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# LX-14 A NEW HIGH-ENERGY PLASTIC-BONDED EXPLOSIVE

J. R. Humphrey

November 21, 1977

MASTER

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## ABSTRACT

LLL has developed and tested LX-14-0, a general purpose, plastic-bonded explosive (PBX) containing 95.5 wt% HMX and 4.5 wt% Estane 5702-F1 polyurethane. This composition has high energy, low sensitivity to handling and shock, and has mechanical properties that are superior to other PBXs now in use.

Six types of sensitivity tests indicated that LX-14-0 is definitely safer to handle than PBX-9404-3 or LX-10-1. In sliding impact (skid) tests, LX-14-0 underwent only a mild, low-order reaction when dropped from a height of 3 m. In contrast, the other two explosives detonated when they were dropped from only 0.38 m.

In the projectile impact (Susan) test, velocities required to obtain *any* reaction from LX-14-0 were 30 to 50% higher than those that caused detonation of PBX-9404-3 and LX-10-1. Even at these higher velocities, the reaction of LX-14-0 did not progress to a high-order detonation. Also, LX-14-0 was less sensitive than the other PBXs in four important small-scale sensitivity tests.

LX-14-0 has better mechanical properties than other PBXs: it is stronger, tougher, and more resistant to creep in tension and compression. It resists cracking caused by strains that result when materials that have different thermal coefficients of expansion are in contact. The new explosive has excellent thermal stability. Only very small quantities of gases evolve from LX-14-0 or from intimate mixtures with polymers during the 22-h/120°C chemical reactivity test.

In unrealistically hostile, long-term, disk-to-disk compatibility tests at 80°C, LX-14-0 has stability and compatibility satisfactory for weapons environments. Despite the evidence that LX-14-0 retained its integrity during long exposures, the Estane binder in this PBX degraded. The relationship between this degradation and the properties of LX-14-0 after long term aging needs further study.



## GLOSSARY

Adiprene (L-100 and L-315)	— Polyurethane, manufactured by E. I. DuPont.
CRT	— Chemical reactivity test.
CTC	— Coefficient of thermal conductivity.
CTE	— Coefficient of thermal expansion.
DTA	— Differential thermal analysis.
Estane 5702-F1	— Polyester polyurethane elastomer, manufacture by B. F. Goodrich.
HE	— High Explosive.
HMX	— 1,3,5,7-tetranitro-, 1,3,5,7-tetraazacyclooctane.
LASL	— Los Alamos Scientific Laboratory, Los Alamos, NM.
LLL	— Lawrence Livermore Laboratory, Livermore, CA.
LX-04-1	— 85 wt% HMX-15 wt% Viton A.
LX-10-1	— 94.5 wt% HMX-4.5 wt% Viton A.
Mulberry	— U-7.5 wt% Ni-2.5 wt% Zr
Mylar	— Polyethylene terephthalate DuPont fiber (also Dacron).
Neoprene B	— Poly(2-chloro-1,3-butadiene) (polychloroprene).
NP	— Nitroplasticizer eutectic composed of a 50/50 wt% bis(2,2-dinitropropyl)acetal/bis(2,2-dinitropropyl) formal.
Pantex	— Mason & Hanger-Silas Mason Co., Inc., Amarillo, TX.
PBX	— Plastic bonded explosive.
PBX-9011	— 90 wt% HMX-10 wt% Estane 5740-X2.
PBX-9404-3	— 94 wt% HMX-3 wt% nitrocellulose (12.0%N)-3 wt% Tris( $\beta$ -chloroethyl)-phosphate.
PBX-9501	— 95 wt% HMX-2.5 wt% Estane 5702-F1-2.5 wt% NP.
Polycarbonate	— plastic, e.g., Lexan, Merlon - Mobay Chemical Co.
Silastic (RTV 93-119)	— Silicone rubber potting compound - Dow Corning.
TMD	— Theoretical maximum density.
Viton A	— Copolymer of hexafluoropropylene/vinylidenefluoride.
Y-3333 } Y-3260 }	— Cellular silicones made by Union Carbide.
Y-3602/B	— Boron metal powder in a silicone binder made by Union Carbide.

# LX-14: A NEW HIGH-ENERGY PLASTIC-BONDED EXPLOSIVE

## INTRODUCTION

Formulation and testing of LX-14-0 containing 95.5 wt% HMX<sup>\*</sup> and 4.5 wt% Estane 5702-F1 polyurethane elastomer was part of our continuing effort to improve the handling safety and thermal stability of high-energy chemical explosives.

LLL and LASL have formulated many useful plastic-bonded explosives such as LX-04, LX-09, LX-10, and PBX-9404 from HMX crystals coated with 5 to 15 wt% of various polymers. During pressing, the polymer coating becomes a matrix which gives mechanical strength to the PBX. The polymer may also function as a lubricant that reduces sensitivity to accidental detonation; presumably by dissipating and distributing the energy of an impact throughout a larger volume of the explosive. Although the strengths and explosive energies of these various compositions were adequate, each of these early PBXs had one or more drawbacks. For example, handling sensitivity was considered too high, there was too much compressive creep or performance in accelerated aging tests was unsatisfactory.

In 1960, LASL observed for the first time that Estane polyurethane binders drastically reduced the impact and shock sensitivity of HMX. Since then, ERDA laboratories have examined many formulations containing HMX coated with Estanes. LASL did extensive testing to qualify two promising candidates, PBX-9011 (90 wt%-HMX 10 wt%-Estane 5740-X2) and PBX-9501 (95 wt%-HMX 2.5 wt%-Estane 2.5 wt% NP),<sup>\*</sup> but neither of these compositions was completely satisfactory. PBX-9011 has about 15% less energy than PBX-9404, and PBX-9501 has poor mechanical properties and possible long-term compatibility problems in some weapon systems.

During this period, LLL made and tested extensively LX-14-0, a simpler composition containing less than 5 wt% Estane, but no plasticizer. This composition was chosen after we systematically compared eight compositions having from 1 wt% to 8 wt% Estane 5702-F1 to find the minimum

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<sup>\*</sup> See Glossary of abbreviations.

amount of Estane that reduced the impact and shock sensitivity of HMX to satisfactory levels.

Judging the suitability of a high-energy explosive demands information about many factors and requires optimization of conflicting attributes. An explosive must be easily and safely prepared, densified, and converted to useful shapes. Designers need extensive knowledge of the mechanical and performance properties. Safety experts require detailed information about thermal stability and about behavior in a variety of shock and impact tests. Different weapons applications raise numerous questions about compatibility and long-term stability in special environments.

For these reasons, qualification of a new explosive composition is usually a long, arduous, and expensive task.

This report presents information available in June 1977 about the properties and performance of LX-14-0. It discusses in detail the formulation of this explosive, its pilot plant preparation, and the production of a 4500-kg (10,000 lb) lot. It records studies of pressing cycles needed to make high-density LX-14-0 billets, and discusses dynamic and quasi-static mechanical properties, shock sensitivity, energy, equation of state, compatibility, thermal stability, and accelerated aging studies.

## FORMULATION AND PRESSING STUDIES

### Formulation by the Slurry Technique

To find out if there is an optimum ratio of HMX to Estane 5702-F1 for giving a PBX of high energy, low sensitivity, and good properties, we made and tested molding-powder beads and billets from eight different compositions that contained from 1 wt% to 8 wt% Estane. Details of slurry processing and pressing are discussed below.

We combined crystals of HMX with Estane 5702-F1 to make molding powder beads, applying the same slurry technique commonly used to coat particles of other explosives with a polymeric binder. *Appendix A* describes this technique in detail.

In this process, fine particle-size HMX and water are agitated rapidly while adding a lacquer made by dissolving Estane in an appropriate solvent. While the solvent is being

removed with continued stirring, the Estane forms a thin layer on the explosive crystals. During further stirring and solvent removal, the dispersed particles agglomerate to form beads. When all the solvent has been removed, the beads are filtered from the water and air-dried to a nominal bulk density of 0.9 to 1.0 Mg/m<sup>3</sup>.

Proper particle-size distribution of HMX crystals is an important factor in making satisfactory LX-10-1 molding-powder beads. In earlier studies<sup>1</sup> of LX-10-1, density during pressing was dependent on the HMX crystal size used for bead preparation. Because we expected the same factors to operate in our experimental compositions, we chose the same LX-10-1 particle-size distribution (65/25/10-Class A grade/LX-04-1 type/Class B grade) for our formulation studies.

## Pressing Studies

Billets are ordinarily made from PBXs by pressing dry molding powder beads using carefully preset conditions. After some experimentation, we selected the following cycle to press the eight experimental HMX/Estane compositions into billets 0.15 m (6 in.) in diameter by 0.15 m (6 in.) long:

1. Preheat the molding powder beads for 4 to 6 h at 120°C.
2. Load beads into a preheated Neoprene rubber bag, place in press and evacuate to 0.13 kPa (1 mm Hg).
3. Press isostatically at 120°C and 138 MPa (20 ksi) for 10 minutes.
4. Release pressure, repressure twice at 120°C and 138 MPa for 10 minutes each time, using the captive water intensification technique (the rubber bag containing the PBX is opened, flooded with water, closed, and returned to the press).

Densities obtained by pressing the eight experimental compositions using this cycle are recorded in Table 1.

We conclude from this table and from other observations that:

- a) Multiple pressings are much more effective in producing high-density billets than single or double pressings with long dwell times.
- b) A density of 1.83 Mg/m<sup>3</sup> (99% of the theoretical material density) was chosen as the minimum acceptable density for LX-14-0 billets.
- c) High densities can be obtained in billets of HMX containing as little as 1% binder. (Note composition RX-04-EH in Table 1.)
- d) At the same HMX content, a composition containing Estane 5702-F1 (a 50/50 blend of low and high intrinsic viscosity Estane 5702) yielded a higher density than we achieved

Table 1. Densities of billets made by pressing HMX/Estane 5702-F1 molding beads into nominal 5.5-kg billets.

RX-04-	HMX <sup>a</sup> , wt%	Density Mg/m <sup>3</sup> after pressings <sup>h</sup>					TMD, <sup>g</sup> Mg/m <sup>3</sup>	% of TMD <sup>g</sup> obtained
		One isostatic pressing		Captive water <sup>c</sup>				
		Rough <sup>b</sup>	Finished	One repressing	Two repressings			
EL	94	90.68	—	—	—	1.822	1.833	99.4
EE	95	92.20	1.821	1.819	1.829	1.830	1.844	99.2
EN <sup>d</sup>	95	92.20	—	—	—	1.826	1.844	99.1
EO <sup>e</sup>	95	92.20	—	—	—	1.825	1.844	99.0
EQ <sup>f</sup>	95.5	92.95	—	—	—	1.835	1.849	99.2
EK	96	93.71	—	—	—	1.833	1.855	98.8
EP	96.2	94.02	—	—	—	1.835	1.857	98.8
EM	96.6	94.64	—	—	—	1.839	1.861	98.8
EF	97	95.21	1.828	1.830	1.840	1.844	1.865	98.9
EG	98	96.82	1.828	1.831	1.845	1.852	1.877	98.7
EH	99	98.40	1.835	—	1.845	1.855	1.886	98.4

<sup>a</sup>The HMX particle-size distribution was 65/25/10 - Class A grade/LX-04-1 Type/Class B Grade.

<sup>b</sup>Isostatic pressing - 120°C preheat, 10 min dwell/138 MPa.

<sup>c</sup>Captive water repressing - ambient temperature 10 min dwell/138 MPa.

<sup>d</sup>Low intrinsic viscosity Estane 5702.

<sup>e</sup>High intrinsic viscosity Estane 5702-F2.

<sup>f</sup>Nominal LX-14-0.

<sup>g</sup>TMD = Theoretical material density.

<sup>h</sup>Density data in each horizontal row are the cumulative results of successive pressing steps.

with either RX-04-EN made from the low intrinsic viscosity Estane 5702 component or RX-04-EO made from the high intrinsic viscosity Estane 5702-F2 component.

e) Stress relaxation did not occur in billets after isostatic pressing and machining.

f) The dye penetrant test showed that a hot, isostatically pressed billet of LX-14-0 did not crack when removed from the press and shock-cooled by plunging immediately into cold water.

g) Using water at 92°C, instead of water at ambient temperature,

during captive water repressing did not raise the density of the billets.

### LX-14-0 Specifications

Based on these results and on sensitivity tests described later, we chose a composition containing 95.5 wt% HMX and 4.5 wt% Estane

5702-F1 for extensive evaluation and pilot-plant production.

Material Specification RM 253683 describes acceptable LX-14-0 and RM 253782 describes Estane 5702-F1.

A 4500-kg production lot made at the Holston Army Ammunition Plant satisfied these specifications as shown in *Appendix A*.

## SENSITIVITY

The sensitivity of any explosive is of primary concern to the handler, his co-workers and immediate community. New formulations, which could introduce unknown risks, demand special attention. In order for a new HE to be of practical use, it must have safety characteristics that allow it to be routinely formulated, pressed, machined, and handled in useful sizes and shapes. It must also withstand accidental heat-producing stimuli such as shock, mechanical impact, and friction.

At LLL, we use six important sensitivity tests to evaluate HEs:

- (1) Plant handling safety — skid test.
- (2) Confined impact sensitivity — Susan test.
- (3) Sensitivity to shock — gap, wedge, and rifle bullet impact tests.
- (4) Unconfined impact sensitivity — drop hammer.

### Skid Test

The most reliable method of evaluating the hazards associated with plant handling of large billets of HE is the sliding impact test (skid test).

In the LLL-Pantex version of this test, a 10.4-kg (23-lb) hemispherical explosive billet, supported on a pendulum device, swings down from a preset height and strikes a sand-coated steel target plate. The impact angle is the angle between the tangent of the arc the billet travels and the horizontal target surface. The spherical surface of the billet serves to concentrate the force of the impact in a small area and the pendulum arrangement gives the impact a sliding or skidding component as well as a vertical one. We also tested a few 136-kg (300-lb) and 45-kg (100-lb) billets.

The results of the test are expressed in terms of the type of chemical event produced by the impact as a function of impact angle and vertical drop. The events are classified by intensity on a scale ranging from no reaction (0) to high order detonation (6). Sliding impact test results are applicable to plant handling safety because the drop heights and impact angles used in the test are within the limits one might find for the accidental dropping of an explosive billet, e.g., from a workbench or from the tailgate of a truck.

#### LLL-Pantex (Standard) Skid Test

Figures 1 and 2 compare skid test data<sup>1,2,3</sup> for the four PBXs: LX-14-0, LX-10-1, LX-04-1, and PBX-9404-3. Figure 1 shows that when tested at a 14 deg angle (our most severe test), PBX-9404 and LX-10 detonate after a

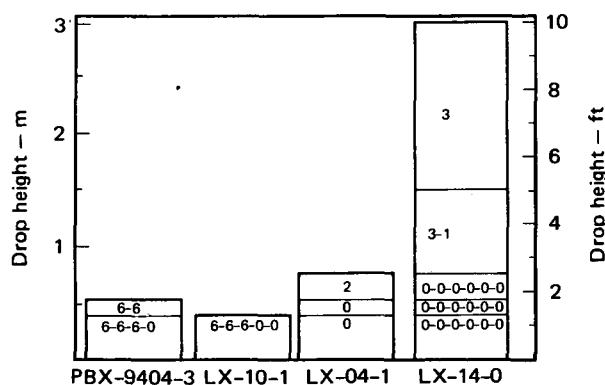


Fig. 1. Comparison of LLL-Pantex skid-test results of various PBX formulations impacted at 14 deg. The intensity scale ranges from 0 = no reaction to 6 = high order detonation.

fall of 0.38 m. In contrast, even after LX-14-0 falls 3 m, it undergoes only a mild, low-order reaction that produces only flame or light. The reaction at impact breaks and scatters the charge.

Of special importance is the observation that LX-14-0 does not undergo *any large* reaction, even when dropped from a height of 1 m, the normal working height encountered in plant handling.

When the four explosives are tested at a 45 deg striking angle (Fig. 2), PBX-9404-3 and LX-10-1 show a detonation threshold after falling only 1 m. LX-04-1 shows only a mild No. 3 threshold reaction after falling 1.5 m. LX-14-0 is definitely less sensitive than any of these explosives, since it exhibits only a mild No. 3 threshold reaction even after falling 3 m.

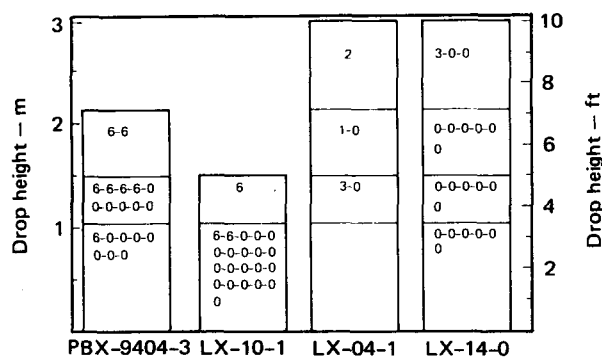


Fig. 2. Comparison of LLL-Pantex skid-test results of various PBX formulations impacted at 45 deg. 10.4-kg billets were tested and the intensity scale is the same as in Fig. 20.

Several HMX/Estane compositions were formulated and skid tested to find the minimum amount of Estane binder that would desensitize the HMX. The compositions and the densities of the billets are listed in Table 1 and the skid test results<sup>3</sup> are shown in Figs. 3 and 4. RX-04-EP (96.2% HMX) was slightly more sensitive than LX-14-0 (95.5% HMX), but much less sensitive than either PBX-9404-3 or LX-10-1. RX-04-EF (97% HMX) and RX-04-EG (98% HMX) had no sensitivity advantage. When only 1% binder was present, as in RX-04-EH, the composition could not be skid tested because the parts were so weak that they

cracked when they fell on the target.

Since the Estane 5702-F1 used for making LX-14-0 is a blend of equal amounts of the low and high viscosity Estanes 5702 and 5702-F2, we tested each component separately. RX-04-EN, RX-04-E0, and RX-04-EE (see Table 1) were formulated with 95% HMX and compared in the skid test. The Estane 5702-F1 blend used in RX-04-EE reduced sensitivity *more* than either the low or the high intrinsic viscosity Estanes used in RX-04-EN and RX-04-E0. While we do not understand this result, it would seem that there is some optimum lubricity and/or elasticity that best desensitize this

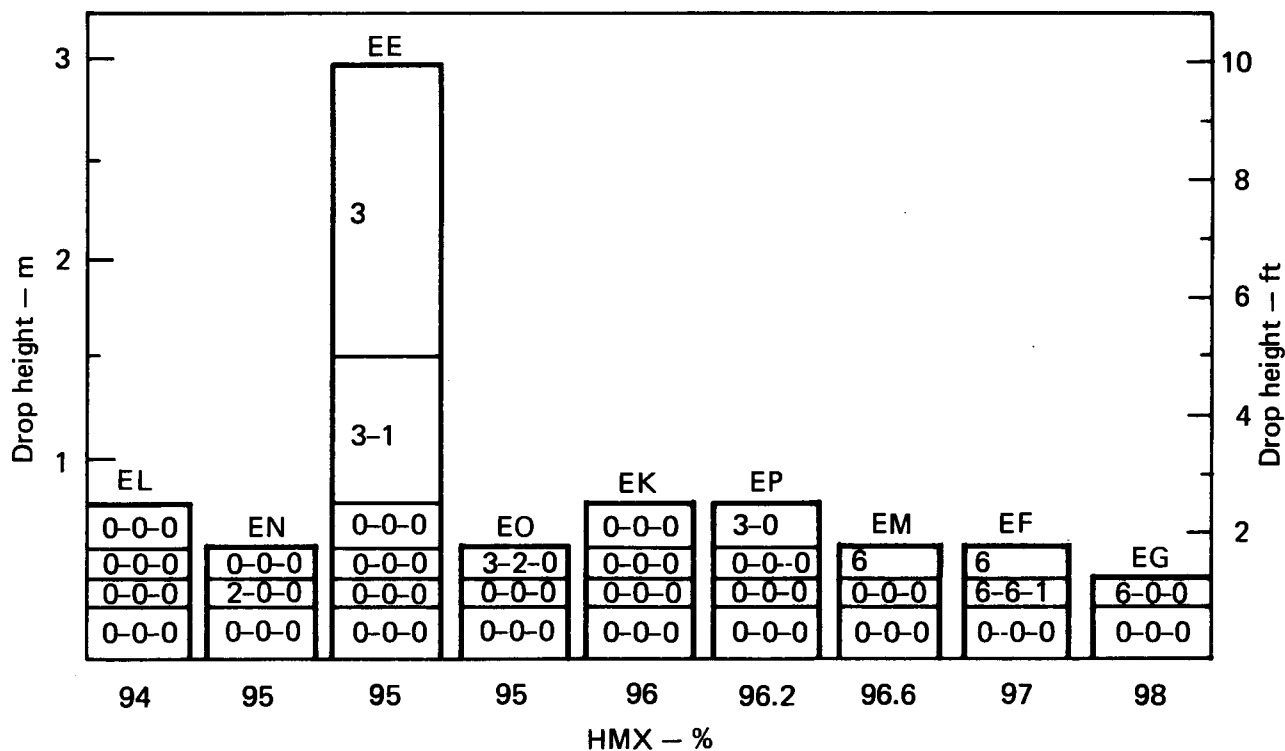


Fig. 3. LLL-Pantex skid tests of HMX/Estane formulations impacted at 14 deg. 10.4-kg billets were tested and the intensity scale is the same as in Fig. 2. Letters above each stack denote samples.



