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Produced by the NASA Center for Aerospace Information (CASI)
Project No. 6031.2

1. Contractor's Name and Address:

Springborn Laboratories, Inc.
Department of Analytical Chemistry
Ten Springborn Center
Enfield, CT 06082

2. Title of Report:

Development of Acceptance Criteria for Batches of Silane Primer for External Tank Thermal Protection System Bonding Applications

March 2, 1985 - May 24, 1985

3. Date of Publication:

April 10, 1985

4. Type of Report and Contract Number:

11th Progress: NAS8-35818

5. Authors:

F. Mikes, C. Mowrey, E. Reis

6. Prepared For:

C. Marshall Space Flight Center, AL 35812

cc: AP-29-F - 1x
AS24D - 3x
AT01 - 1x
EM13B-18/Belwins - 1x
EH33/Morris - 10x
NASA Scientific & Technical Info. Facility - 1x + Repro.


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N85-26995

JUN 1985
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**TABLE I**

DC 1200 Primer Lots Used in Project Program

<table>
<thead>
<tr>
<th>Springborn Labs ID #</th>
<th>DC Primer Lot #</th>
<th>Date Received at Springborn</th>
<th>Additional Description and Further Sample Labeling</th>
</tr>
</thead>
<tbody>
<tr>
<td>A**</td>
<td>QL033703</td>
<td>5/11/83</td>
<td>red opaque</td>
</tr>
<tr>
<td>B**</td>
<td>QL071621</td>
<td>11/29/83</td>
<td>red, leaked</td>
</tr>
<tr>
<td>C**</td>
<td>QL093752</td>
<td>11/29/83</td>
<td>clear, sealed</td>
</tr>
<tr>
<td>1***</td>
<td>EN057367</td>
<td>3/30/84</td>
<td>clear</td>
</tr>
<tr>
<td>2***</td>
<td>QL033705</td>
<td>3/30/84</td>
<td>red</td>
</tr>
<tr>
<td>3***</td>
<td>063711</td>
<td>3/30/84</td>
<td>red, S/L Exp 6/84, Temp 50/90F</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>RS3.900324, 7/83 MMSK343A025 83G382</td>
</tr>
<tr>
<td>4***</td>
<td>093713</td>
<td>3/30/84</td>
<td>red, S/L Exp 09/84, Temp 50/90F,</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>RS3.794481 09/83 MMSK343A025 83G529 (hold for J Mills)</td>
</tr>
<tr>
<td>5***</td>
<td>093733</td>
<td>3/30/84</td>
<td>red, S/L Exp 09/84, Temp 50/90F,</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>RS3.794481, 09/83 MMSK343A025 83G529</td>
</tr>
<tr>
<td>6***</td>
<td>071620</td>
<td>3/30/84</td>
<td>red, S/L Exp 7/82, Temp 50 to 90F,</td>
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<td></td>
<td></td>
<td></td>
<td>RS1:705481, 08/81 MMSK343A025 81G464</td>
</tr>
<tr>
<td>7+</td>
<td>QL103753</td>
<td>1/10/85</td>
<td>red</td>
</tr>
</tbody>
</table>

* A sample identification table is included as the first page in each report.

** Three (3) DC 1200 Primer Lots acquired by Springborn Laboratories for initial tests (see monthly reports 1, 2 and 3).

*** Six (6) DC 1200 Primer Lots received from NASA, Management Division Bldg. 4471, on March 30, 1984.

+ DC 1200 Primer Lot received from NASA, Bldg. 4612, Mr. Don Morris, Project Monitor.
1. SHEAR TESTS - VALIDATION STUDY

From a series of 10 DC 1200 primers two silane primers (#3 and #7) were selected for validation studies. Recent FTIR analyses (5/16/85, Figure 1) indicated that sample #3 contains the highest OH-absorption area (74) and sample #7 the smallest OH value (12)*.

