CONFIDENTIAL

MONSANTO CHEMICAL COMPANY
LION OIL COMPANY DIVISION
Research Department
El Dorado, Arkansas

Interim Report

on

DEVELOPMENT OF FIRE RESISTANT ASPHALT BASE MASTIC COATINGS

2057
Project No.

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Report No.

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I. INTRODUCTION

This work was undertaken because there was an ever increasing consciousness of the need for fire resistance in mastic coatings. This included mastic coatings with many end uses, such as protective coatings for metals, moisture and vapor proof barriers over insulation and in roof construction. The Asphalt and Protective Coating Research and Development group received numerous reports of fires originating from bituminous mastics. Examples were fires resulting from: (1) spraying the mastics on hot vessels (usually above 700°F); (2) dropping hot welding rods onto surfaces coated with uncured mastics. Also, it was known that conventional asphalt coatings in a cured state would contribute materially to a fire once ignited. Roofing asphalt was the main contributing fuel in the multi-million dollar loss suffered by General Motors at their Detroit automatic transmission plant.

It was becoming apparent that a mastic coating's fire resistance would be an ever increasingly important factor governing its acceptability for use in industrial plants. Also, the fire resistance of mastic coatings used was beginning to affect insurance rates.

II. SUMMARY

Initial work on this project was directed toward developing a suitable test for fire resistance. In the second phase asphalt base mastics employing added material to make them more fire resistant were tested. Various inorganic salts and Montars (still bottoms from Aroclors) were the additives tested. Several experimental mastics were subjected to standard performance tests such as: (1) accelerated weathering; (2) salt spray protection; (3) low temperature adhesion; (4) corrosion of metals; and (5) moisture vapor transmission rate.

Practically all the materials tested improved fire resistance. However, each fire retarding agent also reduced the level of performance in one or more of the areas listed above. It was found that small variations in the percentages of solvent retained in the coating at time of fire testing markedly affected the fire test results.

III. CONCLUSIONS

Work covered by this report indicates it would be most difficult to formulate a quality, combination protective coating-moisture vapor barrier mastic employing Mastars or inorganic salts. Good fire resistance can be attained but serious deficiencies in other performance areas would have to be overcome.

IV. RECOMMENDATIONS

Work should continue toward developing a formulation which is fire resistant and also has the other necessary properties required in good protective coatings and moisture vapor barriers. The asphalts now available from the propane decarbonization unit should be investigated to determine if they might have some special utility in this application. A wider range of fire retarding agents should be investigated, including the new Birum compounds now available from the R & E Division.

V. PATENT STATUS
(Prepared by H. B. Roberts)

It appears that this report does not contain subject matter of a patentable nature.

VI. NOTEBOOK REFERENCES

Work covered by this report is recorded in Ilion Refinery and Research Notebooks as follows:

Refinery Notebooks 390, 396, 402, 404, 414, 419, 420, 426, 436 and 438.

Research Notebooks 550 and 654.

VII. EXPERIMENTAL - DESCRIPTION AND DISCUSSION OF THE WORK

Fire Tests

A number of tests were run to determine the effect of the state of cure on free burning time (burning time after test flame goes out) of Nokorode Seal Kote and a Foster cutback mastic. A solids content of 97.0% appeared to be a critical point for both mastics. No free burning was experienced in either mastic when cured to a solids content greater than 97.0% by wt.

The results on these coatings and on a Foster clay emulsion are shown in Table 1.

Several inorganic salts were added to the basic Seal Kote formula and fire tested. Most of these were tested at two states of cure. In each case a Seal Kote control was run and the reduction in burning time given by the experimental formulation was recorded. Sodium bicarbonate, aluminum sulfate, ammonium phosphate and borax were some of the salts employed. All were effective in reducing the free burning time. The use of these materials produced coatings of fair fire resistance at a solids content of 92% by wt. This is in contrast to 97% by wt. solids required in Seal Kote to obtain no free burning in the fire test. Detailed test results are shown in Table 2.

Mastics of the basic Seal Kote formula were prepared using Montars 3, 4 and 5 in concentrations of 4 to 25% based on the asphalt content. Concentrations as high as 10% were not effective in reducing flammability. All three Montars were effective at concentrations of 25%. Test results are shown in Table 3.