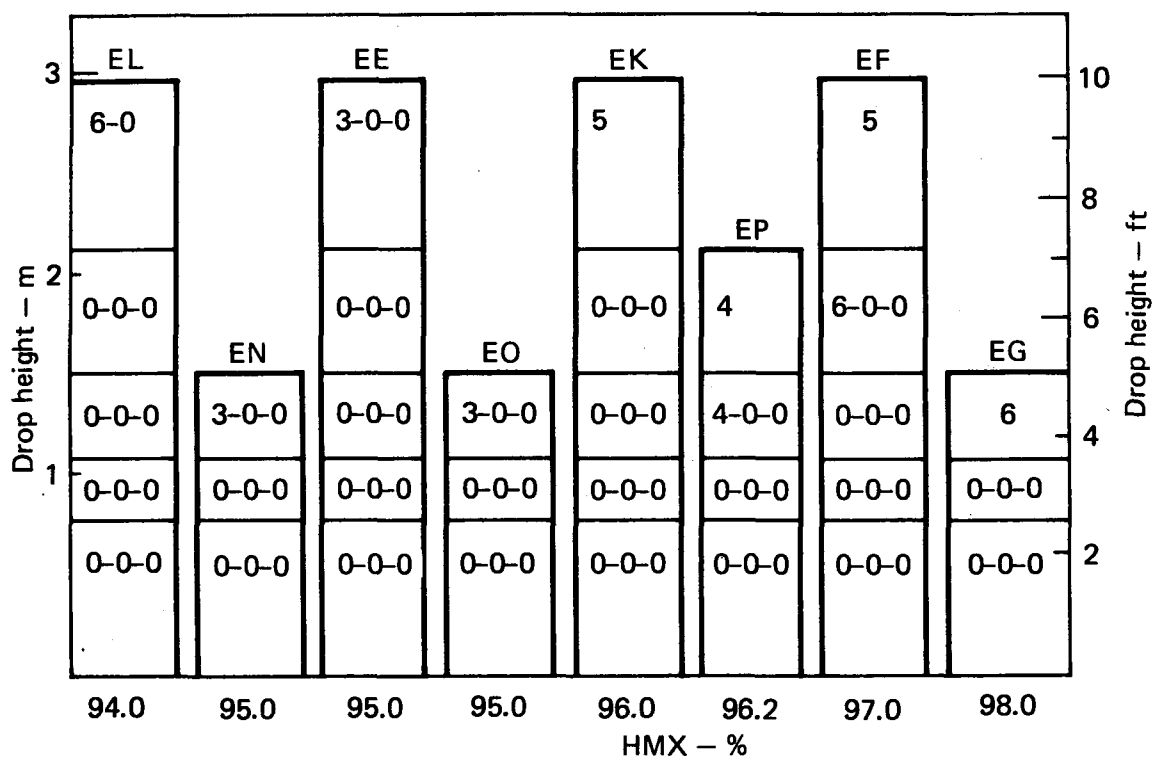


Fig. 4. LLL-Pantex skid tests of HMX/Estane formulations impacted at 45 deg. 10.4-kg billets were tested and the scale is the same as in Fig. 2.

HE. Since the three Estanes were the same except for molecular weight distribution, we advise LX-14-0 producers to use Estane 5702-F1.

#### LASL Skid Test

In their version of the pendulum skid test, LASL tested LX-14-0 on a quartz pad target. This LASL test differs from the LLL-Pantex test in that the HE billet used is a composite with a 38-mm (1.5 in.) thick, hollow hemisphere of HE bonded to a solid hemisphere of mock HE. The composite is slightly smaller than the Pantex billet, weighing only 9.1 kg instead of 10.4 kg (20 vs 23

lb). The explosive shell weight is 5.9 kg (13 lb). Los Alamos determines a 50% height for reaction and the overpressures are measured. The LASL test is limited to a maximum drop height of 4.3 m (14 ft).<sup>4</sup>

The LASL results<sup>4</sup> are in Table 2. If we assume that LX-14-0 would have reacted in all drops at 6.1 m (20 ft), (the worst case possible), the  $H_{50}$  for this test would be >4.3 m (14 ft) at 3.4 kPa (0.5 psi). In contrast, PBX-9404-3 had an  $H_{50}$  of 0.73 m (2.4 ft) at 55 kPa (8 psi) in this test. As in the LLL skid tests, LX-14-0 exhibits very moderate reactions compared to PBX-9404-3.

Table 2. LASL Pendulum Skid Test<sup>6</sup> of LX-14-0<sup>a</sup> (9.1 kg billets).

Drop height, m (ft)	Reaction overpressures, <sup>b</sup> kPa			
4.3 (14)			0	0
3.1 (10)		2.5	0	0
2.1 ( 7)	4.3	0	0	
1.5 ( 5)		0		

<sup>a</sup>Method of testing: if a billet reacts to a drop, the next billet is dropped through a smaller distance. If there is no reaction, the next drop is from a higher point.

<sup>b</sup>Overpressures in kPa.

#### Experimental Skid Tests with Large Billets

To gain more insight into the hazards of handling large charges of PBXs, we extended the scope of the skid test to 136 kg (300 lb) and 45 kg (100 lb) billets. Billets this large have not been tested before, so there are no earlier data for comparison.

We made 136-kg and 45-kg hemispheres from LX-14-0, PBX-9404-3, and LX-14-0 by pressing billets to densities of 1.850, 1.872, and 1.833 Mg/m<sup>3</sup>, respectively, and machining them to shape.

To determine mechanical response of these large billets during impact testing, we dropped them from various heights onto smooth, horizontal steel plates. These drops did not cause detonation because so little friction is involved, but the hemispheres were temporarily deformed during impact. The mark made by the PBX on the steel

plate is called the spot size and the deviation from spherical shape after the hemisphere has recovered for 24 h is the permanent deformation.

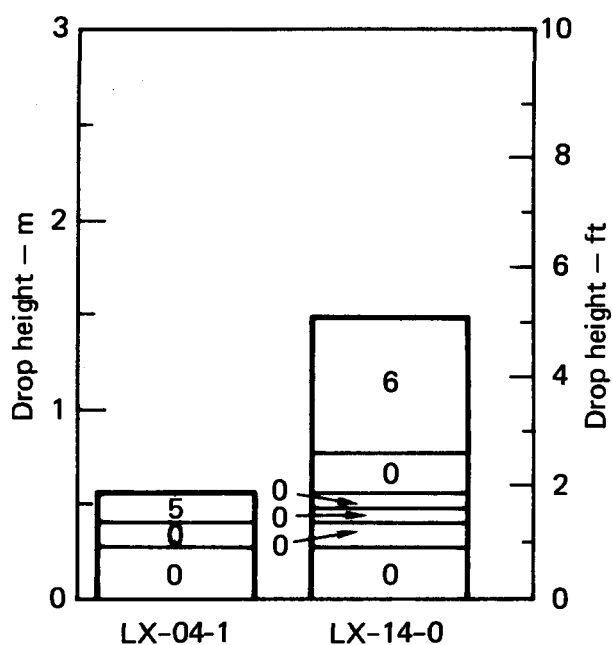
From data<sup>5</sup> in Table 3, we concluded that 136-kg hemispheres of LX-14-0 deform less when dropped than LX-04-1 or PBX-9404-3; but from spot size data, the deformation of LX-14-0 is between the other two PBXs. PBX-9404-3 is mechanically brittle; a hemisphere cracked after dropping 0.305 m.

Figure 5 shows skid-test results from a test in which a 136-kg billet was dropped onto a sanded steel plate at a 45 deg angle.<sup>5</sup> All of the spots of unexploded LX-14-0 hemispheres showed melting at the point where the hemisphere struck the steel plate; melting absorbs energy and may have helped to inhibit an explosive reaction. LX-04-1 melted slightly after a fall of 0.38 m.

Table 3. Spot diameters and permanent deformation caused by drop tests of nominal 136-kg (300-lb) hemispheres of PBX-9404, LX-04-1, and LX-14-0. Test conditions: hemispheres dropped vertically onto smooth steel.

Composition	Drop height, mm	Spot size, mm	Permanent deformation, <sup>a</sup> mm
PBX-9404	19.6	30.0	0.10
	40.1	35.8	0.18
	76.2	41.7	0.23
	304.8	56.4	0.56
LX-04-1	21.3	34.0	0.10
	38.4	39.1	0.20
	77.5	46.5	0.25
	304.8	63.5	0.58
LX-14-0	19.1	32.3	0.05
	35.6	36.3	0.10
	78.2	44.7	0.15
	304.8	63.5	0.48

<sup>a</sup>Permanent deformation was measured with a spherometer 24 h after test.



These 136-kg billets of LX-14-0 detonated when dropped from a height (1.53 m) that would cause only a mild reaction from the standard 10.4-kg LX-14-0 billets. Similarly, the large hemispheres of LX-04-1 also reacted more vigorously than standard LX-04-1 billets; after a drop of

Fig. 5. Nominal 136-kg (300 lb) skid tests of LX-04-1 and LX-14-0 impacted at 45 deg. The intensity scale is defined in Fig. 2.

Table 4. Spot diameter and permanent deformation of nominal 45-kg (100-lb) hemispheres of PBX-9404, LX-04-1, and LX-14-0 dropped vertically onto smooth steel.

Composition	Drop height, mm	Spot size, mm	Permanent deformation, <sup>a</sup> mm
PBX-9404	19.1	22.6	0.02
	36.8	24.1	0.10
	74.9	30.7	0.20
	305.0	40.6	0.38
LX-04-1	19.6	23.6	0.08
	39.4	29.2	0.13
	76.2	33.5	0.18
	305.0	45.5	0.41
LX-14-0	18.5	22.4	0.05
	38.9	26.9	0.08
	76.2	31.8	0.13
	305.0	43.4	0.28

<sup>a</sup>Permanent deformation was measured with a spherometer 24 h after test.

0.53 m, the large LX-04-1 billet deflagrated, whereas the smaller one underwent only a small reaction. The data in Fig. 5 reinforce the conclusion derived from smaller billets that LX-14-0 is *less* sensitive than LX-04-1 in the skid-test.

Table 4 lists the results of dropping the 45-kg (100-lb) skid test hemispheres vertically onto smooth steel.<sup>6</sup> The same mechanical property responses were observed as in the 136-kg test, except that the PBX-9404-3 did not crack in the

0.305-m drop. The LX-14-0 spot size data again were between those of LX-04-1 and PBX-9404-3; LX-14-0 also showed the least amount of permanent deformation.

Figure 6 shows results from the 45-kg skid test.<sup>9</sup> The 45-kg billets of LX-04-1 generated the same reaction level (3) from 1.53 m as the 10.4-kg billets did earlier. However, this was *not* true for the other two HEs. The 45-kg PBX-9404-3 billets gave a level-6 reaction (detonation) when dropped from a slightly lower

height (0.76 m vs 1.07 m) than in the 10.4-kg test.

Surprisingly, the LX-14-0 results were radically different. Figure 7 shows that 45-kg billets of LX-14-0 actually detonated (6 reaction) after dropping only 0.76 m, whereas, in the 10.4-kg test, the billets had *only* a level-3 reaction from 3.06 m. One would conclude from this test that 45-kg billets of LX-14-0 are *more* sensitive than LX-04-1 and comparable to PBX-9404-3.

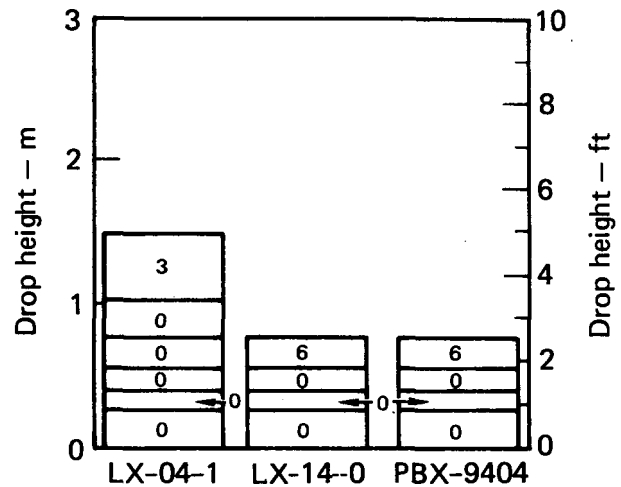


Fig. 6. Nominal 45-kg skid-test results for LX-04-1, LX-14-0, and PBX-9404-3 impacted at 45 deg. The intensity scale is defined in Fig. 2.

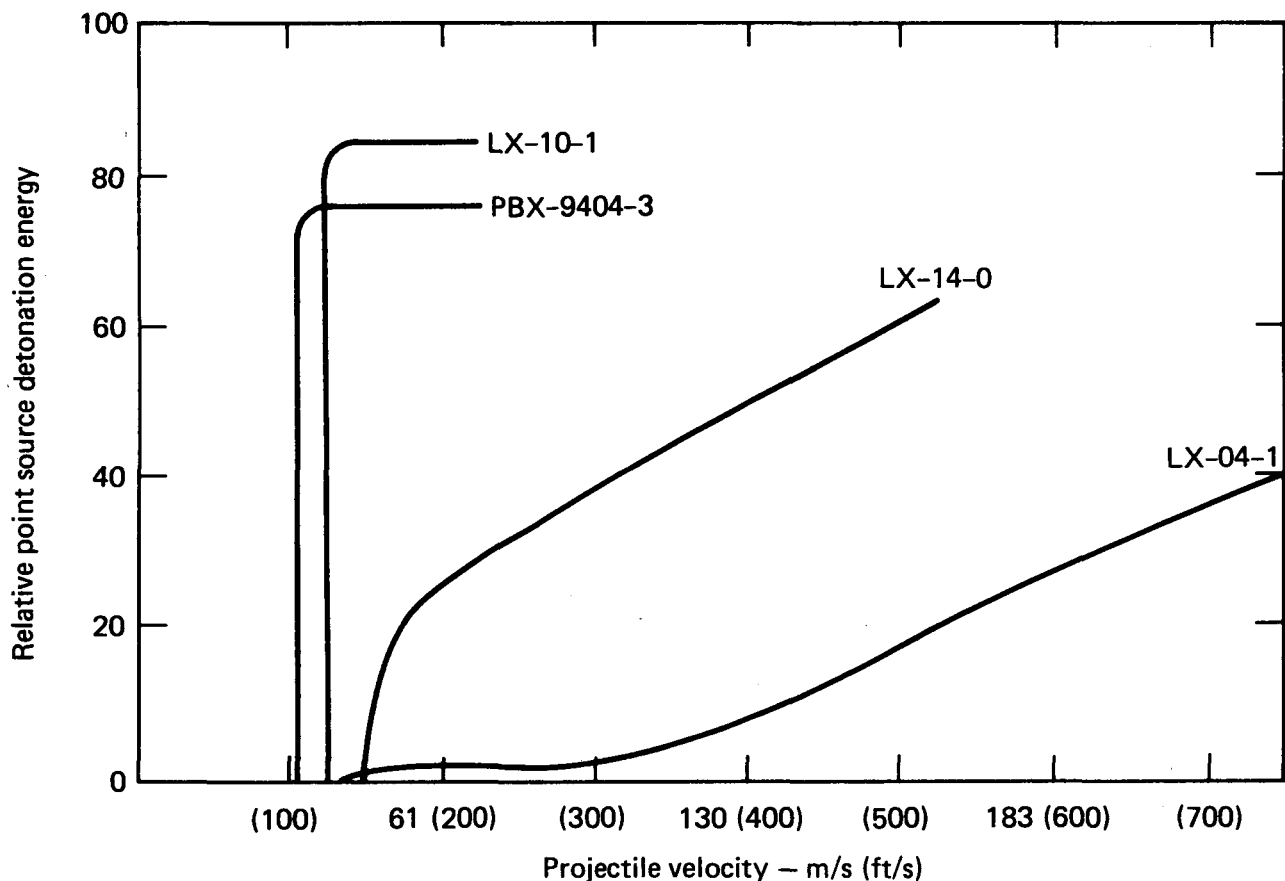


Fig. 7. Summary of results from Susan tests of PBX-9404-3, LX-10-1, LX-14-0 and LX-04-1. The curves show that LX-14-0 has a higher threshold velocity than either PBX-9404-3 or LX-10-1 and reacts less violently with increasing impact velocities.

Additional testing will be required to establish a predictable behavior pattern for these large billets.

### Susan Test

The projectile impact (Susan)<sup>7</sup> test assesses the hazard in dropping an encased HE system from a great height, as in accidental release from an airplane. In this test, about 0.5 kg of PBX is encased in an aluminum cup and fired from an air gun into a reinforced concrete-backed armor-plate steel target. As it strikes, the explosive undergoes severe crushing, shearing, impact, and extrusion forces. The projectile impact velocity is intentionally increased by degrees until a chemical reaction occurs in the explosive.

Impact sensitivity is judged by plotting the energy released by the explosive after the impact as a function of projectile velocity. Pressure gages calibrated with a series of point-source detonations of PBX-9404 at the target allow the projectile's energy release to be calculated. Relative detonation energy is derived from overpressure and transit-time measurements of the air shock wave, from the impact point to the measuring point. The scale used for total energy release ranges from 0 for conditions of no chemical reaction to 100 for detonation-like reactions in which

all of the explosive is consumed.

Table 5 and Fig. 7 show the relative energy release from LX-14-0, PBX-9404, LX-10-0, and LX-04 in the Susan test as a function of the impact velocity.<sup>1,8,9</sup> The data indicate that LX-14-0 has a higher threshold velocity (velocity which just produces a reaction) than LX-10-0 or PBX-9404.

LX-14-0 ignited after about 25 mm (1.0 in.) of projectile deformation, which is consistent with its moderate threshold velocity of about 48 m/s (158 ft/s). In contrast, PBX-9404 ignited after only 8.9 mm (0.35 in.) of deformation of the nose cap, consistent with its very low threshold velocity to ignition of 32 m/s (105 ft/s). Figure 7 shows that LX-14-0 and LX-04-1 require about the same impact velocity, 48 m/s and 43 m/s (158 ft/s and 140 ft/s) to ignite them, though the reaction levels are very low in LX-04-1 up to about 107 m/s (350 ft/s).

