

Monsanto

POLYSULFIDE

FROM (NAME & LOCATION)

A. W. Morgan, D. S. Moorman, St. Louis Research

DATE

October 8, 1969

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SUBJECT

WR 69-22, CaCO₃
in Thiokol Sealants

REFERENCE

TO

C. Paton - CPATO

At your request an extensive evaluation of Monsanto plasticizers in Thiokol sealants filled with calcium carbonate has been completed. Testing was done according to the supplied Thiokol test conditions whenever possible. Volatility measurements were done at 70°C. only. Equipment problems prevented a second run at 86°C. Evaluations were made with catalysts utilizing Santicizer 160 and Santicizer 902 as dispersants. The following formulations were run:

General	PHR	Formulation
LP-32	100	
Plasticizer	15 and 40	100
Non-coated CaCO ₃	40	
(Catalyst System)		
Lead Dioxide	50	
Stearic Acid	5	15
Santicizer 160 or Santicizer 902	45	
Sulfur		0.1

Sample Identifications

<u>Sample Number</u>	<u>Plasticizer</u>	<u>Parts</u>
1	Control	-
2	S 278	15
3	S 261	15
4	S 160	15
5	S 902	15
6	Aroclor 1260	15
7	Aroclor 1254	15
8	Chlorowax 500	15
9	HB-40	15
10	S 278	40
11	S 261	40
12	S 160	40
13	S 902	40
14	Aroclor 1260	40
15	Aroclor 1254	40

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<u>Sample Number</u>	<u>Plasticizer</u>	<u>Parts</u>
16	Chlorowax 500	40
17	HB 40	40
18	bis(4-chlorobutyl) adipate	15
19	bis(4-chlorobutyl) phthalate	40
20	bis(4-chlorobutyl) adipate	15
21	bis(4-chlorobutyl) phthalate	40

Sample Preparation:

All samples were paint milled. Catalyst additions were made by shearing in polystyrene cups. No sample was molded for physical testing for at least 48 hours after catalyst addition. All moldings were made at 300°F. for 10 minutes. Volatility samples were prepared according to the Thiokol specification.

Compatibility: Thiokol Miscibility Test

All plasticizers tested, except Chlorowax 500, were miscible at levels up to 1/1.

Compatibility: Sheet Test

Heavy exudation was seen with HB-40 at 40 phr.

Volatility: Thiokol Method, 1 week 70°C. (Santicizer 902 Catalyst)

Table One

<u>Sample</u>	<u>Plasticizer</u>	<u>Phr</u>	<u>% wt. lost</u>	<u>Hardness(Shore / after volatility)</u>
1	Control	-	1.40	38
2	S 278	15	1.90	27
3	S 261	15	1.73	26
4	S 160	15	1.98	24
5	S 902	15	1.75	25
6	Aroclor 1260	15	1.01	21
7	Aroclor 1254	15	1.85	31
8	Chlorowax 500	15	1.90	28
9	HB-40	15	2.87	21
10	S 278	40	2.10	15
11	S 261	40	1.92	13
12	S 160	40	2.05	14

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Table One (contd)

<u>Sample</u>	<u>Plasticizer</u>	<u>Phr</u>	<u>% wt. lost</u>	<u>Hardness(Shore A) after volatility</u>
13	S 902	40	2.17	18
14	Aroclor 1260	40	2.02	14
15	Aroclor 1254	40	2.04	21
16	Chlorowax 500	40	3.28	10
17	HB-40	40	3.34	16
18	Bis(4-chlorobutyl) adipate	15	1.64	15
19	Bis(4-chlorobutyl) phthalate	15	1.68	14
20	Bis(4-chlorobutyl) adipate	40	2.59	5
21	Bis(4-chlorobutyl) phthalate	40	1.99	5

It is obvious that the Thiokol test is much less severe than the charcoal volatility run by Monsanto. In this Thiokol test a 1.5 x 4 x 1/4 inch block is molded, placed on a sheet of metal, and oven aged one week. Weight loss is measured but is not totally plasticizer loss. From the above results no meaningful differentiation can be made. At a higher temperature a more useful set of data might be obtained particularly if combined with fogging. As previously stated, equipment problems have prevented further work.

Physical Testing:

100% Modulus, Tensile and Elongation were determined on all samples. Readings were taken initially and after three and seven days immersion in water at 70°C. The immersed samples were simply wiped off and tested. Any absorbed water was not lost prior to the test.