Mastics were prepared from 25/75 - Montar 4/asphalt base using the Seal Kote formula for fillers. The solvents employed were mixtures of 300/360 b.r. naphtha and three chlorinated solvents. The chlorinated solvents were chlorothene, perchloroethylene and Wyandotte's C-L-2. Mastics in which as little as 20% of the solvent was chlorinated showed measurable improvement in fire resistance. A solvent combination of 25% perchloroethylene, 50% C-L-2 and 25% 300/360 naphtha produced a mastic which showed no free burning when tested with 10.2% of solvent remaining in the film. Test results on these chlorinated solvent blends are shown in Table 4.

Moisture Vapor Transmission

Moisture vapor transmission constants were determined on a series of blends employing Montars and inorganic salts. The inorganic salts increased permeability in general by a factor of two and the Montars by a factor of 10 to 40.

Corrosion of Various Metals

This test as outlined in procedure IV of the Appendix was run on Nokorode Seal Kote and a mastic of the same filler composition as Seal Kote but employing a base composed of 25% Montar #4 and 75% Lion 180/200 roofing asphalt.

Neither mastic caused any of the three metals to corrode.

Low Temperature Performance Test

This test as outlined in procedure V of the Appendix was run on the following coatings:

(1) Lion Asbestos Roof Coating.
(2) Nokorode Seal Kote (medium grade).
(3) Roof Coating C-6-1 (Project 2058, Monsanto Roofing Program).
(4) Mastic from 25% Montar #4 and 75% 180/200 roofing asphalt.

None of the above four mastics showed any signs of failure in this test.

Salt Spray Test

Salt spray tests run according to the procedure outlined in procedure VI of the Appendix were run on six mastics containing inorganic salts and five mastics containing Montars.

All the experimental blends gave poorer performance than did Nokorode Seal Kote. The results are tabulated in Tables 6 and 7.

Weatherometer Tests

Montars Nos. 3, 4 and 5 were blended with 177°F softening point roofing asphalt in a ratio of 1/3 - Montar/asphalt. Accelerated weathering tests of 25 mil films of these materials and the base asphalt were made as described in procedure VII of the Appendix.

All the Montars had a definite detrimental effect on the weatherability of the asphalt. Montar #3 showed the least deleterious effect. The results are tabulated in Table 8.

Uniformity of Montars

As the test program progressed and additional samples of Montars were received, it became evident that the varied widely in composition from batch to batch. Inspection test data showing this variation are given in Table 9 on two Montar #4 samples and four Montar #5 samples.

VIII. ECONOMIC EVALUATION

Fire resistant coatings now available from competition are being specified by some companies in place of Nokorode Seal Kote for certain applications. This is being done even though the cost of the competitive coating is triple that of Seal Kote.

We face an almost certain loss of a considerable volume of business unless we add a fire resistant coating to our line. Its addition to our line should bring new business that we could not otherwise acquire. The emphasis on reduction of fire hazard in industrial plants makes coating cost a secondary consideration. Therefore, a fire resistant coating with a reasonable manufacturing cost can be marketed with a good margin of profit.

IX. ACKNOWLEDGEMENT

To Dr. A. J. Hoiberg for initial guidance.

To Technicians L. L. Simpson, B. F. Fuller and R. A. Purtle for assistance in preparation of basic materials and in testing finished products.

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X. APPENDIX

Table 1

Fire Tests on Mastics at Various States of Cure

(Test Method - Procedure I of Appendix)

Plaque No.		% Solvent	Duration in secs. test flame	Seconds to flash	Seconds to flame	Duration of film burning	Duration of free burning
Con 8	Seal Kote (medium grade)	3.4%	170	90	110	350	180
Con 1	Ditto	8.6	170	75	80	400	230
Con 2	Ditto	10.0	185	90	100	380	205
Con 10	Ditto	3.7	190	85	90	230	40
Con 11	Ditto	2.8	180	80	90	180	0
Con 33	Ditto	6.7	170	90	100	890	720
24	Foster Cutback Mastic	6.5	170	90	100	390	220
23	Ditto	3.0	180	130	N	N	0
10	Ditto	2.7	170	130	N	N	0
16	Ditto	2.4	178	120	N	N	0
9	Foster Clay Emulsion Mastic	2.6	170	170	N	N	0

N - no burning.

