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13. ABSTRACT (Maximum 200 words)

An investigation of LOVA propellant aging was performed to identify the cause of stabilization periods in certain propellant lots. The following results were obtained: (1) the level of residual solvent in propellant grains slowly decreases while in storage; mechanical properties of the grain were qualitatively observed to change in response to the loss of plasticization by residual solvent; the rate at which solvent is lost depends on storage conditions (e.g., free volume in storage vessel, the frequency with which vessel is opened); (2) during the drying process and while in storage, plasticizer "comigrates" with solvent, resulting in plasticizer accumulation at grain surfaces; and (3) during grain storage, residual solvent is oxidized to generate organic acids. Based on these results, it is proposed that stabilization periods result from processes related to migration, evaporation, and oxidation of residual solvent. The effect of solvent content and migration on the relaxation of mixing/extrusion-induced propellant stress was not examined in this study, but is suspected to be significant and is recommended as a topic for future investigation.

14. SUBJECT TERMS

propellant aging, residual solvent, plasticizer migration, solid propellants, FTIR spectroscopy, plasticizers, spectroscopy

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1. INTRODUCTION

During the three-year period prior to the preparation of this report, an investigation of the causes of solid propellant aging was conducted. To date, two technical reports resulting from this effort have been published (Pesce-Rodriguez 1992a, 1992b); both reports deal with the long-term behavior of additives used in some LOVA propellant formations. The stimulus for initiating these investigations was the observation that certain nitramine-based propellants experience a "stabilization period." Stabilization periods can last as long as 20 weeks, and are characterized by measurements of breech pressure which gradually decrease before leveling-off at some constant value. Identification of the cause of the stabilization periods has been hampered by the absence of a coordinated ballistic, mechanical, and chemical testing program. The objective of this investigation was to examine the chemical aspect of the aging problem and, where possible, ascribe the results to potential variations in ballistic and mechanical properties.

To understand LOVA propellant aging, the following questions must be answered:

• Which propellant component(s) is (are) responsible for the chemical and/or physical changes that occur during the stabilization period?

• Why do mixes with identical formulations demonstrate different aging behavior?

To answer these questions, data presented in the two reports referenced previously, as well as previously unpublished results, have been examined and the conclusions based on these examinations are discussed here. The most important observations made were those indicating a correlation between the migration of residual solvent and the migration (and accumulation) of plasticizer. Based on these observations, it is proposed that residual solvent is the source of the LOVA aging problem. A solution to the aging problem therefore appears to depend on the reevaluation of the current drying processes, storage conditions, and standards for permissible residual solvent level.

As a further introduction to the discussion of the effects of storage conditions and residual solvent content on propellant aging, the following information on propellant storage is provided:

The volume of a typical storage drum (a *LEVER-PAC*) is approximately 75 liters, and is filled to approximately 20 cm from the top of the drum with 68 kg (150 lb) of propellant. Drums are covered with a lid that is secured with a "snap-tight" ring. The volume occupied by the 68 kg of the propellant stored in the drum is approximately 40 liters. This leaves 35 liters of free volume in the drum, of which 20 liters is above the stored propellant, and 15 liters is in the space between the grains. In a drum that contains propellant with a residual solvent content of 0.25 wt-% (the MIL-Spec limit for dried propellant), there is approximately 170 g of solvent. Considering the vapor pressure of ethyl acetate (73 mm at 20° C) and the free volume available, approximately 12 g of solvent will be in the vapor phase. Of this, 7 g will occupy the space above the propellant, and 5 g will occupy the space between grains. The remaining 158 g of solvent will remain trapped in the propellant grains. If there is a leak in the drum, or if the drum is opened, solvent vapor will escape from the drum. Eventually, the propellant grains will be depleted of residual solvent.

2. EXPERIMENTAL

Three methods for the characterization of solid propellants were used in this investigation: (1) micro-reflectance Fourier transform infrared spectroscopy (Bergin 1989; Messerschmidt and Harthcock 1988) (FTIR-mic) and (2) photoacoustic-FTIR (Grahm, Grimm, and Fateley 1985; Ferraro and Basile 1982; McClelland 1983; Pesce-Rodriguez and Fifer 1991) (PA-FTIR) spectroscopy for nondestructive examination of chemical composition at propellant surfaces, and (3) desorption-gas chromatography FTIR (D-GC-FTIR) (Griffiths and deHaseth 1986) spectroscopy for monitoring the desorption of volatiles such as residual solvent. PA-FTIR and FTIR-mic spectra were obtained on a Mattson Polaris FTIR spectrometer using First software (Kramers-Kronig transformations [Graf, Koenig, and Ishida 1985; Hadni 1967] were used to correct reflectance spectra). For PA analysis, a helium-purged MTEC Model 100 PA cell was used. The velocity of the moving mirror was 0.316 cm/s. Carbon black (Norit-A) was used to obtain background spectra. FTIR-mic spectra were obtained using the Spectra-Tech IR-Plan® infrared microscope with a MCT detector. The microscope was operated in reflectance mode and aluminum foil was used to obtain background spectra. For all spectra, 32 scans were collected with a resolution of 8 cm^{-1}. Desorption experiments were performed with a CDS Model 122 Pyroprobe® (coil probe, sample in quartz tube) connected via a heated interface chamber to the splitless injector of a Hewlett Packard GC-FTIR system (Model 5890 GC and Model 5965 IRD® with narrow band MCT detector). P-GC-FTIR conditions are provided in Table 1.

