REVIEW OF FORMULATION AND TESTING PROCEDURES
FOR COAL TAR EPOXY (SSPC PAINT 16-68T)

by
A. Beitelman

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# REVIEW OF FORMULATION AND TESTING PROCEDURES FOR COAL TAR EPOXY (SSPC PAINT 16-68T)

The Corps of Engineers uses the Steel Structures Painting Council (SSPC) specification 16-68T for coal tar epoxy paint extensively. Although performance of the material is unquestioned, difficulty in obtaining the coal tar pitch used in its manufacture has been experienced during energy shortages; this problem has stimulated efforts to revise the specification.
This study critically examined the test methods and requirements set forth in the specification and evaluated several pitches as possible replacements. It is recommended that the sag test be simplified without altering the test requirement; that the nonvolatiles requirement be altered if a proposed change in the packaging ratio is implemented; and that coal tar pitch RT-12 be used as a replacement for the pitch now specified--if requirements can be written to provide a pitch with a minimum of water-extractable material, and if the odor requirement of the present specification is eliminated.
FOREWORD

This research was performed by the Paint Laboratory of the Construction Materials Branch, Materials and Science Division (MS), U.S. Army Construction Engineering Research Laboratory (CERL). The work forms a part of Civil Works Research Work Unit 31205. It is funded through the U.S. Army Engineer Waterways Experiment Station under reimbursable order WES RF-76-19. The Technical Monitor is Mr. L. G. Guthrie of the Directorate of Civil Works, Office of the Chief of Engineers.

Mr. P. Howdyshell is Acting Chief of the Construction Materials Branch and Dr. G. Williamson is Chief of MS. COL J. E. Hays is Commander and Director of CERL and Dr. L. R. Shaffer is Deputy Director.
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REVIEW OF FORMULATION AND TESTING PROCEDURES FOR COAL TAR EPOXY (SSPC PAINT 16-68T)

1 INTRODUCTION

Background

The Steel Structures Painting Council's (SSPC) Paint No. 16, Coal Tar Epoxy-Polyamid Black (or dark red) Paint, was originated by the U.S. Army Corps of Engineers Rock Island District in 1962. Later the same year, the first commercial batch of the material, which was given Corps of Engineers specification number C-200, was applied to about 1500 sq ft (139 m²) of upstream skinplate on a Mississippi River tainter gate near Gladstone, IL. In the mid 1960s the SSPC initiated efforts to standardize a formulation for a coal tar epoxy. Formulation C-200 was adopted by SSPC as specification 16-68T (commonly known as Paint No. 16), largely because of the Corps research. After the council's adoption of the formula, the Corps specification C-200 was modified to simply reference the more widely accepted SSPC specification (see Appendix A).

Since its adoption by SSPC, Paint No. 16 has gained wide acceptance throughout the country. Several major coating manufacturers have adopted the formulation for their standard shelf products, making it easily obtainable. Paint No. 16 is specified on numerous construction projects by both the government and private industry, and is used on many manufactured items such as pipes, valves, and storage tanks. It is accepted as a standard to which other products are compared, making it truly a "standard of industry."

Nevertheless, two major obstacles threaten continued use of Paint No. 16. The first obstacle is pollution control regulations, many of which limit the percentage of xylene that may be used in a coating material's solvent blend. The formulation for Paint No. 16 specifies xylene as the only solvent to be used in the material. No solvent or solvent blend has been found which meets air pollution regulations and still gives film properties equal to those found when xylene is used.

The second obstacle was encountered during the energy shortage of 1974-1975, when the coke industry had to use coal tar, which is one of its by-products, as a fuel to maintain its coke ovens. This severely limited the availability of coal tar pitch to the coatings industry. At the peak of the shortage, the premium grade of pitch used in Paint No. 16 could not be obtained from many major suppliers. Although this grade of pitch is now obtainable, its future availability to the coatings industry is in doubt.
Consequently, the Corps, in conjunction with the SSPC Advisory Committee on Coal Tar Epoxy-Polyamide Paint, initiated work toward revising the specification. It was decided that initial work should be directed toward evaluating a replacement pitch for the one presently specified. Criteria established for the replacement pitch are: (1) it should be available from more than one supplier, (2) its availability should be essentially unaffected by any future energy shortage or process change within the coke industry, and (3) the coating produced with the replacement pitch should have working properties and provide performances equal to those of the present material.

The committee suggested that a packaging ratio of four parts of the base component (A) to one part of the liquid epoxy resin (B) might be more economical than the present 3 1/2 to 1 ratio. Additionally, it was suggested that all test methods be critically evaluated and updated if necessary.

**Objective**

The objectives of this study were (1) to critically evaluate the test methods and requirements set forth in the SSPC Paint Specification, SSPC Paint 16-68T, and (2) to evaluate suitable replacement pitches for the coal tar that will be readily obtainable on a long-term basis.

**Approach**

The SSPC committee and the Corps agreed that RT-12 pitch would meet the availability requirements; however, its performance in the present formulation was questioned because of its low softening point. Therefore, samples of formulations using RT-12 and control samples of the standard formulation were obtained from two manufacturers for laboratory evaluation.

Although either of two methods can be used to produce an epoxy resin meeting the requirements of the Paint No. 16 specification, the respective resins do exhibit different properties in some formulations. Therefore, all experimental materials were evaluated with each of the two types of epoxy resin represented by Dow Chemical Company's DER-331 and Shell Chemical Company's EPON-828.
2 TEST PROGRAM

Materials Evaluated

The coal tar epoxy-polyamide paint called for in the specification is a heavy-bodied thixotropic material packaged as two components. The base component (A) contains a refined coal tar pitch, a liquid polyamide resin, a catalyst, a mineral filler, a gelling agent, and volatile thinners. The second component (B) is a liquid epoxy resin which is mixed into the base component just prior to application. Twenty variations of the two components were evaluated. In addition to RT-12 pitch, several other pitches were tested as replacement pitches, in case the preferred RT-12 pitch did not perform adequately. Table 1 gives the characteristics of the pitches used. Variations of Component A evaluated were:

- Material A--standard Paint No. 16 manufactured to meet the existing specification; used as a control for the tests.

- Material B--standard Paint No. 16 produced by a second manufacturer to meet the existing specification; used as a second control for the tests.

- Material C--similar to Paint No. 16 except the coal tar RT-12 replaced the pitch called for in the specification. A 3 1/2 to 1 mixing ratio was used. The composition by weight was:

<table>
<thead>
<tr>
<th>Component</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coal tar RT-12</td>
<td>35.0%</td>
</tr>
<tr>
<td>Polyamide resin</td>
<td>11.2%</td>
</tr>
<tr>
<td>Magnesium silicate</td>
<td>29.1%</td>
</tr>
<tr>
<td>Xylene</td>
<td>19.0%</td>
</tr>
<tr>
<td>Thixotrope and polar additive</td>
<td>4.4%</td>
</tr>
<tr>
<td>Catalyst</td>
<td>1.3%</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>100.0%</strong></td>
</tr>
</tbody>
</table>

- Material D--similar to Paint No. 16 except that coal tar RT-12 replaced the pitch called for in the specification, and the formulation was altered to be used with a 4 to 1 mixing ratio. The composition by weight was:

<table>
<thead>
<tr>
<th>Component</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coal tar RT-12</td>
<td>30.0%</td>
</tr>
<tr>
<td>Polyamide resin</td>
<td>9.6%</td>
</tr>
<tr>
<td>Magnesium silicate</td>
<td>37.5%</td>
</tr>
<tr>
<td>Xylene</td>
<td>17.3%</td>
</tr>
<tr>
<td>Thixotrope and polar additive</td>
<td>4.5%</td>
</tr>
<tr>
<td>Catalyst</td>
<td>1.1%</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>100.0%</strong></td>
</tr>
</tbody>
</table>
Table 1
Pitch Characteristics of Materials Evaluated