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Table 2

Fire Tests on Mastics Containing Inorganic Salts

(Test Method - Procedure I of Appendix for panels SA, SB, 2, 3 and 4
Procedure II for remainder)

Panel No.	Composition	% Solvent	Duration Test Flame seconds	Seconds to flash	Seconds to flame	Duration of film burning	Duration of free burning	Duration free burning of control
Set 1								
SA	72.5% cutback (55% 170/175 SP asphalt, 45% 306/360 naphtha) 15% 7T asbestos 2% sodium bicarbonate 2.5% aluminum sulfate 8% slate flour	4.7	180	140	N	N	N	180
SB	72% cutback 16% 7T asbestos 2% sodium bicarbonate 4% aluminum sulfate 6% slate flour	4.3	175	N	N	N	N	180
2	96% Seal Kote (medium) 4% sodium bicarbonate	4.0	180	100	140	185	5	180
3	96% Seal Kote (medium) 4% aluminum sulfate	5.0	170	110	140	170	N	180
4	94% Seal Kote (medium) 4% sodium bicarbonate 2% aluminum sulfate	5.7	178	100	140	178	N	180
5-2	97% Seal Kote (medium) 3% aluminum sulfate	4.1	180	130	150	180	N	180

(continued)

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Table 2 (continued)

Panel No.	Composition	% Solvent	Duration Test Flame seconds	Seconds to flash	Seconds to flame	Duration of film burning	Duration of free burning	Duration free burning of control
Set 1 (continued)								
7-4	97% Seal Kote (medium) 3% sodium bicarbonate	4.4	180	100	150	180	N	180
7-6	97% Seal Kote (medium) 1% aluminum sulfate 2% sodium bicarbonate	4.5	175	100	120	175	N	180
Con.8	Seal Kote (medium)100%	3.3	170	90	110	350	180	180
Set 2								
10	1201-1:90% Seal Kote 5% ammonium phosphate 5% sodium bicarbonate	13.2	175	80	90	320	145	220
9	1201-1	9.0	180	85	90	190	10	240
12	1201-2:90% Seal Kote 5% aluminum sulfate 5% sodium bicarbonate	12.5	190	80	95	240	50	220
11	1201-2	7.5	185	90	110	190	5	240
14	1201-3:90% Seal Kote 5% ammonium phosphate 5% aluminum sulfate	11.2	170	80	90	350	180	220
13	1201-3	5.7	170	85	100	170	N	240
16	1201-4:88% Seal Kote 5% borax 5% aluminum sulfate 2% zinc oxide	9.2	180	70	80	240	60	220
15	1201-4	7.5	185	70	85	185	N	240

(continued)

Table 2 (continued)

Panel No.	Composition	% Solvent	Duration Test Flame seconds	Seconds to flash	Seconds to flame	Duration of film burning	Duration of free burning	Duration free burning of control
Set 2	(continued)							
8	1201-5:93% Seal Kote 5% borax 2% zinc oxide	12.9	170	80	90	230	60	220
17	1201-5	7.5	185	70	85	185	X	240
C4	Seal Kote(medium)100%	10.9	190	70	80	410	220	220
C3	Seal Kote(medium)100%	6.3	170	80	90	410	240	240
Set 3								
9	94% Seal Kote 4% aluminum sulfate 2% sodium bicarbonate	10.7	175	80	100	340	165	230
10	Ibid. 9	7.7	180	85	111	335	155	205
11	94% Seal Kote 4% sodium carbonate 2% aluminum sulfate	9.9	185	70	90	200	15	230
12	Ibid. 11	9.0	195	85	110	385	190	205
13	96% Seal Kote (medium) 4% sodium bicarbonate	9.5	175	70	90	220	45	230
14	Ibid. 13	9.4	180	70	90	390	210	205
15	96% Seal Kote (medium) 4% aluminum sulfate	6.6	180	65	90	350	170	230
16	Ibid. 15	10.1	187	90	110	400	213	205

(continued)

Table 2 (continued)

Panel No.	Composition	% Solvent	Duration Test Flame seconds	Seconds to flash	Seconds to flame	Duration of film burning	Duration of free burning	Duration free burning of control
	Set 3 (continued)							
6	96% Seal Kote (medium) 2% aluminum sulfate 2% sodium bicarbonate	8.4	180	130	N	N	N	230
5	Ibid. 6	4.1	185	85	N	N	N	205
8	94% Seal Kote (medium) 2% sodium bicarbonate) 2% aluminum sulfate)dry 2% ammonium phosphate)	5.4	175	95	N	N	N	230
7	Ibid. 8	3.3	165	100	N	N	N	205
C-1	Seal Kote 100%	7.7	170	75	80	400	230	230
C-2	Seal Kote 100%	8.0	185	90	100	390	205	205
	Set 4							
4	94-B:94% Seal Kote + 1/4% Redicote E 2% sodium bicarbonate) 2% aluminum sulfate)dry 2% ammonium phosphate)	13.6	165	65	70	440	275	256
5	Ibid. 4	11.8	170	65	70	290	120	210
6	Ibid. 4	10.8	180	60	70	340	160	205
1	94-A:94% Seal Kote + 1/4% Redicote E 2% sodium bicarbonate)in 8 oz. 2% aluminum sulfate)water 2% ammonium phosphate)	24.8	180	60	70	420	240	256
2	Ibid. 1	15.5	185	70	80	250	65	210
3	Ibid. 1	8.9	170	80	90	170	N	205

