MASTER

AGING STUDIES OF LX-14

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E. Kohn

DEVELOPMENT DIVISION

OCTOBER - DECEMBER 1975

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ABSTRACT

A long-term compatibility study (2 years) of LX-14 at ambient temperature and at 50, 60, and 80 C has been initiated and has been partially completed. Analysis of aged LX-14 samples, performed by gel permeation chromatography showed that the Estane degraded. Previous data on the compositional analysis of LX-14 using 1,2-dichloroethane were found to be unreliable and a new method of analysis is being investigated.

DISCUSSION

LX-14 is a plastic-bonded explosive having a nominal composition of 95.5/4.5 HMX/Estane 5702-F1, by weight. The HMX is a mixture, by weight, of 65% Class A, 25% LX-04-01 type and 10% Class B type. The Estane is a polyester-polyurethane elastomer, and is essentially a linear polymer. The molding powder is a 4 to 1 mixture of undyed and dyed pellets. The dying is accomplished by the addition of 0.1% Heliogen Violet during manufacture. Compositional analysis of the LX-14 used in the investigation was 94.9/5.1 HMX/Estane.

Two different configurations designated Sack and Selden are being studied using LX-14. Thirty of each configuration along with twenty-four LX-14 control samples are being aged at ambient, 50, 60, and 80 C. Approximate dates for removal of these containers are given in Table I. All but two containers from the 80 C oven and the 6-month containers from both the 50 and 60 C ovens have been removed along with one of each configuration that has been at ambient temperature for one year. The results of gas analysis along with other tests that are being used for evaluation in this study have been reported(1).

The Estane in LX-14 was analyzed by High Speed Gel Permeation Chromatography (HSGPC). The method of analysis, data reduction and instrumentation have been developed by E. Kohn(2). Tables II, III and IV show the HSGPC data of aged LX-14 samples from the Sack, Selden and HE control containers, respectively.

The HSGPC results show a large decrease in the average molecular weight of Estane as a function of aging time and temperature, thus indicating degradation. Fig. 1 is a plot of the weight average molecular weight (M_w) of Estane in LX-14 versus time at 50, 60, and 80 C. The graph shows that Estane in LX-14, in the first few months of aging, undergoes rapid degradation but then the degradation rate appears to level off. It should be noted that although the Estane was degraded, all aged LX-14 samples were machineable for mechanical testing.

Compositional analyses were run on all LX-14 samples but no changes were detected. Indications are that the present method of analysis, which is an extraction method(3), could give erroneous results due to:

1. All aged LX-14 has turned darker in color and CO_2 and N_2O are evolved indicating decomposition.

Table I. Schedule for the Removal of Containers in the LX-14 Long-Term Compatibility Study

Oven Time (months)	Type of Units	80 C	60 C	50 C	Ambient	Removal Date
1	Sack Selden	F13-F14 F29-F30				01/16/75 01/16/75
3	Sack Selden Control	F15-F16 F31-F32 F52-F53				03/17/75 03/17/75 03/17/75
6	Sack Selden Control Sack Selden	X01-X02 X15-X16 F54-F55	F58-F10 F25-F26	F5-F6 F21-F22		12/14/74 12/14/74 06/16/75 06/16/75 06/16/75
12	Sack Selden Control Sack Selden	X03-X04 X17-X18 F56-F62	F46-F47 F11-F12 F27-F28	F39-F40 F7-F8 F23-F24	X05 X19 F33-F34 F1 F17	06/14/75 06/14/75 12/16/75 12/16/75 12/16/75
18	Sack Selden Control		X06-X07 X20-X21 F48-F49	X11-X12 X25-X26 F41-F43		12/14/75 12/14/75 06/16/76
24	Sack Selden Control Control Sack Selden		X08-X09 X22-X23 F50-F51	X13-X14 X27-X28 F44-F45	X10 X24 F35-F36 F37-F38 F2-F3-F4 F18-F60-F20	06/14/76 06/14/76 12/16/76 12/16/76 12/16/76 12/16/76