<table>
<thead>
<tr>
<th>Material</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
<th>H</th>
<th>I</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ball and ring (B and R)</td>
<td></td>
<td>*</td>
<td></td>
<td>34</td>
<td>*</td>
<td></td>
<td>56</td>
<td></td>
<td>70*</td>
</tr>
<tr>
<td>Softening point (°C)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>75-85</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Benzene insolubles (% wt)</td>
<td></td>
<td>*</td>
<td></td>
<td></td>
<td>11.7</td>
<td>22.3</td>
<td>16.2</td>
<td>20 min</td>
<td>19.5</td>
</tr>
<tr>
<td>Distillation to 300°C (% wt)</td>
<td></td>
<td>*</td>
<td></td>
<td></td>
<td>0.0</td>
<td>**</td>
<td>**</td>
<td>5.8</td>
<td>*</td>
</tr>
<tr>
<td>Softening point of residue (°C)</td>
<td></td>
<td>**</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>**</td>
</tr>
<tr>
<td>Float test</td>
<td></td>
<td>**</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>**</td>
</tr>
<tr>
<td>Specific gravity</td>
<td>1.25</td>
<td>**</td>
<td>**</td>
<td></td>
<td></td>
<td></td>
<td>1.22</td>
<td>1.23</td>
<td>1.24 min</td>
</tr>
<tr>
<td>Total bitumen</td>
<td></td>
<td>**</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>**</td>
</tr>
</tbody>
</table>

* Meets requirement for Paint No. 16 (see Appendix A).
† Manufacturer's product limits. Distillation is run to 400°C.
The manufacturer of material D altered the ratio of pitch to magnesium silicate considerably from the existing Paint No. 16 formulation.

- Material E--similar to Paint No. 16 except that coal tar RT-12 replaced the pitch called for in the specification and the formulation was altered to be used with a 4 to 1 mixing ratio. The composition by weight was:

<table>
<thead>
<tr>
<th>Material</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coal tar RT-12</td>
<td>35.1%</td>
</tr>
<tr>
<td>Polyamide resin</td>
<td>10.3%</td>
</tr>
<tr>
<td>Asbestine 3X</td>
<td>30.0%</td>
</tr>
<tr>
<td>Xylene</td>
<td>19.1%</td>
</tr>
<tr>
<td>Methanol</td>
<td>2.2%</td>
</tr>
<tr>
<td>Bentone</td>
<td>2.2%</td>
</tr>
<tr>
<td>Catalyst</td>
<td>1.1%</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>100.0%</strong></td>
</tr>
</tbody>
</table>

- Material F--manufactured to meet the requirements of Paint No. 16 formulation during the energy shortage. The pitch used had 22.3 percent benzene insolubles, but met the requirements of the specification in all other respects.

- Material G--a specially formulated material designed for a 4 to 1 mixing ratio and manufactured with roofing pitch. The composition by weight was:

<table>
<thead>
<tr>
<th>Material</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Roofing pitch</td>
<td>35.1%</td>
</tr>
<tr>
<td>Polyamide resin</td>
<td>10.3%</td>
</tr>
<tr>
<td>Asbestine 3X</td>
<td>30.0%</td>
</tr>
<tr>
<td>Xylene</td>
<td>19.1%</td>
</tr>
<tr>
<td>Methanol</td>
<td>2.2%</td>
</tr>
<tr>
<td>Bentone</td>
<td>2.2%</td>
</tr>
<tr>
<td>Catalyst</td>
<td>1.1%</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>100.0%</strong></td>
</tr>
</tbody>
</table>

- Material H--similar to Paint No. 16 except that a petroleum-derived pitch was substituted for the specified pitch on a volume basis.

- Material I--similar to Paint No. 16 except that Rule 66* exempt solvents were substituted for the specified xylene on a volume basis.

* Rule 66, enacted by the Los Angeles County Air Pollution Control Board in 1966, is a widely known and used air pollution regulation that (among other things) restricts the concentrations and use of photochemically reactive solvents. Exempt solvent systems are those that meet the Rule 66 requirements.
A Rule 66 exempt thinner was supplied with the material. The solvent blend in the final product by weight was:

<table>
<thead>
<tr>
<th>Component</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>High flash, VM &amp; P naphtha (16 percent aromatic)</td>
<td>30.6 percent</td>
</tr>
<tr>
<td>Cellosolve acetate</td>
<td>30.0 percent</td>
</tr>
<tr>
<td>Isobutyl isobutyrate</td>
<td>34.0 percent</td>
</tr>
<tr>
<td>Ethanol, 95 percent denatured</td>
<td>5.4 percent</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>100.0 percent</strong></td>
</tr>
</tbody>
</table>

The solvent blend for the thinner by volume was:

<table>
<thead>
<tr>
<th>Component</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>High flash VM &amp; P naphtha</td>
<td>90 percent</td>
</tr>
<tr>
<td>Cellosolve acetate</td>
<td>10 percent</td>
</tr>
</tbody>
</table>

Due to a high degree of incompatibility with this thinner, material I was re-evaluated with a thinner having the same solvent blend as that used in the manufacture of the base material. The second thinner/material combination was designated material I'.

Each variation of Component A was combined with both Dow's and Shell's epoxy resins meeting the requirements for Component B of Paint No. 16. A single batch of epoxy resin was procured from each company to be used with Materials A through I'.

**Tests Performed**

The following tests were performed as required by the specification for Paint No. 16 or as outlined below:

1. Brookfield viscosity of Component A (3.5.1).* To provide uniform results, the initial reading was taken after 2 minutes instead of the 1 minute specified.

2. Nonvolatile content of Component A (3.5.2).

3. Sag test (3.6.1).

4. Sag test--multinotch blade (Federal Test Method Standard No. 141, Method 4494). The applicator blade used had notches ranging from 14 to 60 mils (0.4 to 1.5 mm).

* Numbers in parentheses refer to the applicable section of the Paint No. 16 specification (Appendix A).
5. Viscosity characteristics. Materials were thinned as prescribed for the sag test in 3.6.1. Brookfield viscosity curves were plotted using no. 4, no. 5, and no. 6 spindles. The first reading (at 100 rpm) was taken after 2 minutes, and the following readings at successively lower speeds (down to 5 rpm) after 1 minute of stabilizing time.

6. Penetration test (3.6.2). Penetration readings were taken daily for 5 days to observe the rate at which hardness is actually attained.

7. Pot life test (3.6.3).

8. Adhesion test (3.6.4).

9. Odor test (3.6.5).

10. Exposure test. Exposure panels were prepared as follows: coatings were spray-applied using air atomization to a wet film thickness of at least 20 mils (0.5 mm) in one double-spray coat. Dry film thickness measurements showed the thicknesses to be in the 15 to 20 mil (0.4 to 0.5 mm) range with most panels having about 17 mils (0.43 mm) of coating. The edges of all panels were dipped, and each panel was given a single diagonal score approximately 1 1/2 in. (3.8 cm) long. Two atmospheric, two saltwater immersion, two cold tap water immersion, and one reserve panel were prepared. Panels were placed under the following test conditions after a minimum of 5 days cure time:

a. Atmospheric panels were exposed at 45° south at Champaign, IL. (Area is nonindustrialized.)

b. Saltwater panels were immersed in an aquarium containing synthetic seawater as specified in ASTM-1141-52. The water was continuously filtered and aerated.

c. Cold water panels were immersed in a tank fed continuously by city tap water. The temperature of this tank remained at 68° to 70°F (20° to 21°C).

d. The reserve panel was not used during this study.