(continued)

Table 2 (continued)

Panel No.	Composition	% Solvent	Duration Test Flame seconds	Seconds to flash	Seconds to flame	Duration of film burning	Duration of free burning	Duration free burning of control
Set 1 (continued)								
7	96:96% Seal Kote (med.) 2% sodium carbonate)in 8 oz. 2% aluminum sulfate) water	22.4	195	52	60	480	285	256
8	Ibid. 7	15.3	190	55	60	370	180	210
9	Ibid. 7	10.6	170	65	80	185	15	205
Controls								
72	Seal Kote	15.1	169	50	70	425	256	256
74	Seal Kote	9.2	180	50	74	390	210	210
C-2	Seal Kote	8.9	185	90	180	390	205	205

N - No burning.

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Table 3

Fire Tests on Mastics Containing Montars Nos. 3, 4 or 5

(Test Method - Procedure II of Appendix)

Panel No.	Material	Time to flash	Time to Burning	Duration of test flame	Duration of burning	Time of free burning	% Solids at time of test
40	Seal Kote	80 sec	120 sec	170 sec	650 sec	480 sec	97.2
41	Seal Kote	120	150	175	175	N	97.4
3-1	Montar 3 (10%) in base asphalt	85	100	177	510	333	94.7/94.8
4-1	Montar 4 (10%) in base asphalt	90	140	175	450	275	95.6/95.7
5-1	Montar 5 (10%) in base asphalt	90	130	170	390	220	95.6/95.5
Regular Montar							
1	Seal Kote	95	150	180	340	160	98.4/99.0
3	Montar 3 (2% in mastic	100	110	175	290	115	98.7/98.6
Montar 4							
4	Montar 4 (4% in mastic	100	140	180	1.0	N	97.5/97.3
20	Seal Kote	50	60	190	390	200	88.5
21	Seal Kote	60	90	185	415	230	94.3
22	Seal Kote	65	-	165	280	115	95.5
17	Montar 3 (25) in base asphalt	55	70	170	250	80	87.5
18	Ditto	60	80	160	170	10	94.5
19	Ditto	60	90	180	180	N	95.8
14	Montar 4 (25%) in base asphalt	50	60	180	310	130	85.5
15	Ditto	60	80	190	190	N	92
16	Ditto	60	85	170	160	-10	93.4
11	Montar 5 (25%) in base asphalt	50	60	170	170	N	91.8
12	Ditto	70	95	170	160	-10	95.8
13	Ditto	60	100	150	140	-40	96.0

No burning.

Table 4

Fire Tests on Mastics Prepared with Chlorinated Solvent Thinners

(Test Method - Procedure II of Appendix)

Panel No.	Composition of solvent employed		Time of curing	% Solvent at test	Type test flame	Duration in secs. of test flame	Seconds to flash	Seconds to flame	Duration of film burning	Duration of free burning	Comparison control - same cond.
32	Solvent 1	wt.%	18 hrs. room temp.	14 g.	Methanol in 4" trough	175	70	75	300	125	275
	chlorobenzene	10%									
	perchloroethylene	10%									
	300/360 naphtha	80%									
33	Ditto		42 hrs. R.T.	13.2	Ditto	176			310	134	199
34	Solvent 2										
	perchloroethylene	20%	18 hrs. R.T.	15.3	Ditto	182	70	100	290	108	225
	300/360 naphtha	80%									
39	Ditto		42 hrs. R.T.	8.7	Ditto	175	55	75	225	50	199
40	Solvent 3										
	Wyandotte 1,1,CL2	20%	18 hrs. R.T.	14.9	Ditto	175	70	75	300	125	275
	300/360 naphtha	80%									
41	Ditto		42 hrs. R.T.	9.7	Ditto	160	60	70	225	65	199
[illegible]	Control Seal Kote		18 hrs. R.T.	15.6	Ditto	185	70	80	460	275	---
39	Control Seal Kote		42 hrs. R.T.	9.8	Ditto	176	55	65	375	199	---
31	Same as Panel 32 & 33		65 hrs. R.T.	8.8	Ditto	188	80	90	270	82	231
35	Ditto		161 hrs. R.T.	5.8	Ditto	180	85	100	220	40	0