Lap sheer tests were performed on 17 metal strips coated with primers #3 and 13 metal strips coated with primer #7, respectively. All lap shear values obtained are reported with Mean, Standard Deviation and Relative Standard Deviation calculation in graphical and tabular form in Figure 2. The recordings obtained on the Instron instrument are included in Figure 3 (panels L, M, N) and Figure 4 (panels O and P). A detailed description of the shear tests is given in Figure 5a-9b.

2. LAP SHEAR TESTS OF SEVEN NASA SILANE PRIMERS vs. INFRARED TESTS FOR OH - ABSORPTION.

2.1 Silane Aging vs OH-Absorption

A series of DC 1200 primers were analyzed four times during 1984-85 for their OH-Absorption areas and are enclosed in graphic form for each silane in Figures 10 to 19.

Silane samples deteriorated during the year (although covered with a dry nitrogen layer before closing the metal containers, some moisture and air can enter during opening). An OH-absorption value of > 80** indicates almost total hydrolysis of silane batch (i.e. sample slightly opaque if red dye added). The new silane batches OH-absorption values were found to be between 0 and 3. The average values for OH-areas are included in Figures 10 to 19.

The most recent FTIR spectra (May'85) for all ten (10) silane primers are included as Figures 20-29.

* In this report all four OH-absorption band values, given separately in previous reports, were averaged and only one absorption value is given. Areas were calculated using the Trapezoid Rule, where the area $A_{OH}$ is

$$A_{OH} = \int_{a=3470 \text{ cm}^{-1}}^{b=3360 \text{ cm}^{-1}} f(x)dx = \frac{Y_0 + Y_1 + Y_2 + Y_3 + Y_4}{2} \Delta x$$

AND $\Delta x = \frac{b-a}{n} = \frac{110 \text{ cm}^{-1}}{4} = 27.5 \text{ cm}^{-1}$

** Area calculated using Trapezoid Rule.
2.2 Correlation of OH-Absorption of Silane Primers with Lap Shear Tests of Coated Panels

FTIR area values of OH-absorptions recently obtained (5/16/85) were plotted against lap shear values (mean values) of the DC 1200 primer used for adhesive bonding between two aluminum panels (coated with deSoto primer). (The bonding procedure was described in the 10th report, pg. 3).

The graphic presentation of FTIR and shear test values is given in Figure 30. Individual values for each lap shear test are given in tables for test series R to Z in Figures 31a to 39b. The statistical values for panel tests bonded with DC 1200 silane primers #1 to 7 are summarized in the table below.

### Statistical Values for Lap Shear Tests of Seven Silane Primer Lots

<table>
<thead>
<tr>
<th>Silane Lot No.</th>
<th>n (Total of Lap Shear Tests)</th>
<th>M (Mean)</th>
<th>STD (Standard Deviation)</th>
<th>RSTD (Relative STD)</th>
<th>Fig. No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10</td>
<td>136</td>
<td>16</td>
<td>12</td>
<td>5-9, 31, 33, 34</td>
</tr>
<tr>
<td>2</td>
<td>11</td>
<td>82</td>
<td>26</td>
<td>32</td>
<td>31, 32, 35</td>
</tr>
<tr>
<td>3</td>
<td>21</td>
<td>60</td>
<td>22</td>
<td>35</td>
<td>5-9, 39</td>
</tr>
<tr>
<td>4</td>
<td>10</td>
<td>76</td>
<td>16</td>
<td>21</td>
<td>36, 37, 38</td>
</tr>
<tr>
<td>5</td>
<td>11</td>
<td>110</td>
<td>16</td>
<td>15</td>
<td>36, 37, 38</td>
</tr>
<tr>
<td>6</td>
<td>10</td>
<td>162</td>
<td>32</td>
<td>20</td>
<td>32, 33, 34</td>
</tr>
<tr>
<td>7</td>
<td>24</td>
<td>217</td>
<td>60</td>
<td>28</td>
<td>5-9, 35, 39</td>
</tr>
</tbody>
</table>


3. PLAN FOR FUTURE WORK

3.1 GC Analysis of Silane Primers (12th Progress Report)

Complete GC analyses of headspace polar volatiles evaluated for their alcoholic faction. This section is before completion and will be issued as the 12th Progress Report.
3.2 **Final Report**

A Final Report will be issued in June summarizing results obtained during the contract work.