Test Results

Tables 2, 3, and 4 show the test results. Table 2 shows the results of tests 1 through 9. Table 3 gives the penetration test values on a day-to-day basis. These values, although not required by the specification, show the rate at which hardness is obtained with the various formulations. Table 4 evaluates the panels immersed in synthetic seawater.
for 6 months. The panels exposed to the atmosphere and cold tap water were visually examined for failure; the only failures observed were in materials I and I' immersed in cold water.
Table 2
Results of Tests 1 through 9

<table>
<thead>
<tr>
<th>Material</th>
<th>Component A Viscosity ( (N\cdot s/m^2) )</th>
<th>Component A Nonvolatile Content, %</th>
<th>Epoxy Resin Used*</th>
<th>Thinning Required for Sag Test, %</th>
<th>Sag Test**</th>
<th>Leneta Anti-Sag Index</th>
<th>Penetration**</th>
<th>Pot Life**</th>
<th>Adhesion**</th>
<th>Odor**</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>290 (29.0)</td>
<td>81.7</td>
<td>D</td>
<td>5</td>
<td>P</td>
<td>7.4</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>P</td>
</tr>
<tr>
<td>B</td>
<td>150 (15.0)</td>
<td>78.1</td>
<td>D</td>
<td>10</td>
<td>P</td>
<td>11</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>P</td>
</tr>
<tr>
<td>C</td>
<td>156 (15.6)</td>
<td>80.4</td>
<td>S</td>
<td>2</td>
<td>F</td>
<td>4.2</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>F</td>
</tr>
<tr>
<td>D</td>
<td>163 (16.3)</td>
<td>80.7</td>
<td>D</td>
<td>10</td>
<td>P</td>
<td>10.8</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>F</td>
</tr>
<tr>
<td>D</td>
<td>58 (5.8)</td>
<td>77.8</td>
<td>D</td>
<td>4</td>
<td>F</td>
<td>8.0</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>F</td>
</tr>
<tr>
<td>F</td>
<td>259 (25.9)</td>
<td>81.7</td>
<td>S</td>
<td>9</td>
<td>P</td>
<td>9.0</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>P</td>
</tr>
</tbody>
</table>
Table 2 (cont'd)

<table>
<thead>
<tr>
<th>Material</th>
<th>Component A Viscosity, (N•S/m²)</th>
<th>Component A Nonvolatile Content, %</th>
<th>Epoxy Resin Used*</th>
<th>Thinning Required for Sag Test, %</th>
<th>Sag Test**</th>
<th>Leneta Anti-Sag Index</th>
<th>Penetration**</th>
<th>Pot Life**</th>
<th>Adhesion**</th>
<th>Odor**</th>
</tr>
</thead>
<tbody>
<tr>
<td>G</td>
<td>99 (9.9)</td>
<td>79.5</td>
<td>D</td>
<td>7</td>
<td>P</td>
<td>11</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>P</td>
</tr>
<tr>
<td>H</td>
<td>94 (9.4)</td>
<td>82.0</td>
<td>S</td>
<td>7</td>
<td>P</td>
<td>11</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>P</td>
</tr>
<tr>
<td>I</td>
<td>&gt;400 (&gt;40.0)</td>
<td>85.8</td>
<td>D</td>
<td>33</td>
<td>P</td>
<td>11</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>P</td>
</tr>
<tr>
<td>I'</td>
<td>&gt;400 (&gt;40.0)</td>
<td>85.8</td>
<td>S</td>
<td>36</td>
<td>P</td>
<td>11</td>
<td>P</td>
<td>P</td>
<td>p⁺</td>
<td>P</td>
</tr>
</tbody>
</table>

* D = Dow Chemical Company's resin DER-331; S = Shell Chemical Company's EPON-828.
** P = pass specification; F = fail specification.
† No odor from the pitch; however, a pronounced odor from the solvent blend remains.
### Table 3

Penetration Test Results

<table>
<thead>
<tr>
<th>Material</th>
<th>Epoxy Resin</th>
<th>1 day</th>
<th>2 day</th>
<th>3 day</th>
<th>4 day</th>
<th>5 day</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>D</td>
<td>6</td>
<td>5 1/4</td>
<td>3+</td>
<td>1 1/2+</td>
<td>1-</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>7 1/2-</td>
<td>6+</td>
<td>6-</td>
<td>3 1/2</td>
<td>2+</td>
</tr>
<tr>
<td>B</td>
<td>D</td>
<td>2 1/2</td>
<td>1/2</td>
<td>1/4+</td>
<td>1/4-</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>2 3/4+</td>
<td>1/2</td>
<td>1/4-</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>C</td>
<td>D</td>
<td>5 3/4+</td>
<td>5 1/2+</td>
<td>3 1/4-</td>
<td>1 1/2</td>
<td>3/4</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>7 3/4+</td>
<td>6 1/2-</td>
<td>5 3/4-</td>
<td>2 1/4</td>
<td>1</td>
</tr>
<tr>
<td>D</td>
<td>D</td>
<td>5+</td>
<td>2+</td>
<td>3/4-</td>
<td>1/2-</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>5</td>
<td>2+</td>
<td>1/2+</td>
<td>1/4</td>
<td>0</td>
</tr>
<tr>
<td>E</td>
<td>D</td>
<td>3 3/4</td>
<td>3/4</td>
<td>1/2+</td>
<td>1/4</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>2 1/2</td>
<td>3/4+</td>
<td>1/2</td>
<td>0+</td>
<td>0</td>
</tr>
<tr>
<td>F</td>
<td>D</td>
<td>2+</td>
<td>1-</td>
<td>1/2</td>
<td>1/2</td>
<td>1/4-</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>2+</td>
<td>1-</td>
<td>1/2</td>
<td>1/2-</td>
<td>1/4-</td>
</tr>
<tr>
<td>G</td>
<td>D</td>
<td>3+</td>
<td>3/4+</td>
<td>1/4-</td>
<td>0</td>
<td>0</td>
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<tr>
<td></td>
<td>S</td>
<td>3-</td>
<td>1/4</td>
<td>0+</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>H</td>
<td>D</td>
<td>6 1/2</td>
<td>5 3/4</td>
<td>2+</td>
<td>1-</td>
<td>1/2-</td>
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<tr>
<td></td>
<td>S</td>
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<td>3 3/4</td>
<td>2 1/4</td>
<td>1-</td>
<td>1-</td>
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<tr>
<td>I</td>
<td>D</td>
<td>5 1/2</td>
<td>4 1/2</td>
<td>4 1/4</td>
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<td></td>
<td>S</td>
<td>6+</td>
<td>3 3/4</td>
<td>3 1/2</td>
<td>2 1/2</td>
<td>1 1/4+</td>
</tr>
<tr>
<td>I'</td>
<td>D</td>
<td>6 1/2</td>
<td>5 1/2+</td>
<td>5 1/2+</td>
<td>4 1/2-</td>
<td>3 1/2+</td>
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<tr>
<td></td>
<td>S</td>
<td>6 1/2</td>
<td>6 1/2</td>
<td>6 1/2</td>
<td>6+</td>
<td>5+</td>
</tr>
</tbody>
</table>
Table 4  
Evaluation of Salt Water Immersion Panels  

<table>
<thead>
<tr>
<th>Material</th>
<th>Epoxy Resin</th>
<th>Adhesion Loss* (in. [mm])</th>
<th>Order of Water Discoloration</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>D</td>
<td>0</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>D</td>
<td>1/4 (6.4)</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>3/4 (19.1)</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>D</td>
<td>0</td>
<td>1 (darkest)</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>D</td>
<td>D</td>
<td>0</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>1/2 (12.7)</td>
<td></td>
</tr>
<tr>
<td>E</td>
<td>D</td>
<td>1/2 (12.7)</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>1/8 (3.2)</td>
<td></td>
</tr>
<tr>
<td>F</td>
<td>D</td>
<td>1 (25.4)</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>1 1/4 (31.8)</td>
<td></td>
</tr>
<tr>
<td>G</td>
<td>D</td>
<td>3/4 (19.1)</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>1/8 (3.2)</td>
<td></td>
</tr>
<tr>
<td>H</td>
<td>D</td>
<td>1/4 (6.4)</td>
<td>7</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>2 to 4** (5.1 to 10.2 cm)</td>
<td></td>
</tr>
<tr>
<td>I</td>
<td>D</td>
<td>complete</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>complete</td>
<td></td>
</tr>
<tr>
<td>I'</td>
<td>D</td>
<td>complete</td>
<td>8 (clear)</td>
</tr>
<tr>
<td></td>
<td>S</td>
<td>complete</td>
<td></td>
</tr>
</tbody>
</table>

* Values reflect the distance from the score mark, that the coating can be removed using a sharp knife.  
** Poor application and sag properties accelerated failure.
ANALYSIS AND DISCUSSION OF TEST METHODS AND RESULTS

Discussion of Test Methods

Viscosity Test

The original intent of the viscosity requirement was to provide a material fluid enough so that a uniform mixture could be obtained when the components are combined in a normal field application. Additionally, this requirement limits the amount of thinner required to achieve a sprayable viscosity. Initially, materials such as A and F appear to meet the intent of this requirement, but additional factors must be considered.