Table II. HSGPC Analysis of Estane in LX-14 After Aging
in Weapon Configuration

Container & Configuration	Time in Oven (months)	Temperature of Oven (C)	Number of Runs	Peak Average	% Std. Dev.	$\bar{M}_n$	% Std. Dev.	$\bar{M}_w$	% Std. Dev.	MWD	% Std. Dev.
Estane 5702-F1			13	47965	6	19813	11	56979	7	2.91	7
HE Control			5	35833	5	13659	10	54964	8	4.03	7
F-13 (Sack)	1	80	3	18077	11	7962	9	25958	4	3.27	9
F-14 (Sack)	1	80	4	19714	5	8663	3	27521	3	3.18	1
F-15 (Sack)	3	80	4	7046	5	3867	2	8507	3	2.20	2
F-16 (Sack)	3	80	4	7847	8	4069	4	8844	8	2.17	5
X-01 (Sack)	6	80	4	5384	5	3625	2	9568	4	2.64	2
X-02 (Sack)	6	80	3	3756	1	2407	1	7262	12	3.02	11
X-03 (Sack)	12	80	3	3329	2	2217	1	4112	2	1.85	1
X-04 (Sack)	12	80	4	3189	1	2133	2	3908	1	1.83	2
F-58 (Sack)	6	60	3	14668	3	7682	2	19547	2	2.54	1
F-10 (Sack)	6	60	3	14395	2	6958	2	20161	4	2.90	2
F-05 (Sack)	6	50	3	26942	7	10096	4	31742	8	3.15	7
F-06 (Sack)	6	50	6	9112	5	4377	2	10861	8	2.48	7
X-05 (Sack)	12	Ambient	6	36598	7	12782	7	59985	8	4.70	4

Table III. HSGPC Analysis of Estane in LX-14 After Aging
in Weapon Configuration

Container & Configuration	Time in Oven (months)	Temperature of Oven (C)	Number of Runs	Peak Average	% Std. Dev.	$\bar{M}_n$	% Std. Dev.	$\bar{M}_w$	% Std. Dev.	MWD	% Std. Dev.
F-29 (Selden)	1	80	4	21502	5	9182	5	28688	4	3.13	3
F-30 (Selden)	1	80	5	20471	4	9112	5	27804	3	3.06	3
F-31 (Selden)	3	80	5	13422	4	6893	4	25413	6	3.68	4
F-32 (Selden)	3	80	3	7265	4	3874	2	8107	4	2.09	1
X-15 (Selden)	6	80	4	7426	7	3856	5	9307	6	2.42	6
X-16 (Selden)	6	80	4	7440	7	4006	6	8520	5	2.13	4
X-17 (Selden)	12	80	4	3695	1	2397	2	5305	9	2.21	9
X-18 (Selden)	12	80	3	3794	5	2512	4	5727	11	2.28	7
F-25 (Selden)	6	60	4	16915	5	7799	4	22426	7	2.87	4
F-26 (Selden)	6	60	4	15652	4	7441	5	20928	8	2.81	4
F-21 (Selden)	6	50	11	27929	7	10321	6	35579	8	3.44	6
F-22 (Selden)	6	50	11	28826	6	10434	4	35192	6	3.37	3
X-19 (Selden)	12	Ambient	4	40822	6	14466	7	73409	14	5.07	10

Table IV. HSGPC Analysis of Estane in LX-14 After Aging
in Weapon Configuration

<u>Container & Configuration</u>	<u>Time in Oven (months)</u>	<u>Temperature of Oven (C)</u>	<u>Number of Runs</u>	<u>Peak Average</u>	<u>% Std. Dev.</u>	<u>$\bar{M}_n$</u>	<u>% Std. Dev.</u>	<u>$\bar{M}_w$</u>	<u>% Std. Dev.</u>	<u>MWD</u>	<u>% Std. Dev.</u>
HE Control			5	35833	5	13659	10	54964	8	4.03	7
F-52 (HE Control)	3	80	3	9687	4	4646	3	11645	5	2.51	2
F-53 (HE Control)	3	80	5	7417	6	3935	3	9127	8	2.32	8
F-54 (HE Control)	6	80	3	4650	2	3100	5	6261	5	2.02	3
F-55 (HE Control)	6	80	4	4572	3	3062	6	6051	6	1.98	1

Fig. 1. Weight Average Molecular Weight of Estane After Aging in Selden Configuration at Temperatures of 50, 60 and 80 C Versus Time

2. The more discolored LX-14 samples are more difficult to dissolve in tetrahydrofuran (THF) (solvent used for the HSGPC), but the Estane in aged samples is soluble in 1,2-dichloroethane (DCE) which is used for the compositional analysis.
3. HSGPC does show that the Estane in LX-14 at elevated temperatures is degrading.

