest whole number.)

4.7 Specific Gravity

Three specimens, approximately $1 \times 2 \times 0.030$ inch, shall be prepared and fully cured. The specimens shall be weighed in air and then in water by means of an analytical or Jolly balance. The specific gravity shall be computed by means of the following formula:

Specific Gravity =
$$\frac{Wair}{Wair - Wwater}$$

 W_{air} - Weight in air

 W_{water} - Weight in water

4.8 Resistance to Hydrocarbon Fluids

4.8.1 Weight Loss

Cast the mixed sealant into a $6 \times 4 \times 1/8$ inch thick slab by pressing between two plates separated by a parting agent such as polyethylene sheet (Class C only), or by extruding into a closed mould coated

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with a parting agent. Open the mould after 72 hours and allow the cast sealant to cure for 5 weeks at standard conditions. For Class E-2. Extrude an excess of the mixed sealant into an open mould and work with a spatula to eliminate voids. Allow 2 hours for solvent evaporation before closing the mould, exercising care to avoid air entrapment. Allow to cue for 48 hours.

Cut three specimens, 5 x 1 inch, from the cured slab and weigh on an analytical balance (to three decimal places). Using aluminum wire, suspend the specimens in a one quart screw top jar containing approximately 1 liter of TT-S-735, Type III test fluid. Cover the jar opening with aluminum foil, screw the lid on tightly and place it in a convection oven controlled at 120° F. Remove the specimens from the jar after 7 days, place them on paper toweling and allow them to dry (in the oven) for 48 hours at 120° F. Remove the specimens from the oven and allow them to cool to room temperature (1 to 2 hours) in a desiccator before reweighing. Calculate the weight loss as follows:

Weight Loss (%) =
$$\frac{Wb - Wa}{Wb}X100$$

Wb - Weight before exposure

Wa - Weight after exposure

4.8.2 Flexibility

Bend the three weighed specimens 180 degrees over a 1/8 inch radius mandrel and examine the stressed are for evidence of cracks.

4.9 Resistance to Heat

Coat three 6 x 1 x 0.06 inch 2024-T3 aluminum panels conforming to QQ-A-250/12, with the catalyzed sealant, and cure as specified. Place the cured specimens in an air circulating oven for 48 hours at 200° F. Remove the specimens at the end of the exposure period and allow to cool in a desiccator. Bend the specimens 180 degrees over a 1/8 inch radius mandrel and examine for conformance to **Para.3.3.4**.

4.10 Shear Strength

Pretreat two 9 x 5 x 0.06 inch 2024-T3 aluminum test pieces per MIL-C-5541, Class 1A (Alodine 1200), prime with epoxy primer per DHMS C4.01, Type 2 and allow to cure as specified. As shown in **Figure 2**, apply the sealant to a one inch wide strip at the ends of one surface of both test pieces and parallel to the 9 inch dimension. Bond the mating surfaces to produce a one inch overlap. Place a packer plate (spacer of 0.06" T) under the top plate, apply a compressive load of 2 lb to the bond are and allow the test panel to cure for six weeks at $77 \pm 5^{\circ}$ F and $50 \pm 5\%$ relative humidity

Machine eight 9 x 1 inch wide test pieces (normal to the overlap). Expose three specimens to TT-S-735, Type III fluid as described in **Para.4.8** and immerse three specimens in a one quart jar containing 3% saline solution, seal as described in **Para.4.8** and expose for 168 hours at 120° F. Retain the remaining two specimens at standard conditions, i.e. for control purposes. Remove the jars from the heat source and allow them to cool to ambient conditions, prior to removing the test specimens. Perform the shear test described ASTM D 1002, using a jaw separation of 2 inches per minute. Measure the dimensions of the bond areas of each specimen and calculate the shear strength in psi. Record the mode of failure as a percentage of the bond area.

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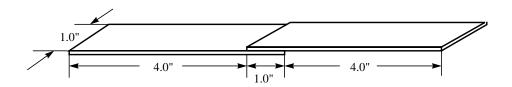


FIGURE 2. Test Panel Configuration for Shear Strength

4.11 Storage Life

A one quart sample of sealing compound and the specified amount of curing agent shall be conditioned at 25° F for 6 months. If the curing agent shows signs of setting or hardening, it shall be stirred. Failure of the curing agent to return to a smooth workable consistency shall be construed as a failure. The stirred curing agent and the base shall be mixed and tested for conformance with **Para.3.3.6**.