The viscosity of coatings containing gelling agents often changes after extended storage. The degree of change often varies from batch to batch depending on the incorporation technique used by the manufacturer. The change can range from a slight loss in viscosity to an increase of more than 100 percent after 1 year's storage; the typical change is an increase of 30 to 40 percent. Such an increase does not render a material having an initial viscosity of 160 unworkable even at low temperatures; however, if the initial viscosity is in the 260 to 290 range, the material may become very difficult to mix in low-temperature field conditions. Based on these observations, it would seem appropriate to leave the viscosity requirement unchanged.

Sag Test

Because experience has indicated that the sag requirement of Paint No. 16 is the most difficult requirement for manufacturers to meet, considerable work was done on the sag test and related gel properties of the materials evaluated. It was assumed as a starting point that the requirement for no sagging of a 14-mil (0.5 mm) wet film at the prescribed viscosity is both necessary for a practical field application and practical for a manufacturer to produce. Experience has shown that both these assumptions are basically true.

Use of a Brookfield viscometer to observe gel properties of the tested formulations, which were non-Newtonian materials, would seem to be logical. The materials were all mixed and thinned as prescribed by the specification; several variations of the test procedure were then used. The specification calls for the use of a no. 4 spindle turning at 100 rpm and having a reading of not less than 99. Moreover, it calls for taking five readings at 1-minute intervals. This procedure appears to be overly lengthy and awkward. Although the viscosity does decrease over the entire 5-minute period, the major decrease occurs during the first 2 minutes, after which the viscosity levels off to a
decrease of only 1 or 2 units per minute. Thus, to save time, the initial viscosity reading was taken and recorded after a 2-minute interval. This reading was found to be quite reproducible.

The necessity of obtaining a reading of 99 at 100 rpm also makes the test lengthy and awkward. The scale limit of the machine is 100. During the thinning process, when a reading is taken there is no way of knowing how much more thinning will be required to obtain a reading of 99. Simply lowering the speed to 50 rpm does not solve the problem, for it was observed that a material producing a reading of 99 at 100 rpm can produce readings ranging from 60 to 80 at 50 rpm, depending on the gel property of the material (see the viscosity curves provided in Appendix B).

Viscosity measurements obtained using no. 4, no. 5, and no. 6 spindles at various speeds were compared. The viscosity measurements obtained using the various spindles at a given speed for a given material compare very favorably. The measurements are especially reproducible between the no. 4 and no. 5 spindles. Consequently, using a no. 5 spindle to obtain the viscosity required for the sag test seems more appropriate. This would enable the tester to observe the viscosity change produced by a given amount of thinning and judge how much additional thinning would be required to obtain the specified viscosity.

The use of a multinotch blade to evaluate the sag properties of this material proved to be of little if any benefit. Although the blade can dramatize the effect of exceptionally poor or exceptionally superior sag properties, its results in marginal areas are not consistent with the specification test. This is because a doctor blade of a given gap will apply varying thicknesses of material depending on the gel properties of the material. Thus, a gap of 35 mils (0.9 mm) may apply a 14-mil (0.4 mm) film of one material but only 12 mils (0.3 mm) of a similar material with different gel properties. The specification test takes this fact into consideration by requiring a wet film measurement rather than specifying the gap of the doctor blade.

Penetration and Pot Life Tests

The penetration and pot life tests required by the specification effectively insure that the coating cures in an acceptable length of time. Failure of the penetration test indicates that the coating will not obtain a sufficient hardness to withstand the abuse dealt many coatings within the first several weeks after their application. Failure of the pot life test indicates that when combined, the components react so rapidly that there is insufficient time to apply a normal-size batch before it becomes too viscous to use. Both tests give reproducible results and should remain a part of the specification.
Adhesion Test

The adhesion test insures that the coating will adhere both to steel and to itself. Both of these capabilities are essential to coating performance. The test is very practical and should remain unchanged in the specification.

Odor Test

The examination for odor is rather subjective. When the original specification was written, the odor test was important because a major use for Paint No. 16 was inside potable water tanks and pipes. It is highly doubtful that this use can continue. The Food and Drug Administration, Division of Regulatory Guidance, has ruled that all coal tars contain polynuclear aromatics and are therefore carcinogenic. They have further stated that coal tar shall not be used in any location where it can potentially contaminate potable water. Tests indicate that coated steel panels immersed in beakers of distilled water for 6 months did impart some taste to the water in all cases. Although often very minor, this is obviously contamination and would therefore come under the regulation of the Food and Drug Administration.

Discussion of Test Results

Table 2 indicates that four of the base materials evaluated had viscosities over 160 poise (16.0 N·s/m²) and would therefore fail the specification requirement. Of these four, the viscosity of material D was well within experimental error and should be considered as meeting the specification requirement. Material I, which had a viscosity of over 400 poise (40.0 N·s/m²), is judged to be too viscous to be mixed and handled in a normal field application. Materials A and F, although more viscous than the specification limit, were not found to be difficult to mix and did not require an excessive amount of thinner.

Table 2 shows that the nonvolatile content requirement for Component A was met by all the materials tested.

Five of the materials failed the sag test performed using the cold-rolled steel panels and masking tape as called for in the specification (Table 2). Results obtained using the multinothal blade were not consistent with the results of the masking tape test. It is thought that the gel properties of the material adversely affect the reliability of the blade.

The penetration values recorded daily (Table 3) indicate that except for the Rule 66 material thinned with strong solvents (material I'), all the materials passed this test. It is of some interest that half
the materials met the requirement in 2 days. In addition, materials containing the softer pitches hardened at about the same rate as did the standard materials.

All materials passed the pot life and adhesion tests.

All the tested materials containing the softer pitches retained a faint odor of pitch both at the end of the odor test and again after 6 months on the immersion panels and the atmospheric-exposed panels of materials C, D, E, G, and H.

The atmospheric panels were evaluated after 6 months' exposure. All panels showed varying degrees of white or brown chalking. The hypothesis that the degree of chalking could be attributed to a particular type of pitch was not found to be true. Neither was the degree of chalking found to be attributable to the source of the pitch. Although the pitches for materials B, E, and G were all produced by a single manufacturer, B and E showed the least amount of chalking, whereas G showed the greatest amount. There is very good reproducibility of chalking characteristics between panels of a single base material, indicating that the method of manufacture of the epoxy resin has no effect on chalking. The adhesion test performed on the atmospheric panels in the area of the score resulted in no failures.

The cold water panels were evaluated after 6 months' immersion. The only materials which failed the adhesion test after immersion were I and I', with I' being the poorer of the two. The loss of adhesion occurred between the material and the steel and included the entire immersed area. A brownish color was noted below the waterline on panels coated with materials C, D, and I.