(continued)

Table 4 (continued)

Panel No.	*Composition of solvent employed		Time of curing	% Solvent at test	Type test flame	Duration in secs. of test flame	Seconds to flash	Seconds to flame	Duration of film burning	Duration of free burning	Comparison control - same cond.
37	Same as Panels 34 & 36		65 hrs. R.T.	7.8	Methanol in 4" trough	170	80	85	230	60	231
42	Ditto		161 hrs. R.T.	6.3	Ditto	188	85	100	188	0	0
43	Same as Panels 40 & 41		65 hrs. R.T.	7.4	Ditto	185	60	80	180	0	231
44	Ditto		161 hrs. R.T.	6.0	Ditto	170	85	90	192	22	0
45	Seal-Kote		65 hrs. R.T.	8.0	Ditto	170	80	85	401	231	---
46	Seal-Kote		161 hrs. R.T.	5.0	Ditto	180	80	85	180	0	---
62	Solvent 6A	wt.%									
	chlorothene	15%	42 hrs.	7.8	Ditto	180	56	69	180	N	219
	perchloroethylene	25%									
	300/360 naphtha	60%									
63	Ditto		90 hrs.	5.7	Ditto	170	90	113	160	-10	308
64	Ditto		160 hrs.	5.0	Ditto	186	67	N	N	N	0
68	Solvent 6B										
	perchloroethylene	25%	42 hrs.	8.8	Ditto	171	58	70	204	33	219
	"CL2"	15%									
	300/360 naphtha	60%									
69	Ditto		90 hrs.	6.3	Ditto	173	61	73	160	-13	308
70	Ditto		160 hrs.	4.6	Ditto	183	84	N	173	-10	N

(continued)

Table 4 (continued)

Panel No.	*Composition of solvent employed		Time of curing	% Solvent at test	Type test flame	Duration in secs. of test flame	Seconds to flash	Seconds to flame	Duration of film burning	Duration of free burning	Comparison control same cond.
65	Solvent 6C	wt.%									
	perchloroethylene	25%	42 hrs.	10.2	Methanol in 4" trough	194	52	73	194	N	219
	"Cl2"	50%									
	300/360 naphtha	25%									
66	Ditto		90 hrs.	4.9	Ditto	165	74	N	157	-8	308
67	Ditto		160 hrs.	3.2	Ditto	191	78	N	186	-5	
59	Seal Kote		42 hrs.	9.8	Ditto	169	58	71	388	219	
60	Seal Kote		90 hrs.	4.7	Ditto	178	68	99	446	308	
61	Seal Kote		160 hrs.	3.9	Ditto	173	80	90	173	N	

N = No burning.

*The mastic composition was:

wt. %	
27.0	solvent indicated
50.0	base containing 25% Montar #4
14.0	7T asbestos
9.0	325 mesh slate flour.

Table 5

Moisture Vapor Transmission Constants

Composition		MVT Constant times 10^8
Montar 3 blend		14 28
Montar 4 blend		11 9
Montar 5 blend		30 48
1210-1	5% ammonium phosphate 5% sodium bicarbonate 90% Seal Kote (medium)	2.8 2.2 2.3
1210-2	5% ammonium phosphate 5% sodium bicarbonate 90% Seal Kote (medium)	2.2
1210-3	5% ammonium phosphate 5% aluminum sulfate 90% Seal Kote (medium)	2.2 8.1
1210-4	5% Borax 5% aluminum sulfate 2% zinc oxide 88% Seal Kote (medium)	2.8 1.6
1210-5	5% Borax 5% sodium bicarbonate 2% zinc oxide 88% Seal Kote (medium)	6.3
	2% aluminum sulfate 4% sodium bicarbonate 94% Seal Kote (medium)	2.0 2.4
	4% aluminum sulfate 2% sodium bicarbonate 94% Seal Kote (medium)	2.2 2.7
	4% aluminum sulfate 96% Seal Kote (medium)	1.6 1.6
	4% sodium bicarbonate 96% Seal Kote (medium)	2.0 1.6
	Seal Kote (medium)	1.5 1.3 0.9

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Table 6

Salt Spray Cabinet Test on Mastics Employing Inorganic Salts(1)