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Table 1. P-GC-FTIR Conditions

Condition	Value
Pyroprobe Conditions	
Pulse temperature:	100° C
Pulse duration:	20 s
GC Conditions	
Column type:	Quadrex capillary
Column dimensions:	0.32 mm × 25 m
Stationary phase:	3 μm OV-17 film
Temperature program:	50° C for 3 min 50 → 200° C @ 10°/min 200° C for 10 min
Injector temperature:	200° C
Interface temperature:	100° C
Light pipe temperature:	200° C
Transfer lines temperature:	200° C
FTIR Conditions	
Sampling rate:	3 interferograms/s
Resolution:	8 cm^{-1}

The composite propellant grains used in this investigation were cylinders having a length and diameter of approximately 1 cm. The four formulations examined in this investigation were all RDX/CAB/NC-based. Other additives include stabilizer, plasticizer, and a proprietary processing aid. Two different plasticizers (P1 and P2) were used; both are energetic materials with proprietary structures. Table 2 lists the plasticizer(s) used in each formulation. The other propellant ingredients are RDX (1,3,5-trinitro-1,3,5-triazacyclohexane), CAB (cellulose acetate butyrate), and NC (nitrocellulose). All propellant grains were processed using a mixture of ethyl acetate and ethyl alcohol.

To examine the effect of grain geometry and drying conditions on plasticizer level, six specially processed sets of formulation F4 samples were used. Table 3 gives a description of these samples.

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Table 2. Plasticizers Used in Propellant Formulations

Formulation	Plasticizer
F1	P1 only
F2	P1 and P2 (2:1)
F3	P1 and P2 (1:1)
F4	P2 only

Table 3. Description of Specially Processed F4 Samples

Sample	Number of Perforations	Perforation size (mm)	Processing conditions[a]
A	0	—	—
B	1	3.0	—
C	19	0.38	—
D	19	0.38	mixing time = "std" + 1 hr
E	19	0.38	mixing temperature = "std" + 15° C
F	19	0.38	drying time @ 60° C = "std" + 1 week

[a] Other than proprietary "standard" conditions.

Formulations F1, F2, and F3 were all processed under "standard" conditions. Except for "undried" F3, all grains were dried for two days at ambient temperature, then two additional days at 60° C, and then stored under ambient conditions prior to analysis. Analysis by FTIR-microscopy was initiated one week after completion of the 60° C drying process; samples were subsequently kept in unsealed vials during the 10-month examination period. Table 4 describes the storage conditions of samples analyzed by D-GC-FTIR (formulation F4 only).

For analysis of plasticizer levels at interior "bulk" surfaces by FTIR-mic, grains were cross-sectioned by cleaving them lengthwise through the center row of perforations (see Figure 1). The surfaces examined did not come in contact with the blade used to cleave the grains. Measurements at the interior bulk

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Table 4. Storage Conditions for F4 Samples Analyzed
by Desorption-GC-FTIR

Set Number	Days stored in sealed bottle[a]	Days since removal from sealed bottle[a,b]
1	3	158
2	153	8
3	161	0

[a] All samples stored at ambient temperature, except for sample F (of each set), which was stored uncovered in a 60° C oven.

[b] After removal from sealed bottle, samples were stored uncovered at ambient temperature.
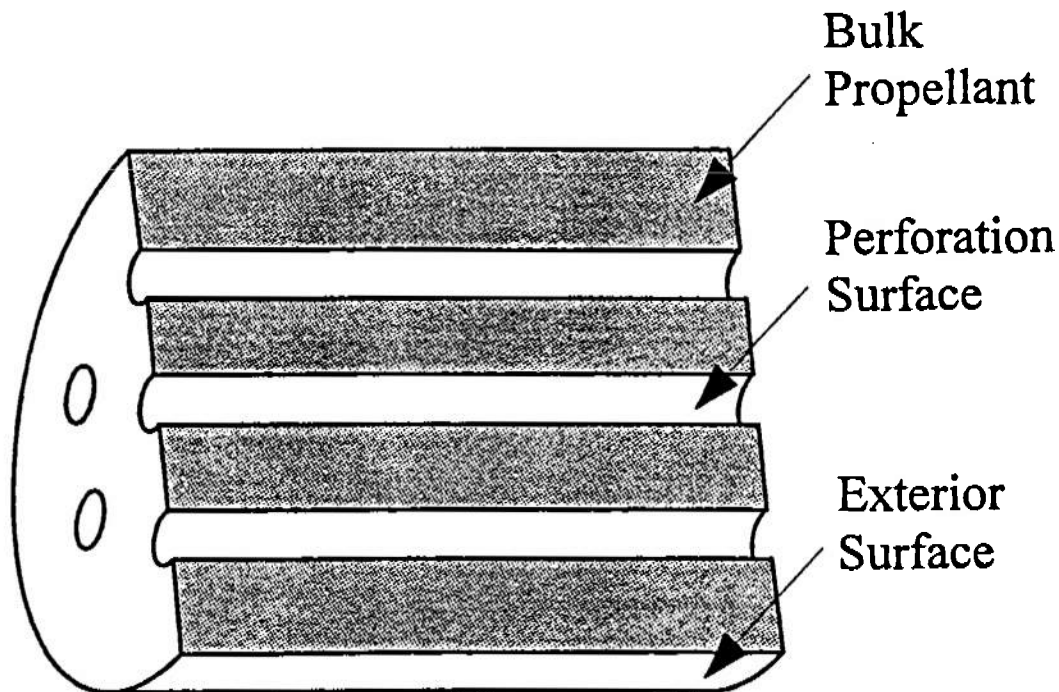

