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TRANSITIONS IN THE LFT-10 GAS GENERATOR
(U)

by

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U. S. NAVAL ORDNANCE TEST STATION

AN ACTIVITY OF THE BUREAU OF NAVAL WEAPONS

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FOREWORD

The major purpose and direction of the study in this report was to ascertain the cause of the grain growth of the gas generator LFT-10 and the extent to which the binder participated in this phenomenon. Areas of investigation were entered only to the extent of their pertinence to the problem and often not dealt with as completely as the investigators may have desired.

The study was initiated on 7 July 1964 under Bureau of Naval Weapons Task Assignment RM 3731-007/216-1/W120-00-01 and research on the fundamental aspects was completed under Bureau of Naval Weapons Task Assignment RMSP 22-066/216-1/R001-06-01. The completion date was 30 September 1964.

This report was reviewed for technical accuracy by Ronald A. Henry and William R. McBride.

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TABLE 1. Ammonium Nitrate Transitions[a]

Phase reaction	Temp., °C	Vol. change,[b] ml/g
I ⇕ II	125.2	0.013
II ⇕ III	84.2	-0.098
III ⇕ IV	32.3	0.0221
IV ⇕ V	-18	-0.016, -0.018
II ⇕ IV	45 to 51	.....

[a] The data are taken from Ref. 1.
[b] Sign is attributed to lower arrow transition.

EXPERIMENTAL

All propellant samples used in our experiments were received in the form of 1/2-inch cubes that were trimmed to fit the dilatometer.

The sample of LFT-10 was manufactured by the Propellants Division of Amoco Chemical Corporation, Chicago, and its composition is given in Table 2. The ingredients are mixed at 90°C, poured into molds at 105-110°C, evacuated for 1 minute, and then pressed for 2 minutes at approximately 4,400 psi. The density as measured in n-hexane at 25°C was 1.514 g/ml (theoretical density 1.56 g/ml). Two determinations were made with this material.

To evaluate the effect of a eutectic composition upon the transitions of ammonium nitrate, two propellants were prepared in a common binder. The sample PL3204-R contained a mixture of ground ammonium nitrate and ground guanidine nitrate in a 70:30 ratio. The sample PL3205 contained a 70:30 melt, which is close to the eutectic composition (74:26; Ref. 4). The melting point of the fused mixture was 128°C and that of the eutectic is 127.7°C (Ref. 4). The melt was ground to 100-micron particle size. The binder for both samples was a Butarez Type II carboxyl terminated polybutadiene and was cured with HX-868 (Minnesota Mining & Manufacturing Co. designation for trimesoyl-trismethylaziridine) crosslinking agent. Both lots were made up in 300-gram batches, mixed in 1-pint Baker-Perkins vertical mixers, and cured for 4-5 days at 70°C. The densities as measured in n-hexane at 25°C for PL3205 and PL3204-R were 1.3554 g/ml and 1.3496 g/ml, respectively (theoretical density 1.44 g/ml). The propellants were cut into 1/4-inch-strips.

The ammonium nitrate was a sample of J. T. Baker Analyzed Reagent. It was dried for 2 days under vacuum at 70°C. The density was taken as 1.725 g/ml at 18°C (Ref. 1).