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5 MATERIAL QUALIFICATION REQUIREMENTS

5.1 Request For Qualification

All requests for qualification to this specification shall be addressed to Bombardier Aerospace Materials Technology Engineering department for approval.

All material qualification shall be site specific.

An audit of the manufacturers and/or test facilities by Materials Technology Engineering may be necessary prior to approval.

5.2 Qualification testing

Potential suppliers shall submit a written qualification test report based on 3 batches/lots of materials showing compliance with the requirements contained in section 3. The test report shall contain actual numerical test values, average test results as well as failure modes where applicable.

A sample shall be submitted for testing at the discretion of Bombardier Aerospace Materials Technology for evaluation.

5.3 Qualification by Similarity

Where a product has been qualified to another similar specification, the supplier may submit the qualification data applicable to this specification for consideration. The similar specification may be a government, company, or other specification where the requirements are similar to this specification.

5.4 Process Control Document

- 5.4.1 The manufacturer shall develop and maintain a Process Control Document (PCD). The PCD shall define the manufacturing and quality control requirements and procedures for assuring consistent, uniform and compliant products. The PCD shall identify baseline chemical constituents, in-process test procedures and requirements, and manufacturing procedures. All specifications and test procedures employed during the process shall also be listed and issue/date controlled.
- When qualification has been granted, the PCD shall be signed by the supplier and Bombardier Aerospace Materials Technology Engineering and shall not be changed without prior written approval.
- **The PCD** and all production data shall be available to any Bombardier Aerospace auditors when requested.

5.5 Qualification

- 5.5.1 Upon review of supplier's data, PCD and de Havilland tests, the supplier will be advised either of product qualification or reasons for disqualification. Products that are qualified will be listed in the Qualified Products List of this specification.
- | 5.5.2 No changes in the method of manufacture and/or formulation shall be made without notification and prior written approval of Materials Technology Department.

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Re-qualification of the product may be requested by the Bombardier Materials Technology if there any changes in the method of manufacture and/or formulation.

6 QUALITY ASSURANCE REQUIREMENTS

6.1 Manufacturer Batch/Lot Acceptance Tests

- 6.1.1 The manufacturer/supplier is responsible for the performance of all sampling, inspection and testing of each batch/lot as specified in <u>Table 3.</u>
- 6.1.2 The manufacturer/supplier shall issue with each batch of product one copy of an Acceptance Test Report showing actual test data conformance to the acceptance tests specified in <u>Table 3</u>. The report shall include the supplier's batch identification, materials specification and date of testing.
- 6.1.3 Bombardier Aerospace Materials Technology Engineering reserves the right to perform any or all of the tests set forth in this specification to ensure that the product continues to meet specification requirements. Any product not meeting the requirements of this specification will be returned to the supplier at the supplier's expense.
- 6.1.4 The manufacturer/supplier shall certify with a Certificate Conformance that each batch of each shipment meets the requirements of this specification.

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6.2 Purchaser Batch/Lot acceptance tests

The purchaser/user is required to perform of all sampling, inspection and testing of each batch/lot as specified in **Table 3**.

6.3 Definitions

- 6.3.1 <u>Batch</u> is defined as the end product of all the raw materials mixed and/or manufactured at the same time and place. The weight and volume may vary, depending upon the capacity of the manufacturers facilities.
- 6.3.2 <u>Lot</u> is defined as the total quantity of product in a shipment taken from the same batch.