Figure 1. Cross-sectioned solid propellant grain.

surface and exterior "circumferential" surface were made on three different grains of each type and then averaged. The same 18 grains (3 grains of each of the 6 types) were analyzed during the course of the FTIR-mic investigation. To aid in consistently analyzing the same area on these samples, measurements were made in the centers of small circles (approximately 1 mm in diameter) outlined with a pencil on each of the samples.

3. RESULTS AND DISCUSSION

3.1 <u>D-GC-FTIR Investigations</u>. Figure 2 shows chromatograms generated via FTIR detection of volatile components desorbed from the propellant samples; note that the data in set No. 1 was obtained approximately 22 weeks *after* the data in set No. 3. Identities of the peaks were determined by on-line searches of spectral databases and are summarized in Table 5. Suspected sources of the observed peaks are also given in Table 5. Note that due to its low vapor pressure and vaporization rate, desorbed plasticizer is not observed in any of the chromatograms.
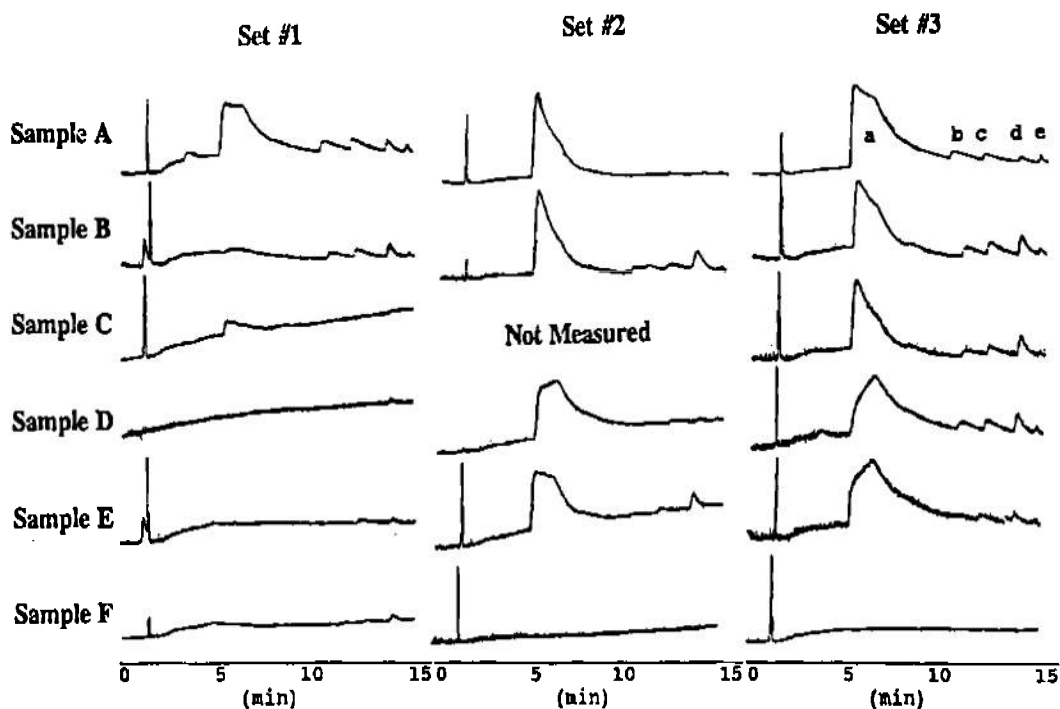
Figure 2. <u>Desorption-GC-FTIR results, sample F4. Relative IR response vs. retention time. See Tables 3 and 4 for sample descriptions and storage conditions: (a) ethyl acetate; (b) acetic acid; (c) a carboxylic acid; (d) isooctanol; and (e) isooctanoic acid.</u>