The synthetic seawater immersion panels showed considerable variation in adhesion after 6 months. The variation ranged from no loss of adhesion using materials A and C, to complete loss of adhesion and blistering below the waterline when materials I and I' were used. The reproducibility between duplicate panels is excellent; however, definite conclusions cannot be drawn relative to the effects of the pitch or epoxy used. Materials A and B, which were control materials from the two suppliers, exemplified the results noted. Material A had no loss of adhesion at the score when combined with either epoxy resin, whereas material B showed a 1/4-in. (6.4 mm) loss of adhesion using Dow's epoxy and a 3/4-in. (19.1 mm) loss using Shell's. The areas where loss of adhesion occurred at the scores of the various systems are shown in Table 4, and the following generalizations appear appropriate:

1. Materials I and I' show obvious adhesion failure. Initial blistering was noted after about 1 month in immersion. The panels prepared with material I' have a higher degree of blistering and poorer adhesion to steel than panels prepared with material I.
2. Panels coated with Material F, produced with a high benzene insoluble pitch, had a notably larger area of poor adhesion than the control panels. This pitch would therefore seem to be an inappropriate replacement for the pitch now specified.

3. With the above-mentioned exceptions, all remaining pitches appear to perform as well as the control pitch. These include materials produced with RT-12 pitch, roofing pitch, and petroleum pitch.

4. The test results do not show any consistent results favoring either of the epoxy resins.

An unanticipated phenomenon occurred during the exposure test. The saltwater panels were originally immersed in a single aquarium containing the synthetic seawater. After several weeks it was noted that the water was becoming increasingly dark. To locate the panel or panels responsible for the problem, single panels representing each material were removed from the aquarium and placed in individual beakers containing synthetic seawater. After 2 months, the water in the beakers was observed and the relative amounts of discoloration noted as shown in Table 4.

Care must be taken that the data in Table 4 are not misinterpreted. The data indicate the order of discoloration, but because the variation between the orders was not consistent, they do not necessarily indicate relative amounts of discoloration. Observed without comparison, orders of 5 and greater would probably be considered clear. To note any differences, the materials had to be observed in a column 4 in. (10.2 cm) tall. On the other hand, materials with orders of 1 and 2 had a distinctive brown color which could be easily observed in a column only 1 in. (2.5 cm) tall. This test has clearly shown that materials C and D, which contained RT-12 pitch from a single supplier, were chiefly responsible for the discoloration noted in the aquarium. Material E, which contained RT-12 pitch from another source, exhibited far less discoloration.
4 CONCLUSIONS AND RECOMMENDATIONS

The tests performed led to the following conclusions and recommendations relative to the validity and practicality of the specification:

1. The test for viscosity of the base material in the present specification gives reproducible results. Although a higher viscosity limit could be tolerated at normal ambient temperatures, the present limit of 160 poise (16.0 N·s/m²) is necessary to provide a manageable material at low ambient temperatures. No change in this test or specification limit is recommended.

2. The total nonvolatiles test in the present specification gives reproducible results and requires no alteration. The specification requirement would need revision if compositional changes in the formulation were made to provide 4 to 1 packaging ratio.

3. The sag test is a practical requirement; however, the test procedure could be simplified by requiring a Brookfield reading of 50, using a no. 5 spindle at 100 rpm. This change could simplify the thinning procedure without changing any of the other test requirements. The use of a multinoth blade to test sag properties is not practical for this material.

4. The penetration test requirement can be met without difficulty even when a softer pitch is employed in the manufacture of the material. This test is of increased importance when Rule 66 exempt solvent systems are used.

5. The pot life test or test requirement need not be altered for the proposed formulation changes.

6. The adhesion test or test requirement need not be altered for the proposed formulation changes.

7. The odor requirement will have to be dropped if RT-12 pitch is used in the material.

8. The existing specification requirements for the epoxy resin are sufficiently restrictive to provide a material with reproducible performance irrespective of the method of resin manufacture.

9. Any reference pertaining to the use of Paint No. 16 in potable water tanks and lines should be removed from the specification.

10. The RT-12 pitches from the two manufacturers contained different amounts of water-extractable materials. The pitch manufacturers
should therefore be contacted to determine the nature of the extractable materials and possible methods of control in order to insure that a uniform material is obtained. If a uniform material can be obtained and the odor requirement is dropped from the specification, RT-12 pitch can be used as an acceptable replacement for the pitch now specified in Paint No. 16.
APPENDIX A:

EXISTING SPECIFICATION FOR PAINT NO. 16
STEEL STRUCTURES PAINTING COUNCIL
PAINT SPECIFICATION
No. 16, Coal Tar Epoxy-Polyamide
Black (or Dark Red) Paint

1. SCOPE
1.1 This specification covers a two-component coal tar-epoxy black (or dark red) paint which employs a co-reacting polyamide resin and an aromatic tertiary polyamine catalyst as curing agents. It is self-priming and exhibits good adhesion to clean structural steel and concrete surfaces. It has excellent water resistance, good chemical and oil resistance and is not significantly affected by continuous exposure to direct sunlight.

2. DESCRIPTION
2.1 This paint is a heavy-bodied, thixotropic material which is usually spray applied in two heavy coats to produce a dry film having a minimum thickness of 16 mils. At normal temperatures it dries dust free in about four hours and becomes thoroughly hardened after 3 to 5 days of curing.

2.2 The base component (A) of this two-part paint contains a refined coal tar pitch, a liquid type polyamide resin, a polyamine catalyst to accelerate curing rate, a mineral filler, a gelling agent to induce thixotropic, anti-sag properties and volatile thinners. The second component (B) is a liquid type epoxy resin which is mixed into the base component just prior to application. After mixing, the paint remains usable with thinning for upwards of four hours at normal ambient temperatures, but its viscosity will show a gradual rise during this period. The storage properties of each component are excellent. However, some separation of the ingredients in the base component may take place during prolonged storage and its apparent viscosity may increase. Hand stirring should readily restore the material to its original homogeneity, but to break down the thixotropic gel formed during storage requires vigorous power stirring.

2.3 Steel surfaces which are to receive this coating should be prepared by blast cleaning to a commercial grade (SSPC-SP 6-63). Where the surfaces are to be subjected to very severe chemical exposures, blasting to the near-white grade (SSPC-SP 10-63T) should be specified. The adhesion of the coating to smooth surfaces such as cold-rolled steel or intact mill scale has been found to be substantially inferior to that attained on blast roughened surfaces. Although experience with this specific coal tar epoxy paint over pickled surfaces is lacking, it is believed that performance would be very satisfactory.

2.4 This paint is suitable for use on steel surfaces subject to fresh water immersion, salt water immersion, tidal and splash zone exposures, chemical exposures, buried exposure, and for interiors of tanks and lines containing crude oil, salt brine, and petroleum products. The cured coating imparts little or no odor and taste to water and, with respect to toxicity, has been approved by the U.S. Public Health Service for the interior surfaces of potable water tanks.

2.5 The mixed paint, prior to any addition of field thinners, contains approximately 75% by volume of nonvolatile, film forming solids.

3. REQUIREMENTS
3.1 Composition. Coal tar epoxy polyamide paint shall contain ingredients conforming to the requirements specified in paragraph 3.7 and shall have the composition shown in Table 1.
No. 16 COAL TAR EPOXY-POLYAMIDE BLACK (OR DARK RED) PAINT

TABLE 1
COAL TAR EPOXY-POLYAMIDE COATING

Component A

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Formula % by Weight*</th>
<th>Range Lbs. for 3.5 Gallon Batch</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coal Tar Pitch</td>
<td>35.0</td>
<td>34.0 - 36.0</td>
</tr>
<tr>
<td>Polyamide Resin</td>
<td>11.5</td>
<td>11.0 - 12.0</td>
</tr>
<tr>
<td>Magnesium Silicate**</td>
<td>31.0</td>
<td>30.0 - 32.0</td>
</tr>
<tr>
<td>Xylene***</td>
<td>18.7</td>
<td>18.0 - 21.0</td>
</tr>
<tr>
<td>Ethyl Alcohol (95% Denatured)</td>
<td>1.0</td>
<td>0.9 - 1.1</td>
</tr>
<tr>
<td>Gelling Agent C or D (Approximate Amount)</td>
<td>1.5</td>
<td>1.5</td>
</tr>
<tr>
<td>Catalyst</td>
<td>1.3</td>
<td>1.2 - 1.3</td>
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<td></td>
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</tr>
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</table>

Component B

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>1 gallon Lbs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid Epoxy Resin</td>
<td>9.70 Lbs.</td>
</tr>
</tbody>
</table>

*Column 1 gives the nominal formula, and Column 2 shows the maximum range permitted for individual batches.