4. **FINANCIAL STATUS**

The total cumulative expenditure incurred as of report dated 1/3/85 to 92% of the total budget.
FIGURE 1

VALUES ONLY FOR TOP LIQUID LAYER (SAMPLES HYDROLYZED)

AREA OH ABSORPTION
IR SPECTRUM VS SHEER TEST

Values:
\[ n = 13 \]
Mean: 21
SD: ± 67
Rel. SD: ± 31%

Values:
\[ n = 17 \]
Mean: 64
SD: ± 15
Rel. SD: ± 24%

LOT #3
LOT #7

UNITS (AREA OH/PSI)

DC 1200 LOT #

AREA-OH
MEAN PSI

ORIGINAL PAGE OF POOR QUALITY

FIGURE 2
### LAP SHEAR TESTS

<table>
<thead>
<tr>
<th>Panel # (ID)</th>
<th>DC1200 ID</th>
<th>lbs</th>
<th>PSI</th>
<th>% Adhesive Failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>L-1</td>
<td>3</td>
<td>36</td>
<td>72</td>
<td>90%</td>
</tr>
<tr>
<td>L-2</td>
<td>3</td>
<td>36</td>
<td>72</td>
<td>80%</td>
</tr>
<tr>
<td>L-3</td>
<td>5</td>
<td>38</td>
<td>76</td>
<td>75%</td>
</tr>
<tr>
<td>L-4</td>
<td>7</td>
<td>112</td>
<td>224</td>
<td>70%</td>
</tr>
<tr>
<td>L-5</td>
<td>7</td>
<td>90</td>
<td>184</td>
<td>70%</td>
</tr>
<tr>
<td>L-6</td>
<td>7</td>
<td>136</td>
<td>272</td>
<td>80%</td>
</tr>
<tr>
<td>L-7</td>
<td>7</td>
<td>172</td>
<td>344</td>
<td>50%</td>
</tr>
</tbody>
</table>

**Comments:**
<table>
<thead>
<tr>
<th>Panel # (ID)</th>
<th>DC1200 ID #</th>
<th>lbs</th>
<th>PSI</th>
<th>% Adhesive Failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>M - 1</td>
<td>3</td>
<td>42</td>
<td>84</td>
<td>100%</td>
</tr>
<tr>
<td>M - 2</td>
<td>3</td>
<td>41</td>
<td>82</td>
<td>100%</td>
</tr>
<tr>
<td>M - 3</td>
<td>3</td>
<td>39</td>
<td>78</td>
<td>90%</td>
</tr>
<tr>
<td>M - 4</td>
<td>3</td>
<td>38</td>
<td>76</td>
<td>95%</td>
</tr>
<tr>
<td>M - 5</td>
<td>7</td>
<td>93</td>
<td>186</td>
<td>70%</td>
</tr>
<tr>
<td>M - 6</td>
<td>7</td>
<td>74</td>
<td>148</td>
<td>50%</td>
</tr>
<tr>
<td>M - 7</td>
<td>7</td>
<td>81</td>
<td>162</td>
<td>50%</td>
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</table>

**COMMENTS:**
**TEST SERIES**

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<tr>
<th>Panel # (ID)</th>
<th>DC1200 ID 0</th>
<th>lbs</th>
<th>PSI</th>
<th>% Adhesive Failure</th>
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</thead>
<tbody>
<tr>
<td>N-1</td>
<td>3</td>
<td>38</td>
<td>76</td>
<td>100%</td>
</tr>
<tr>
<td>N-2</td>
<td>3</td>
<td>32</td>
<td>64</td>
<td>100%</td>
</tr>
<tr>
<td>N-3</td>
<td>3</td>
<td>35</td>
<td>70</td>
<td>100%</td>
</tr>
<tr>
<td>N-4</td>
<td>3</td>
<td></td>
<td></td>
<td>Broken during handling</td>
</tr>
<tr>
<td>N-5</td>
<td>7</td>
<td>58</td>
<td>116</td>
<td>50%</td>
</tr>
<tr>
<td>N-6</td>
<td>7</td>
<td>118</td>
<td>236</td>
<td>50%</td>
</tr>
<tr>
<td>N-7</td>
<td>7</td>
<td>136</td>
<td>272</td>
<td>50%</td>
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</table>

