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PARTITIONING OF RESIDUAL D-LIMONENE CLEANER VAPOR AMONG ORGANIC MATERIALS IN WEAPONS

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D-limonene is a replacement solvent selected by Sandia and Allied-Signal to clean solder flux from electronics assemblies in firesets and programmers. D-limonene is much slower drying than the solvents it has replaced and this has raised concerns that residual quantities of the cleaner could be trapped in the electronics assemblies and eventually carried into warhead assemblies. This paper describes a study designed to evaluate how vapors from residual d-limonene cleaner would be partitioned among typical organic materials in a Livermore device. The goal was to identify possible compatibility problems arising from the use of d-limonene and, in particular, any interactions it may have with energetic materials. To predict the partitioning behavior of d-limonene, a simple model was developed and its predictions are compared to the experimental findings.

Experimental

The partitioning experiment was performed in the High Explosives Application Facility (HEAF) at LLNL. Four identical containers of materials were exposed to different concentrations of d-limonene vapor at 0, 2, 4 & 8 grams per 15L (the approximate free volume in the device). Table I lists the materials tested. Included were three energetic materials: LX-13, LX-16, and pressed ultrafine TATB. LX-17 was not tested due to explosive quantity limits imposed by the facility, so an appropriate quantity of its binder, the copolymer Kel-F 800, was used in its place. This was not considered a significant compromise because TATB, the other and primary ingredient in LX-17, has been determined to be completely insoluble in d-limonene.[1]

Table I. Materials and Test Specimen Quantities.

Material	Mass (g)	Material	Mass (g)
UF-TATB	8.26	EPDM O-Ring	1.20
LX-13	0.33	Halthane 88-3	1.33
LX-16	0.023	MVP-11 Acrylic	0.27
Kel-F 800	80	Polycarbonate	0.67
Molded Desiccant	100	Polyolefin HS	1.51
Pyralux (as Det	0.86	Polysulfide,	1.33
PUR Lock Foam	25.7	PUR, EN-7 & EN-8	0.64
Silicone Stress	7.73	PUR Sleeving	0.42
Silastic Adhesive	1.87	HVLI Gasket	0.13
Adiprene L-100 +	5.47	Sylgard 184	1.13
EMR Silicone	4.39	Y-9469 Acrylic	0.50

Test specimens were dried over desiccant for at least two weeks before testing. Each specimen was weighed immediately upon removal from the desiccator, then quickly assembled into sealed test containers in a 10% R.H. dry room. Care was taken to arrange the specimens to facilitate transport of d-limonene, which was introduced at the top of the container to minimize complications due to

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vapor stratification. The containers were subjected to a cyclic time-at-temperature sequence to accelerate equilibrium dissolution and partitioning of d-limonene vapor. The total time at the maximum test temperature (~16 hours at 68°C) was estimated by Sandia to be equivalent to 1 stockpile life for diffusion of d-limonene vapor out of the fireset.[2] The test was concluded by opening the containers under dry nitrogen and quickly bottling the specimens for final weighing.

Results & Discussion

Figure 1 shows the measured weight changes in the test materials. These values are "adjusted" using the weight changes from the control container to account for non-d-limonene induced weight changes, for example, those due to movement of water vapor and other volatiles among the test materials. As expected, some materials exhibited a greater affinity for d-limonene vapor than others. The greatest affinity for d-limonene was exhibited by four classes of materials: *1) elastomeric polyurethanes* -- Halthane 88-3, EN-7/8, Adiprene, and sleeving material, *2) elastomeric silicones* -- stress cushions, Sylgard 184 silicone potting compound, and APC-300 silastic adhesive, *3) hydrocarbon polymers* -- EPDM O-Rings and polyolefin heat-shrink sleeving, and *4) acrylic* transfer tape. In general, the weight changes measured for the energetic materials were very small («1 %). There were no surprises in these results.