Figure 7, also shows that not only do LX-10-1 and PBX-9404 have very low threshold velocities for reaction, but there is rapid buildup to violent detonation. This means that *any* mechanical ignition of these PBXs has a large probability of building to violent deflagration or detonation. On the other hand, LX-14-0 has a moderately low probability of building to a violent reaction or detonation

Table 5. Susan Test summary of the relative energy release of PBX-04-4-3, LX-10-1, LX-04-1, and LX-14-0.<sup>a</sup>

m/s	Velocity,	<u>PBX-9404-3</u>	<u>LX-10-1</u>	<u>LX-04-1</u>	<u>LX-14-0</u>
	ft/s				
30	( 98)	0			
31	(100)	0			
31	(102)	53			
31	(103)	0		0	
32	(105)				0
36	(116)	74			
36	(119)	55			
37	(120)		0		
38	(123)				0
38	(127)	0			
41	(134)		0.1		
42	(136)	71			
44	(144)	76		1	0
45	(147)		84		0
46	(150)	74			
48	(158)				1
56	(183)				24
62	(205)	53		1	
71	(232)	26			
73	(240)		92		
77	(252)				31
82	(268)			1	
96	(316)			2	
107	(350)				63
115	(380)			8	
129	(423)			9	
147	(483)			23	
173	(569)				54
237	(778)			41	

<sup>a</sup>Total energy release values on an arbitrary scale from 0 = no chemical reaction to 100 = detonation that consumes all of the explosive.

in encased systems. Also, the threshold velocity for LX-14-0 is 50% higher than PBX-9404 and 30% higher than LX-10-0.

HMX/Estane 5702-F1 compositions ranging from 94% to 97% HMX (see Table 1) were fired in the Susan test to determine the minimum level of Estane 5702-F1 required to desensitize HMX. The data<sup>5,10</sup> in Table 6 indicate that only compositions containing 3% Estane or less (97% HMX or more) have the low, potentially dangerous threshold velocities like PBX-9404.

Thus both the skid and Susan test results indicate that, if the manufacturing specifications for LX-14-0 are met (within plus or minus 0.6% HMX/batch and plus or minus 0.5% HMX/lot), this PBX has significant advantages over PBX-9404 and LX-10-1 in plant handling and encased impact sensitivity. LX-14-0 that is not within specification is sensitized and approaches PBX-9404 and LX-10-1 in sensitivity.

### Rifle Bullet Impact Test

Weapons made from PBXs must be insensitive to high speed impacts, such as those from a typical rifle bullet.

The Pantex version of the Picatinny Arsenal rifle bullet impact test has always differentiated quantitatively

between very energetic PBXs like PBX-9404-3 and less energetic HEs like LX-04-1. In the Pantex version, a 50.8-mm-diameter by 76.2-mm-long cylinder of explosive is encapsulated in Adiprene adhesive in a capped 50.8-mm (2 in.) schedule-40 pipe nipple that has a cold-rolled steel plate 76 × 76 × 3.2 mm welded on the target end. When a 30-06 rifle bullet is fired at 836 m/s through the flat plate, the cylinder of PBX ignites. Overpressures and time-to-response are recorded.

Rifle bullets were fired into three PBX formulations containing 94, 95, and 96 wt% of HMX and 6, 5, and 4 wt%, respectively, of Estane 5702-F1. Table 7 and Fig. 8 show results from this test and from earlier similar tests with PBX-9404-3, LX-10-1, and LX-04-1.<sup>11</sup>

LX-10-1 produced consistently high overpressures in this test with very short response times. PBX-9404-3 produced high overpressures 80% of the time, but in only one instance was the response time shorter than that of LX-10-1. In every case the HMX/Estane formulations generated much lower overpressures than either of these HEs and took longer to respond. LX-14-0 responses would be between those of RX-04-EE and EK, and generally, like those of the less energetic formulation LX-04-1.



Table 6. Susan tests of HMX/Estane formulations.

		<u>Relative energy release RX-04 series (% HMX)</u>			
<u>Velocity,</u>					
<u>m/s</u>	<u>ft/s</u>	<u>EL (94.0)</u>	<u>EE (95.0)</u>	<u>EK (96.0)</u>	<u>EF (97.0)</u>
27	(87)				0
29	(96)				0
31	(101)				72
32	(105)		0		
35	(115)				0
35	(115)				92
38	(123)		0		
40	(131)			0	
43	(142)	0			
44	(144)		0		
45	(148)				58
45	(149)		0	0	
46	(151)	0			
47	(155)			0	
48	(158)	1		1	
54	(176)	1			
56	(183)		24		
57	(186)			10	
59	(195)	2			
69	(227)	27			
75	(247)			40	
77	(252)		31		59
79	(260)	7			
98	(323)	40			
107	(350)		63		
107	(352)			45	
119	(391)				83
162	(532)	39			
173	(569)		54		

Table 7. Rifle bullet test summary.<sup>c</sup>

<u>Explosive tested</u>	<u>Number of samples reported</u>	<u>Sample density, Mg/m<sup>3</sup></u>	<u>%<sup>b</sup> TMD</u>	<u>Reaction overpressure, kPa</u>	
				<u>Low</u>	<u>High</u>
LX-10-1	9	1.857	97.9	24.573	33.591
PBX 9404-3	8	1.842	98.8	23.821	41.362
LX-04-1	9	1.865	98.8	3.778	12.121
RX-04-EL <sup>a</sup>	10	1.824	99.5	1.827	12.535
RX-04-EE <sup>a</sup>	24	1.832	99.3	5.578	12.845
RX-04-EK <sup>a</sup>	9	1.836	99.0	9.770	14.617

<sup>a</sup>HMX/Estane 5702-F1: -EL, 94/6; -EE, 95/5; -EK, 96/4. LX-14-0 would be between EE and EK.

<sup>b</sup>TMD = Theoretical Material Density.

<sup>c</sup>Pantex version of the Picatinny Arsenal Test, using 30.06 cal. rifle bullet with a muzzle velocity of 836 m/s.

High overpressures ruptured the pipe containers into fragments, but low overpressures merely split open the pipe. Fragmentation was typical when testing LX-10-1 and PBX 9404-3, where response times were short.

Overpressures from PBX-9404-3 were in the high end of the test range, from 23 to 41 kPa. LX-10-1 also produced pressures in this range (24-33 kPa), indicating rapid decomposition, but HMX/Estane compositions decomposed much more slowly producing overpressures in the low range (4-15 kPa), as shown in Table 7.

The test confirms the same lower sensitivity for LX-14-0 that we noted in the skid and Susan tests.

### Wedge Test

To be of practical value, a new explosive must be initiated easily by conventional detonators. Important parameters in judging this are the speed of buildup to detonation and the relationship of that speed to input shock pressures.

One way to determine the shock sensitivity of an explosive is to measure the rate of buildup to a steady-state detonation after an explosive is subjected to an incident shock wave. This rate of buildup (the excess transit time) can be expressed as a function of the incident pressure and of, either, the time

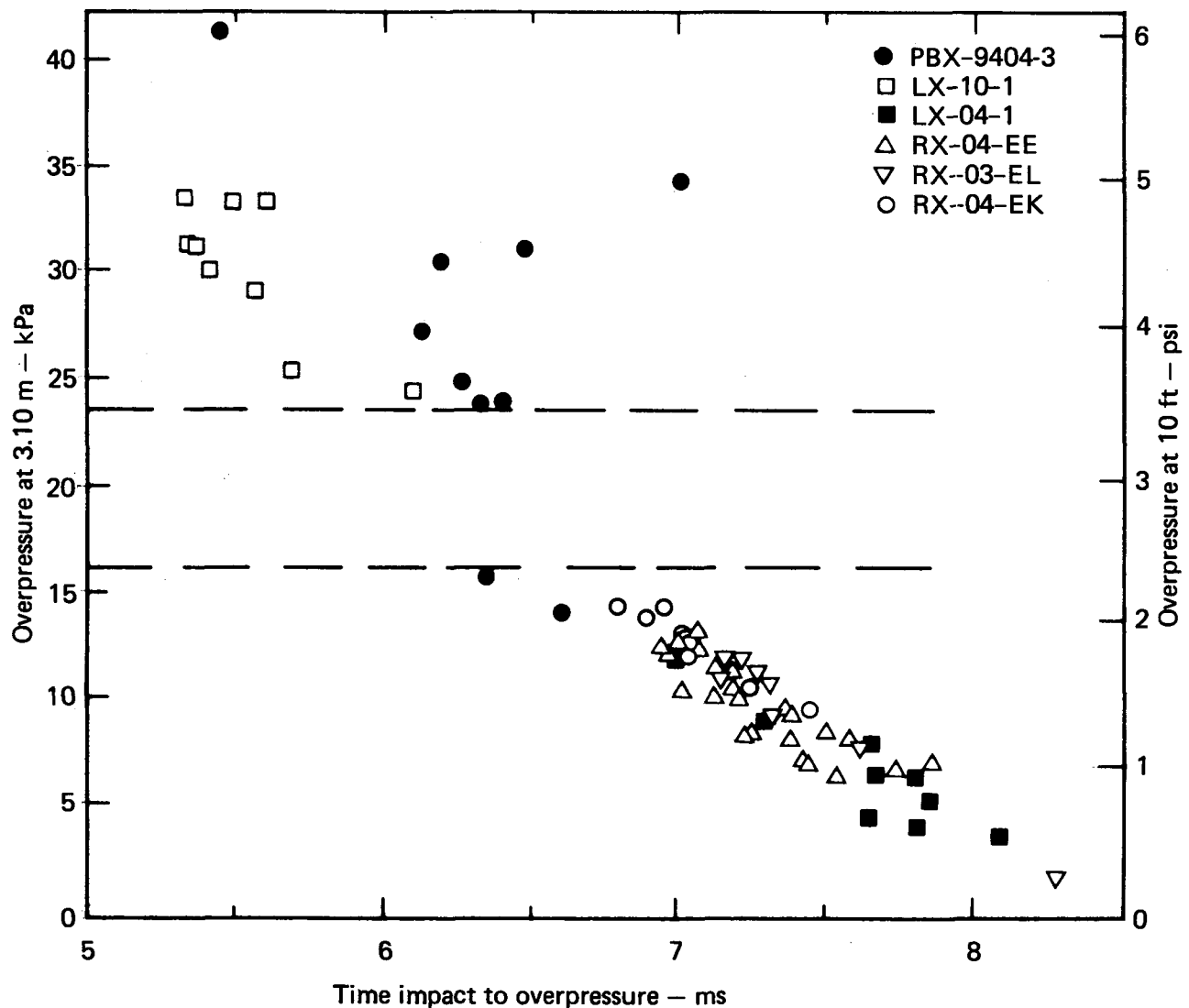


Fig. 8. Pantex version of the Picatinny Arsenal rifle bullet impact test for several high energy PBXs. PBX-9404-3 and LX-10-1 are much more sensitive to this test than LX-04-1 and HMX/Estane systems.

or the distance that the shock wave travels (i.e., the observed shock transit time minus the calculated transit time) before reaching a steady-state detonation. Ramsey and Popolato<sup>12</sup> have shown that, for a given explosive, the relationship is nearly linear between the logarithm of this measured distance (the excess transit time or XSTT) and the loga-

rithm of the entering shock pressure. Therefore, only a few experiments are required to characterize the relationship of the initiating pressure to the distance the shock wave has progressed into a particular explosive before detonation. For a plot of entering shock pressure vs distance to be useful, the shock profiles must be similar in shape and of

sufficient duration to be considered sustained loading during the various time periods of interest.

We used the wedge technique<sup>13,14</sup> to study the shock initiation of LX-14-0. In this test a 50.8 by 50.8 mm wedge of LX-14-0 was subjected to a known planar pressure

input created by an attenuator on an explosive pad on a P-080 plane wave lens. Figure 9 shows the wedge test set-up in detail. The desired initiation pressures met the criteria of pulse shape and duration needed to define the excess transit time (XSTT). Table 8 summarizes the wedge test

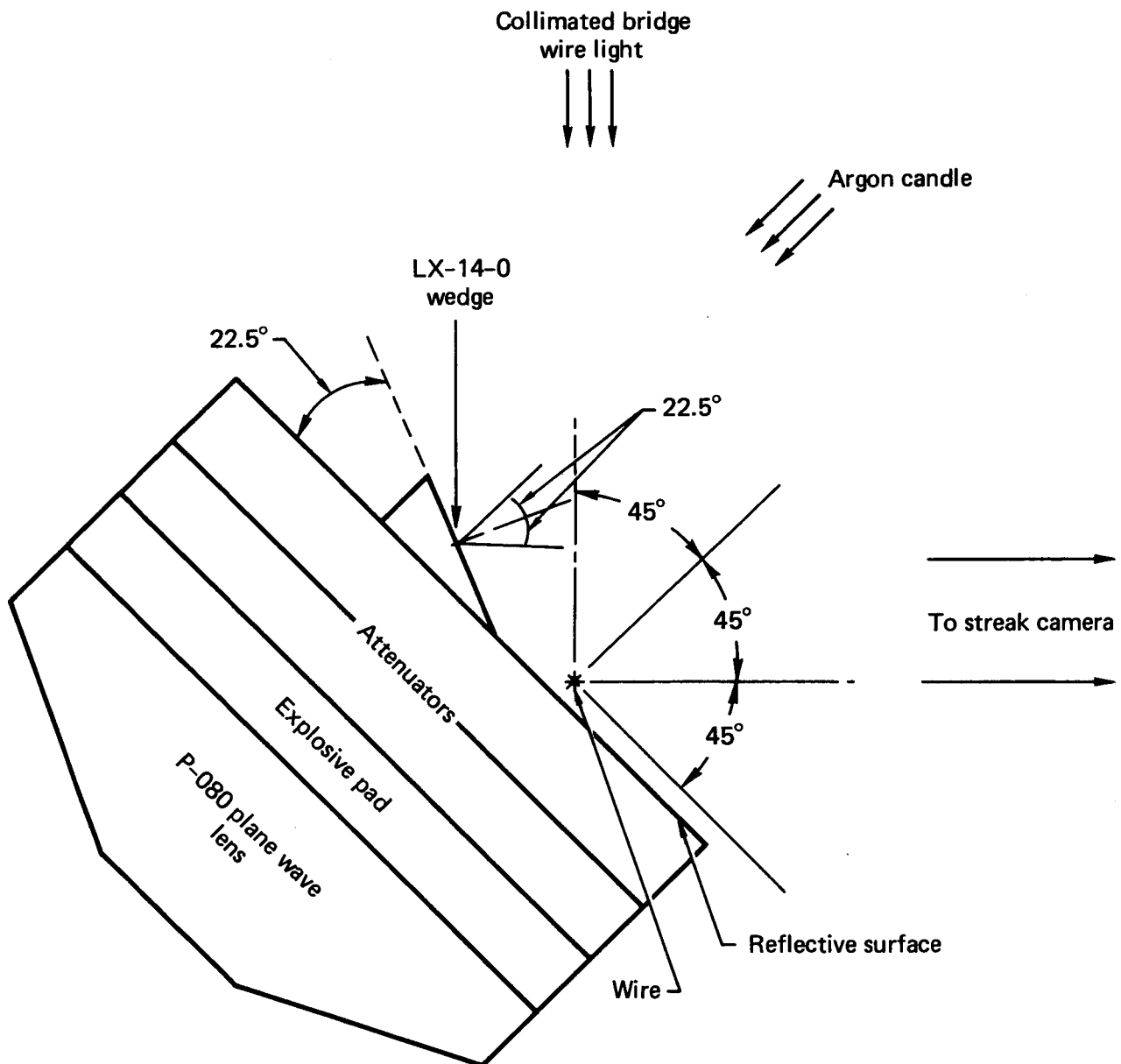


Fig. 9. Schematic of a typical Wedge test assembly used to correlate excess transit time with initiating pressure for LX-14-0.

Table 8. Shock initiation of LX-14-0.

Density of explosive, Mg/cm <sup>3</sup>	Pressure transmitted into explosive, <sup>a</sup> GPa	Distance to detonation, <sup>b</sup> mm	Time to detonation, μs	XSTT, μs
1.833	9.0	2.5	0.48	0.20
1.833	5.9	5.2	1.08	0.49
1.833	5.0	6.6	1.47	0.71

<sup>a</sup> 1 GPa = 10 kbar.

<sup>b</sup> Distance the shock wave travels in the PBX before detonation occurs.

data<sup>5</sup> obtained with LX-14-0 and Fig. 10 compares these results with LX-10-1 and LX-04-1.<sup>15,16</sup>

We conclude from these data that conventional detonators will initiate

LX-14-0 without difficulty because the shock initiation behavior differs so slightly from other HMX PBXs.

### Gap Test

The small-scale gap test<sup>17</sup> is a rapid and inexpensive way to measure the relative shock sensitivity of as little as 5 g of an explosive. The test uses pellets 12.7 mm (0.50 in.) in diameter by 38.1 mm (1.5 in.) long. An inert spacer (thin brass shim) is placed between the test explosive and the standard detonating charge. When the test explosive detonates, it makes a dent in a 10.2-cm (2-in.) thick witness plate.

The shock sensitivity of the test explosive is expressed as the thickness of spacer needed to reduce the probability of detonation of that test explosive to 50%. For this reason, the larger the gap, the more sensitive the explosive.

In this test, LX-14-0 detonated when the gap (thickness) was 1.78 mm,

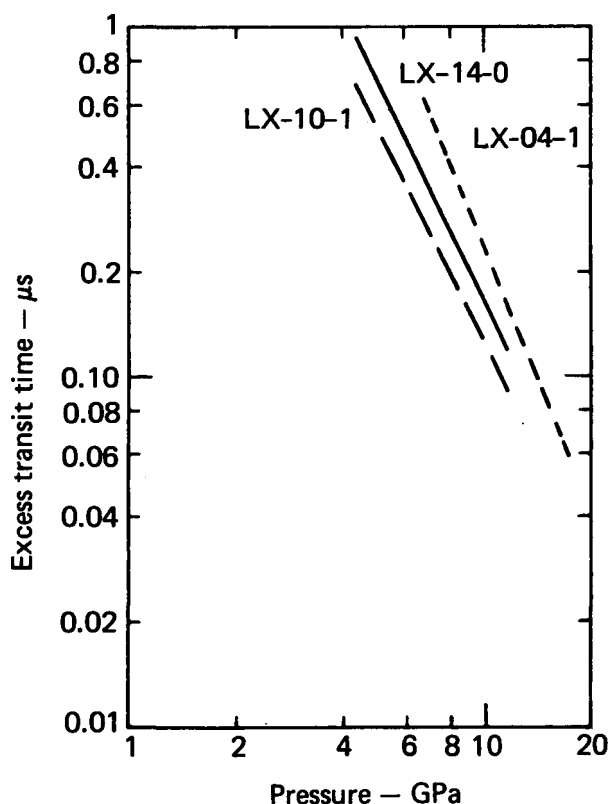


Fig. 10. Correlation of excess transit time (XSTT) with initiating pressure for LX-14-0, LX-10-1, and LX-04-1.

which means that it is more sensitive in this test than LX-04-1 at 1.27 mm and less sensitive than PBX-9404 at 2.46 mm.

### Drop Hammer Test

To evaluate the *relative* impact sensitivity of a new explosive at the earliest stage possible — even before gram quantities are made — we use the drop-weight machine (drop hammer). This machine compares the impact sensitivity of a new explosive with well-known standards. A new HE must have a drop hammer value comparable to HEs in common use before larger scale tests HE can proceed.