**COMMENTS:**
NASA-MSFC 6031.2

LAP SHEAR TESTS

<table>
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<th>Panel # (ID)</th>
<th>DC1200 ID</th>
<th>Ibs</th>
<th>PSI</th>
<th>% Adhesive Failure</th>
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<tr>
<td>0 - 1</td>
<td>3</td>
<td>42</td>
<td>84</td>
<td>100%</td>
</tr>
<tr>
<td>0 - 2</td>
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<td>44</td>
<td>88</td>
<td>100%</td>
</tr>
<tr>
<td>0 - 3</td>
<td>3</td>
<td>48</td>
<td>96</td>
<td>100%</td>
</tr>
<tr>
<td>0 - 4</td>
<td>7</td>
<td>154</td>
<td>308</td>
<td>88%</td>
</tr>
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<td>0 - 5</td>
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<td>164</td>
<td>328</td>
<td>88%</td>
</tr>
<tr>
<td>0 - 6</td>
<td>7</td>
<td>112</td>
<td>224</td>
<td>80%</td>
</tr>
<tr>
<td>0 - 7</td>
<td>7</td>
<td>102</td>
<td>204</td>
<td>20%</td>
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COMMENTS:
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<th>lbs</th>
<th>PSI</th>
<th>% Adhesive Failure</th>
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<tbody>
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<td>P-1</td>
<td>7</td>
<td>127</td>
<td>254</td>
<td>100%</td>
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<tr>
<td>P-2</td>
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<td>100%</td>
</tr>
<tr>
<td>P-3</td>
<td>7</td>
<td>60</td>
<td>190</td>
<td>96%</td>
</tr>
<tr>
<td>P-4</td>
<td>3</td>
<td>24</td>
<td>48</td>
<td>100%</td>
</tr>
<tr>
<td>P-5</td>
<td>3</td>
<td>22</td>
<td>44</td>
<td>100%</td>
</tr>
<tr>
<td>P-6</td>
<td>3</td>
<td>23</td>
<td>46</td>
<td>100%</td>
</tr>
<tr>
<td>P-7</td>
<td>3</td>
<td>19</td>
<td>38</td>
<td>100%</td>
</tr>
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</table>

COMMENTS:
AGING OF DC 1200 LOT #1

NASA-MSFC 6031.2
AGING OF DC 1200 LOT # 2

NASA-MSFC 6031.2

AREA-OH ABSORPTION

DATE

3-30-84  10-8-84  3-14-85  5-16-85

2  18  55  56
FIGURE 12

AGING OF DC 1200 LOT # 3

NASA-MSFC 6031.2

AREA-04 ABSORPTION

DATE

3-14-85

3-30-84

3-16-85

3-8-84

18-8-84

74

82

26

1
AGING OF DC 1200 LOT # 4

NASA-MSFC 6031.2

FIGURE 13

DATE

AREA-CH ABSORPTION

3-30-84
3-15-84
3-14-85
5-16-85

3
15
53
55

DATE
AGING OF DC 1200 LOT # 5

NASA-MSFC 6031.2

AREA-OH ABSORPTION

DATE

3-30-84  10-8-84  3-14-85  5-16-85

0  25  45  48
AGING OF DC 1200 LOT # A

NASA-MSFC 6031.2

AREA-CH ABSOPPTION

150 -
120 -
90 -
60 -
30 -
0 -

old Sample

41

67

Not Analyzed

96

Liquid (top)
layer
only

sample hydrolyzed

DATE

3-30-84
18-9-84
3-14-85
5-16-85

FIGURE 17
AGING OF DC 1200 LOT # C

NASA-MSFC 6031.2

AREA-OH ABSORPTION

150

120

90

60

30

0

133

134

102

Liquid (top)
layer only Sample hydrolyzed

3-30-84  10-8-84  3-14-85  5-16-85

DATE

FIGURE 19
NASA-MSFC 6031.2
DC-1200 LOT#6
5/16/85 .2MM CELL
NASA-MSFC 6031.47
DC1200 LOT A-TOP LAYER/DECOMPOSED
5/16/85 .2MM CELL
IR SPECTRA VS SHEER TESTS

