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Ammonium Perchlorate, Friend or Foe?
Part 1: The Influence of this Oxidizer on the Aging Behavior of Propellant Compositions

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Abstract: Propellants containing nitroglycerine and ammonium perchlorate have been reported to have comparatively shorter shelf lives than analogous energetic materials without this oxidizer. However, investigation into the aging behavior of three compositions containing polyethylene glycol and nitroglycerine revealed that the propellant which included ammonium perchlorate degraded at a slower rate compared with the other materials. It was suggested that ammonium perchlorate might act as an oxygen inhibitor reducing the oxidation rate of the polyethylene glycol binder so decreasing the rate of the propellant decomposition. In addition, at temperatures of 80 °C or lower, ammonium perchlorate initially appears to hinder acid hydrolysis of nitroglycerine which also slows down the degradation of polyethylene glycol based propellant.

Keywords: Ammonium perchlorate · Nitroglycerine · Polyethylene glycol · Propellants · Aging

1 Introduction

Ammonium perchlorate (AP) is an oxidizer in a vast range of propellant compositions. These include materials which are classified as composite, high energy and composite modified cast double base (CMCDB) propellants [1].

Although there is a drive to replace this compound with more environmentally friendly oxidizers, AP is still used in a large number of compositions and so the need to understand and predict how such propellants degrade or age under a range of conditions remains. The behavior of AP in compositions containing nitrate esters is of particular interest as this oxidizer can accelerate the decomposition of nitrate esters once degradation has started, to the extent where the material can eventually ignite [2, 3].
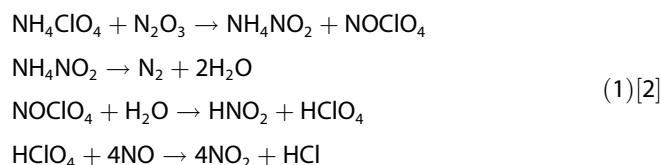
It has been noted that if Composition A (Table 1), containing polyethylene glycol (PEG), AP and nitroglycerine (NG), is subjected to the silver vessel test in which approximately 100 g of material is heated at a fixed temperature of 80 °C in a bespoke glass vessel, there are no brown fumes evolved or any gradual temperature rise which would indicate the decomposition of the NG. Instead, the propellant suddenly and violently ignites without prior warning.

This behavior has been attributed to the fact that at elevated temperatures, in the presence of NG, AP can degrade to form perchloric acid. This causes the rapid hydrolysis of the nitrate ester and can directly react with the oxides of nitrogen evolved as NG undergoes degradation (Equation set 1) [2–4].

$$NH_4ClO_4 + N_2O_3 \rightarrow NH_4NO_2 + NOClO_4$$
$$NH_4NO_2 \rightarrow N_2 + 2H_2O$$
$$NOClO_4 + H_2O \rightarrow HNO_2 + HClO_4 \qquad (1)[2]$$
$$HClO_4 + 4NO \rightarrow 4NO_2 + HCl$$

It is clear, from the literature, that AP has an important role in the degradation of propellants containing nitrate esters. A number of authors have studied the rate of stabilizer depletion and the time to ignition of the propellant. For example, Asthana, Divekar et al. [5] looked at the stability, auto ignition and stabilizer depletion of CMCDB propellants containing NG and AP. They highlighted, at the time

Table 1. Composition details.

Composition Name	% AP	% Nitramine	% Al	% NG/PEG binder [a]
A	15	42	18	25
B	0	57	18	25
C	0	0	0	100

a) Binder also contains 2-nitrodiphenylamine (2-NDPA) and paranitromethylaniline (pNMA) which act as stabilizers.

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of publication, there was limited information regarding the shelf life of these propellants. It was noted that the inclusion of AP increases the autocatalytic behavior of CMCDB propellants over time. However, the rate of stabilizer depletion in the propellants studied followed a trend in agreement with that for double base propellants.