In a drop hammer test, a 2.5-kg weight drops on a small (about 35-mg) sample of explosive. The drop is re-

peated from a series of heights. We judge whether explosion has occurred by the measured sound produced. When enough data have been taken the height at which the probability for explosion is 50%,  $H_{50}$ , is calculated. In general, values below 25 cm indicate relative sensitivity to impact, values between 26 and 70 cm indicate moderate sensitivity, and those above 71 cm signal relative insensitivity. Data are, of course, only *approximate* indications of sensitivity, and data from different laboratories do not always correlate.

The  $H_{50}$  values of PBX-9404 and LX-10 were 40 cm, whereas that of LX-14-0 was 51 cm, using roughened steel tooling (type 12B) as a surface. In contrast to larger scale tests, the drop hammer test characterizes LX-14-0 like the other two PBXs, i.e., moderately sensitive.

## MECHANICAL PERFORMANCE

### Tensile and Compressive Performance

Figure 11 shows the effects of various temperatures at constant strain rates on the stress and strain of LX-14-0 specimens.<sup>5,18</sup> Data were measured at temperatures from -37°C to 74°C at a strain rate of  $1.25 \times 10^{-5}$ /s. Stress-strain data at con-

stant crosshead speed are compared with LX-10-1 data<sup>5,18</sup> in Fig. 12.

Uniaxial tensile data<sup>1</sup> at ambient temperature and several strain rates<sup>1,19</sup> are plotted in Fig. 13. The data show that LX-14-0 is tougher in tension (area under the curve) than LX-10-1 over the strain rates tested. Tensile data at high strain

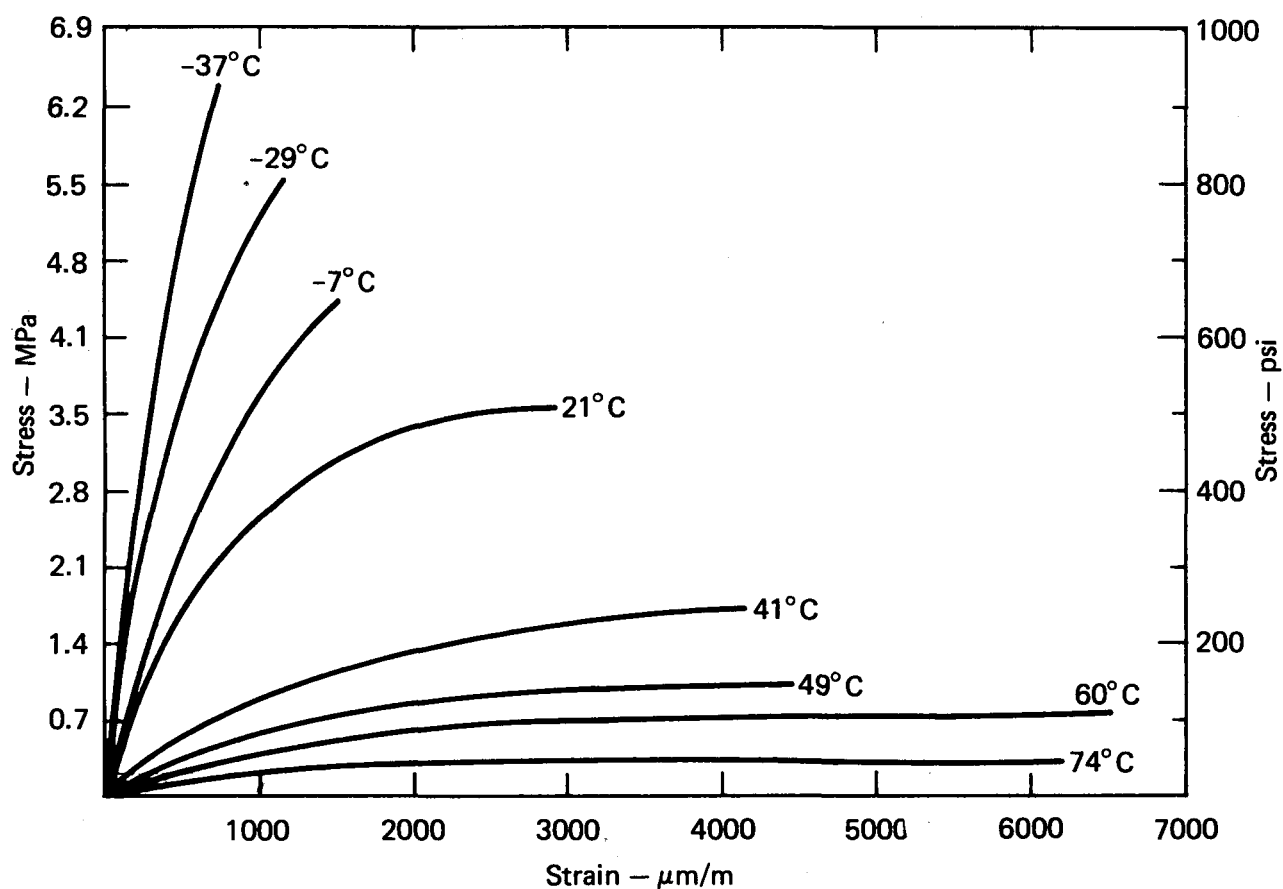


Fig. 11. Tensile tests of LX-14-0 at constant strain rate ( $1.25 \times 10^{-5} \text{ s}^{-1}$ ) and at various temperatures.

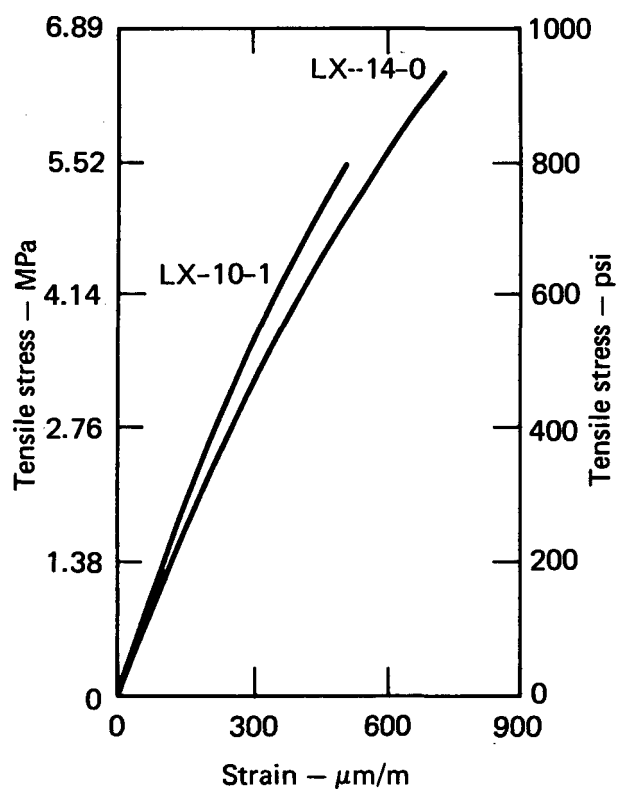


Fig. 12. Tensile tests of LX-14-0 and LX-10-1 at constant crosshead speed and at  $-37^\circ\text{C}$  ( $-35^\circ\text{F}$ ) and  $0.127 \text{ mm/min}$  ( $0.005 \text{ in./min}$ ).

rate conditions could not be obtained because the specimens broke just behind the bond line to the grips.

Figure 14 shows the effects of temperature at constant strain rate on the compressive stress and strain of LX-14-0.<sup>18</sup> Data were measured at temperatures from 21 to 60°C at a strain rate of  $1.25 \times 10^{-5}/s$ .

Figure 15 shows the compression results from uniaxial testing in an Instron machine<sup>1</sup> and with a Hopkinson split-bar gun<sup>19</sup> for LX-14-0 and LX-10-1. It is evident that the strength of LX-14-0 at high strain rates is approximately four times its strength at low strain rates.

### Tensile Failure Envelope

Figure 16 shows the results of uniaxial tensile tests on LX-14-0 at constant strain rate from -37°C to 74°C. We use data in Fig. 16 to show the tensile failure envelope and compare it with envelopes<sup>1,20</sup> for LX-10-1 and PBX-9404. It is evident from these data that LX-14-0 is the strongest and toughest of the three PBXs.

Initial moduli derived from the tensile and compression data<sup>18</sup> are in Fig. 17. Data for LX-10-1 and PBX-9404-3 are included for comparison.<sup>1,20</sup> These HEs are temperature dependent.

### Creep Performance in Tension and Compression

LX-10-1 was the most creep resistant HMX-based explosive available in 1972.<sup>1</sup> We chose it as the standard for comparisons in tests of LX-14-0.

When tested at 21°C and tensile stress levels of 1.24 vs 1.38 MPa (180 vs 200 psi) (see Fig. 18), LX-10-1 and LX-14-0 had similar strains at rupture, but the LX-10-1 specimens broke after only 0.2 h. Specimens of LX-14-0 resisted failure longer, breaking after 62 h.<sup>5,21</sup>

Similar tensile creep behavior was observed<sup>5,21</sup> at 49°C (see Fig. 19). At this temperature the LX-14-0 resisted rupture for 39 h, but the LX-10-1 specimens broke after 16 h.

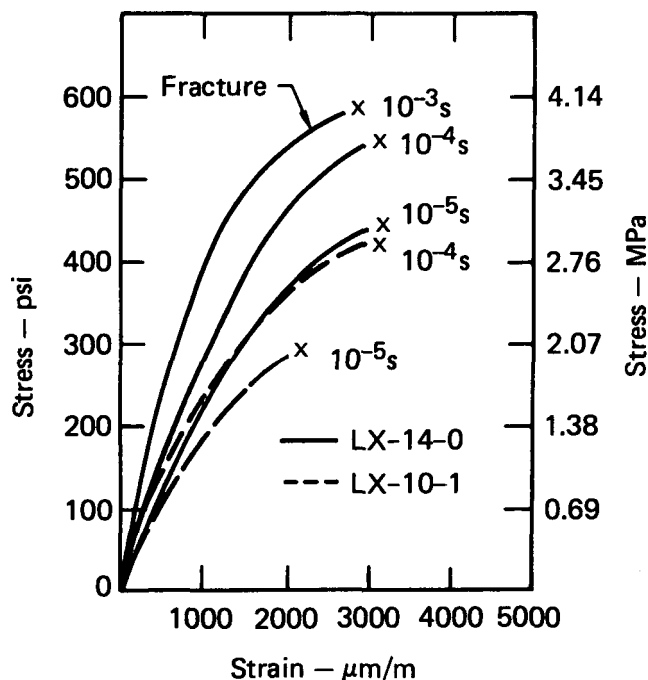


Fig. 13. Uniaxial tensile data of LX-14-0 and LX-10-1 at ambient temperature and indicated strain rates.



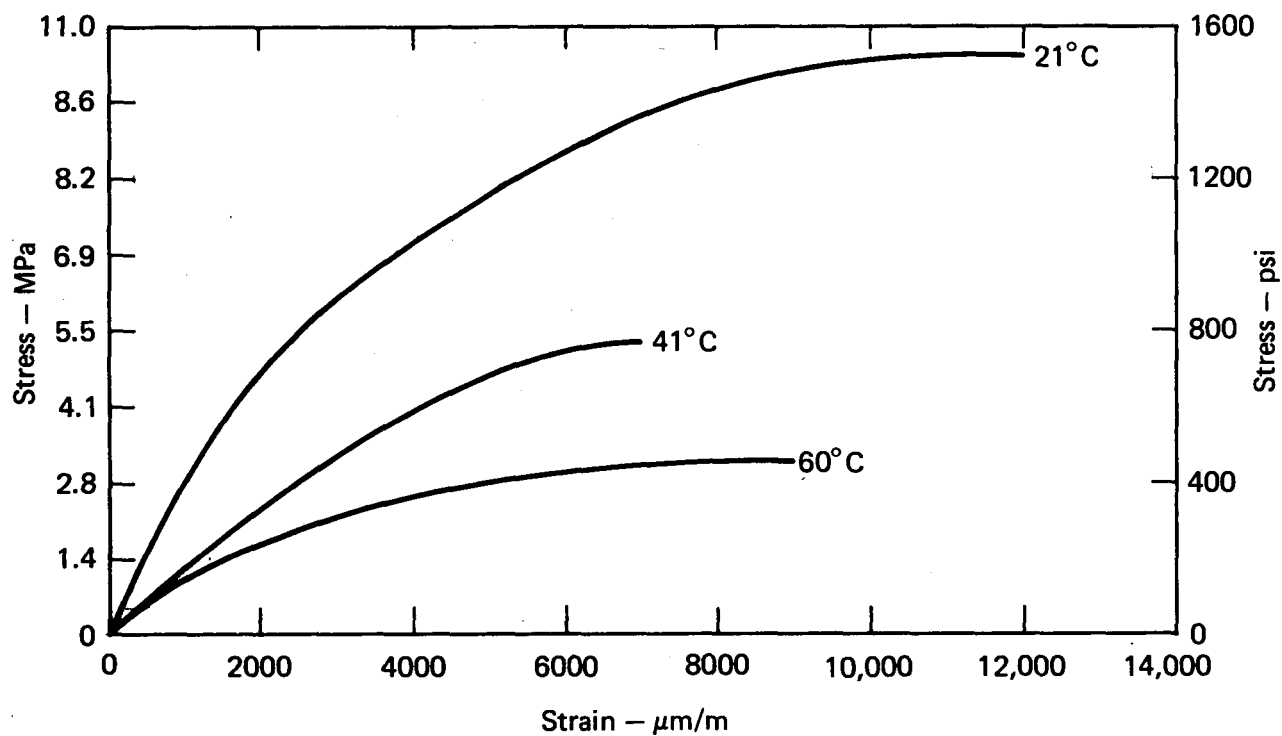


Fig. 14. Constant strain-rate ( $1.25 \times 10^{-5}$  s) compression curves for LX-14-0 at various temperatures.

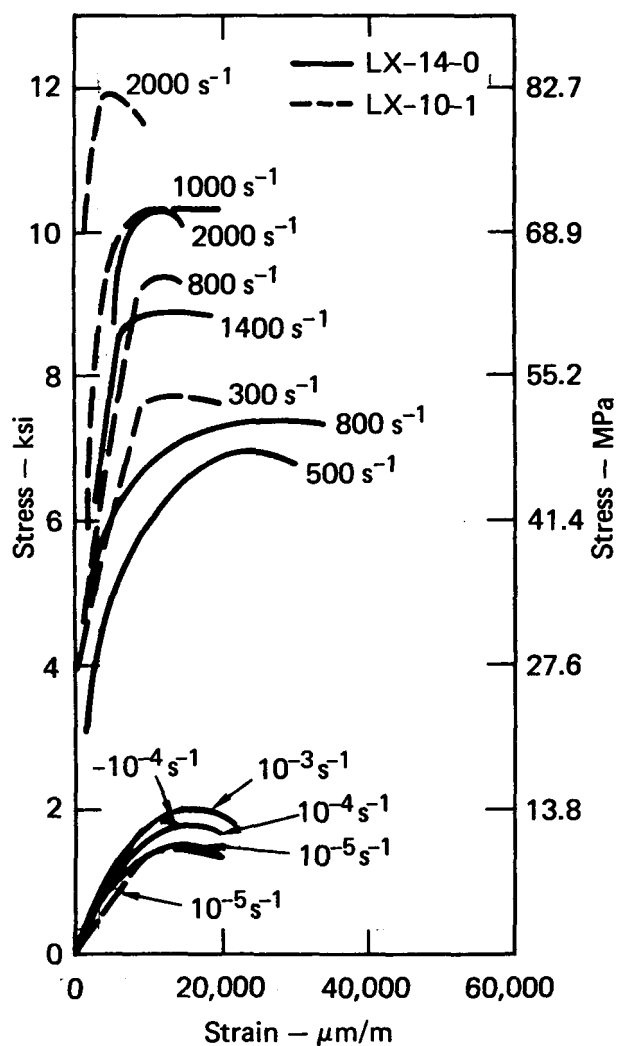


Fig. 15. Uniaxial compression data of LX-14-0 and LX-10-1 at ambient temperature and indicated strain rates.

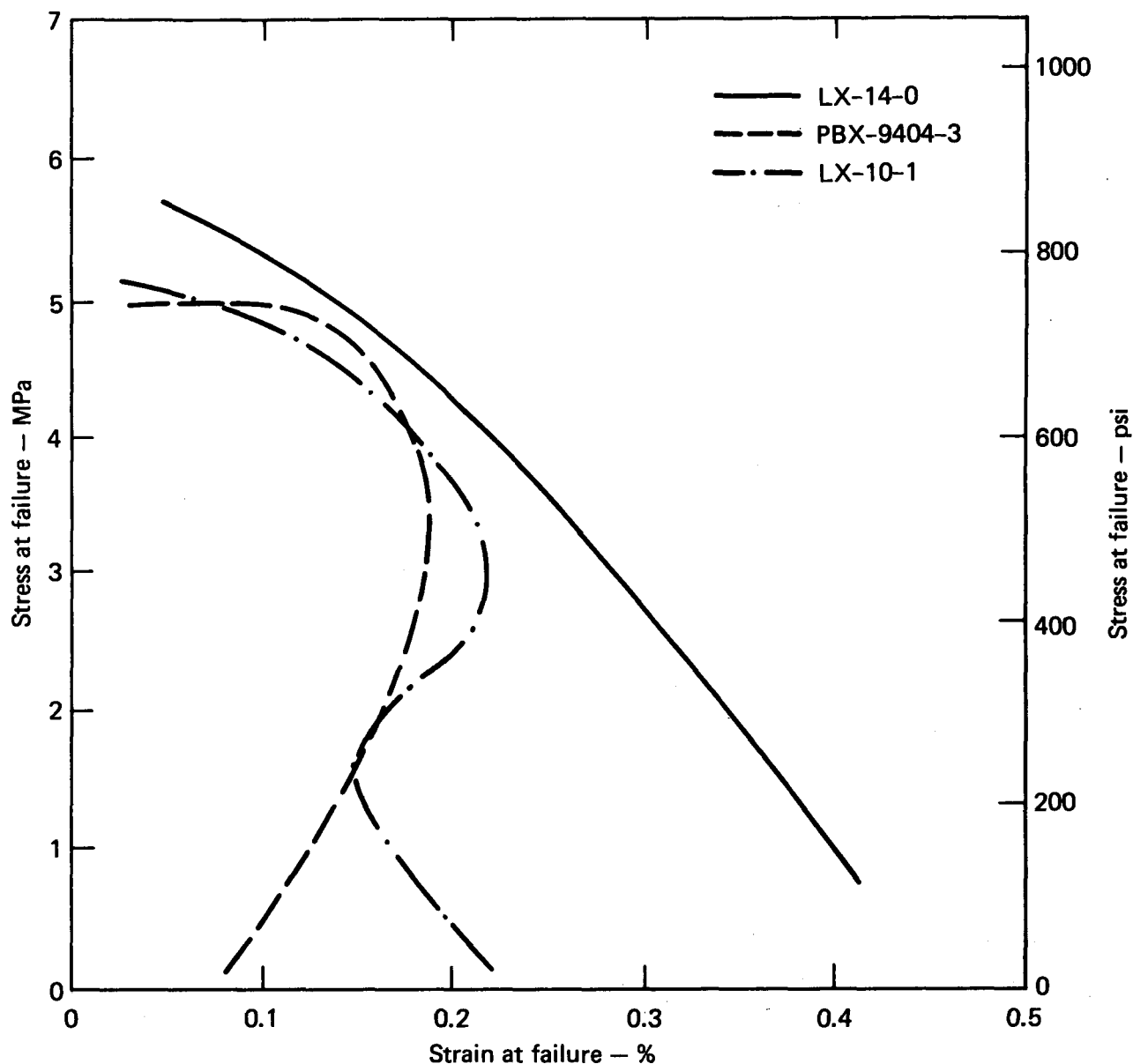


Fig. 16. Tensile failure envelope for LX-14-0, PBX-9404-3, and LX-10-1 at constant strain rate.