NASA-MSFC 6031.2

UNITS (OH AREA/PSI)

250
200
150
100
50

LOT #1  LOT #2  LOT #3  LOT #4  LOT #5  LOT #6  LOT #7

DC 1200 LOT #
<table>
<thead>
<tr>
<th>Panel (ID)</th>
<th>DC1200 ID 0</th>
<th>lbs</th>
<th>PSI</th>
<th>% Adhesive Failure</th>
</tr>
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<tbody>
<tr>
<td>R-1</td>
<td>1</td>
<td>72</td>
<td>144</td>
<td>100%</td>
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<tr>
<td>R-2</td>
<td>1</td>
<td>80</td>
<td>160</td>
<td>100%</td>
</tr>
<tr>
<td>R-3</td>
<td>1</td>
<td>72</td>
<td>144</td>
<td>100%</td>
</tr>
<tr>
<td>R-4</td>
<td>2</td>
<td>52</td>
<td>104</td>
<td>100%</td>
</tr>
<tr>
<td>R-5</td>
<td>2</td>
<td>60</td>
<td>120</td>
<td>100%</td>
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<td>R-6</td>
<td>2</td>
<td>59</td>
<td>118</td>
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<td>R-7</td>
<td>2</td>
<td>52</td>
<td>104</td>
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**COMMENTS:**
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<th>PSI</th>
<th>% Adhesive Failure</th>
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<tbody>
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<td>T-1</td>
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<td>44</td>
<td>88</td>
<td>100%</td>
</tr>
<tr>
<td>T-2</td>
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<td>37</td>
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<td>100%</td>
</tr>
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<td>T-3</td>
<td>2</td>
<td>30</td>
<td>60</td>
<td>100%</td>
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<td>T-4</td>
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<td>28</td>
<td>56</td>
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</tr>
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<td>T-5</td>
<td>6</td>
<td>58</td>
<td>114</td>
<td>90%</td>
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<td>T-6</td>
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<td>68</td>
<td>136</td>
<td>75%</td>
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<tr>
<td>T-7</td>
<td>6</td>
<td>93</td>
<td>186</td>
<td>90%</td>
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**Comments:**
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<td>Panel # (ID)</td>
<td>DC1200 ID #</td>
<td>lbs</td>
<td>PSI</td>
<td>% Adhesive Failure</td>
<td></td>
<td></td>
</tr>
<tr>
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<td>-------------</td>
<td>-----</td>
<td>-----</td>
<td>--------------------</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S-1</td>
<td>6</td>
<td>56</td>
<td>112</td>
<td>103.2%</td>
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<td></td>
</tr>
<tr>
<td>S-2</td>
<td>6</td>
<td>70</td>
<td>144</td>
<td>103.9%</td>
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<tr>
<td>S-3</td>
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<td>86</td>
<td>172</td>
<td>106.2%</td>
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<td></td>
</tr>
<tr>
<td>S-4</td>
<td>6</td>
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**COMMENTS:**
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X
### Lap Shear Tests

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Comments:
## Test Series

### Lap Shear Tests

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**COMMENTS:**
## LAP SHEAR TESTS

### TEST SERIES X

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**COMMENTS:**
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**COMMENTS:**

**FIG 392**
NASA GO312
ASTM D1002 (mod)
LAP SHEAR TEST
CROSS HEAD SPEED 0.05"/MIN
CHART SPEED 0.5"/MIN
5-30-85 HKP