The chromatograms in Figure 2 indicate that compared to perforated grains (samples B–F), unperforated grains (sample A) retain more desorbable materials. This is the result of the longer distance through which the species must diffuse before reaching a surface from which they can evaporate. The effect of ambient aging is demonstrated by a comparison of the levels of the five desorbable species across

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Table 5. Identity and Source of Peaks in Figure 2

Retention Time (min)	Identity	Suspected Source
5.2	ethyl acetate	processing solvent
10.1	acetic acid	oxidation of ethanol and/or ethyl acetate (processing solvents)
10.1	a carboxylic acid	oxidation of a processing aid component
13.6	isooctanol	solvent/carrier for processing aid
14.6	isooctanoic acid	oxidation of isooctanol

a given row. Such a comparison indicates that when stored in an unsealed vial, most residual solvent escapes from the propellant grain. The only sample still possessing a significant level of residual solvent after 22 weeks in an unsealed vial is sample A, the unperforated grains. Considering that grains are usually kept in sealed drums during storage, it is suspected that the required time for complete loss of residual solvent would be relatively long (i.e., solvent must first evaporate from the propellant, and then escape from the drums). The consequences of trapping residual solvent in sealed containers is discussed below.

Although mechanical properties were not determined for the samples used in this investigation, it was observed that as the level of residual solvent dropped, the propellant grains tended to become more brittle, indicating the loss of plasticization by the solvent.

3.2 PA-FTIR Investigation. Relative P1 levels for three propellant formulations were obtained from the PA-FTIR spectra (not shown) of three propellant formulations. This was accomplished by taking the ratio of the intensity of the P1 absorption at 2,100 cm^{-1} to that of the CAB absorption at 1,754 cm^{-1}. Unlike plasticizer P2, which does not appear to evaporate from propellant formulations (see section 3.1), plasticizer P1 does evaporate. Furthermore, from a comparison of the slopes of the lines in Figure 3, it appears that the evaporation of plasticizer P1 is suppressed by the presence of P2. The effect of residual solvent is demonstrated by inspection of the data obtained for "dried" and "undried" samples formulation F3 (hereafter referred to as $F3_D$ and $F3_U$, respectively). The former had been subjected to
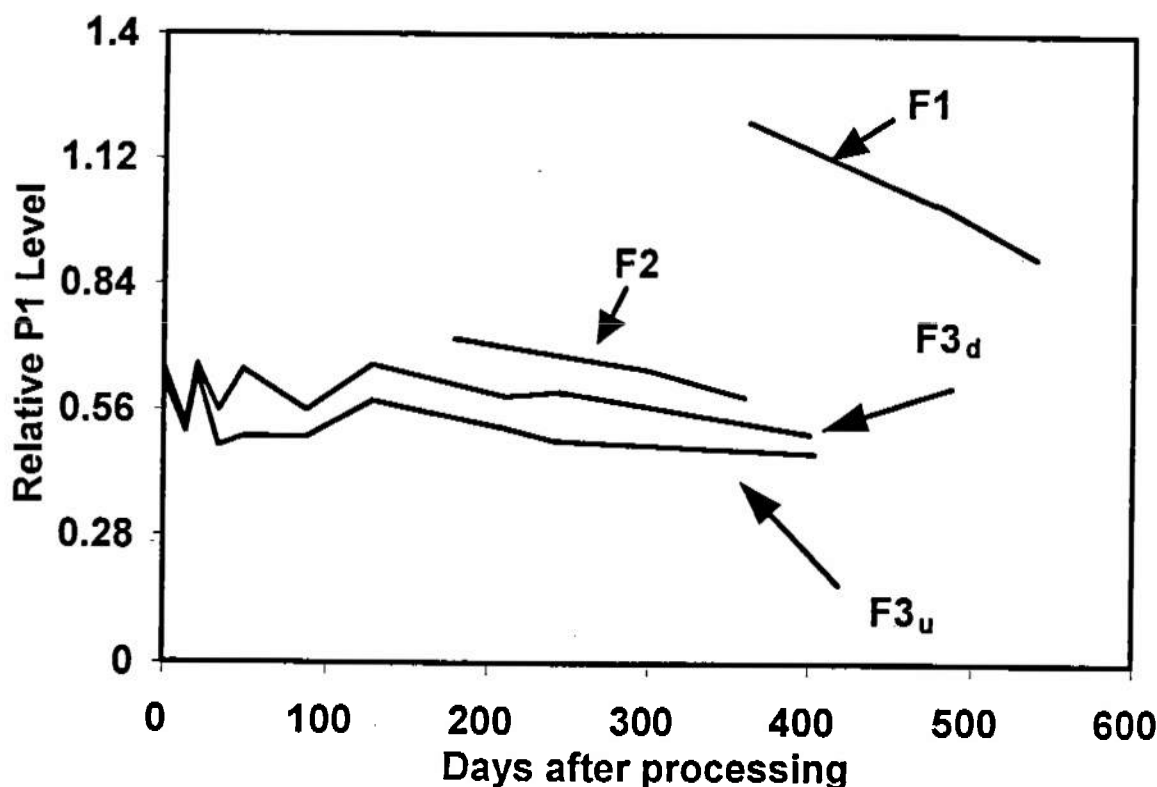
Figure 3. Relative levels of plasticizer P1 in formulations F1, F2, and F3.