Figure 20 shows the compressive creep compliance of these two PBXs at  $49^{\circ}\text{C}^{5,21}$  up to 24 h. In this comparison, although LX-14-0 was tested at an 80% higher stress than LX-10-1, 1.24 vs 0.69 MPa (180 vs 100 psi), it

still showed a lower creep compliance.

We concluded from these data that LX-14-0 has excellent resistance to creep compared with LX-10-1, within the load and temperature ranges examined.

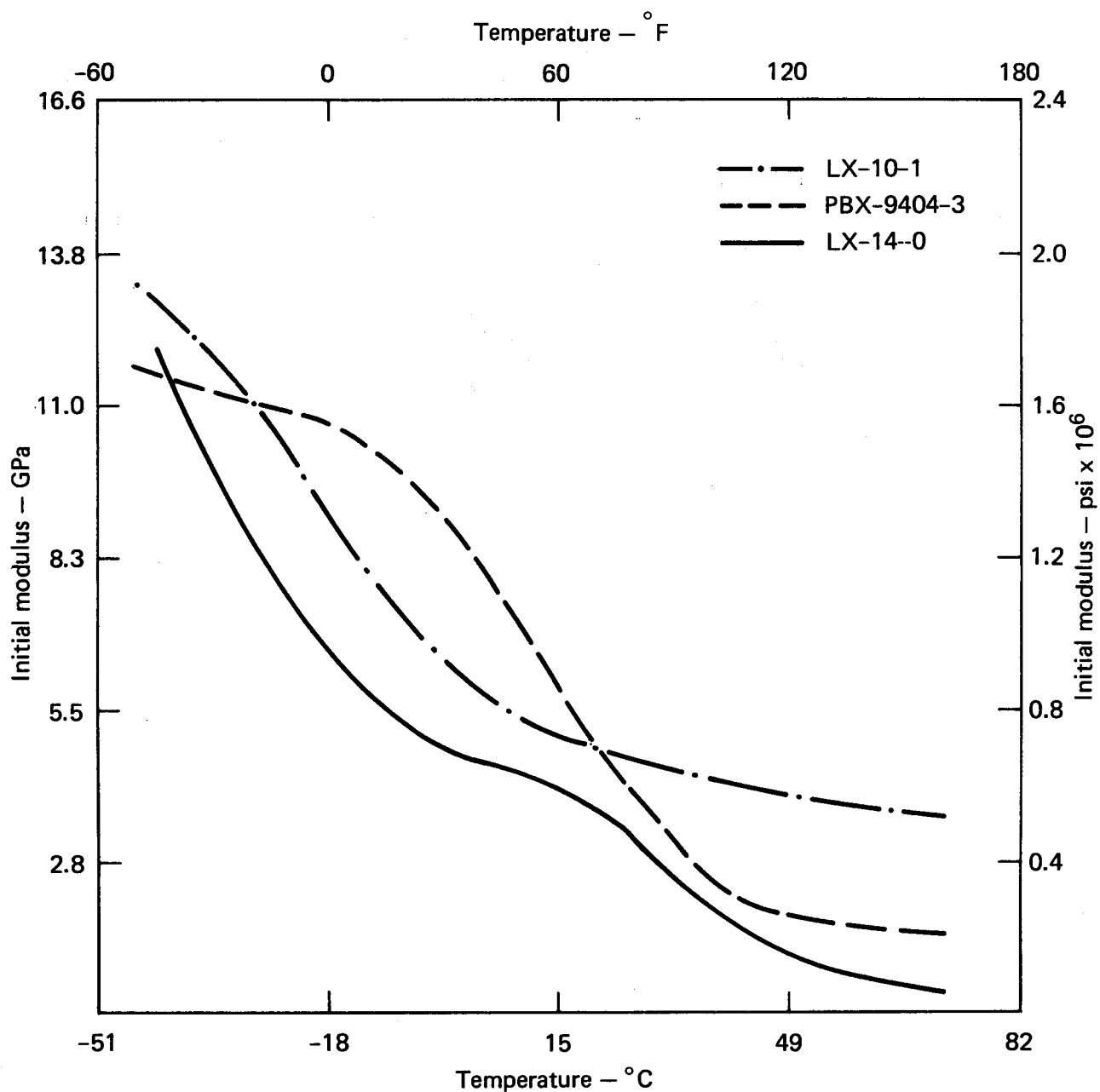


Fig. 17. Initial moduli of LX-14-0, LX-10-1, and PBX-9404-3.

### Simulated Weapons Confinement Test

We have noted in the past that when an HE is thermally cycled under confinement before testing, it increases in density and is less compliant. Also, there is a dramatic increase in

tension-creep rupture life.<sup>1</sup> In contrast, thermal cycling an HE without confinement causes an insignificant change in density and produces specimens that rupture sooner and at higher strains during tension-creep tests than control specimens. These tests

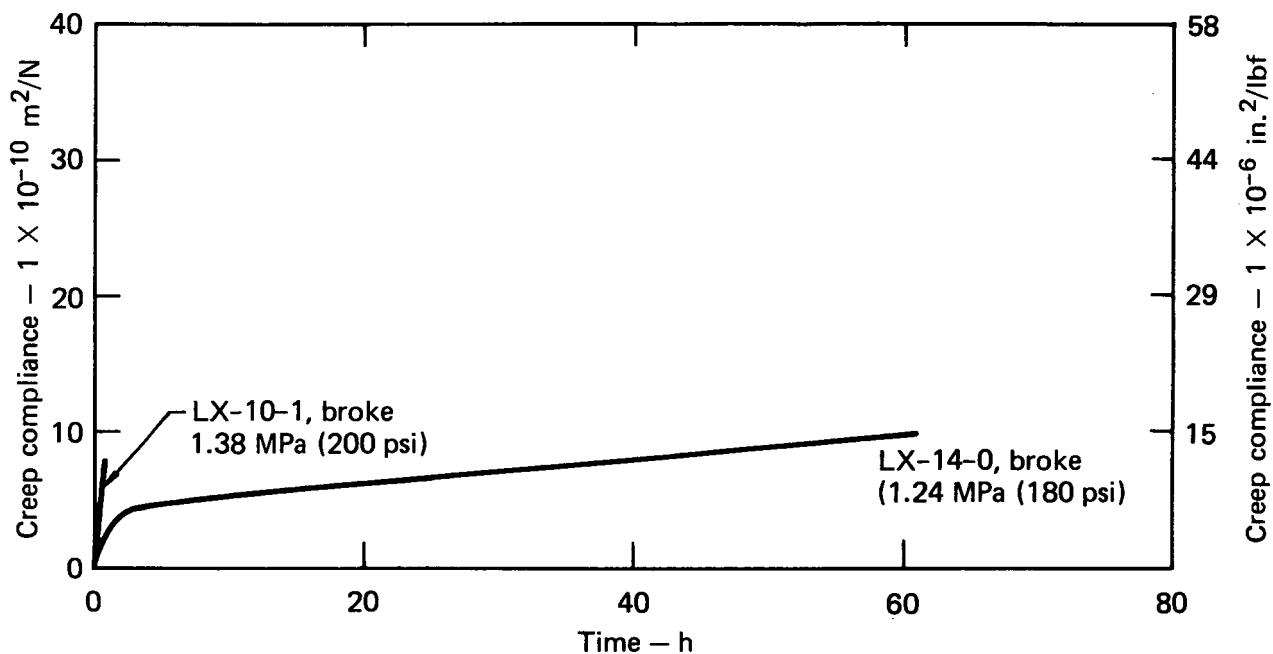


Fig. 18. Tensile creep compliance ( $J(t) = \epsilon(t)/\sigma$ ) vs time for LX-14-0 and LX-10-1 at 21°C (70°F). Note that the LX-10-1 stress is 10% higher than that of LX-14-0.

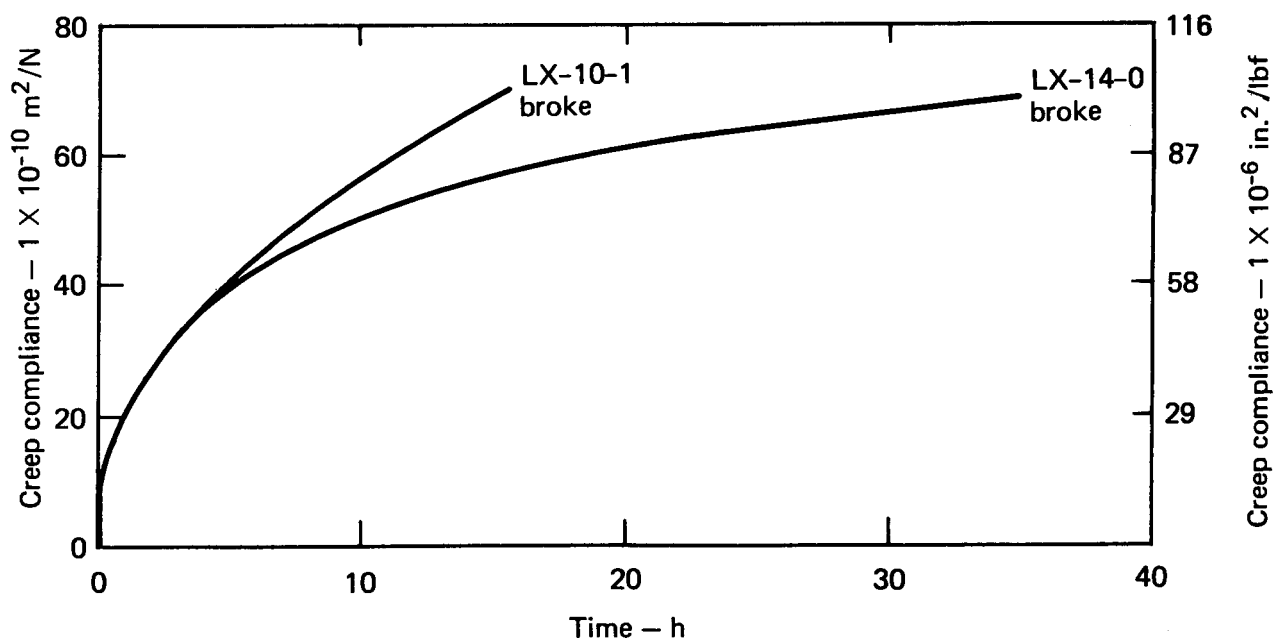


Fig. 19. Tensile creep compliance ( $J(t) = \epsilon(t)/\sigma$ ) vs time for LX-14-0 and LX-10-1 at 49°C and 0.35 MPa (120°F and 50 psi).

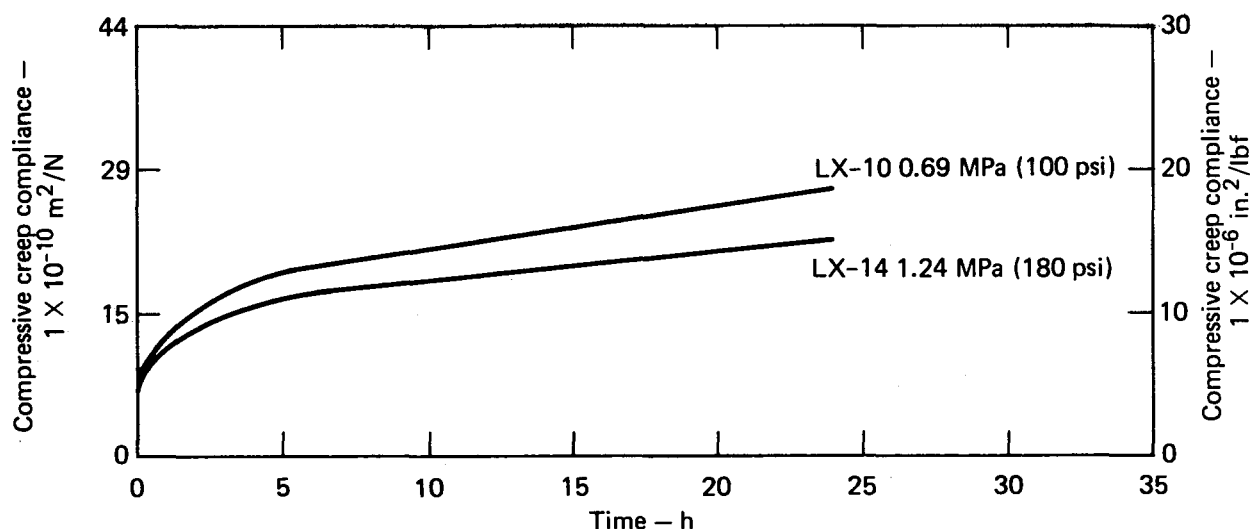


Fig. 20. Compressive creep compliance ( $J(t) = \epsilon(t)/\sigma$ ) of LX-14-0 and LX-10-1 at 49°C.

were done to study the affect of densification when HE is confined in a weapon exposed to normal temperature excursions.

The effects of confinement and thermal cycling of LX-14-0 specimens are similar to those of LX-10-1 tested previously.<sup>1</sup> Billets of LX-14-0 were pressed and divided into sections that were then pretreated in several ways (see Table 9). Control specimens are coded C. Another group of specimens (coded TU) were thermally cycled unconfined from 21 to 74 to 21°C once a day for 15 days. A third group was confined while thermally cycled (coded TC) once a day for 30 days. A fourth group (coded TC + TU) was cycled under confinement once a day for 30 days followed by thermal cycling without

confinement once a day for an additional 15 days.

Table 9 lists the densities of the samples after treatment and shows that LX-14-0 densifies when thermally cycled if confined, but not when thermally cycled unconfined.<sup>22</sup> Unconfined cycling after confined cycling lowers the density below the maximum achieved by confinement. As reported earlier,<sup>1</sup> LX-10-1 showed similar behavior.

Figure 21 shows the tensile-creep curves of control and thermally treated specimens.<sup>22</sup> Confinement during thermal cycling greatly lengthens the time before creep-rupture and reduces the compliance, perhaps because the Estane wets the crystals of HMX better after treatment, and the treatment annealed out microstrains. When

Table 9. Density of LX-14-0 after thermal treatment.

<u>Specimen</u>	<u>Disposition</u>	<u>Density, Mg/m<sup>3</sup></u>
(C) Control	No thermal treatment	1.828
(TU) Unconfined	(Thermally cycled from 21°C to 74°C to 21°C once per day for 15 days.)	1.828
(TC) Confined	(Thermally cycled under pressure once per day for 30 days)	1.840
(TC+TU) Confined plus Unconfined	(Thermally cycled under pressure once per day for 30 days, followed by thermally cycled once per day for 15 days.)	1.835

not confined during thermal treatment before testing, specimens ruptured sooner than the control did. Specimens that had been confined during cycling, then cycled unconfined, showed increased compliance in the creep test and reduced time to rup-

ture. These samples had less compliance and longer creep lives than the control.

Results<sup>22</sup> from compressive creep tests of thermally treated LX-14-0 and control specimens are plotted in Fig. 22. These tests were

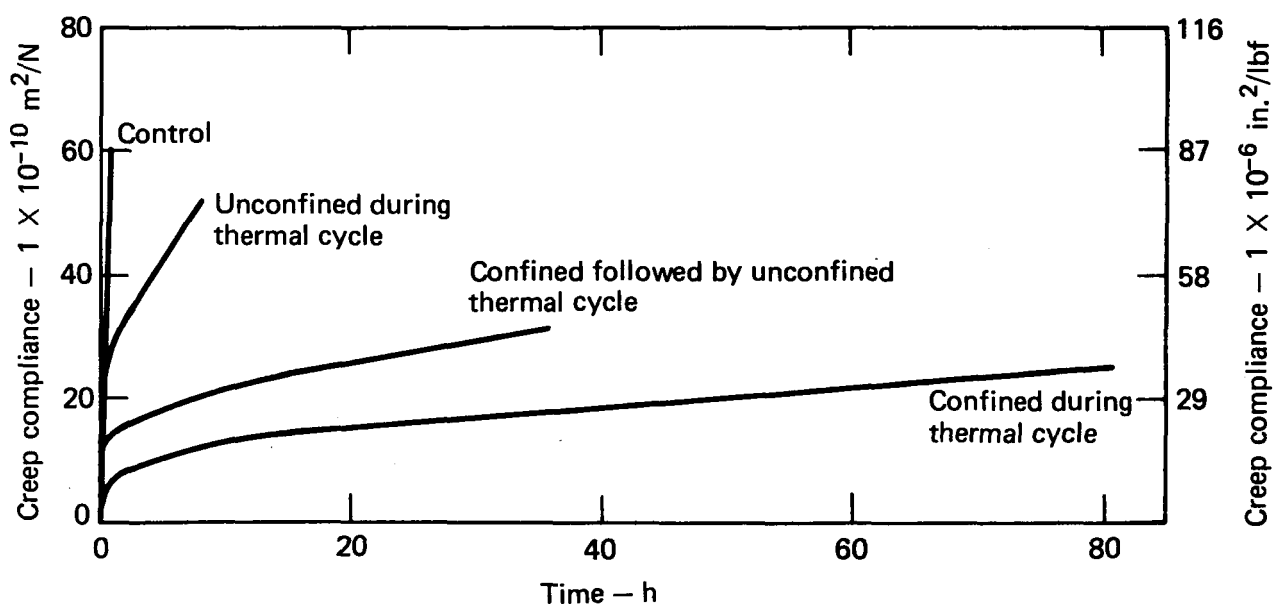


Fig. 21. Tensile creep compliance ( $J(t) = \epsilon(t)/\sigma$ ) vs time for thermally treated LX-14-0. Measurements at 49°C and 0.621 MPa (120°F and 90 psi).