**When so specified by the purchaser, a dark red oxide colored paint shall be furnished in which 50% or more (by volume) of the magnesium silicate is replaced by synthetic red iron oxide. The color shall approximate that of Chip No. 20061 of Federal Color Standard No. 595 unless more precisely prescribed by purchaser and agreed to by the manufacturer. The red paint shall meet all of the test requirements prescribed for the black, except that the non-volatile content of Component A shall be higher by an amount reflecting the greater specific gravity of the iron oxide pigment.

***In those cases where the specified volatiles are not permitted under local air pollution regulations the volatile portion of the coal tar-epoxy paint shall be replaced with exempt material to the extent necessary to assure compliance with the applicable regulation.

3.2 Manufacture. The constituent raw materials of Component A shall be mixed and thoroughly dispersed by such methods as will provide a product which is uniform, stable, free from lumps and grit, and entirely suitable for the purpose intended. A freshly opened container of Component A shall be capable of being hand-stirred to a homogeneous mixture of uniform consistency even after 12 months in storage. With respect to processing of the base component, it is suggested that the following points be kept in mind: (1) anti-sag characteristics of the mixed paint are largely determined by the degree of dispersion of the gelling agent. Dampening of the gelling agent with the alcohol prior to milling is believed to be helpful in obtaining a thorough dispersion. Although either gelling agent C or D is effective, it is believed that less of the material and less work is required with D to attain the desired paint properties; indeed in order to make most efficient use of agent D it would appear that high shear paint making operations should largely precede the addition of the gellant. (2) The use of a low oil absorption type of magnesium silicate may facilitate the attainment of a balance of properties such that viscosity and anti-sag requirements will be met; and (3) in order to provide some margin of processing freedom, the minimum nonvolatile content specified in paragraph 3.5 and the range in composition shown in Table 1 have been established at levels which permit an appreciable variation in the amount of xylene used in making up the base component. Also, the amount of gelling agent to be used is specified only on an approximate basis.

3.3 Packaging and Labeling

3.3.1 Three and one-half (3.5) gallons of Component A shall be packaged in a standard five-gallon container with lug type removable lid. Component B shall be packaged to the full mark in a one-gallon tin can with friction lid. Other forms of packaging which provide the required ratio of components are permissible unless otherwise specified by the purchaser.
3.3.2 Each container of each component shall be suitably labeled with the following information, filling in indicated items:

Name: Coal Tar Epoxy-Polyamide
Black (or Dark Red)
Paint Component ............................................................
Specification: SSPC-Paint 16-68T
Lot Number: ......................................................................
Stock Number: ...............................................................
Date of Manufacture: ........................................................
Quantity of Component in Container: ...................................
Information as may be required by Federal and State Laws: ............................................................
Manufacturer’s Name: .......................................................
Address: ......................................................................  

3.4 Directions for Use. The following directions for use shall appear on, or accompany each container (or unitized group of containers) of Component A.

Directions for Use of Coal Tar Epoxy-Polyamide Paint

This paint is primarily intended for use both as a primer and finish coat(s) over clean steel which has been blast cleaned. It may be used on steel in chemical or marine environments, on buried tanks and pipes on steel immersed in fresh or salt water, in tidal, splash and weather zone exposures and for interiors of tanks and lines containing crude oil, salt brine or caustic soda. It is suitable for use in potable water tanks and lines.

To prepare the paint for application, add the entire contents of the epoxy resin (Component B) container to the previously stirred contents of the related container of base (Component A) and mix vigorously for at least two minutes with a power agitator equipped with a 3" or longer blade. Some thinning may be desirable for spray application. Only xylene may be used as a thinner and not more than one-half gallon to a 4.5 gallon batch should be added. Apply the paint as soon after mixing as practicable since the material will thicken substantially over a two-hour period and may set up in the paint tank within two to four hours during very warm weather unless cooled prior to and/or after mixing.

This coating is usually applied by spray in two coats to a dry film thickness of 16 mils at its thinnest spots. This requires a spreading rate of 60 square feet per gallon of unthinned paint. A uniformly applied wet film of slightly more than 10 mils will produce an 8-mil dry film. However, in actual practice, upwards of 12 mils of wet paint will probably be required for each coat to obtain the desired minimum thickness. Allow a minimum of 12 hours and a maximum of 72 hours drying time between coats under normal painting conditions. Long drying times between coats will probably cause poor intercoat adhesion and it is advisable in warm weather to reduce the maximum interval between coats. In very hot weather with surfaces exposed directly to the sunlight, it may be necessary to limit the intercoat drying period to 24 hours or less.

The paint may be applied to large, flat or nearly flat surfaces such as tanks by high-pressure airless spray. For application to complex surfaces, use heavy-duty conventional air atomization spray equipment. If the application is by brush, apply with a stiff brush heavily loaded with paint; apply quickly and smoothly and avoid excessive brushing.

Do not apply this coating when the receiving surfaces or the ambient temperatures are below 50°F., nor unless it can reasonably be anticipated that the average ambient temperature will be 50°F. or higher for the five-day period subsequent to the application of any coat.

Clean all equipment immediately after use with a suitable solvent. Such cleaning solvents as high-flash naphtha, xylene or toluene are satisfactory for clean-up, but can be improved by adding about 10-20 percent of methyl isobutyl ketone and 10 percent isopropyl or normal butyl alcohol.

3.5 Quantitative Requirements.

3.5.1 The viscosity of Component A shall not exceed 160 poises when tested as follows:

Fill a container having a diameter and a height of not less than 3 and 3¾ inches respectively to a depth of not less than 3 inches with a representative sample of Component A. Set up a Model RVT or RVF-100 Brookfield Synchro-Electric Viscometer with a No. 7 spindle and with guard removed. Bring the sample to (and thereafter maintain) a temperature of 25°C. and stir vigorously for two minutes with a stiff spatula. Immediately after stopping the motor, lower the viscometer until one-half of the “neck” mark on the spindle is covered. Run the viscometer at 100 r.p.m. for one minute and take a reading of the position of the pointer on the dial. If the dial reading is 40 or less, the viscosity shall be considered to be 160 poises or less. If the reading is over 40, immediately start the motor and take additional readings at one-minute intervals. If one or more readings of 40 or less are obtained out of 10 readings, taken at one-minute intervals, the viscosity of the material shall be considered to be within specification limits.
3.5.2 The non-volatile content of Component A shall be not less than 77 percent (by weight) when tested as follows:

Place a stirrer made of stiff wire into a small disposable aluminum dish of about two inches in diameter and weigh to the nearest 0.1 milligram. As rapidly as possible, place between two and three grams of Component A into the dish and weigh immediately to the nearest 0.1 milligram. After weighing, spread the material over the bottom of the dish. Heat the dish, wire and contents in a well-ventilated convection-type oven maintained at \(105^\circ\pm 2^\circ\text{C.}\) for three hours. After the material has been in the oven for a few minutes, and periodically thereafter, stir the material. Cool in a desiccator, weigh to the nearest 0.1 milligram and calculate the percentage non-volatile on a weight basis.