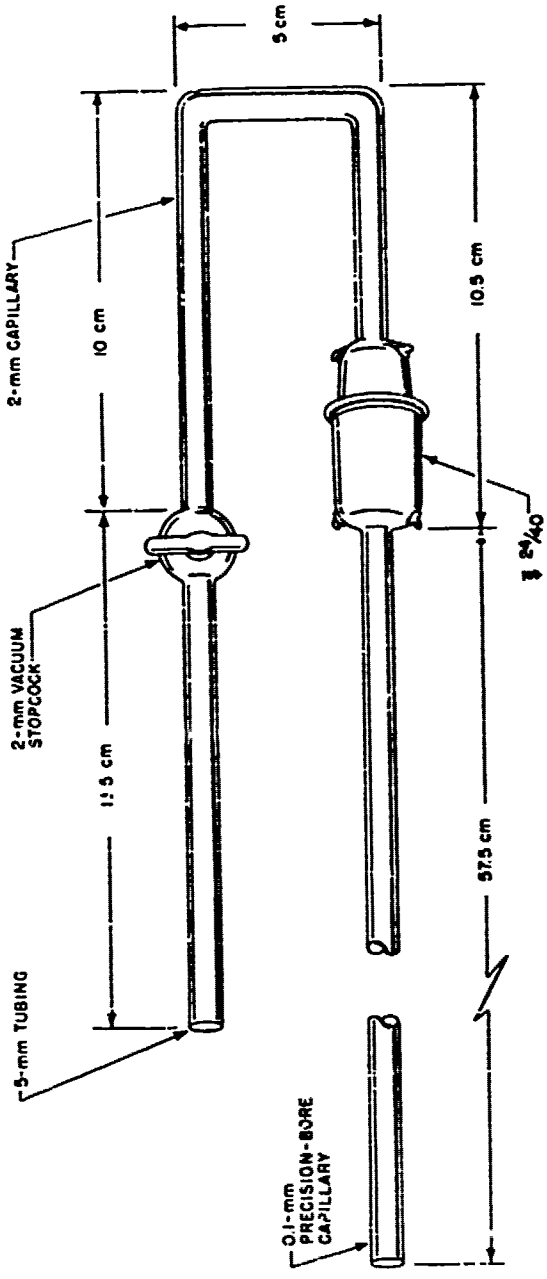

FIG. 1. Dilatometer.

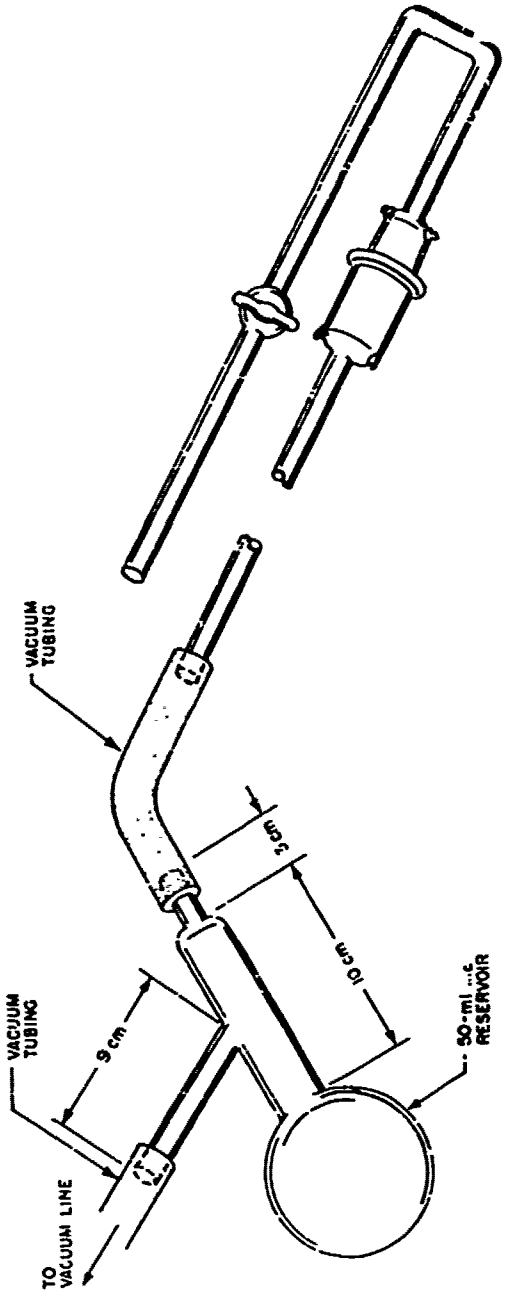

FIG. 2. Method of Filling the Dilatometer.