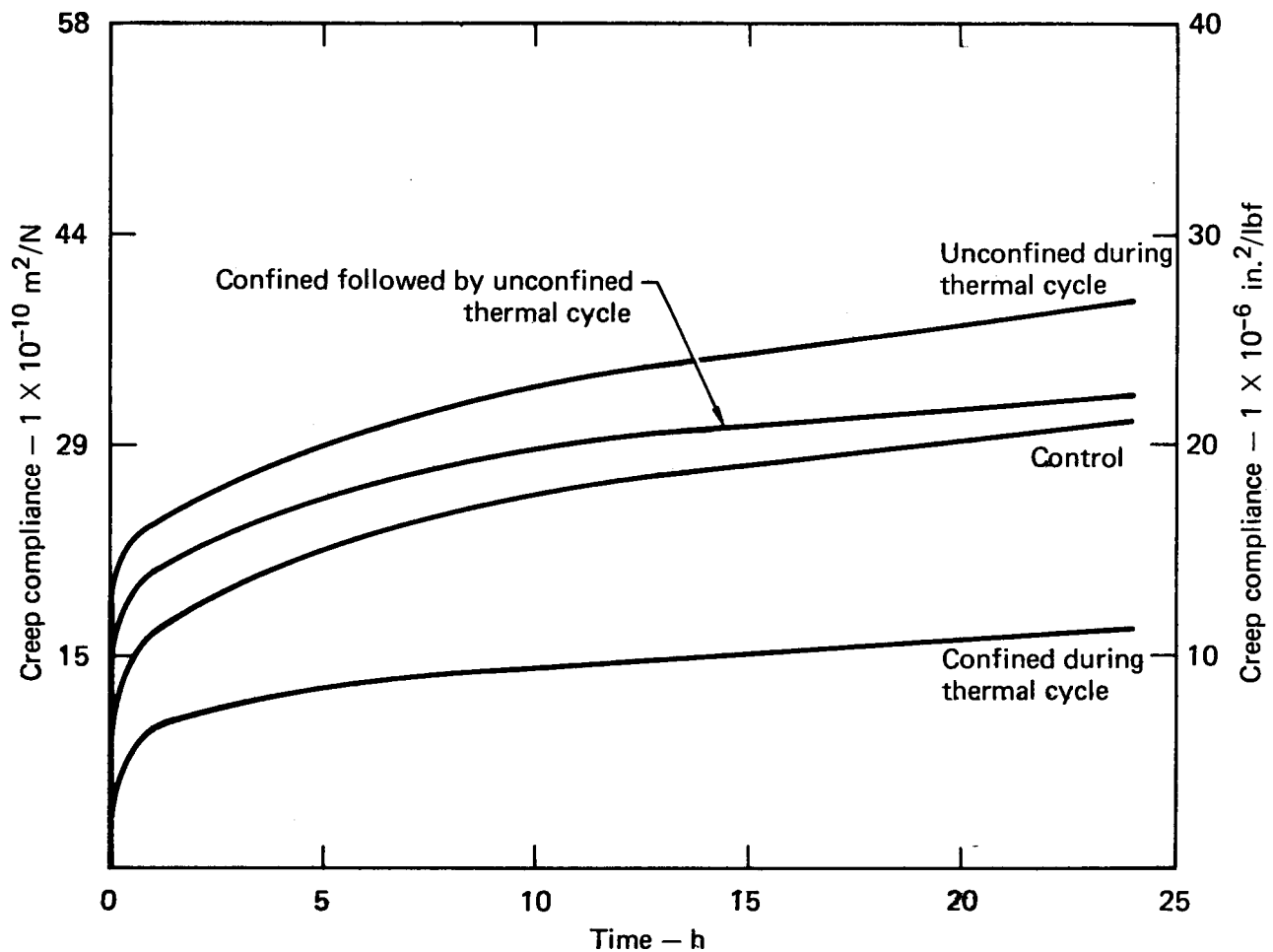


Fig. 22. Compressive creep compliance ( $J(t) = \epsilon(t)/\sigma$ ) of thermally treated LX-14-0 at 49°C and 1.24 MPa (120°F and 180 psi).

discontinued at 24 h. As expected from the tensile-creep results, cycling under confinement produced a marked reduction in the strain that occurred during the creep test. This was not true for either the unconfined ther-

mally cycled specimens or the confined thermally cycled plus unconfined thermally cycled specimens. Thus, the effect of thermal cycling on tension-creep properties is about the same as on other PBXs.

## THERMAL PROPERTIES

We use several tests at LLL to determine whether or not thermal properties of an explosive are

suitable for weapons application. Differential thermal analysis (DTA) provides information about the

kinetics of decomposition, the phase changes, melting points, and thermal stability of an explosive. A chemical reactivity test<sup>23</sup> (CRT) determines the inherent reactivity of an explosive, by itself, and when intimately mixed with other materials. A vacuum stability test measures the total volume of gases evolved from a sample; it is sometimes used in addition to the CRT. A Henkin Test measures the thermal stability of a lightly confined explosive. We determine the coefficient of thermal expansion (CTE) and the coefficient of thermal conductivity (CTC) for engineering design purposes.

A DTA thermogram<sup>24</sup> of LX-14-0 shows the normal HMX phase transition endotherm at 185°C, followed by decomposition beginning at 250°C. The usual heating rate, 10°C/min was used in this test.

The LLL chemical reactivity test examines the gases evolved from a 0.25-g test sample heated in tubes filled with inert gas at 120 and 140°C for 22 h. The gases pass through a two-stage chromatography unit, which measures the individual volumes of N<sub>2</sub>, NO, CO, N<sub>2</sub>O, and CO<sub>2</sub> generated during the test. Table 10 shows volumes of gases evolved from LX-14-0, LX-10-1 (our most thermally stable HE), and PBX-9404-3. Table 11 also includes results from the vacuum stability test in which 1 g of explosive is heated for 48 h at 120°C. Only insignificant quantities of gas are evolved from LX-14-0 and LX-10-1, but PBX-9404-3 generates a significant gaseous product.

Table 11 compares the thermal stability of Estane 5702-F1 and LX-14-0 under CRT conditions.<sup>24</sup> The quantity of gas evolved from the

Table 10. Thermal stability of LX-14-0, LX-10-1, and PBX-9404-3.

Formulation	LLL chemical reactivity test (0.25 g for 22 h)		Vacuum stability test (1.0 g for 48 h)	
	Temp, °C	Gas STP, <sup>a</sup> cm <sup>3</sup>	Temp, °C	Gas STP, <sup>a</sup> cm <sup>3</sup>
LX-14-0	120	0.02	120	0.03
	140	0.04		
LX-10-1	120	0.02	—	—
	140	0.04		
PBX-9404-3	120	0.38	120	3.2-4.9
	40	1.39		

<sup>a</sup>For reference purposes, if 1 cm<sup>3</sup> of gas at standard temperature and pressure, is evolved per gram of explosive, this represents about 0.2% decomposition.



Table 11. Thermal stability of Estane 5702-F1 and LX-14-0 (0.20 g sample).

Material	Temperature, °C	Time, h	cm <sup>3</sup> gas/0.25 g sample
Estane 5702-F1	100	22	0.02
	120	22	0.06
	140	22	0.21
LX-14-0	100	24	0.01
	100	48	0.02
	120	24	0.01
	120	48	0.03
	120	72	0.04
	140	24	0.05
	140	48	0.08
	140	72	0.08

sample containing only Estane 5702-F1 almost doubled for each 10°C rise in temperature (the 100°C gas value was rounded to 0.02 from 0.015 cm<sup>3</sup>). However, gas evolution from LX-14-0

(4.5% wt Estane) does not double, indicating that HMX is stable in this environment. All three tests, the DTA, CRT, and vacuum stability, confirmed the excellent thermal stability of LX-14-0 and indicated that LX-14-0 is as stable as LX-10-1.

Table 12. Henkin thermal stability test data for LX-14-0 and LX-10-1 (0.080 g sample confined and heated to explosion).

Temperature, °C	Time to detonation, s (Harmonic mean)	
	LX-14-0	LX-10-1
220	845	
230		489
232	405	
238		235
246	155	193
255	76	
258		73

In the Henkin thermal stability test, 80 mg of confined explosive is heated at preset temperatures until explosion occurs. Table 12 and Fig. 23 compare Henkin results for LX-14-0 and LX-10-1. As shown in Fig. 23, the slope of the curves of the time to explosion vs the reciprocal of the temperature are the same, suggesting that LX-14-0 and LX-10-1 have similar activation energies. This test also confirmed the excellent thermal stability of LX-14-0.

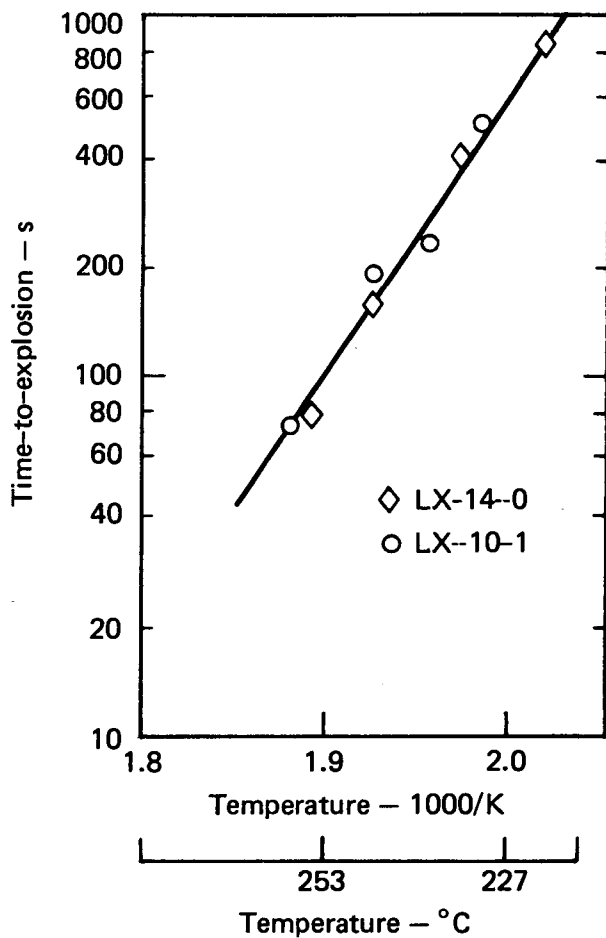


Figure 24 and Table 13 show that the coefficient of thermal conductivity (CTC) and the coefficient of thermal expansion (CTE) of LX-14-0, LX-10-1, and PBX-9404-3 are all acceptably low.<sup>2,25</sup>

Fig. 23. Time-to-explosion vs reciprocal of the temperature for LX-14-0 and LX-10-1 (Henkin test).

Table 13. Coefficient of thermal expansion of LX-14-0, LX-10-1, and PBX-9404.<sup>4</sup>

Temperature range	$\mu\text{m}/\text{m}^{\circ}\text{C}$ ( $10^{-6}$ in./in. - $^{\circ}\text{F}$ )		
	LX-14-0	PBX-9404	LX-10-1
-54 to -34 $^{\circ}\text{C}$ (-65 to -30 $^{\circ}\text{F}$ )	45.7 (25.4)	50.6 (28.1)	—
-34 to 74 $^{\circ}\text{C}$ (-30 to 165 $^{\circ}\text{F}$ )	56.9 (31.6)	58.0 (32.2)	—
-54 to -28 $^{\circ}\text{C}$ (-65 to -18 $^{\circ}\text{F}$ )	—	—	44.6 (24.8)
-28 to 74 $^{\circ}\text{C}$ (-18 to 165 $^{\circ}\text{F}$ )	—	—	47.0 (26.2)

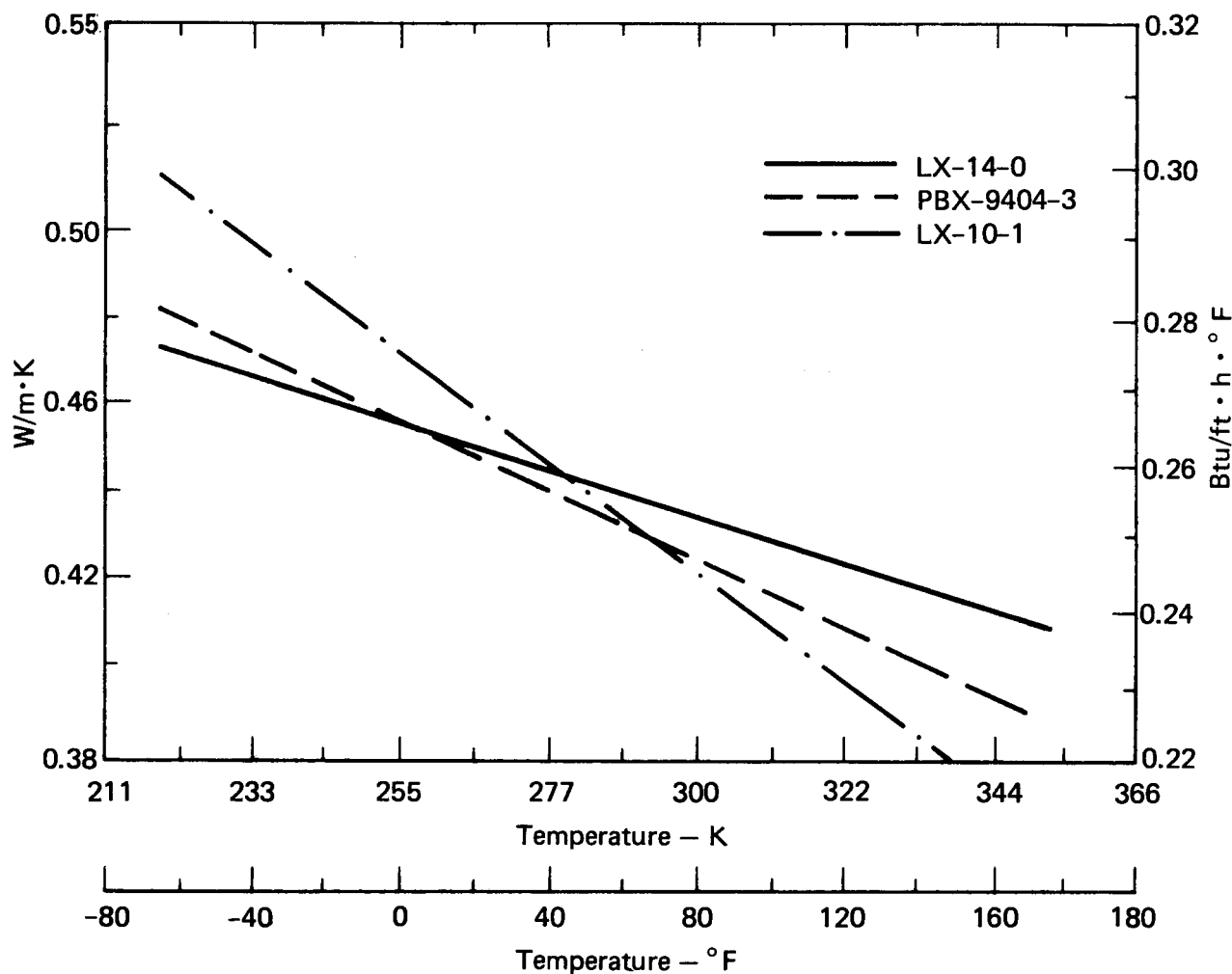


Fig. 24. Coefficient of thermal conductivity of LX-14-0, LX-10-1, and PBX-9404-3.

## COMPATIBILITY

To be compatible in weapons environments, a PBX should not react with or be affected by other materials present in a way that will adversely affect the performance of the system. Since experience shows that changes in both HE and other materials do occur, testing is required to demonstrate that the changes are not deleterious to the system. We assess

the magnitude of changes that occur with two types of tests:

1. The chemical reactivity test<sup>23</sup> (CRT): in this test, intimate mixtures of the PBX and materials whose mutual compatibility is to be examined are heated at 120°C for 22 h in tubes filled with inert gas. The identities and quantities of gases evolved are signals of chemical reactions.

2. Long-term core sample compatibility tests: in this test (see Figs. 25 and 26), disks of explosive touch or are in close proximity to various materials. The test assemblies are heated in a closed 304 stainless steel container at constant temperature in an inert gas atmosphere for periods up to 2 years.<sup>26</sup> Constant test temperatures used are from ambient to 100°C.

Two configurations of the long-term test assemblies that test several materials simultaneously are illustrated in Figs. 25 and 26. The terms Silastic and Adiprene refer to the spacing rings that separate materials under test in the assembly. After

heating, we assess compatibility of the materials by analysis of the gases evolved, by using mass spectrometry, by changes in appearance, by physical and mechanical properties tests, by molecular weight changes of polymers, etc.

### Thermal Stability of Polymer /LX-14 Mixtures

Table 14 shows the excellent compatibility of intimate mixtures of LX-14-0 with various polymeric materials in the chemical reactivity tests at 120°C for 22 h. Only very small quantities of gases evolved from any sample during the test period.

Table 14. Chemical Reactivity Test (CRT) of LX-14-0 with various polymeric materials. Test Conditions: 22 h at 120°C in a tube filled with inert gas.

<u>Material</u>	<u>Gas evolved, cm<sup>3</sup></u>
LX-14 <sup>a</sup>	0.02
Cured Adiprene (L-100) <sup>a</sup>	0.02
Silastic (93-119) <sup>a</sup>	0.01
Y3602/B <sup>a</sup>	0.02
Modified Neoprene B <sup>a</sup>	0.02
LX-14/L-100 <sup>b</sup>	0.04
LX-14/93-119 <sup>b</sup>	0.01
LX-14/Y3602/B <sup>b</sup>	0.02
LX-14/Modified Neoprene B <sup>b</sup>	0.08

<sup>a</sup>Samples weighed 0.25 g.

<sup>b</sup>Samples were intimate mixture of 0.25 g HE + 0.25 g polymer.

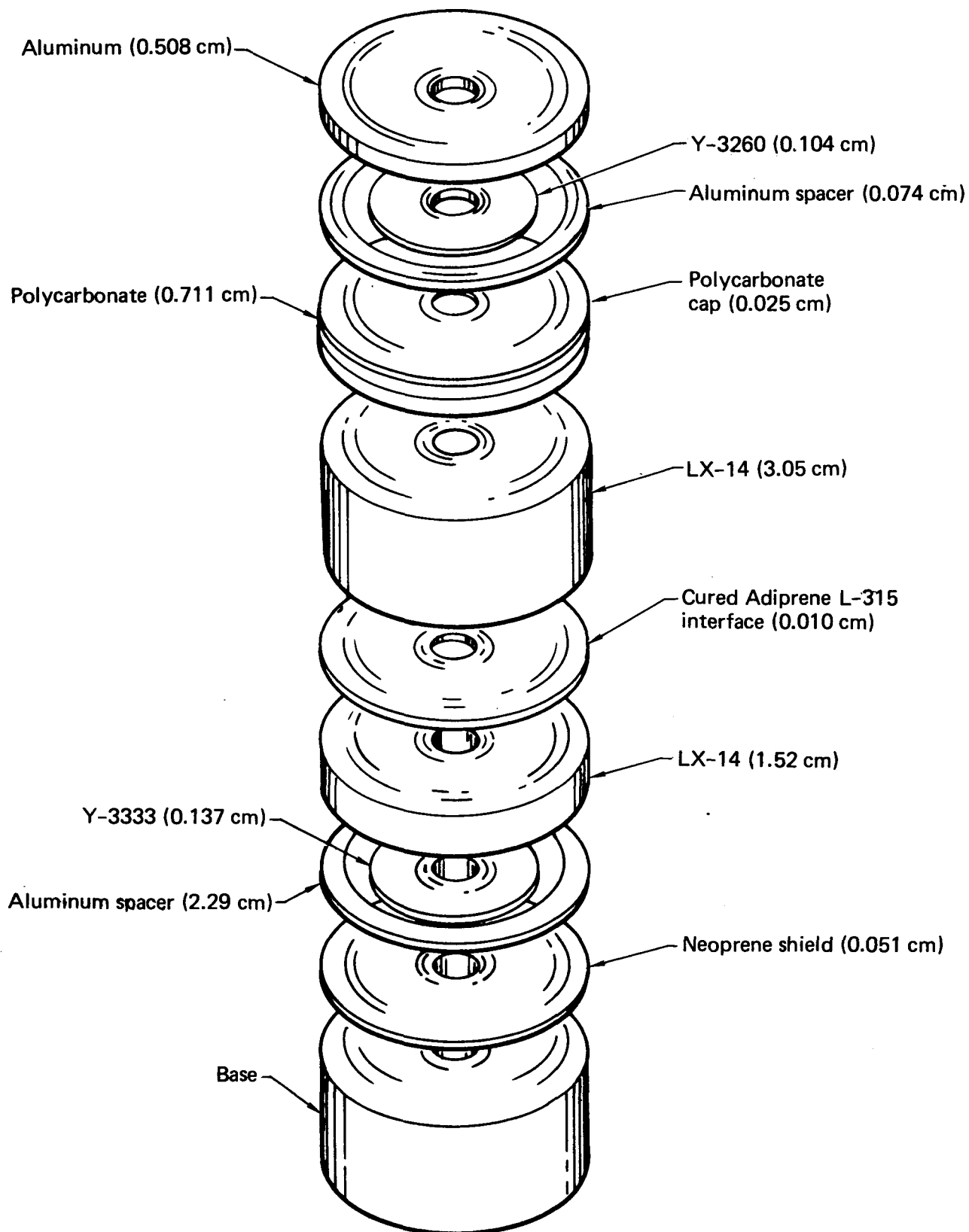


Fig. 25. Typical Adiprene L-315 compatibility core assembly.