3.6 Quantitative Requirements. The paint shall meet the following quantitative requirements:

3.6.1 Sag. The paint shall not sag when tested as follows:

Prepare approximately one pint of the material by thoroughly mixing 100 ml. of Component B into 350 ml. of Component A. Determine its viscosity immediately after mixing, using the same procedure as for Component A (paragraph 3.5.1) but employing a No. 4 spindle. If all of five readings taken at one-minute intervals are above 99, reduce the viscosity by adding xylene in small increments until a reading not greater than 99 is obtained. Press a strip of one-inch masking tape across the full width of a solvent-cleaned 3" x 6" cold-rolled steel panel. The tape should be parallel to and centered on the shorter axis of the panel. Within five minutes after making the final check of viscosity, apply the material to the panel at a wet film thickness of at least 14 mils as determined by an Interchemical wet film thickness gage. The application may be made with a doctor blade having a gap of approximately 25 mils or by brush. Immediately after applying the material, carefully remove the masking tape and stand the panel in a vertical position (with the bare strip horizontal) in a draft-free, \(77^\circ\pm 3^\circ\text{F.}\) location. Examine the panel after four hours. Sagging or running of the coating into the base area shall constitute failure of the material to pass the sag test. Save the mixed paint for the penetration test and adhesion test described below.

3.6.2 Penetration. The paint shall pass the following test:

Select and solvent spray-clean two 3" x 6" cold-rolled steel panels in accordance with Method 2011 of Federal Test Method Standard 141. Draw down in accordance with Method 2161, a coat of the paint mixed (including any thinning) for the sag test. Allow the film to dry 18 to 24 hours in a horizontal position at \(77^\circ\pm 3^\circ\text{F.}\) and at a relative humidity of not over 60 percent. Apply a second coat over and at right angles to the first, using freshly mixed paint prepared identically to that used for the first coat. (Save both paints for preparing adhesion test panels.) The drawdown applicator(s) shall be such as to provide a total dry-film thickness for the two coats of 23 \(\pm\) 3 mils and the coats shall be of approximately equal thickness. Allow the second coat to dry in a horizontal position at \(77^\circ\pm 3^\circ\text{F.}\) After 120 hours of curing, clamp the panel onto the table of penetrometer (ASTM D5-61) so that the needle is over an area which is within the prescribed thickness range (as measured by Federal Test Method 6181) and determine the penetration, using a total load of 200 grams applied for five seconds at \(77^\circ\text{F.}\) The average of the three lowest out of five penetration readings, all taken within a one-centimeter square, shall not exceed 3/100 of a centimeter after 120 hours of curing.

3.6.3 Pot Life. The paint shall pass the following test:

Mix 100 ml. of Component B into 350 ml. of Component A, both of which have been brought to a temperature of \(77^\circ\pm 3^\circ\text{F.}\) before mixing. Pour the material at once into a pint tin can, seal tightly and store at \(77^\circ\pm 3^\circ\text{F.}\) Examine the material four hours after it was mixed. For its pot life to be considered satisfactory, the mixed material must still be in a fluid condition, and when thinned, with no more than 100 ml. of xylene shall be lump-free and brushable.

3.6.4 Adhesion. The paint shall pass the following test:

Sand blast two steel panels (similar to those used in the penetration test) with a clean, 30 to 50 mesh non-metallic abrasive until a uniform, gray-white surface, with well developed anchor pattern, is achieved. Blow off dust with a clean air blast. Brush-apply one coat of the mixed paint used for the penetration test panels, allowing the first coat to dry 18 to 24 hours at \(77^\circ\pm 3^\circ\text{F.}\) before applying the second. Each coat shall be applied at a wet film thickness of between 10 and 14 mils. After the final coat has cured for 120 hours at \(77^\circ\pm 3^\circ\text{F.}\), test the adhesion of the coating to the metal with a sharp knife. It shall strongly resist being removed from the metal. Also test the intercoat adhesion by attempting to separate the coats with the knife. Any delamination of the two coats shall constitute failure.

3.6.5 Odor.

Examine the paint film on one of the adhesion panels for odor after the second coat has been cured for 48 hours. The film shall be free of any odor except for a faint odor of xylene.
3.7 Specifications for Ingredients. The ingredients shall meet the listed requirements:

3.7.1 Coal Tar Pitch.
(a) B and R softening point, degree C 70 min. 75 max.
(Method: ASTM D316-62)
(b) Ash, percent by weight 0.5 max.
(Method: ASTM D271-58)
(c) Benzene insolubles, percent by weight 18.9 max.
(Method: ASTM D367-49)
(d) Volatiles, percent by weight
   Under 250°C 0
   Under 300°C 5.0 max.
(Method: ASTM D20-56)

3.7.2 Epoxy resin shall be a di-epoxide condensation product of bisphenol A and epichlorohydrin with terminal epoxide groups and shall have the following properties:
(a) Epoxy equivalent
   (Method: ASTM D1652-62T) 180 min. 200 max.
(b) Non-volatile content, percent
   (1-2 gram sample after one hour @ 105° ± 2°C) 99 min.
(c) Color (Gardner)
   (Method: ASTM D1544-63T) 5 max.
(d) Specific gravity, 25°C/25°C 1.15 min. 1.18 max.
(e) Viscosity, 25°C, poises (Brookfield) 100 min. 160 max.

3.7.3 Polyamide resin shall be a condensation product of a dimerized fatty acid and polyamines and have the following properties:
(a) Amine value 330 min. 360 max.

Amine value is defined as the milligrams of potassium hydroxide equivalent to the amine alkalinity present in one gram of sample. It is determined by a potentiometric titration with standard perchloric acid according to the following General Mills method:

-1- Weigh the approximate amount of well mixed resin to give a titration in the range of 12-18 ml. into a tared 200 ml. Berzelius tall form beaker on an analytical balance. Cover the beaker with aluminum foil to minimize contact with air.

-2- From a graduated cylinder, carefully add 90 ml. of nitrobenzene, insert a stirring bar, cover the beaker with foil and stir on a magnetic stirrer to dissolve the sample. Add the nitrobenzene immediately after weighing the sample. Nitrobenzene is highly toxic and a fume hood should be used for all operations.

-3- From a graduated cylinder, add 20 ml. of glacial acetic acid to the sample solution and stir for several minutes.

-4- Immerse the electrodes into the sample solution, stir for two minutes and titrate potentiometrically with 0.1 N perchloric acid using the millivolt scale. Record the millivolt readings every 0.1 ml. Plot a graph showing the millivolts against the titration. The endpoint is the midpoint of the inflection on the titration curve.

-5- Conduct a blank determination on 90 ml. of nitrobenzene and 20 ml. of acetic acid. The blank need only be determined once for each lot of nitrobenzene used. On the majority of lots used, the blank has been found to be zero.

-6- Calculate the amine value:

\[
\text{AMINE VALUE} = \frac{\text{Sample Titration} - \text{Nitrobenzene Blank}}{\text{Weight of Sample}} \\
\times \text{Normality} \times 36.1
\]

(a) Non-volatile content, percent 97 min.
   (1-2 gram sample after one hour @ 105° ± 2°C)
(b) Color (Gardner) 12 max.
   (Method: ASTM D1544-63T)
(c) Specific gravity 25°C/25°C 0.96 min. 0.98 max.
(d) Viscosity, 75°C, poises (Brookfield) 7 min. 9 max.

3.7.4 Magnesium silicate shall conform to ASTM Specification D605-53T.

3.7.5 Xylene shall conform to ASTM Specification D364-61 (Industrial Grade).

3.7.6 Ethyl alcohol (95% denatured) shall conform to Federal Specification O-E-760, ethyl alcohol (Ethanol); denatured alcohol, and proprietary solvent, grade 3 or 4.

3.7.7 Gelling agent C shall be an organic derivative of magnesium montmorillonite for use in low polarity hydrocarbons. It shall be a creamy white powder having a bulk density of 15 ± 0.2 pounds per gallon and a water content of 3.0 percent maximum. (Bentone 38, National Lead Company, is such a product.)