the standard drying procedure (including cycles at 25° C and 60° C), while the latter was allowed to dry very slowly at ambient conditions in loosely capped vials. It is noted that formulation F3 generally experienced little or no loss of P1 over the course of the investigation. The exception to this generality occurred for $F3_U$ during the first few weeks after processing. Initially the P1 level in $F3_D$ and $F3_U$ were nearly equal. After the third week, the P1 level in the $F3_U$ decreased slightly and then remained approximately constant for the remainder of the monitoring period. Two interpretations for this observation are offered. The first interpretation is that rapid drying made the extruded surface of $F3_D$ less permeable to plasticizer than did slow drying. The second interpretation is that although P1 appears to have little tendency to evaporate when in the presence of P2, P1 levels decreased as a result of "comigration" and "coevaporation" with solvent; i.e., solvent evaporation facilitated the evaporation of P1, and evaporation of P1 ceased after most of the solvent had evaporated. Both explanations are plausible, and both point to the potential importance of residual solvent.

3.3 FTIR-MIC Investigations. The effect of ambient aging on plasticizer P2 levels was studied by monitoring the six types of grains described in Table 3. Typical FTIR-mic spectra (1,500–1,800 cm^{-1})

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of bulk and extruded exterior surfaces of a F4 grain are shown in Figure 4. Absorptions assigned to RDX, CAB, NC, and P2 are labelled accordingly. Comparison of these spectra indicates that the composition of interior "bulk" material is significantly different from that of the extruded surface (i.e., the extruded surface is rich in polymeric binder and plasticizer P2). Scanning electron microscopy indicates that this binder-rich layer is several microns thick (Lieb 1989). Figure 5 shows plasticizer levels for the interior bulk and extruded exterior surface of propellant grains as a function of time. Relative P2 levels were calculated by taking the ratio of the intensity of the P2 absorption band at 1,570 cm^{-1} and that of the RDX absorption band at 1,600 cm^{-1}. Two interesting observations can be made from these results. The first is that in all but the unperforated grains (sample F4/A), neither migration nor evaporation of P2, appears to occur to any significant extent during the time frame of this investigation. Migration in sample F4/A is evidenced by the increase in the "exterior" plasticizer level relative to the "bulk" plasticizer level at approximately 100 days. The plasticizer levels in samples B–F vary somewhat over time, but experience no net change. The possibility of a steady-state diffusion/evaporation process can be ruled out based on results of desorption experiments (see section 3.1) which indicate no evaporation of plasticizer even at temperatures as high as 100° C. The second observation that may be made from examination of Figure 5 is that the initial "bulk" plasticizer level for sample A is approximately twice that of samples B–F (i.e., 0.8 vs. 0.4).
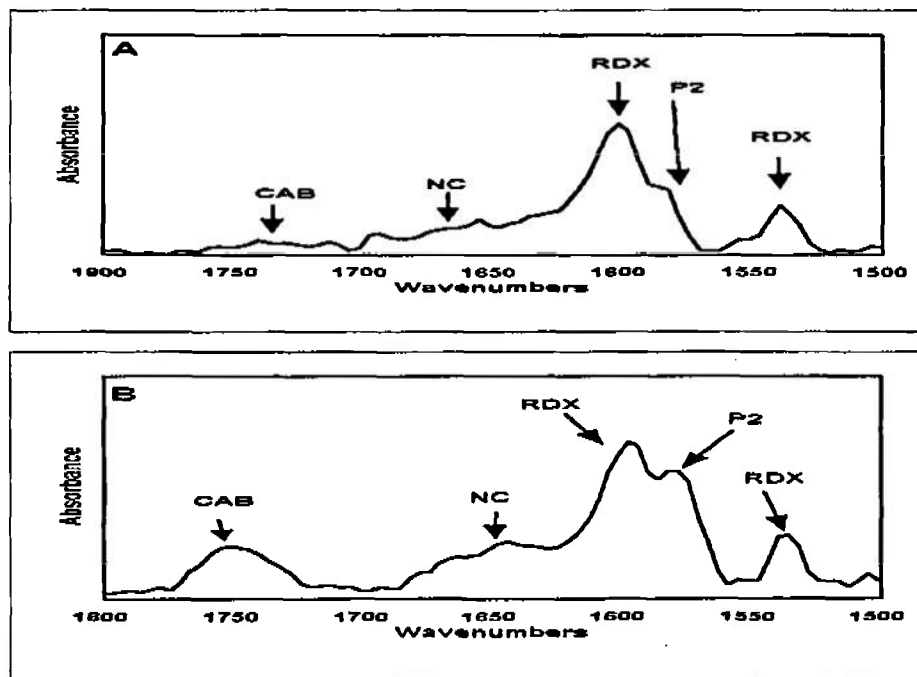