We defined partitioning as the fraction of the total mass of d-limonene available that was measured in each material. It is important to note that high affinity for d-limonene does not alone determine the partitioning of d-limonene vapors -- the mass of each material also must be taken into account. Figure 2 shows that three-fourths of the available d-limonene vapor was partitioned among only five materials: silicone stress cushions (~26% of the available d-limonene vapor), Adiprene polyurethane (~19%), Halthane 88-3 polyurethane (~13%), APC-300 silastic silicone (8%), EPDM O-rings (~8%). Very little d-limonene appeared to be incorporated in the energetic materials. Figure 2 also shows that the partitioning results were relatively independent of vapor concentration over the range tested.

As noted above, it was expected that the measured weight changes would be complicated by the elimination (or pickup) of water vapor and other volatiles. Unfortunately these changes could not be totally accounted for by adjusting the measurements with the control as described above. In fact, we

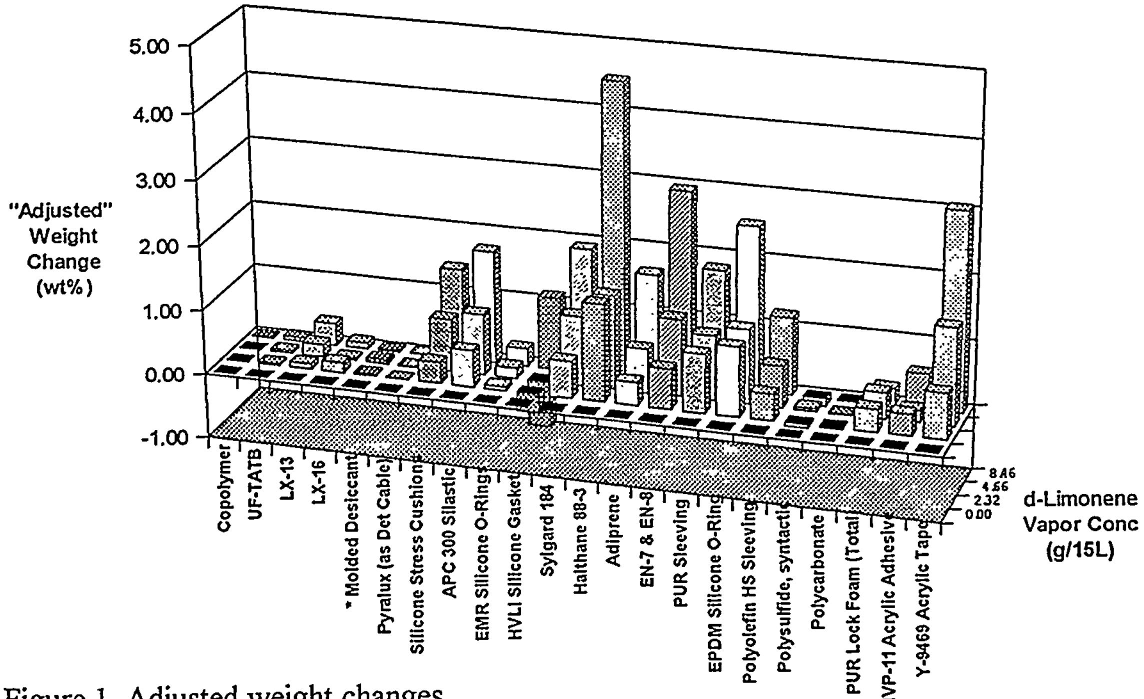

Figure 1. Adjusted weight changes.

found that each container experienced a net weight *gain;* (i.e., a positive apparent d-limonene mass balance). Subsequent experimentation revealed that two of the test materials were to blame, the molded desiccant and the PUR lock foam. After their initial weights were recorded and while the test container was being assembled, these materials picked up additional and unaccounted for water vapor from the 10% R.H. atmosphere. The partitioning analysis could be made consistent only by ignoring the apparent weight gains in both of these highly hygroscopic materials.