For this work readings were made on samples prepared from both Santicizer 160 and Santicizer 902 catalyst pastes. Results are shown in Table Two. From these results the following points are to be considered:

1. A general trend is noticed in which modulus decreases with immersion time, tensile increases and elongation also increases. This result would indicate a fair amount of water is retained and is aiding in lower modulus. The tensile would ordinarily be lower. Its increase may be caused by an orientation phenomenon.

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2. A slight difference is seen between catalysts. Santicizer 902 pastes seem to give generally lower tensile and elongation but higher modulus. A consistent exception occurs when HB-40 or Santicizer 160 is the rubber plasticizer.

3. No system tested showed exceptional water resistance. Santicizer 278 and Chlorowax 500 appeared to be the least affected.

Water Sensitivity

Water sensitivity was run on the samples with the following results:

<u>Sample</u>	<u>Plasticizer</u>	<u>Catalyst</u>	<u>% Solids Lost</u>	<u>% H₂O Absorbed</u>
1	Control	S-160	0.41	0.86
2	S-278	S-160	0.35	1.06
3	S-261	S-160	0.32	1.43
4	S-160	S-160	0.28	1.14
5	S-902	S-160	0.38	1.14
6	Aroclor 1260	S-160	0.28	1.10
7	Aroclor 1254	S-160	0.28	1.04
8	Chlorowax 500	S-160	0.36	1.01
9	HB-40	S-160	0.38	1.09
10	S-278	S-160	0.28	0.99
11	S-261	S-160	0.34	0.96
12	S-160	S-160	0.36	1.45
13	S-902	S-160	0.43	0.90
14	Aroclor 1260	S-160	0.32	0.60
15	Aroclor 1254	S-160	0.38	0.73
16	Chlorowax 500	S-160	0.39	1.17
17	HB-40	S-160	0.60	0.68
18	Bis 4-chlorobutyl adipate	S-160	0.40	1.59
19	Bis 4-chlorobutyl phthalate	S-160	0.53	1.56
20	Bis 4-chlorobutyl adipate	S-160	0.51	1.89
21	Bis 4-chlorobutyl phthalate	S-160	0.29	1.23

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<u>Sample</u>	<u>Plasticizer</u>	<u>Catalyst</u>	<u>% Solids Lost</u>	<u>% H₂O Absorbed</u>
1	Control	S-902	0.80	1.20
2	S-278	S-902	0.41	1.00
3	S-261	S-902	0.48	1.20
4	S-160	S-902	0.47	2.05
5	S-902	S-902	0.29	0.96
6	Aroclor 1260	S-902	0.29	0.78
7	Aroclor 1254	S-902	0.35	1.09
8	Chlorowax 500	S-902	0.71	1.29
9	HB-40	S-902	0.57	1.22
10	S-278	S-902	0.35	2.05
11	S-261	S-902	0.39	2.07
12	S-160	S-902	0.39	2.74
13	S-902	S-902	0.53	2.23
14	Aroclor 1260	S-902	0.54	1.25
15	Aroclor 1254	S-902	0.59	1.51
16	Chlorowax 500	S-902	(0.93)	(1.43)
17	HB-40	S-902	3.30	0.93
18	Bis(4-chlorobutyl) adipate	S-902	0.52	2.72
19	Bis(4-chlorobutyl) phthalate	S-902	0.40	2.31
20	Bis(4-chlorobutyl) adipate	S-902	0.94	6.12
21	Bis(4-chlorobutyl) phthalate	S-902	0.33	1.82

It is interesting to note that, with few exception, less weight is lost with a plasticizer than without. This observation would be consistant if the plasticizer coats the CaCO₃ in the formulation and thus reduces its water solubility. Little difference is seen with catalyst changes and no plasticizer is seen which is truly outstanding. Santicizer 278 again appears superior at higher levels.