Figure 4. Typical FTIR-mic spectra of propellant F4 grain surfaces: (A) interior "bulk" surfaces of cross-sectioned grain and (B) exterior extruded surface.
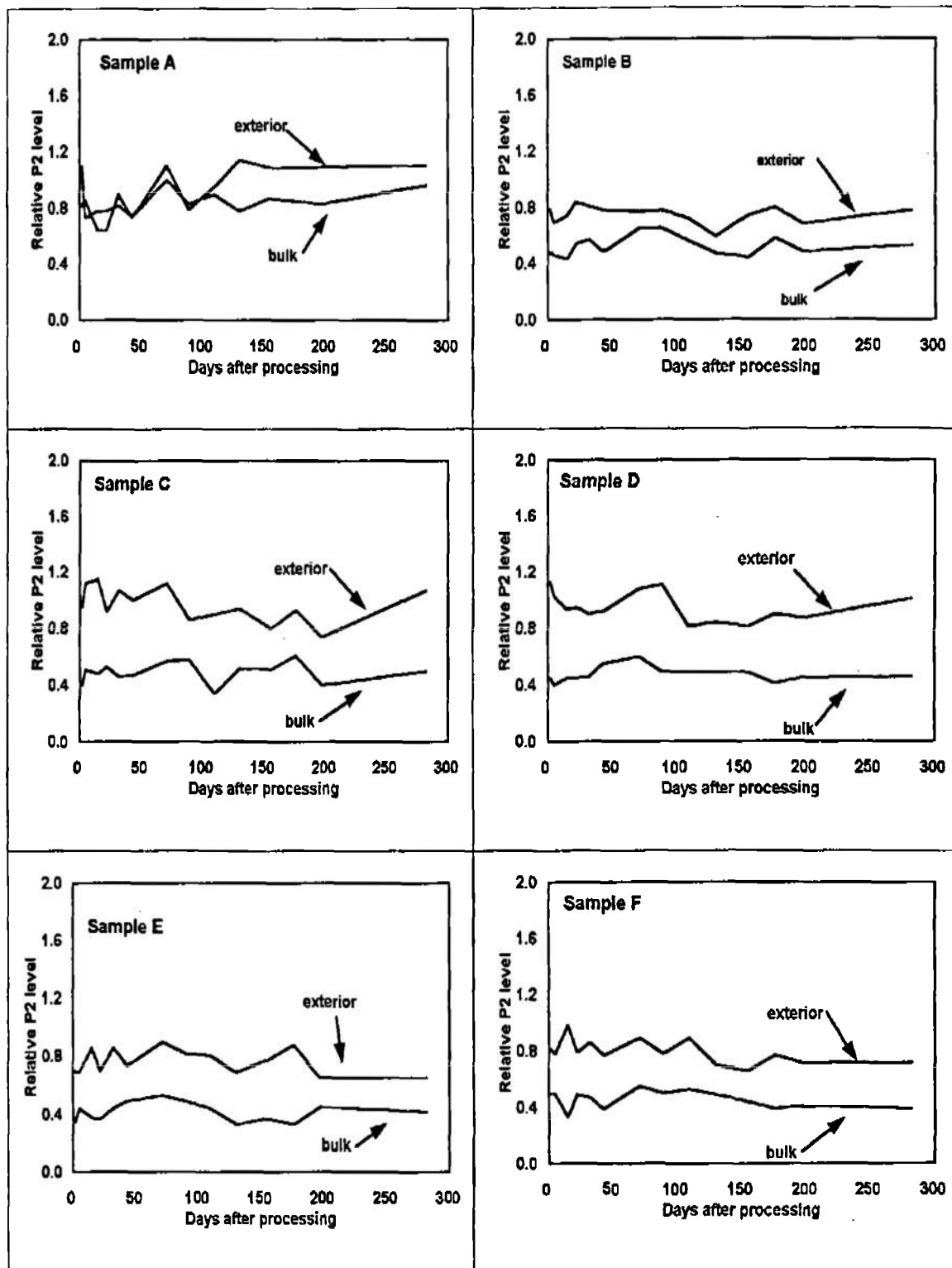
Figure 5. P2 levels at exterior extruded surface and interior bulk surface of F4 grains vs. time. See Table 2 for sample descriptions.

There are two new questions that must now be answered:

- What characteristic of the unperforated grains makes them the only grains to experience significant plasticizer migration?

- Why is the initial bulk plasticizer level in unperforated grains so much higher than in perforated grains?