It was postulated that the DCE used in determining compositional analysis may contain HCl, which could catalyze the hydrolysis of Estane and give spurious results. To investigate this a sample of Estane aged at 80 C for 10.5 months in a polyethylene bag was refluxed in THF and DCE. Samples were also dissolved in both solvents at room temperature. These samples of Estane were then analyzed by HSGPC.

The HSGPC results showed that Estane refluxed in either THF or DCE underwent degradation. Estane did not degrade when dissolved in THF and stored at room temperature. Data for Estane dissolved in DCE at room temperature are forthcoming; however, preliminary indications are that the Estane is degrading. This supports the above hypothesis that DCE causes Estane hydrolysis and thus erroneous results in the compositional analysis of LX-14. A new method, liquid chromatography, is being investigated.

To study the effect of temperature and aging on Estane alone, samples of the elastomer were sealed in polyethylene bags and aged at ambient temperature and at 50, 60, and 80 C. These samples, held at elevated temperatures, showed increased molecular weights which suggests cross-linking of the Estane molecules.

Because the molecular weight of Estane alone increased while the Estane in LX-14 showed a decrease the possible effect of air was evaluated. Estane was therefore sealed in containers, backfilled with helium and aged at ambient, 50, 60, and 80 C, and these are being periodically sampled. Table V gives the HSGPC results of the samples which have been removed to date. The data show that Estane aged up to 7 months at 50, 60, and 80 C in the absence of air exhibits little change in average molecular weight. Thus, it appears that Estane crosslinking is dependent upon its environment.

CONCLUSIONS

HSGPC analysis of Estane 5702-F1, aged at 50, 60, and 80 C showed either degradation or crosslinking depending upon the conditions employed. Estane aged in LX-14 decreased in molecular weight, while Estane alone in metal containers backfilled with helium showed little change in molecular weight. However, the Estane showed an increase in molecular weight when aged in polyethylene bags, which were permeable to the atmosphere.

Table V. HSGPC Analysis of Estane 5702-F1 Samples
(Conditioned at 50, 60 and 80 C in a Helium Atmosphere)

Time in Oven (months)	Temperature of Oven (C)	Number of Runs	Peak Average	% Std. Dev.	$\bar{M}_n$	% Std. Dev.	$\bar{M}_w$	% Std. Dev.	MWD	% Std. Dev.
Estane Control										
		13	47965	6	19813	11	56979	7	2.91	7
Container No. 26										
1	50									
2	50	3	50721	5	20675	3	60573	5	2.93	5
3	50	3	53079	4	22111	12	65075	3	2.97	10
4	50	3	53179	7	20200	9	65958	5	3.27	4
5	50	3	45744	7	20307	13	54730	6	2.71	8
6										
7	50	3	51547	7	19948	7	61681	5	3.10	6
Container No. 25										
1	60									
2	60	2	54917	5	23439	5	68226	2	2.92	7
3	60	6	54438	7	22017	6	71454	7	3.25	5
4	60	4	51751	3	21393	2	64765	3	3.03	2
5	60	3	49244	3	21029	7	60391	3	2.88	5
6	60									
7	60	3	51437	4	19786	6	61415	5	3.11	8
Container No. 10										
1	80									
2	80	2	49017	6	20169	2	57497	6	2.85	3
3	80	4	49083	4	19075	12	57026	4	3.00	7
4	80	4	47432	8	17885	3	55816	7	3.12	8
5	80	3	46969	5	19419	10	54965	6	2.84	4
6	80									
7	80	3	45358	1	18341	1	52539	2	2.87	2
Container No. 49										
1	Ambient									
2	Ambient	3	51068	9	20300	2	61053	8	3.01	10
3	Ambient	3	51319	6	21865	7	61328	6	2.81	6
4	Ambient	8	49533	5	21229	7	60320	6	2.85	5
5	Ambient	4	53574	5	23880	3	67503	4	2.83	4
6	Ambient									
7	Ambient	4	53797	6	22972	7	66905	3	2.92	9

REFERENCES

1. P. A. Foster, "Long-Term Compatibility of LX-14," MHSMP-74-35, July - September 1975 (CRD).
2. E. Kohn, R. W. Ashcraft and P. A. Foster, "Development of a GPC Analysis Method for Estane in LX-14," MHSMP-76-5H.
3. Walter Selig, "The Analysis of Explosives Containing 1,3,5-Tetra-nitro-1,3,5,7-Tetrazacyclooctane (HMX), Energetic Plasticizers, and Various Plastics," UCID-15025 Rev. 1 (April 22, 1968).