Table 3: Qualification and Batch Acceptance Tests

Properties	Paragraph	Qualification	Acceptance (Supplier/User)
Non-Volatile Content	<u>Para.3.2.3</u>	х	
Initial Viscosity	<u>Para.3.2.4</u>	X	X
Application Time	Para.3.2.5	X	
Squeeze Out Life	<u>Para.3.2.6</u>	X	x
Curing Time	<u>Para.4.5</u>	х	x 1
Hardness	Para.4.5.3	х	x ²
Soluble Chromate Content (Type I only)	<u>Para.3.3.1</u>	x	
Specific Gravity	Para.3.3.2	X	
Resistance to Hydrocarbon Fluid	<u>Para.3.3.3</u>	x	
Resistance to Heat	<u>Para.3.3.4</u>	X	
Shear Strength	Para.3.3.5	X	
Storage Life	<u>Para.3.3.6</u>	x	
Workmanship	<u>Para.3.3.8</u>	х	

^{1.} For batch acceptance test, it is acceptable to use accelerated cure. for class C

^{2.} For Class C only

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7 ORDERING DATA

7.1 Prerequisite

Material furnished under this specification for production use shall be qualified and listed on the Qualified Products List prior to issuing a Purchase Order.

8 PROCUREMENT DOCUMENTS

Procurement documents shall specify the following:

- Title, Number, Issue and Amendment Number of this specification.
- Type or Size of Cartridges (Imperial or U.S. measure)
- Total Quantity (Imperial or U.S. measure)
- Acceptance Test Report

9 PREPARATION FOR DELIVERY

9.1 Preservation and Packing

The sealant shall be packed in such a manner as to assure that, during shipments and storage, the product will be protected against damage from exposure to hazards which would affect adversely the property conformance to <u>Section 3</u> of this specification.

9.2 Packaging

For pre-mixed frozen sealant packaging, the supplier must ensure that the sealant is maintained at a temperature below -40^{0} F during transit.

9.3 Marking

Each container shall be legibly marked with the following information:

- Sealing Compound, Fay Surface
- DHMS S3.06 C-80 or E-2
- Manufacturer's Name and Product Identification
- Date of Manufacture
- Date of Mixing
- Cartridge Size
- Expire Date
- Batch Number

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- Net Quantity (Imperial or U.S. measure)

9.4 Shipping Documentation

Shipping document shall show:

- Purchase Order Numbers
- DHMS S3.06 C-80 or E-2
- Number of Cartridges
- Batch Number
- Total Quantity (Imperial or U.S. measure)
- Batch Acceptance Test Report

Each shipment shall contain a copy of the Material Safety Data Sheet.

10 HEALTH AND SAFETY DATA

When supplying samples for qualification per <u>Para.5.2</u>, the supplier shall submit a Material Safety Data sheet (MSDS) complying with the "Controlled Products Regulations" of the Hazardous Products Act (also known as W.H.M.I.S. Regulations). The document must state all hazardous ingredients, safe-handling procedures, first-aid measures, fire and explosion data, re-activity data, physical properties, preparation information and procedures for storage and disposal.

This MSDS must then be supplied with a completed DH 4339 form, "Application To Introduce A New Material", to the Material Safety Committee.

Upon receipt of DH 4340 form, "Recommendation", that approves the use of the material, it can then be included on the Qualified Products List.

NOTE: Any changes in the formulation of the material require a re-submission of the MSDS.

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QUALIFIED PRODUCTS LIST

MANUFACTURER'S PRODUCT IDENTIFICATION NO.	SAFETY DATA QU	ALIFICATION	DATE OF PRODUCT APPROVAL
Type I, Class C-80			
P/S 870 C-80 Part A Part B	31/90 32/90	PQS #1	Dec. 2, 1993
Type I, Class E-2			
PR 1436G E-2 Part A Part B	0092 0093	PQS #2	Dec. 2, 1993
	PRODUCT IDENTIFICATION NO. Type I, Class C-80 P/S 870 C-80 Part A Part B Type I, Class E-2 PR 1436G E-2 Part A	PRODUCT IDENTIFICATION NO. SAFETY DATA QUESTION NO. Type I, Class C-80 SHEET NO P/S 870 C-80 31/90 Part A 32/90 Type I, Class E-2 0092 Part A 0093	PRODUCT IDENTIFICATION NO. Type I, Class C-80 SAFETY DATA QUALIFICATION SHEET NO'S P/S 870 C-80 SHEET NO SHEET NO'S Part A Part B 32/90 PQS #1 32/90 Type I, Class E-2 O092 PQS #2 PQS #2 Part A 0093

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