The answer to both of these questions appears to be related to the presence of residual solvent. The absence of perforations in sample F4/A restricts the evaporation of residual solvent (compare ethyl acetate levels in Figure 2, set No. 1). The observed increase in plasticizer level at exterior surfaces (i.e., at the "circumferential," perforation, and end surfaces) is the result of comigration of solvent and plasticizer to those surfaces. Once at the surface, solvent evaporates and plasticizer accumulates. After all the residual solvent has migrated and evaporated, accumulation of plasticizer ceases. An explanation as to why unperforated grains have a relatively high level of plasticizer in the bulk is that since solvent cannot migrate to perforation surfaces, less plasticizer is transported away from the bulk.

To confirm that solvent actually can transport plasticizer through a propellant grain, the following experiment was performed: a dry, perforated propellant grain was placed in an atmosphere saturated with ethyl-acetate vapor for several hours. As might be expected, exposure to the vapor resulted in a softening of the propellant grain, indicating the plasticizing ability of ethyl acetate. After cross-sectioning the grain, analysis was performed by micro-reflectance FTIR. The spectrum obtained from the bulk of the grain is presented in Figure 6d. Also shown in Figure 6 are typical spectra for bulk and exterior surfaces (circumferential and end) *before* exposure to ethyl acetate vapor. The spectra in Figure 6 indicate that solvent vapor permeates through the plasticizer-rich exterior of the dry grain, carrying plasticizer with it. The result is a redistribution of plasticizer throughout the entire grain. If the "solvent migration/plasticizer accumulation" theory is correct, then the only difference between the composition of the end and bulk surfaces before exposure to solvent vapor should be that the end surface, which is a nonextruded exterior surface, should be richer in plasticizer than the bulk as a result of plasticizer deposition during drying; the spectra should be identical after exposure to solvent vapor (and before reevaporation of solvent) as a result of redistribution of plasticizer. A comparison of spectra b–d in Figure 6 confirms that both requirements are met.
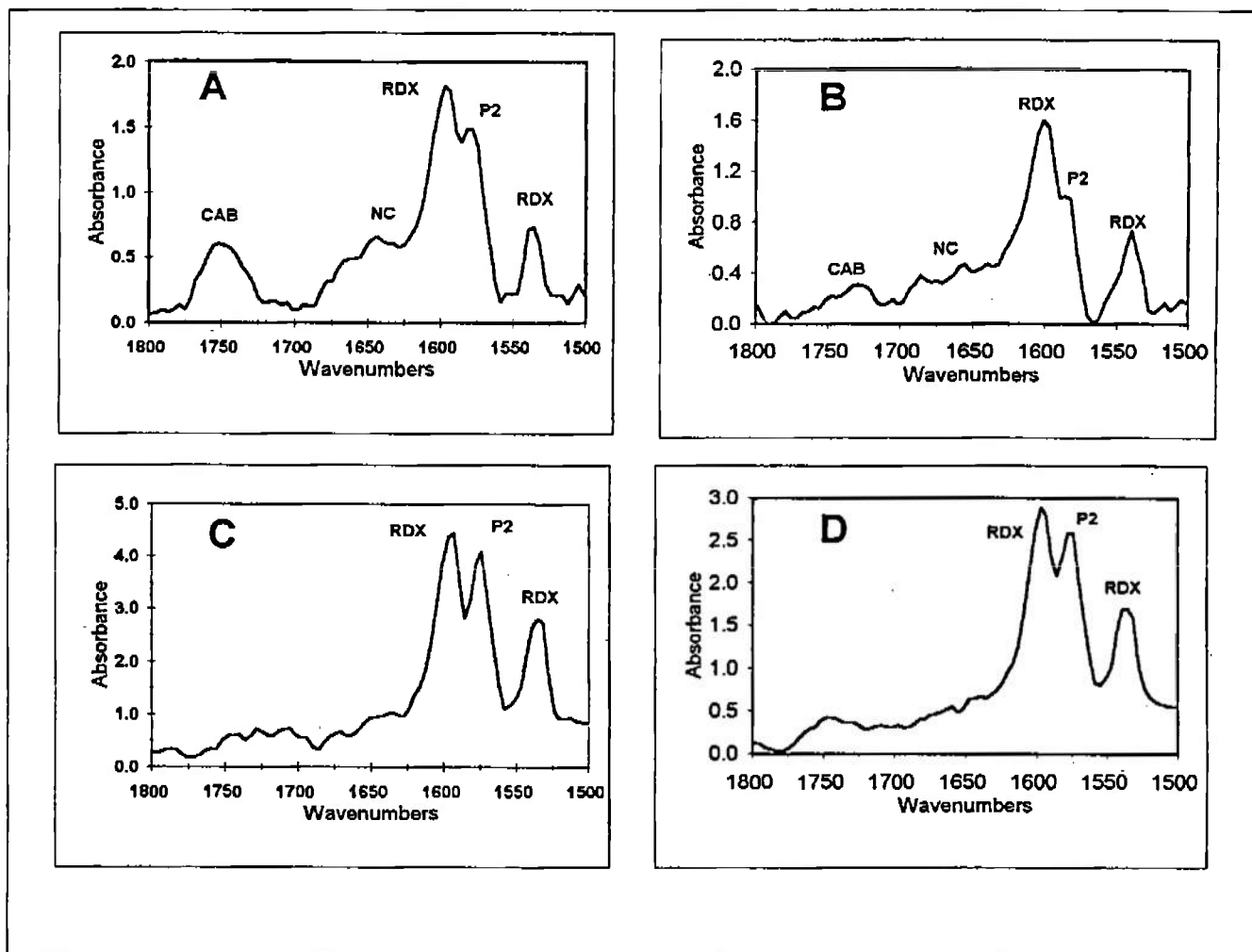
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Figure 6. Microreflectance FTIR spectra of propellant grain: (a) exterior extruded surface; (b) interior "bulk" surface of cross-sectioned grain; (c) end surface; and (d) interior "bulk" surface after exposing whole grain the ethyl-acetate vapor.