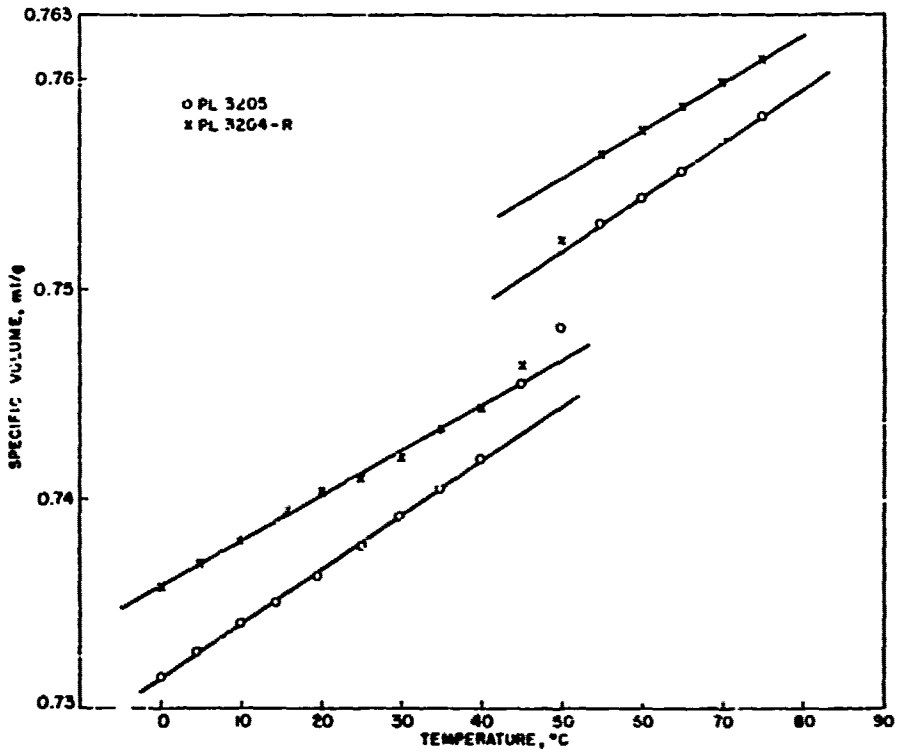

FIG. 5. Specific Volume-Temperature Relationship for PL3205 and PL3204-R.

An increase of 0.64% in specific volume was observed between 50 and 52°C for LFT-10. X-ray diffraction data obtained at room temperature and at 70°C indicate that this transition is the IV → II transition of ammonium nitrate and will be the subject of further discussion. On cooling, this transition does not occur until approximately 47°C.

The results for ammonium nitrate are given in Fig. 4 and Table 3. Only the higher temperature behavior was studied and the experiment was started at 0°C. No transition was observed between 30 and 35°C, but a broad transition did occur between 40 and 50°C. This was shown to be the IV → III transition from X-ray data obtained at room temperature and at 70°C. The transition temperature was shifted to an elevated temperature because of the partial drying of the ammonium nitrate.

The specific volume-temperature data for PL3205 and PL3204-R are given in Fig. 5. The results are similar to those obtained with ammonium nitrate. No transition was observed between 30 and 35°C, but a broad

transition appeared between 40 and 50°C for both PL3205 and PL3204-R. This transition is deduced to be the IV → III transition of ammonium nitrate. The plots and the expansion coefficients indicate that the use of a mixture near the eutectic composition of ammonium nitrate and guanidine nitrate does not alter the ammonium nitrate transition.

The differential thermal analysis results for LPT-10 and ammonium nitrate are quite similar. Ammonium nitrate had endotherms at 46, 92, 130, and 173°C. The LPT-10 had endotherms at 46, 87, and 127°C. No transition is observed at 173°C, the melting point of ammonium nitrate. The endotherm at 87°C for LPT-10 was smaller, however, than the one at 92°C for ammonium nitrate.

It must be concluded from these results that the thermal behavior of LPT-10 from -31 to 75°C is essentially due to the ammonium nitrate. The second order transition of the cellulose acetate binder, which is well plasticized by the acetyl triethyl citrate, must either be below -32°C or is too small to be measured by this technique.

It has been shown from the analysis of the PL3205, PL3204-R, and ammonium nitrate experiments that the observed transition was the IV → III ammonium nitrate type, while in the LPT-10, the main transition was the IV → II transition. This difference in behavior might be explained as follows: the propellant, LPT-10, is processed and the grain pressed at a temperature well above the III → II transition temperature causing most of the ammonium nitrate to be in the II form. When the grain is cooled down, the ammonium nitrate passes directly into the II form since there is very little moisture present. Upon heating the grain to 74°C in the aging test, the II form predominates. However, according to the results of the differential thermal analysis at 87°C it appears that some of the III form is present. These crystals may facilitate conversion of II to the more stable III upon absorption of small amounts of moisture. This conversion gives rise to a volume increase. The PL3204-R, PL3205, and ammonium nitrate samples were never heated to a temperature above the III → II transition temperature and probably contained some moisture. Therefore, these samples undergo the IV → III transition.

Taking the volume of the gas generator grain as 80.80 $in.^3$, the increase in volume of the grain would be 2.15 $in.^3$ when the temperature is increased from 25 to 75°C. If the ammonium nitrate is then converted into the III form on standing as suggested the volume would increase even more.

It is suggested that if the LPT-10 grains are prepared while the ammonium nitrate is in the III form, the severe growth problem would be diminished.

The results also indicate that the forming of a eutectic with guanidine nitrate has very little effect upon the ammonium nitrate transition, and that the Butarez binder has very little effect upon either the transition of the eutectic or the mixed crystal system.

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nitrate and guanidine nitrate. The metastable transition between forms IV and II of the ammonium nitrate oxidizer in the LFT-10 is observed between 50 and 52°C, but the IV → III transition is observed between 40 and 50°C in the other samples. The growth problems obtained in the aging of LFT-10 at 74°C are explained on the basis of these observations.

A detailed account of the dilatometric procedure is also given.

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