The authors used the auto ignition test to characterize a selection of CMCDB and double base propellants. This involved subjecting the propellant to a fixed temperature, for example, 130.5 °C, and measuring the time to ignition. Using this technique they showed that CMCDB samples ignited more rapidly than double base material suggesting that the decomposition kinetics in the CMCDB propellants were faster than in double base analogues. Consequently, CMCDB propellants possess a lower shelf life than their double base counterparts.

Bromberger, Conduit et al. [6] studied a range of CMCDB compositions using the silver vessel test. The materials all contained nitrocellulose (NC), NG, AP, aluminum (Al) and the stabilizer 2-Nitrodiphenylamine (2-NDPA). The concentration of 2-NDPA appeared to have no effect on the test results. It was also concluded that due to the violent nature of ignition of small amounts of hybrid CMCDB propellants, any heating trial should be carried out with caution.

Further research by Conduit [7] showed that CMCDB propellants containing AP and NG had shorter lives than double base propellants and when subjected to the silver vessel test tended to ignite very quickly after the initial onset of self-heating.

Asthana, Ghavate et al. [8] also looked at the effect of high energy materials – cyclo-1,3,5-trimethylene-2,4,6-trinitramine (RDX), pentaerythritol tetranitrate (PETN) and AP – on the shelf life of CMCDB propellants. When AP was present, the RDX and PETN containing CMCDB propellants would undergo self-ignition in a much shorter time. This was attributed to the AP causing auto decomposition of the RDX and PETN in the double base matrix.

These authors concluded that AP containing compositions have a much shorter self-life compared with RDX and PETN based propellants. Nitramine double base propellants, that do not contain AP, have relatively good thermal stability and this has been highlighted by Bunyan [4] and Bhalerao, Gautam et al. [9].

From the literature, it might be expected that a composition containing AP would degrade at a faster rate than an analogous material which did not contain this oxidizer. To investigate this further, three energetic compositions were subjected to aging regimes and the results compared. The details of the materials are shown in Table 1. Ideally, all three compositions should have been subjected to exactly the same aging regimes. However, it soon became apparent that Compositions B and C degraded more rapidly than expected. The reasons for this were investigated with particular attention paid to the role of AP and its interaction with PEG and NG.

2 Experimental

2.1 Analytical Methods

It should be noted that with the exception of accelerating rate calorimetry (ARC), in all cases, duplicate samples were used. Where applicable, the data are reported as an average of the results.

ARC was carried out in a Euro ARC or an ES ARC, both manufactured by THT instruments. A hastelloy C bomb was used with a sample mass of 0.2 ($\pm$ 0.01) g. Testing was performed in heat/wait/search mode in 5 °C steps from 30 °C.

Acidity analysis was carried out using a similar method to that of McDonald [10]. For the compositions, the sample mass was 0.5 ($\pm$ 0.01) g.

Gel Permeation Chromatography (GPC) was carried out on an Agilent 1200 instrument fitted with a refractive index (RI) detector. The columns used were PL gel 3 μm 100 Å with a guard.

Heat Flow Calorimetry (HFC) was conducted in a 2277 instrument manufactured by Thermometric (now part of TA instruments). Approximately 2.5–3 g of each composition was cut into small (2 mm) cubes and weighed into 3 cm^3 vials which were hermetically sealed with a Teflon lined lid. The test was carried out at 80 °C in accordance with the principles of STANAG 4582 [11].

High Performance Liquid Chromatography (HPLC) was performed on a Dionex HPLC Ultimate 3000 instrument. The column used was a Lichrosorb RP_18 (5 μm) fitted with a Lichrospher 100 RP_18 (5 μm) guard.

Nuclear Magnetic Resonance (NMR) was performed using a Bruker Avance 400 spectrometer, proton frequency 400 MHz, fitted with a 5 mm QNP probe. The samples were extracted in a deuterated solvent and quantification was achieved through the addition of tetrakis(trimethyl)silane (TTMS) which acted as an internal standard.

