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ISOTHERMAL AGING EFFECTS ON EXTEX-TETRYL MIXTURES

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ABSTRACT

* An isothermal aging study of a 50/50 weight mixture of Extex (80% PETN, 20% Sylgard) and Tetryl (2,4,6-trinitrophenylmethylnitramine) was undertaken to determine its stability at elevated storage temperatures (60 and 100 C) for extended periods (100 days), to simulate long storage. The original components and mixture, along with the isothermal residues underwent various analyses including infrared spectral and differential thermal analyses, weight loss monitoring at selected intervals up to 100 days, and photomicrography.

Changes at 60 C were limited and very small (weight loss which occurred at the onset of test), some DTA changes in the endotherm, and some color change. At 100 C the last two changes were also seen, and weight loss appears to be significant.

DISCUSSION

The Extex used was ground under liquid nitrogen to mixable particle size. A mixture of the Extex and Tetryl was weighed in tubular aluminum cylinders to a density of 1.32 g/cc. One system was closed by use of an aluminum plug set in place with a pellet press, while the other was run as an open system through removal of the closure plug after density attainment. The resultant sample systems were placed in analytical ovens maintained at 60 and 100 C. Systems were run in quadruplicate. ~~General physical and molecular characteristics of the components and preaged mixture are shown in Figs. 1, 2 and 6.~~

Infrared spectral analysis of the system was through KBr pellet techniques (Perkin Elmer 21). Differential thermal analysis were obtained using a DuPont Model 900.

EXPERIMENTAL

The effects of isothermal aging on weight stability are shown in weight loss data presented in ~~Tables I and II~~ and Fig. 3. There appears to be a very small (< 0.1%) weight loss at 60 C over the time period studied, which occurs at the onset of aging, not in a linear or stepwise behavior. This loss is possibly due to moisture and/or occluded solvent present in the original mixture. The weight loss pattern is the same for both the open and the closed systems. ~~Fig. 4, Spectra A, B and C~~ ^{Fig. 4, Spectra A, B and C} do not indicate any changes occurring in the molecular functionality. ~~Differential thermal analysis of these residues~~ (Fig. 5) shows slight modification of the endothermal pattern at ~ 100-150 C, with no observed variations in the exothermal behavior (~ 205-210 C). ~~Photomicrographs of the system residues~~ ^{Photomicrographs of the system residues} gave no visual indication of significant change save for some slight color lightening of the open compact residue, ~~Fig. 6~~ ^{Fig. 6}.

^{approx. change in 100 C systems.}
^{60C}
^(being in mind the ~~data~~ ^{data} line 6, 2-176)

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Weight losses (~~Table II, Fig. 3~~) are found to be relatively significant at 100 C, undergoing an initial loss during the first 7-8 days ($\sim 0.3\%$) followed by a gradual leveling for a significant period ($< 0.5\%$ @ 50 days). Following this somewhat static behavior, the weight loss increased significantly at 100 days; 1.67% in the open system, 1.38% in the closed system. ~~Infrared spectral results (Fig. 4, Spectra A, D and E) do not indicate any significant molecular changes in the residues, bearing in mind the limits of detection of the infrared method ($\sim 2-4\%$). Differential thermal analyses of the 100 C residues (Fig. 7) show a similar endothermic pattern modification as found with the 60 C system ($\sim 105-150$ C), with no indication of exothermic pattern behavior change ($\sim 205-210$ C). Photomicrographs of these residues (Fig. 6) show a more appreciable color change from that of the original than found in the 60 C system.~~

SYSTEMS COMPARISON

Comparison of weight loss data from the 60 and 100 C systems indicate a real and a stepwise loss at 100 C with time, while losses seen in the 60 C systems are essentially immediate preceding a static condition, probably due to moisture and/or solvent and not a composite loss, (~~Tables I and II, Fig. 3, Fig. 4~~)

Infrared spectral data do not indicate any changes or anomalies occurring among the various systems, qualified by the limits of detection of the method (~~Fig. 4~~)

Differential thermal analysis of all systems show a similar modification of the endothermic pattern (~~relative to the preaged mixture~~) with no apparent change in the exothermic pattern (~~Fig. 8, Fig. 7~~)

Photomicrographs of the thermal residues ^{do} not show any variations in the physical shape of the mixtures but ^{do} show significant color changes relative to the original mixture, particularly those aged at 100 C (~~Fig. 6~~).

The results of this study do indicate some apparent changes occurring in the systems under these storage conditions. However, whether these are minimal ineffective occurrences or actually detrimental to employed systems would rest upon performance data appraisal of the aged residues.

Table I. Isothermal Weight Loss of Tetryl/Extex Systems.

Relative (%)

~~(60°C)~~ *Relative loss (%) at 60°C*

~~Elapsed Time (days)~~ *60 C*

<u>System</u>	<u>7 day</u>	<u>14</u>	<u>28</u>	<u>56</u>	<u>100</u> days
Open Compact	0.10	0.10	0.10	0.10	0.10
Closed Compact	0.04	0.04	0.02	0.03	0.04

~~Table II. Isothermal Weight Loss of Tetryl/Extex Systems.~~

~~(100°C)~~

~~Elapsed Time (days)~~ *100 C*

<u>System</u>	<u>7</u>	<u>14</u>	<u>28</u>	<u>56</u>	<u>100</u>
Open Compact	0.20	0.31	0.32	0.66	1.67
Closed Compact	0.31	0.25	0.26	0.37	1.38

don't need
2

Don't need
9

Fig. 1. Infrared Spectra of Unaged Tetryl/Extex System

- A. Tetryl
- B. Extex
- C. Tetryl/Extex Mixture (50/50)

Use -9

Fig. 2. DTA Thermograms of Original Components

WSE-8

Fig. 3. Isothermal Weight Loss of Extex-Tetryl Mixtures (1.32 g/cc)

Fig. 4. Infrared Spectra of Isothermal Tetryl/Extex Residues

don't need 2

Fig. 5. DTA Thermograms of Extex-Tetryl Isothermal Residues (100 days @ 60 C)

Tetryl

Extex

Tetryl/Extex (50/50)

Tetryl/Extex; 60 C, Open Compact

Tetryl/Extex; 100 C, Open Compact

Tetryl/Extex; 100 C, Closed Compact

Fig. 6. Photomicrographs of the Tetryl/Extex System

Fig. 7. DTA Thermograms of Extex-Tetryl Isothermal Residues (100 days @ 100 C)

Fig. 8. DTA Thermograms of Extex-Tetryl Isothermal Residues (100 days)