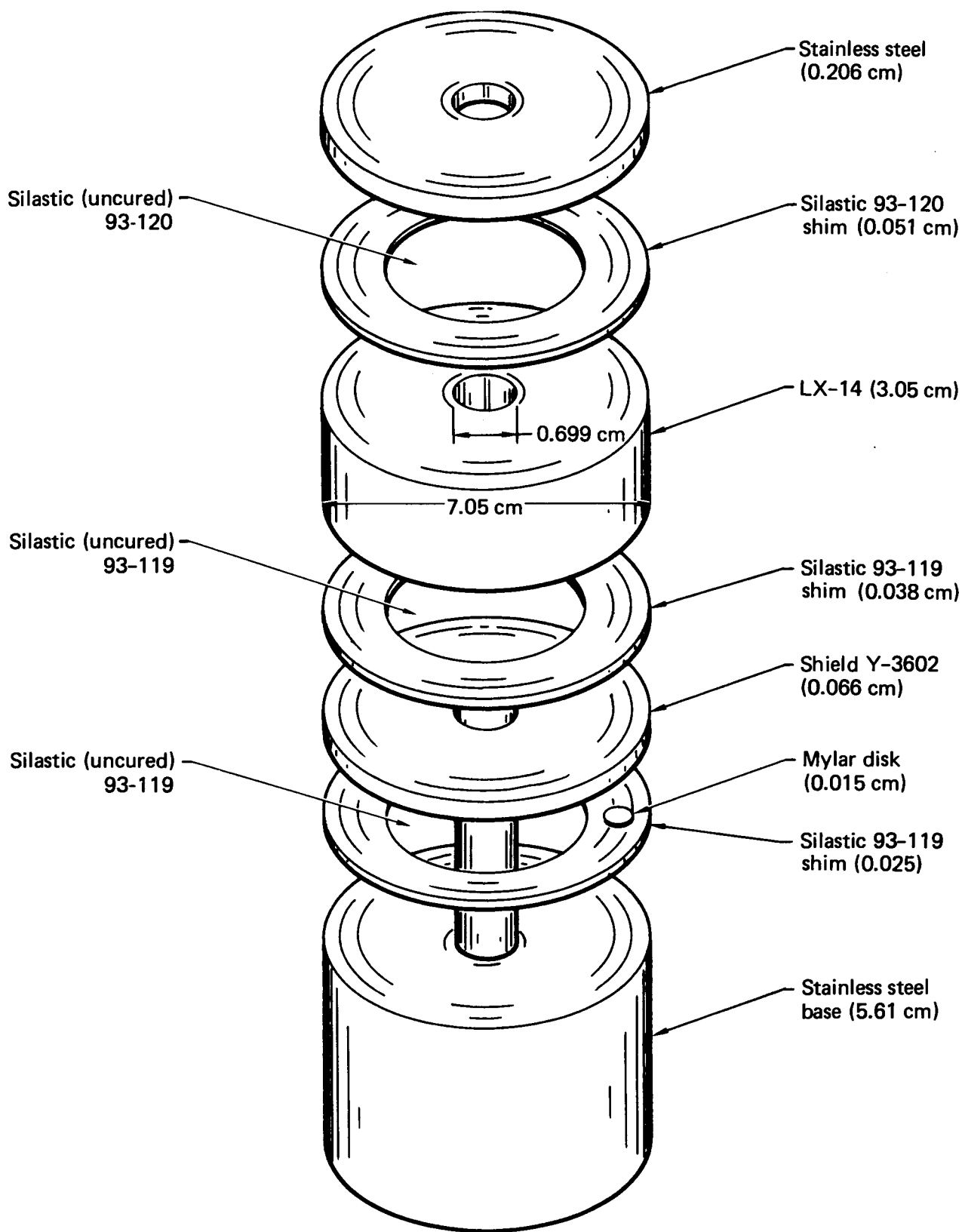


Fig. 26. Typical Silastic compatibility core assembly. Uncured Silastic is poured into the space indicated at the time of assembly. At disassembly the extent of cure is measured to see if there are cure inhibitors in the HE.

## Long-Term Compatibility Studies

We examined the compatibility of a number of materials in disk-to-disk contact or in close proximity with LX-14-0 in both types of assemblies shown in Figs. 25 and 26. These assemblies fit tightly in small flanged vessels so that there is minimum free volume and little gaseous communication around the circumference or the center extractor post. A valve in the cover permits evacuation, purging, and backfilling with an inert gas at the beginning of the test.

Test periods ranged from 1 to 24 months at temperatures of ambient (about 23°C), 50, 60, and 80°C. Separate test assemblies contained separate control specimens not in contact with other materials.

When a container under exposure was selected for examination, we pumped out and examined the gases inside, inspected the test disks carefully for visible changes, and measured the physical and mechanical performance of the polymers and the LX-14-0 specimens. In general, all the disks were in excellent condition after exposure, though the outer few millimeters of LX-14-0 tested at 80°C had an amber color.

### Gas Evolution

Figure 27 shows that only small amounts of gases ( $N_2O$ , NO, CO,  $CO_2$ ),

presumably from LX-14-0, evolved from any of the test assemblies at the three lower temperatures — ambient, 50, and 60°C. At 80°C all assemblies generated much more gas. These data agree with the finding of Matuzak and Taylor<sup>27</sup> that the rate of Estane decomposition increases between 60 and 80°C. Using data from LLL assemblies, Foster<sup>26</sup> plotted  $\ln k$  vs  $1/T$  and calculated an activation energy of decomposition for LX-14-0 of about 58.6 MJ/mol (14 000 calories/mol).

Even at 80°C the volumes of gases evolved during these compatibility tests are not excessive for weapons use, i.e.,  $<0.4 \text{ cm}^3/\text{g}$  of LX-14-0/year at 80°C for this particular assembly configuration. At 60°C, less than  $1.3 \times 10^{-2} \text{ cm}^3$  of gas/g of LX-14-0/

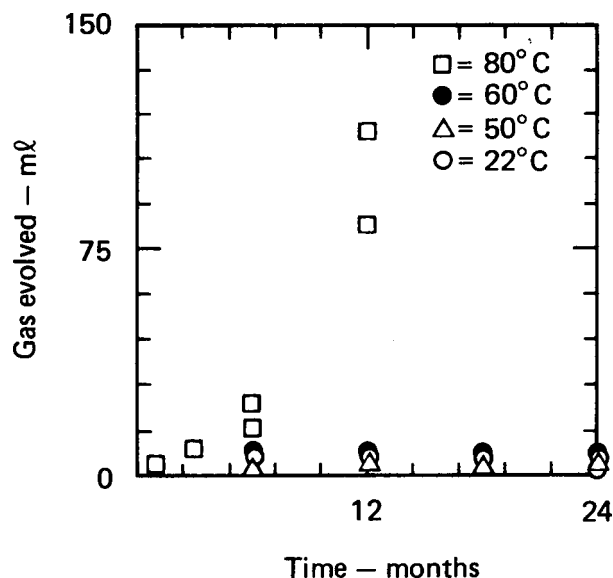


Fig. 27. Gas ( $N_2O$ ,  $CO_2$ , NO, and CO) evolved from core sample compatibility assemblies during aging.

year was generated; this quantity is considered insignificant. One cm<sup>3</sup> of evolved gas/g of HE represents about 0.2% decomposition.

### Tensile Properties of Aged LX-14-0

Because the HE specimens used for compatibility testing under inert gas in closed containers were too small to be examined by usual tension or compression tests, we used an indirect tension (Brazilian) test, evaluated by Pantex.<sup>28</sup> Brazilian tests showed that the excellent mechanical performance of LX-14-0 was not degraded by aging. This test is *not* recommended for deriving engineering data for design purposes, but is acceptable for obtaining comparative data for small specimens, since the mode of failure during testing is always the same.

The Brazilian (indirect tensile) test is described in detail in *Appendix B*. In this test, a disk is placed on edge and squeezed between two platens; the load-deformation characteristic is measured by an extensometer.

Table 15 compares the indirect tensile strength (stress and strain) of four PBXs that have not been aged. The data rank these HEs in the same order as do full-size uniaxial tensile tests.

Table 15. Indirect tensile data from plastic-bonded explosives.<sup>a,c</sup>

Material	Stress, MPa (psi)	Strain, %
LX-09-1	0.62 (90)	0.61
LX-04-1	0.93 (135)	0.91
LX-10-1	1.47 (214)	0.23
LX-14-0 <sup>b</sup>	2.10 (305)	0.44

<sup>a</sup>Indirect (Brazilian) tensile test (see *Appendix B*).

<sup>b</sup>Development lot of LX-14-0.

<sup>c</sup>All tests are of unaged samples at ambient temperature.

Table 16 contains indirect tensile test data measured on specimens machined from LX-14 disks that had been heated for various periods in long-term compatibility test containers. Though the indirect tensile strength of these specimens was unchanged after aging at ambient, strengths fell as exposure temperature and time increased. The largest strength loss under the highest test temperature was only 30%. Strength loss is expected from observations of the change in the molecular weight of the Estane binder during the same exposures (see next section). Percent strain at break also falls as exposure time and temperature are increased; the maximum decrease was 40%.



Table 16. Indirect tensile data on LX-14-0 specimens from compatibility tests<sup>a</sup> at 21°C.

Temperature, °C	Time, mo.	Stress, MPa (psi)	Strain, %
Ambient	0	1.72 (250)	0.51
	12	1.98 (287)	0.50
	18	1.78 (258)	0.52
	24	1.93 (280)	0.52
50	12	1.76 (255)	0.41
	18	1.72 (250)	0.41
	24	1.69 (245)	0.45
60	12	1.48 (215)	0.35
	18	1.58 (229)	0.38
	24	1.50 (218)	0.39
80	3	1.53 (222)	0.37
	6	1.21 (176)	0.30
	12	1.23 (178)	0.30

<sup>a</sup>Indirect (Brazilian) tensile test (see *Appendix B*).

Because of the greater decrease in both stress and strain after exposure at 80°C than at lower temperatures, one concludes that more degradation takes place at this temperature. During exposure at this temperature, indirect ultimate tensile strength and strain seem to decline for six months and then level off.

An important point is: *even after exposure for 2 years at 60°C*, an exposed LX-14-0 specimen still has as high an indirect tensile strength as unexposed LX-10, our most creep resistant and one of our strongest HMX-PBXs.

### Molecular Degradation of Estane Binder

For more than 15 years, ERDA laboratories have used Estane polyurethane elastomers as binders to reduce the shock and impact sensitivity of HMX compositions. LASL incorporated Estane 5740-X2 (an experimental polymer now called 5703-F2) at the 10% level in PBX-9011; weapons using that PBX have been in service for 14 years without problems arising.

Experimental PBX compositions have been made from several types of Estane polyurethanes. The molecular

chains of Estane types 5701, 5702, and 5703 are composed of polyester blocks connected by urethane linkages; but in Estane 5714, the polymeric blocks have a polyether structure rather than polyester. Both types fulfill the basic function of reducing the shock and impact sensitivity of HMX.

LASL found that although both types of Estane were stable when subjected to long-term aging in inert gas, they both suffered severe molecular degradation when heated at 75°C for 16 weeks in intimate contact with HMX or with HMX and nitroplasticizer, as in PBX-9501. However, at 55°C, degradation was relatively slow in LASL tests.<sup>29</sup>

The mechanism of degradation under inert gas is not completely understood. Because CO<sub>2</sub> is evolved in appreciable quantities, Wewerka et al.<sup>29</sup> theorized that nitrogen oxides evolved during long heating of HMX or the decomposition products of the nitro-plasticizer attack the urethane groups of the binder, breaking its molecular chains into short fragments of low strength. According to LASL data, after exposures at 74°C, these fragments had molecular weights about the same as those of the short polyester or polyether blocks in the original Estane molecules.

## LLL-Pantex Exposures of LX-14-0 Under Inert Gas

As shown in Fig. 28 and in agreement with Wewerka's results, we found that Estane 5702-F1 binder in LX-14-0 suffered molecular degradation in LLL core compatibility assemblies under inert gas during long-term exposures of disks at all temperatures above ambient. In Fig. 28, the average molecular weight of the Estane extracted from the exposed LX-14-0 with 1,2-dichloroethane is plotted as a function of exposure time. Since the degradation is a function of both time and temperature, Foster<sup>26</sup> assumed this relationship:

$$M_x = M_f + (M_o - M_f)e^{-RT},$$

where

$M_x$  = molecular weight at time X

$M_o$  = original molecular weight

$M_f$  = final molecular weight

R = rate constant

T = temperature.

$M_f$  is the final molecular weight measured on samples that were exposed for 2 years.  $M_f$  differs for each temperature, as does the rate constant, R, and was obtained by performing a least-squares curve fit, which assumed a zero rate for the ambient data.

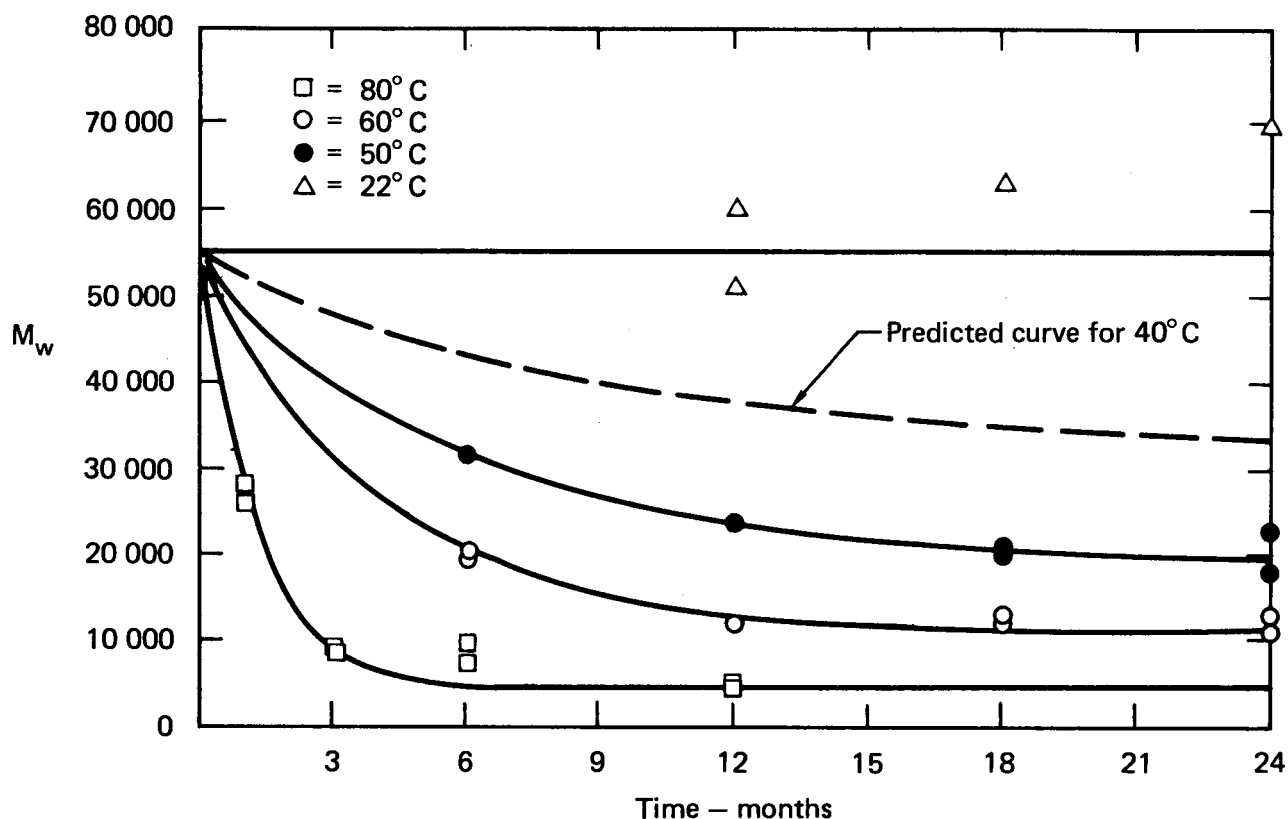


Fig. 28. Weight-average molecular weight of soluble Estane recovered from exposed LX-14-0 compatibility core assemblies.

The fact that Estane 5702-F1 binder degrades when in intimate contact with HMX at 40 to 80°C in compatibility assemblies is disturbing. Estanes themselves (no HMX) are stable under these exposure conditions.<sup>29</sup> At ambient temperature (about 23°C) Estanes in LX-14-0 changed in molecular weight very little, if any, over 2 years time. There may even be some crosslinking by reaction at exposed amine sites with the formaldehyde that evolves from decomposing HMX; this would explain the slight increase in

molecular weight (Fig. 28) noted after exposure at ambient temperature.

At progressively higher temperatures, degradation mechanisms seem to prevail, though crosslinking may also occur. At 80°C, the highest exposure temperature, the molecular fragments isolated after 6 months exposure had molecular weights of 6000. This can be compared to a molecular weight of 56,000 for the Estane and 2000 for the Estane prepolymer. These data agree with those of Wewerka and strongly suggest that in the 80°C test,

practically all of the urethane linkages in the original Estane were destroyed by attack of nitrogen oxides.

### LASL Exposures of LX-14-0 Sealed in Air

LASL aged LX-14-0 specimens (4.1 cm diameter by 4.1 cm long) sealed under air at ambient, 60, and 74°C for 2 years.<sup>30</sup> After aging, they determined the HMX/Estane ratio of the samples and analyzed Estane-soluble portions of the sample by gel permeation chromatography. In Table 17, Estane-soluble samples are labeled

inner and outer depending on whether they were removed from the surface or interior of the specimens.

The peak molecular weight of the Estane recovered from the interior of LASL aged LX-14-0 was essentially unchanged after 2 years at ambient conditions. However, some degradation may have occurred on the surface of these specimens, in view of the slightly lower molecular weight of the recovered Estane.

But during 2 years at 60°C in air, degradation took place, even in the interior of the LASL specimens.

Table 17. LASL molecular weights of aged LX-14-0 specimens sealed in air.

<u>Sample P-36040</u>	<u>% Estane</u>	<u>Time, yr</u>	<u>Temp., °C</u>	<u>Estane mol. wt.<sup>a</sup></u>
-01 Inner	4.45	0	Ambient	63,000
-01 Outer	4.45	0	Ambient	63,000
-02 Inner	4.35	0	Ambient	63,000
-02 Outer	4.45	0	Ambient	63,000
-03 Inner	4.45	0	Ambient	63,000
-03 Outer	4.42	0	Ambient	63,000
-24 Inner	4.36	2	Ambient	63,000
-24 Outer	4.35	2	Ambient	56,000
-27 Inner	4.30	2	60	52,000
-27 Inner	4.25	2	60	43,000
-30 Inner	4.07	2	74	41,000
-30 Outer	3.74	2	74	35,000

<sup>a</sup> LLL and LASL obtain polystyrene-equivalent molecular weights from GPC treatment using a Q factor of 41.4 instead of 20 that Pantex uses. We have adjusted LLL and LASL data by dividing by 41/20 to obtain Estane weight-average molecular weights.

Aging at 74°C for 2 years caused more deterioration, though the final molecular weight of these LASL specimens aged and sealed in air were much higher than those of LLL specimens aged in closed vessels under inert gas. Exposed LASL samples all had higher molecular weights inside than outside, presumably reflecting moisture or possibly oxygen attack on the surface and slow diffusion inward.