Also shown in Figure 2 are the predictions of a simple partitioning model that is defined below. The model assumes: 1) d-limonene vapor is partitioned among test materials in proportion to each material's affinity and capacity for saturated d-limonene vapor, and 2) each material's capacity for d-limonene is unaffected by presence of other materials.

$$\text{Partitioning[\%]} = \frac{\text{spl capacity}}{\text{system capacity}} = \frac{m \times S_{eq}^{sat'd}}{\sum\left(m \times S_{eq}^{sat'd}\right)_i} \times 100\%$$

Where: m = sample mass [grams]

$S_{eq}^{sat'd}$ = equilibrium solubility of saturated d-limonene vapor [wt%] in material

Figure 2 shows that this model does a reasonable job of predicting the distribution of d-limonene among most of the test materials; however, it overestimates the fraction of d-limonene absorbed by the energetic materials.

References

1. Memo from G. Moody (LLNL), April 14, 1992.
2. Provided by C. Pretzel (SNL/CA).

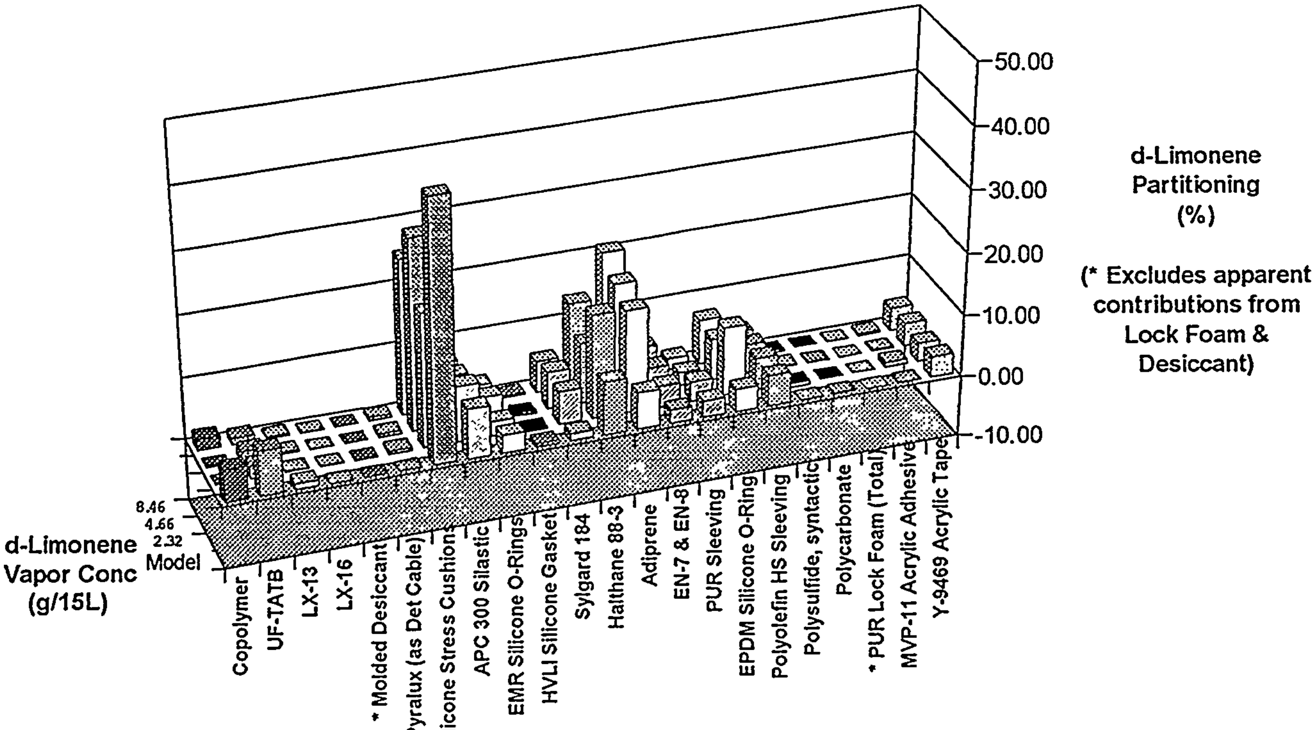

Figure 2. Partitioning of d-limonene vapor.