Panel	Composition	Grade
1	98% Seal Kote, 2% aluminum sulfate	3
2	Ditto	1
3	90% Seal Kote, 5% am. phosphate, 5% sod. bicarbonate	5
4	Ditto	3
5	90% Seal Kote, 5% al. sulfate, 5% sod. bicarbonate	7
6	Ditto	3
7	90% Seal Kote, 5% am. phosphate, 5% al. sulfate	1
8	Ditto	2
9	88% Seal Kote, 5% borax, 5% al. sulfate, 2% zinc oxide	5
10	Ditto	7
11	88% Seal Kote, 5% borax, 5% sod. carbonate, 2% zinc/ oxide	4
12	Ditto	2
13	Seal Kote	7
14	Ditto	8

(1) Reference - "A Visual Rating System for Rusted Steel Specimens", ASTM Bulletin No. 154, October, 1948.

Table 7

Bend, Cold Adhesion and Salt Spray Tests on Montar Mastics

	Composition	Bend Test	Cold Adhesion Test	Salt Spray 500 hrs.	Description	Grade[1]
Blend 1	70.5% plastic cement vehicle (38.7% solid content) and 0.6% solution of potassium hydroxide & water. 12.9% 7T asbestos 8.2% spent clay 7.4% milled Montar 4	Passed 1 cracked. No separation.	Passed	Panel 1 Panel 2	(Many rust) (spots)	0.5 1.0
Blend 2	64.2% plastic cement vehicle (38.7% solid content) 0.6% solution of potassium hydroxide. 16.9% 7T asbestos 10.9% spent clay 7.4% milled Montar 5	Failed 2 cracked. No separation.	Passed	Panel 1 Panel 2	(Thoroughly) (rusted)	0.5 0.0
Blend 2A	Blend 2 - 99.5% Petronate-L 0.5%	Failed 3 cracked 2 separated.	Passed	Panel 1 Panel 2	Over 32 rust spots Many dark spots on field of rust	0 0
Blend	68% plastic cement vehicle (38.7% solid content) 12% 7T asbestos 8% spent clay 12% Montar 5 (crushed)		Passed	Panel 1 Panel 2	32 rust spots 1 rust area Over 32 rust spots, 5 rust areas	1.0 1.0
Blend 4	68% Seal Kote vehicle (34.3% solid content) 12% 7T asbestos 8% spent clay 12% Montar 5 (crushed)		Passed	Panel 1 Panel 2	10 small rust areas, rust dots avg. 12 to in² 8 small rust areas over 50 rust dots	1.0 1.0
	Seal Kote (medium grade)	Passed No cracks No separation.	Passed	Panel 1 Panel 2	1 rust dot Perfect	9 10

(1) Reference - "A Visual Rating System for Rusted Steel Specimens" ASTM Bulletin 154, October 1958.

Table 8

Weatherometer Tests on Base Asphalt and Montar Blends with Base Asphalt

Cycle: 51 minutes continuous light, max. temp. 140° ± 5°F. 9 minutes water spray and light. Water at 20 psi. and 45° ± 5°F. 24 hour runs.

Composition	Light hours to failure	Average
Montar 3:25%, 177 S.P. base 75%	1 = 327.7 2 = 537.9	432.8
Montar 4:25%, 177 S.P. base 75%	1 = 770.4 2 = 770.4	770.4
Montar 5:25%, 177 S.P. base 75%	1 = 1,291.0 2 = 1,128.2	1,209.6
177 S.P. base	1 = 2,225.5 2 = 2,640.1	2,437.8

Table 9

Inspection Tests on Montar Samples

Montar No.	Sample Date	Softening Point (R&B), °F	Ash Content, wt. percent
4	October, 1958	164	--
4	November, 1958	440	32.5
5	March, 1958	363	6.5
5	September, 1958	242	26.0
5	October, 1958	370	5.5
5	November, 1958	344	10.0

Procedure I

Fire Testing of Asphaltic Coatings (Subcommittee T-X, ASTM Committee C-16, October, 1955)

This study represents results of initial trials for devising test procedure to determine the relative fire resistance of films of asphaltic base coatings supported on insulation. The suggestions during previous T-X meetings, March 3, 1955, as reported in the minutes, were followed in regard to test panel preparation and in varying the solids content of the film to be tested.

Coatings tested, with the exception of film of one panel, were supplied by the Benjamin Foster Company as Type E emulsion mastic and Type C cutback mastic. These are identified in Table No. 1 as E and CF respectively, CX of Test 9 representing a film from an additional cutback mastic.