Table 18 contains compression strength data measured on the exposed LASL specimens. For samples sealed in air for 2 years, compressive strength decreased 14 and 22% for samples aged at 60 and 74°C, respectively, but both modulus and proportional limit increased.

We conclude that none of the changes that LASL found in their aged specimens sealed in air indicate gross changes in this PBX, nor would the changes affect the performance of LX-14-0 in weapons environments.

In general, the data indicate that LX-14-0 has satisfactory thermal stability and long-term compatibility for weapons uses. Only small quantities of gases evolve when this PBX is exposed in contact with various substrates so long as the continuous exposure temperature does not exceed about 60°C. Although LX-14-0 that is exposed continuously at 80°C evolves 10 to 15 times as much gas as at lower temperatures, this condition is more severe than any expected weapons environments.

Molecular weight data indicate that Estane is not degraded during the process of preparing the PBX billets from HMX and Estane.

The importance of the degradation of Estane binder molecules during long term exposure is not known. LX-14-0 does not undergo a catastrophic loss in tensile or compressive strength during exposure as long as 2 years. During this time the Estane

Table 18. Compression values of LX-14-0 specimens aged 2 years (sealed in air)<sup>a,b</sup>

<u>Time, yr</u>	<u>Temp, °C</u>	<u>Prop. limit, MPa (psi)</u>	<u>Comp. strength, MPa (psi)</u>	<u>Modulus, GPa (ksi)</u>
2	Ambient	3.6 (521)	13.7 (1992)	1.5 (219)
2	60	3.9 (565)	11.9 (1721)	1.6 (237)
2	74	4.5 (651)	10.7 (1560)	2.1 (298)

<sup>a</sup>LASL data.

<sup>b</sup>Constant crosshead speed of 30.5 mm/s (0.020 in./min) at 21°C.

can be slightly affected or essentially completely degraded, depending on exposure conditions. Even after exposure in the LLL-Pantex compatibility core test under inert gas for 2 years, indirect tensile tests indicate that LX-14-0 is still as strong as unexposed LX-10, previously our most creep resistant and one of our strongest PBXs. Also, these disks of exposed LX-14-0 were strong enough to easily machine to make specimens for indirect tensile testing.

The presence or absence of air during exposure seems to affect degradation rates, though the mechanism is not understood. LLL exposures were done in inert gas, whereas LASL exposed specimens sealed under air in glass tubes. After 6 months at 80°C at LLL, the Estane was reduced almost completely to low molecular weight fragments ( $M_w \sim 6000$ ); but exposure at LASL at 74°C for 2 years reduced the average molecular weight of

Estane by only a third to a half (from 63000 down to between 35000 and 43000).

Degradation under the two types of conditions at lower temperatures followed the same pattern. At 50 and 60°C in LLL-Pantex Test, degradation was slower than at 80°C, though still moderately severe, whereas in specimens exposed at LASL, molecular weights fell only 15 to 28% during 2 years. At ambient temperatures no degradation occurred during 2 years in LLL tests; in fact, molecular weights rose slightly, suggesting crosslinking. But under LASL conditions, molecular weight dropped 11% at the outer and was unchanged at the inner surface in 2 years.

Also, LASL is currently disassembling a weapon containing PBX 9011 (90 wt% HMX, 10 wt% Estane). Information from their observations may help to resolve questions about the effects of Estane degradation on LX-14-0.

## ENERGY AND EQUATION OF STATE

To be an improved and useful explosive, a new PBX must provide a very high energy density when detonated. Experimentally, we measure an HE's energy density in a cylinder test, and use the test data to derive an equation of state that describes the

pressure-volume-energy behavior of the explosive products.

### Cylinder Test

In the cylinder test, a billet of LX-14-0 (25.4 mm diam. and 0.31 m

long) was packed tightly in a copper tube 2.54 mm thick. The explosive was initiated at one end and the radial motion of the cylinder wall measured using a streaking camera.

In a typical experiment, there are six shorting pins equally spaced around a circular holder near the top of the cylinder (see Fig. 29). Six more pins, paired with those at the top, are located 203 mm from the top of the tube. This spacing allows the shock wave to stabilize for three-cylinder diameters before it contacts

the first pins. From the transit time measured by the six pairs of pins, we can calculate the average detonation velocity with a high confidence level.

A detonation velocity of 8.33 km/s was measured for the following LX-14-0 formulation:<sup>31</sup>

HMX, wt%	95.5
Estane, wt%	4.5
Density, Mg/m <sup>3</sup>	1.835
TMD, %	99.2
HMX, vol%	92.2
Estane, vol%	7.0
Air, vol%	0.8
(by difference)	

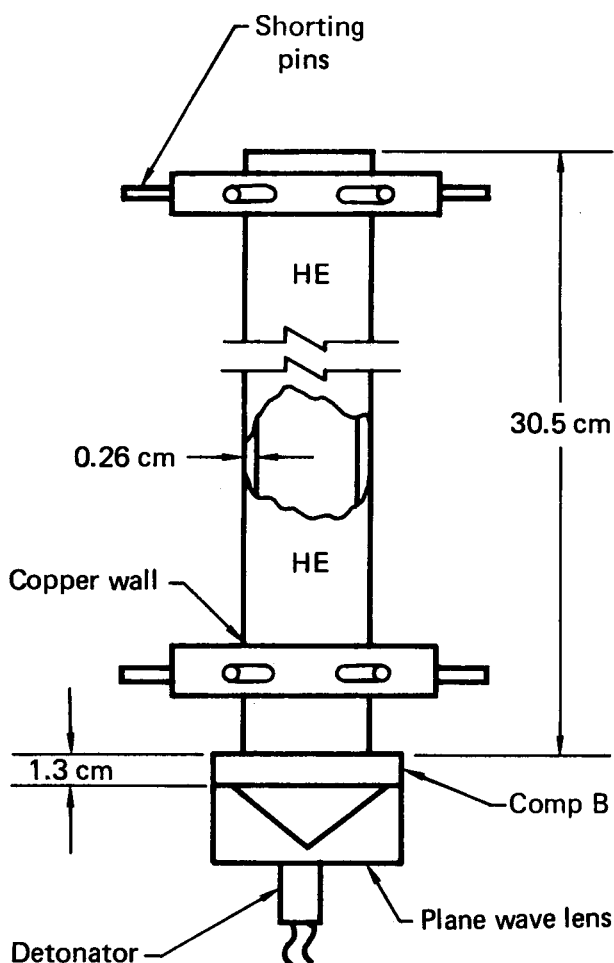


Fig. 29. Schematic of cylinder-test hardware.

The kinetic energy that an explosion imparts to a copper wall in a fixed geometry leads to a simple way of expressing the performance of the explosive. Two extreme geometric arrangements have been considered for the transfer of explosive energy to adjacent metal in the range of mass ratio of explosive to metal encountered in this cylinder test: 1) detonation that is normal to the metal, and 2) detonation that is tangential to the metal.

The effective explosive energy frequently differs in the two directions, even on a relative basis, because of the effects of the equations of state of the detonation products. The cylinder test provides a measure of the relative effective explosive

energy for both head-on and tangential detonation. The radial wall velocity at 5-6 mm wall displacement, expressed as volume-ratio  $V/V_0 = 2$ , is indicative of explosive energy in "normal" geometry. The wall velocity at 19 mm displacement  $V/V_0 = 7$  is a direct measure of performance in tangential geometry.

Figure 30 shows the cylinder test results for LX-14-0 plotted relative to PBX-9404. The specific wall kinetic energy at 6 mm wall displacement was 98.5% of the energy of PBX-9404; at 19 mm wall displacement, it was 98.7% of the energy of exploding PBX-9404.

### J-W-L Equation of State

The Jones-Wilkins-Lee (JWL) equation of state accurately describes the pressure-volume-energy behavior of the detonation products of explosives in applications of metal acceleration.<sup>32,33</sup>

The form of the equation is

$$P = A \left( 1 - \frac{\omega}{R_1 V} \right) e^{-R_1 V} + B \left( 1 - \frac{\omega}{R_2 V} \right) e^{-R_2 V} + \frac{\omega E}{V},$$

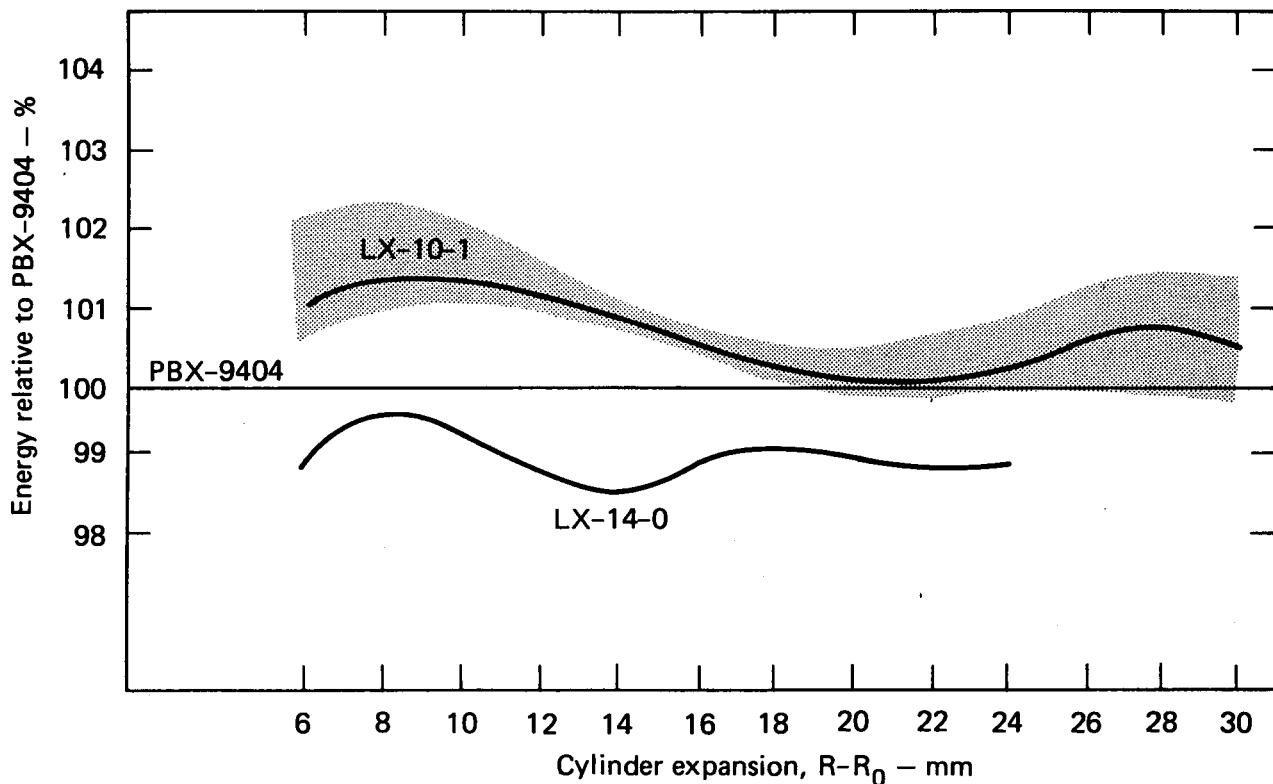


Fig. 30. Specific wall kinetic energies of LX-10-1 and LX-14-0 relative to PBX-9404 (from cylinder test data).



where A, B,  $R_1$ ,  $R_2$ , and  $\omega$  are constants, E is the energy in the explosive, and V is the relative volume  $V/V_0$ , the ratio of the volume of gaseous detonation products to the volume of the unreacted explosive. These terms are called the Jones-Wilkins-Lee coefficients. The equation for an adiabat (isentropes) is given by

$$P(\Delta) = Ae^{-R_1 V} + Be^{-R_2 V} + \frac{C}{V^{\omega+1}},$$

where C is a constant that defines the locus of the isentropes.

The LX-14-0 coefficients in Table 19 were calculated by matching the J-W-L equation with experimental Chapman-Jouget conditions, calorimetric data, and expansion behavior.

Table 19. Calculated J-W-L coefficients and C-J parameters of LX-14-0.

<u>J-W-L coefficients</u>		<u>C-J parameters</u>
A	8.261	Density, $\rho_0 = 1.835 \text{ Mg/m}^3$
B	0.1724	$P_{CJ} = 37.0 \text{ GPa (0.370 Mbar)}$
C	0.01296	$D = 8.83 \text{ km/s (8.83 mm/}\mu\text{s)}$
$R_1$	4.55	$E_0 = 10.2 \text{ GPa cm}^3/\text{cm}^3 \text{ (0.102 Mbar/cm}^3/\text{cm}^3)$
$R_2$	1.32	$\Gamma^a = 284.1 \text{ GPa cm}^3/\text{cm}^3 \text{ (2.841 Mbar cm}^3/\text{cm}^3)$
$\omega$	0.38	

<sup>a</sup> $\Gamma$  = polytropic gas parameter,  $\left( \frac{d \ln P}{d \ln V} \right)_S$

## APPENDIX A: SLURRY TECHNIQUE FOR PLANT PRODUCTION OF 240-KG BATCHES OF LX-14-0 MOLDING POWDER BEADS

1. Prepare a lacquer by dissolving 11.2 kg of Estane 5702-F1 in 125 kg of water-saturated methylethylketone. Agitate for 1 h at ambient temperature to complete dissolution.
2. Preload the 1893-litre (500 gal) vacuum still with 473 kg of water and 73 kg of water-saturated methylethylketone. Then add HMX of three different particle sizes, using 155.0 kg Class A grade, 23.8 kg Class B grade, and 59.6 kg LX-04 grade.
3. Heat the HMX slurry to 65°C while agitating it rapidly.
4. Add the premixed lacquer and agitate for 5 minutes.
5. Distill off the solvent to a head temperature of 100°C, continuing fast agitation, and hold at this temperature for 5 minutes.
6. Cool the beads to 50°C, drop them on a filtering nutsche, and wash them with fresh water at ambient temperature.
7. Distribute the molding powder beads 1-in. deep on aluminum trays and dry them in a forced-air oven at 90°C. The LX-14-0 can be dried to 0.02% moisture content in 2 hours.
8. Store the dried beads in fiberboard drums.

The process for making pilot plant batches at the Pantex plant differed from the Holston production process as follows<sup>34,35</sup>:

1. The solvent used to dissolve the Estane was 1,2-dichloroethane rather than MEK.
2. Deionized water was the carrier rather than plant water.
3. Toluene was added as an antifoaming agent and to aid granule formulation.
4. The solvent was removed by vacuum distillation.
5. The last traces of solvent were removed after dewatering the molding powder beads.
6. Pilot plant batches ranged in size from 2.0 to 250 kg.
7. Holston added Heliogen Violet Toner to some of its 250 kg batches.

Processing details can be changed to control certain factors. For example, out-of-specification production batches of molding powder beads (wrong composition, beads too small, etc.) can be reworked to produce acceptable material. Bead size can be reduced by using less solvent, or the bulk

density of the molding powder beads can be increased by using more solvent during the bead-forming process.

The specification for LX-14-0 is  $95.5 \pm 0.6\%$  HMX for any 250-kg batch, ( $05.5 \pm 0.5\%$  HMX for a 4500-kg lot). The production lot acceptance statistics in Table A1 show that this composition specification was easily met.<sup>34</sup> The batches with the lowest and highest concentrations of HMX had 95.47 and 95.63 wt% HMX, respectively; the HMX composition of 16 of the batches were within  $\pm 0.06\%$  of the 95.5% nominal. Close compositional tolerance is typical of two component systems that contain small quantities of binder, such as LX-14-0. These data are important, because they assure us that it is unlikely any 4500-kg lot of LX-14-0 would be sufficiently out of specification to significantly alter sensitivity, safety, or performance properties.

Heliogen Violet Toner (0.01 wt%) was added to 4 of the 19 batches to color code blended LX-14-0 violet with a white background. Color coding was uniform throughout the appropriate batches and proceeded without difficulty.

During the development of LX-14-0, we made approximately 2500 kg of various pilot plant HMX/Estane formulations in batch sizes from 2 to 250 kg. This pilot plant experience is important because it allows formulation and plant handling history to accumulate before production begins. Most of the 250-kg size pilot plant batches were used for skid testing.

Table A1. Production batch acceptance report for 4500 kg (10 000 lb) Holston Lot of LX-14-0.

Allowed by specifications		HMX, wt%	Volatiles, wt%	Color	Bulk density, Mg/m <sup>3</sup>	Foreign matter	Granulation - Cumulative % retained on USSS series			
							5/16	4	50	80
Batch <sup>a</sup>	Min	94.9	—		—	None	—	—	—	—
	Max	96.1	0.10							
Lot <sup>b</sup>	Min	95.0	—		0.85	None	—	—	95	98
	Max	96.0	0.10		—		0	1	—	—
<u>Individual batch properties</u>										
L-1		95.52	0.02	White	0.98	None	0	0	100	100
L-2		95.47	0.04	White	1.01	None	0	0	100	100
L-3		95.63	0.03	Violet	0.96	None	0	0.1	100	100
L-4		95.55	0.03	Violet	1.01	None	0	0	99.9	100
L-5		95.53	0.02	Violet	1.01	None	0	0	99.8	99.9
L-6		95.49	0.02	Violet	1.01	None	0	0	99.9	100
L-7		95.52	0.02	White	0.99	None	0	0	100	100
L-8		95.51	0.02	White	0.98	None	0	0	100	100
L-9		95.56	0.01	White	1.00	None	0	0	100	100
L-10		95.50	0.03	White	1.04	None	0	0	99.8	99.8
L-11		95.52	0.02	White	0.96	None	0	0	99.9	100
L-12		95.56	0.01	White	0.97	None	0	0	99.8	99.9
L-13		95.53	0.03	White	0.99	None	0	0	99.8	99.9
L-14		95.51	0.03	White	0.90	None	0	0	99.8	99.9
L-15		95.59	0.06	White	1.04	None	0	0	99.8	99.9
L-16		95.61	0.09	White	0.95	None	0	0	99.8	99.9
L-17		95.54	0.03	White	0.95	None	0	0	99.9	100
L-18		95.56	0.04	White	0.96	None	0	0	99.8	99.9
L-19		95.56	0.01	White	1.00	None	0	0	99.9	100

<sup>a</sup>Batch weight is 250 kg (550 lb).

<sup>b</sup>Lot weight is about 4500 kg (10 000 lb).

## APPENDIX B: THE "BRAZILIAN" INDIRECT TENSILE TEST<sup>36</sup>

Pantex has evaluated the feasibility of this test<sup>28</sup> for measuring the mechanical properties of HE compatibility coupons too small for normal tensile testing. They concluded that data from this test are acceptable for comparing changes in mechanical properties of HEs, but they recommend that the test not be used to develop basic data used for design engineering.

In this test, a disk 20 mm diameter by 8 mm thick is compressed on its edge between two platens. Stress analysis models show that lines of stress develop as equilateral hyperbolas that are symmetrical to the center of the disk and are aligned with the compressive force (platens). As the disk is loaded, it elongates parallel to the compressive force creating a state of biaxial stress across the face of the disk.

A special extensometer measures the horizontal movement of the deflecting disk as force is applied to the platens. A valid stress-strain curve may be plotted for linear elastic materials, provided that the strain is in tension at the center of the disk in the horizontal direction and is divided by two.<sup>37</sup> However, it may be more difficult to relate uniaxial and biaxial data for viscoelastic materials (PBXs) because of the time-dependent nature of HEs. Myers and Johnson<sup>28</sup> calculated the stress for the indirect tension specimens according to the equation derived by Frydman as follows:

$$S = 2f/\pi dt,$$

when

S = stress (Pa),

f = applied force (N),

d = diameter (mm),

t = thickness (mm),

using the dimensions of the test specimen, the equation simplified to:

$$S = f(3.97887).$$

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