2.2 Propellant Aging

The initial intention was to heat Compositions A, B and C for up to 25 days at 80 ($\pm$ 0.3) °C and analyze the samples taken at regular intervals. However, it was found that Composition C liquefied after 11 days and after 21 days, Composition B had also lost its mechanical integrity. Therefore these were the maximum aging periods used for these two compositions.

2.3 Aging of Compositions B and C with Inorganic Compounds

Composition B was aged in contact with a range of inorganic compounds to see if these would arrest or slow down the decomposition processes. The samples were prepared by mixing 1.4 ($\pm$ 0.1) g Composition B with 1.4 ($\pm$ 0.1) g of the inorganic compound in a weighing boat. The resultant mixture was carefully transferred to a 3 cm^3 glass

vial and hermetically sealed with a Teflon lined lid. The samples were heated for up to 11 days in a block bath maintained at 80 ($\pm$0.1)°C before analysis.

The following inorganic compounds were aged in contact with Composition B:

1. AP, as received, stored over silica gel in a desiccator.
2. AP dried under vacuum at 100°C for six hours.
3. Potassium perchlorate ($KClO_4$).
4. Ammonium carbonate (($NH_4)_2CO_3$).
5. Sodium chloride (NaCl).
6. Calco 2246. This compound is used as an antioxidant in composite propellants.
7. Resorcinol (1,3-benzenediol). This compound is incorporated into some nitrate ester containing propellants as it acts as a stabilizer.

Composition C was also aged in contact with AP in the same manner. The experiments used the same quantities by mass of Composition C and AP. For the uncured material, very little sample was available and so just 0.2 g was subjected to aging whereas for cured Composition C, a total sample mass of approximately 2 g was heated.

2.3 Aging of PEG

PEG with a molecular weight of 4000 Da was also subjected to aging in glass vials. The samples were PEG and PEG mixed with the solid filler particles which comprise Composition A – Al, AP and a nitramine. The samples contained equal masses of each component and the total sample mass was 2 g. Heating was carried out for four days at 80 ($\pm$0.1)°C in a block bath.

3 Results and Discussion

3.1 Aging Behavior of Compositions A, B and C

Results from ARC testing on the unaged compositions are shown in Figure 1. For Compositions B and C, exothermic data are collected as the materials start undergoing decomposition at 125–128°C with self-heating at a rate greater than 0.02°C min^{-1}. This accelerated until the reactants were consumed. In the case of Composition B, three separate reactions can be seen. The gaps in the data are where the instrument is heating the sample up to the next temperature if self-heating from the sample is not detected.

For Composition A (data in red in Figure 1), no exothermic data was collected as once a temperature of 128°C was reached, there was a sudden onset of self-heating, of the order of tens of degrees per minute, The ARC cannot maintain adiabatic conditions and the propellant probably ignited.

The exotherm for Composition C and the first thermal event for Composition B are thought to be due to the decomposition of NG in the presence of stabilizer. For Com-
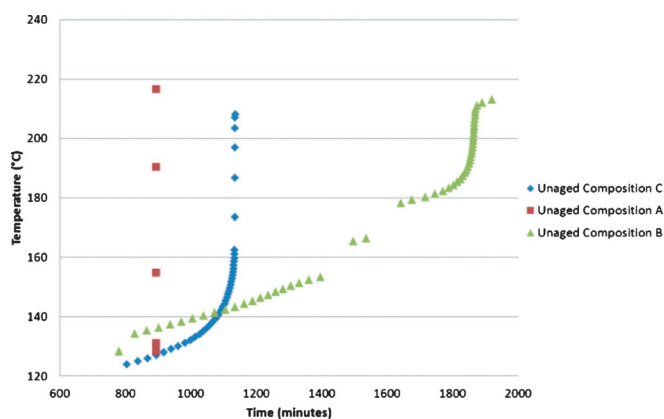