The test panels were calcium silicate blocks with three pieces, each 1 1/2" x 6" x 24", cemented together with "Franset" and adhered to a transite panel with an asphaltic adhesive. The films were "doctor-bladed" on the calcium silicate blocks with guide bars, the cured film thickness being given in Table No. 1. The coated panels after film curing were placed in a frame constructed of angle iron and supported on legs six inches above the base of the hood. Immediately before the flame test, strips of film approximately 1/4" x 3" long were taken from near the edges for measurements of film thickness and content of solids.

Testing was in a hood twenty-five inches wide with the window opening during panel testing approximately two inches to give approximately the same draft for all tests. Observations were made through small side openings. To furnish the flame methyl alcohol was burned in a V-trough constructed of 18 gauge black iron with 2" x 2" ends, one side vertical and the three inch inclined side being diagonal across the end. The top opening was two inches wide and sixteen inches long. The trough was located with the vertical side next to the film and the top above the bottom edge of the film by about one-half inch and with the outside edge one-sixteenth inch away from the film to avoid heat conduction by the metal.

Preliminary trials indicated that the flame from methyl alcohol in the trough would contact from 20 to 30% of the film area if the panel was tipped approximately 4° forward (1.78" from vertical in 24"). Trial of varying amounts of methyl alcohol indicated that flame duration of three minutes was a severe condition and would indicate relative fire resistance of films. The alcohol used was of synthetic origin with 99.85% methanol content.

In testing, the stop watch was started when the alcohol was ignited. Recorded were the alcohol burning time, time for first flash of flame from film, time when the film started to burn steadily, and time the film flame went out. At the conclusion of the test the total area affected by flame and the area showing charring were measured and other phenomena occurring were recorded.

Procedure II

Modified Fire Testing Procedure for Asphaltic Coatings

Panels were prepared according to method already described in procedure I. To facilitate handling and to increase the number of tests in the prescribed time or condition, the substrate was composed of Kaylo blocks 12" x 6" x 1".

Fuel used for the test flame was sufficient methanol needed to produce a full flame for the duration of 180 seconds. In order that 20-30% of film area would be contacted by the test flame, panels were tilted at an angle of 4° toward the testing trough. Changes in quantity of fuel used were often necessary because of variations in circumambient temperatures and barometric pressures.

The test troughs, in which the fuel was burned, were constructed of 18 gauge black iron. The dimensions of the troughs were 4" x 2", one side vertical with the other side being 2 3/4" wide and sloping at 45° angle from the top edge of trough at front to bottom edge of trough at back. In testing, the vertical side of this trough was placed 1/32" from the face and 1/2" above the bottom edge of the film being tested.

Circumambient temperature, quantity of fuel, seconds to flash, seconds to flame, duration in seconds of test flame, duration in seconds of film burning and duration in seconds, when occurring, of free burning were the items recorded.

Procedure III

Moisture Vapor Transmission Test

Preparation test sample

Dextrin coated paper 6" x 18" was attached to Kaylo blocks (6" x 12" x 1"), coated side exposed.

The coating to be tested is mixed thoroughly and used to coat the dextrin paper. An even coating (3/32" wet measure) was obtained by using metal guide bars and striking off surface with a rigid screed bar. The material is dried at room temperature overnight, removed from the Kaylo block and then placed in a force-draft oven to cure.

Curing temperature - 140-150°F
Curing time - 7-10 days

Three discs are cut from each sample. To obtain satisfactory discs, the top flanged rim of the individual cup was used as a template. After cutting, each disc is calipered for thickness. The average of six measurements was accepted as the thickness to be used in subsequent calculations.

These discs are spark-tested before attaching to diffusion cups. Three holidays cause rejection, otherwise one or two holidays are sealed with paraffin wax.

Testing

Diffusion cups are coated inside with roofing grade asphalt and spark-tested - no holidays are tolerated. Approximately thirty mils of a saturated solution of sodium carbonate with 10-15 gms. solid sodium carbonate are placed in the cup. The disc is then attached to the flanges of the cup. An aluminum template of same area as the bottom of the cups is centered on top of disc. Molten paraffin wax is used to edgeseal. As soon as wax solidifies, remove template. The purpose in using a template is to give a constant area for all specimens being tested.

The prepared samples are placed in a desiccator within force-draft oven maintained at 100°F ± 0.1°.

After the system has had sufficient time to reach equilibrium, weighings are recorded at intervals of seven days until a straight line curve is established.