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The experiment described previously indicates that the distribution of plasticizer in a propellant grain is controlled by solvent migration. Plasticizer is added to propellant formulations in such a way that it be uniformly distributed throughout the grain. Based on the results discussed previously it appears that this is defeated by comigration of solvent and plasticizer to grain surfaces.

The following points have been discussed previously, and are summarized here:

- Residual solvent is oxidized to generate organic acids.

- Residual solvent plasticizes propellant grains.

- In the absence of solvent, plasticizer migration is insignificant.

- Plasticizer migration occurs simultaneously with solvent migration.

- Accumulation of plasticizer at the exterior surfaces of propellant grains is the result of co-migration with solvent.

- In the presence of added solvent vapor, plasticizer accumulated at exterior surfaces of propellant grain will re-enter the bulk of the grain and be redistributed throughout the grain.

- The rate of residual solvent evaporation is dependent on propellant storage conditions.

The next step in resolving the LOVA aging problem is to relate the findings summarized previously to propellant stabilization times. Since ballistic and mechanical properties of the samples analyzed in this investigation were never made, it is difficult to directly ascribe "cause and effects." It is recommended that a concerted analysis of the chemistry, ballistics, and mechanical properties of solid propellants be undertaken to get to the root of the aging problem. For the present time, speculation as to the effects of residual solvent on propellant aging must suffice. Suggestions as to what these effects might be are as follows:

- While in storage, trapped residual solvent may provide enough solvating effect to permit the relaxation of polymer chains experiencing stress induced by the mixing and extrusion processes.

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- Even after the relaxation of stresses, residual solvent can have plasticizing effects. The degree of plasticization will deteriorate as solvent evaporates and escapes from the grains' storage drum.

- Migration of plasticizer *to* grain surfaces and *away from* the bulk of the propellant during drying and storage effects the propellant's physical properties, and therefore, its ballistic properties.

Stated most generally, the cause of LOVA propellant aging appears to be the changes in propellant properties caused by the presence, the migration, and the evaporation of residual solvent. The effect of solvent oxidation products (organic acids) on propellant aging is suspected to be of little significance.

A proposed solution to the propellant stabilization period problem is the development of a new drying cycle. For example, a drying cycle could be developed that removes all residual solvent before grains are put in storage. If such a cycle were adopted, it is likely that the composition, as well as the mechanical and ballistic properties, of the propellant should remain constant for the lifetime of the propellant (assuming that the propellant does not undergo "unusual" temperature cycling while in storage). While driving off all residual solvent may not result in propellant with *optimal* mechanical and ballistic properties, the resultant propellant should have more *predictable* properties than those of propellant that is dried and stored using current procedures. If it is true that physical and chemical changes occurring during the stabilization period result in a decrease in mechanical stresses induced by the mixing and extrusion processes, then it might be best to dry the propellant slower than is currently being done. To avoid the stabilization period, the slow-drying cycle should probably be designed to be long enough to ensure complete removal of residual solvent; the propellant that is eventually placed in storage should be free of residual solvent.

4. CONCLUSIONS

A study of the effect of residual solvent on LOVA propellant aging was conducted. The most significant finding of the study was that the distribution of plasticizer in propellant grains depends strongly on solvent content (i.e., in "wet" propellant, plasticizer is distributed relatively uniformly throughout the grain; in dry propellant, plasticizer is accumulated at the surfaces of the grain). In most of the samples examined, the residual solvent content was already quite low, such that little plasticizer migration was observed. It is proposed that "real" propellant grains do experience plasticizer migration while in storage.

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Furthermore, it is suspected that residual solvent trapped in grains during storage provides enough solvation, and therefore enough segmental mobility for polymeric binder, to permit the propellant to relax from stresses induced during mixing and extrusion. Based on the data obtained in this study, it is tentatively concluded that the LOVA stabilization period is a result of plasticizer migration and/or grain relaxation. To confirm these tentative conclusions, a concerted aging study including the analysis of the ballistic and mechanical properties, as well as the composition, of freshly processed propellant (stored under actual storage conditions) should be performed.

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