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Hardness Shore A
After Molding

<u>Samples</u>	<u>S-902 Catalyst</u>	<u>S-160 Catalyst</u>	(S902) <u>After Volatility from Table One</u>
1	42	38	38
2	26	27	27
3	29	29	✓26
4	28	30	✓24
5	31	31	✓25
6	26	28	✓21
7	33	33	31
8	31	28	28
9	31	28	21
10	17	16	15
11	10	12	13
12	18	18	14
13	21	17	18
14	23	24	✓14
15	22	21	21
16	14	12	10
17	10	11	✓16
18	26	24	✓15
19	19	20	✓14
20	9	10	5
21	12	12	5

The above hardness data again indicates that very slight differences exist between S-902 and S-160 catalyst pastes. It is interesting to note that the hardness after molding is generally higher than the hardness after volatility. At low plasticizer levels little change is seen with Santicizer 278, Aroclor 1254, or Chlorowax 500. At higher levels the preceding are again very similar. This indicates that the plasticizer is very permanent (S278 and Chlorowax 500) or quite volatile (Aroclor 1254 and S-261 and S-902 at higher levels). Medium volatility plasticizer results show that the volatility test was less severe than the molding cycle.

All samples have shown good heat and light stability. If we may be of further service please call.

A W Morgan
A. W. Morgan

D S Moorman
D. S. Moorman

Time: 120 hrs.
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S-160 Cat.

Sample	Plasticizer	100%			Tensile			Elong.		
		A	C	B	A	C	B	A	C	B
1	Control	110	112	86	181	230	228	353	506	623
2	S-278	58	60	60	155	200	197	633	823	833
3	S-261	69	66	57	134	164	170	386	543	653
4	S-160	83	70	64	153	228	175	333	630	599
5	S-902	75	68	69	129	206	192	349	706	719
6	Aroclor 1260	87	78	66	125	148	161	209	373	513
7	Aroclor 1254	94	84	770	121	143	150	176	316	473
8	Chlorowax 500	82	771	51	121	167	171	246	489	570
9	HB-40	81	72	68	90	142	114	133	333	233
10	S-278	37	34	31	79	77	81	309	459	526
11	S-261	40	35	33	104	106	102	365	643	663
12	S-160	53	41	28	104	114	85.3	433	626	593
13	S-902	38	42	333	96	121	122	579	729	826
14	Aroclor 1260	52	58	45	98	142	139	323	573	659
15	Aroclor 1254	41	43	38	99	134	157	476	736	836
16	Chlorowax 500	22	27	21	79	95	77	819	941	936
17	HB-40	23	26	23	81	89	86	586	673	803
18	Bis(4-chlorobutyl)adipate	71	64	47	92	78	71	163	236	279
19	" " phthalate	35	34	37	88	111	103.7	589	773	823
20	" " adipate	27	25	19	56	58	51.4	319	466	503
21	" " phthalate	26	25	22	72	74	69.4	533	766	749

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S-902 Cat.

Sample	Plasticizer	100% Modulus (PSI)			Tensile (PSI)			Elongation (Percent)		
		Fresh	3 days 70°C. in H ₂ O	7 days 70°C. in H ₂ O	Fresh	3 days	7 days	Fresh	3 days	7 days
1	Control	108	112	92	187	284	265	369	636	659
2	S-278	82	69	44	138	144	103	256	396	523
3	S-261	79	67	57	105	120	119	209	336	449
4	S-160	73	58	49	176	193	180	426	729	806
5	S-902	97	75	64	117	101	107	139	213	323
6	Aroclor 1260	104	78	112	141	133	110	198	292	319
7	Aroclor 1254	111	80	75	138	158	164	214	349	456
8	Chlorowax 500	85	70	61	138	154	147	315	506	543
9	HB-40	65	61	53	94	114	123	229	376	506
10	S-278	43	31	29	69	63	72	233	389	446
11	S-261	22	21	17	59	77	65	559	819	909
12	S-160	30	22	23	86	97	94	706	819	970
13	S-902	42	44	35	83	118	125	373	613	766
14	Aroclor 1260	50	52	44	95	123	156	386	593	799
15	Aroclor 1254	42	45	42	89	136	122	418	680	689
16	Chlorowax 500	30	30	23	77	92	83	603	803	849
17	HB-40	18.7	25	25	55	68	62	559	699	640
18	Bis(4-chlorobutyl) adipate	18.7	47	42	52	107	109	559	493	616
19	Bis(4-chlorobutyl) phthalate	43	33	28	106	79	72	543	566	656
20	Bis(4-chlorobutyl) adipate	33	18	15	82	60	56	534	793	853
21	Bis(4-chlorobutyl) phthalate	39	27	23	65	59	44	241	386	383

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