Calculations are made according to formula:

$$K = \frac{\text{wt. loss (grams)} \times \text{avg. thickness (cm.)}}{\text{area (cm.}^2\text{)} \times \text{V.P. (mm Hg)} \times \text{time (hrs.)}}$$

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Procedure IV

Corrosion of Various Metals Test

Prepared panels of copper, aluminum and medium steel (see Section II, Preparation of Polished Panels, Instruction Sheet II, Refinery Products Group, Aug. 4, 1958).

Imbedded duplicate sets of each metal in test mastic. Sixteen ounce containers used for each grouping, sealed and placed in 140°F oven for seven days. Metals were then thoroughly cleaned and inspected.

Procedure V

Low Temperature Performance Test

Prepared panels according to Section II, Instruction Sheet II, Refinery Products Group.

Coatings 3/32" thick wet measure were made on panels using guide bars and screed.

Panels were cured at 140°F for ten days. Set aside for testing.

Test

Panels heated in 140° oven for 12 hours and placed in cold box:

Brought to 0°F and held for 5 mins.
Brought to -20°F and held for 5 mins.
Brought to -40°F and held for 5 mins.
Brought to -60°F and held for 5 mins.

Upon termination of each 5 min. interval, panels were inspected for evidence of disbonding or cracking. All hairline cracks when appearing were spark-tested to determine whether breakthrough occurred.

Procedure VI

Salt Spray Protection Test

Preparation of panels

1. Wet ground steel panels with 80-X belt on Porter-Cable power saw until surface was free of pits.

2. Brought to uniform surface by using 240-X belt.

3. Wiped all material from surface of panels with soft clean rag.

4. Gave panels two washings in 300-360 boiling range naphtha at a temp. of 130-150°F.

5. Hand sanded to mirror finish using 280 grit aluminum oxide paper.

6. Washed in 240-315°F boiling range naphtha holding panels with long tongs and wiping with surgical gauze.

7. Slushed panels in hot methanol (nearly boiling). Flash dried and stored in desiccator until coating.

8. Coated with materials under test, making panels in triplicate. Coatings 3/32" thick wet measure were obtained by using steel guide bars and rigid screed.

9. Panels thoroughly cured at 140°F, Spark-tested. Edge sealed panels by dipping edges in paraffin wax liquid, but not hot enough to blister finger.

10. Placed in salt spray cabinet. Cabinet and mode of operation conforms to Method 4001.1 Protection, Salt spray (fog). Time exposed 14 days.

Procedure VII

Weatherometer Tests

Preparation of panels

Aluminum panels 5 7/8" x 2 3/4" x 0.032" ± 0.002" are cleaned by dipping hot 206/360 naphtha and flash drying. Two procedures are followed, one for hot applied and the other for cold applied coatings.

Hot applied: Panels are attached to absorbent paper sheet 5" x 18" x 0.003" with scotch tape so that bands 1/2" at top and 1/4" at sides and bottom are masked.

The materials to be tested are brought to the temperature which converts material to mobile liquid without oxidizing.

Sufficient liquid is poured onto mounted panel, resting on Atlas trimmer bed, to insure complete coverage upon first pass under trimmer roll. Several passes are made in order to produce a smooth film, free of holidays and conforming to the required 0.025±2" thickness.

Panels are placed immediately in cooling bath, stripped from paper backing, scotch tape removed and then given identifying markings.

Code markings which identify material being tested are placed in upper left hand margin, while the panel number is placed in the upper right hand corner (operator facing coated surface of panel).

Panels are checked for thickness using a desk micrometer and making three measurements on each side 1/2" in from margin of coating. One measure out of tolerance is acceptable, two cause rejection of panel. Before being subjected to weathering, panels are spark-tested. No holidays are tolerated.

Cold application: Panels are cleaned using same method used for hot application. The panels are prepared in pairs by placing them end to end. Outer ends are masked 1/2", inner ends 3/8", edges 1/4", using 3/4" scotch tape.

Prepared panels are placed in recessed bars whose outer guide edges have been built up with masking tape to insure a wet thickness of 3/32". The materials to be tested are troweled onto the panels are brought to desired thickness and finish by judicious use of a rigid screed bar. The prepared panels are dried overnight at room temperature and then placed in 140°F forced-draft oven and thoroughly cured.

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After curing the scotch tape is removed, panels spark-tested, and then marked for identification in same manner as that used for hot applied test panels.

Accelerated Weathering

Specimens are subjected to weathering test as outlined in ASTM D 529-37T - condition A. Visual inspections and spark-testings are made according to recommendations set forth by Sub-committee 8 of D-8, ASTM.