3.7.8 Gelling Agent D shall be a microcrystalline hydrated magnesium silicate powder containing colloidal, rod-shaped, submicron particles obtained by processing naturally occurring chrysotile. Typical properties include: Density—2.2 gm./cc; refractive index—1.5; absorbed water—1%; surface area—68 sq. meters/gm. (Avibest C. FMC Corporation, is such a product).

3.7.9 The Catalyst shall be 2, 4, 6 Tri (dimethyl-amino methyl) phenol. (DMP-30, Rohm and Haas Company, is such a product.)

3.7.10 Synthetic Red Iron Oxide shall conform with Federal Specification TT-P-375.
No. 16 COAL TAR EPOXY-POLYAMIDE BLACK (OR DARK RED) PAINT

4. SAFETY

4.1 This paint contains highly flammable solvents whose vapors are toxic. It also contains other ingredients which are irritants to the skin and eyes in addition to being toxic. It should not be processed or applied near an open flame. When being applied in closed spaces, sufficient ventilation should be provided to insure that the vapor does not reach explosive concentrations. Workmen should avoid breathing its vapors and be extremely careful not to get any of the paint in their eyes. Prolonged contact of the paint with the skin should be avoided. Spray painters in addition to wearing protective clothing and gloves should wear an air-fed hood.

5. INSPECTION

5.1 Samples of any or all ingredients used in the manufacture of this paint may be required by the purchaser, and shall be supplied upon request, along with the supplier's name and identification for the material.

5.2 All manufacturing of paint supplied under this specification, and all materials supplied under this specification shall be subject to inspection by the purchaser or his representative. All parts of the plant actually utilized in the manufacture of the paint shall be accessible to the inspector. The purchaser shall have the right to reject any material supplied under this specification which is found to be defective under this specification. In case of dispute, the arbitration or settlement procedure established in the contract, if any, shall be followed. If no arbitration procedure is established, the procedure specified by the American Arbitration Association shall be used.

APPENDIX

A.1 The recommendations contained in this appendix are believed to represent current good practice, but are not to be considered as requirements of the specifications.

A.2 This coating is capable of providing long term protection to steel surfaces in environments involving fresh or sea water immersion, tidal and splash zone exposure, condensation, burial in soil and exposure to brine, crude oil, alkalis, chemical fumes, chemical splashings and immersion in moderately strong sulfurous and muriatic acids. Its weathering properties, which are good, can be improved by applying a finish coat of a compatible aluminum pigmented paint.

A.3 Coal tar epoxy-polyamide paints exhibit excellent adhesion to other primers such as vinyl butyral wash primers, some types of vinyls and to freshly-applied conventional (without coal tar pitch) epoxy resin coatings. Some types of zinc-rich primers based on vinyl resin or epoxy-polyamide binders appear to have some merit for use with this coating. However, its adhesion to long-cured epoxy resin based coatings, either zinc-rich or conventionally pigmented, is only fair. The highly satisfactory performance of the coal tar epoxy-polyamide paint, when used as a self-primed system, makes it debatable as to whether special primers would contribute significantly to the capabilities of the system.

A.4 Directions for use are summarized briefly in 3.4. Application should be as specified in applicable portions of SSPC-PA 1-64.

A.5 For protection of steel to be subjected to moderately severe or severe exposures, this coating should be applied in not less than two coats and the minimum thickness at any point should be not less than 16 mils. If the system is applied in two coats, the drying time between coats should not be less than 12 hours. If the coating is applied in three coats, two of them may be applied the same day provided at least a few hours of drying is allowed between coats and provided all coats are of approximately equal thickness. Long delays between coats can result in poor intercoat adhesion; consequently, the maximum drying time between coats should not exceed 72 hours under normal conditions and should be much less in very warm weather, particularly if the surfaces are in direct sunlight.

A.6 Airless spraying equipment capable of developing upwards of 2,000 p.s.i. hydraulic pressure is satisfactory for applying this coating to simple flat or near flat surfaces and to medium to large diameter pipes. Conventional air spray should be employed to paint complex surfaces. The conventional equipment should include a bottom withdrawal paint tank equipped with gages, regulators and an air-driven agitator or a material pump capable of delivering the paint to the gun at a uniform pressure without significant pulsation. Material and air hoses to the gun should be kept as short as practicable and should not be less than \( \frac{3}{4} \)" inside diameter. The spray gun should be of the conventional type and have a fluid tip with an orifice of about 0.09 inches in diameter. The air cap should be of the external atomization type with seven holes. It should be designed to employ the atomization air efficiently and to produce a uniform spray pattern. The atomization air to the gun should be clean and dry, and be supplied to the gun at a pressure of not less than 80 p.s.i. The temperature of the two components of this paint at the time of mixing has a great effect on its pot life. They should be stored under ventilated cover during hot weather and protected from the direct sun before and after mixing insofar as practicable. In some cases, it may be desirable to have Component A packaged in white containers in order to assist in the control of heat buildup. During cool weather, provisions should be made to maintain the temperature of the mixed paint at above 60°F., and preferably above 70°F., if satisfactory atomization is to be obtained without excessive thinning.
A.7 The dark red paint covered by the footnote (**) to Table 1 is considered to have essentially the same capabilities as the black when used for the first, second, or both coats of the customary 2-coat system. Inasmuch as the paint does not lend itself to precise color matching or to the attainment of other than dark, dull shades of brownish-red, the purchaser should be careful not to specify a precise color which is impractical to produce. Inasmuch as the black paint may be somewhat less expensive and is the more widely produced and used, it is suggested that it be employed in all situations where there is no color preference.

A.8 The coal tar epoxy resin paint covered by this specification employs a co-reacting polyamide resin and a tertiary polyamine catalyst as curing agents. Coal tar epoxy materials which employ polyamines as the sole curing agent are produced by a number of firms. While the paint covered by the instant specification is suitable for many severe exposures, it is possible that some of the polyamine-cured paints have resistance properties which would enable them to perform better than the former in certain specific chemical environments.

Military Specification MIL-P-23236 (Ships), Class 2, covers a coal tar epoxy paint whose curing component is not limited to any specific type. The specification is primarily of the performance type which requires that the materials meet certain Quality Assurance Provisions, included in which is one based upon an 18 months' service test. The specification is intended primarily to provide coatings which are suitable for

ship tanks used for fuel and salt water ballast, but there is little doubt that coal tar epoxy coatings meeting the specified requirements are of high quality and well suited for the broader applications commonly associated with paints of this type. However, the Military specification does not contain provisions which assure that the paint will necessarily have application, intercoat adhesion, low odor, strength, rapid curing rate, hardness and flexibility properties comparable to those required by the instant specification.

NOTE: Formulations based on compositions of epoxy resin and coal tar are the subject of U.S. Patent 2765288.

A-9 Paint prepared in accordance with this specification does not necessarily comply with Section K, Rule 66, Los Angeles County Air Pollution Control District or other air pollution control requirements.

If the formulation of a paint product is changed, in the solvent portion only with no change in the rest of the formulation, and if the test characteristics (ASTM-B117-64 Salt Spray Testing, hardness, scratch, abrasion, and flexibility) are unchanged, then it may be presumed, pending the completion of long-term service tests, that the substitution of one solvent for another has made no change in the service life of the material. Since coal tar epoxies change greatly in physical properties during the early months, these tests should preferably be run both immediately after application and again one month later. See footnote (***) of Table 1.
APPENDIX B:

VISCOSITY CURVES OF MATERIALS EVALUATED
MATERIAL - B  
EPOXY - DOW
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

POISE

NO THINNING
THIN 10%

RPM

MATERIAL - D
EPOXY - SHELL

#6
#5
#4
NO THINNING

THIN 9%

MATERIAL—F
EPOXY—DOW

POISE

RPM

#4

#5

#6
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