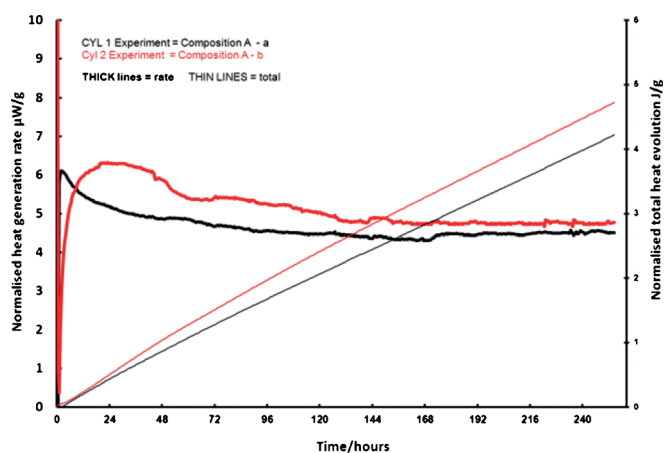**Figure 1.** ARC results from the three compositions.

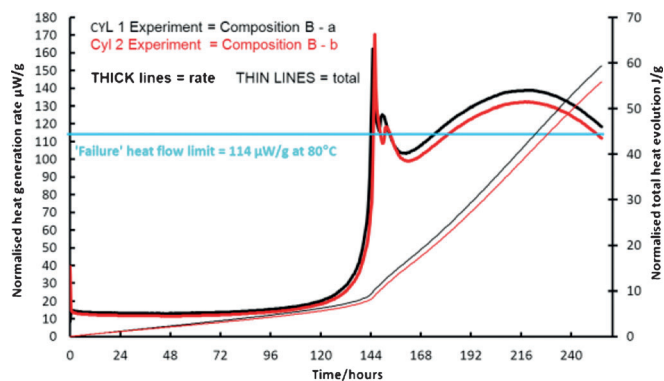**Figure 2.** HFC data for Composition A.

Figure 3. HFC data for Composition B.

position A, however, the dominant reaction is probably that of AP with the degradation products of NG as shown in Equation set 1. It is clear that when Composition A is heated up to a high temperature, it behaves differently to Compositions B and C, due to the presence of AP.

The three materials were also subjected to HFC. The data are shown in Figure 2, Figure 3, and Figure 4.
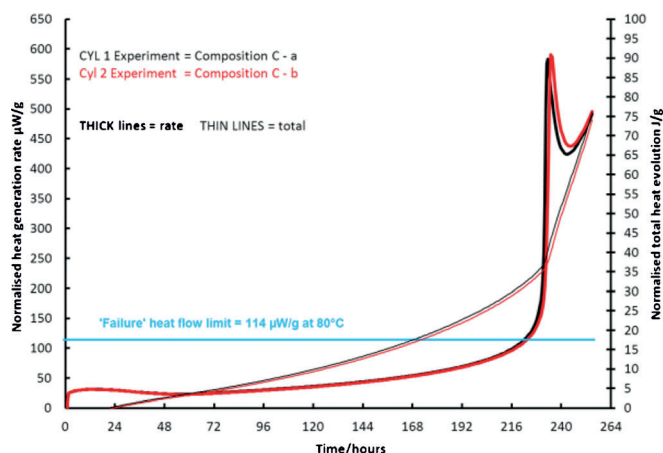
Figure 4. HFC data for Composition C.

Table 2. NMR and acidity results for samples after HFC testing.

Composition	% Soluble PEG	% 1,2-DNG	Change in pH relative to unaged material
A	1.23	0.15	−2.8
C	23.62	1.12	−5.5

Comparing Figure 2, Figure 3, and Figure 4 it can be seen that Compositions B and C exhibit very different thermal behavior compared with Composition A. The heat evolved from the Composition A samples was very low as after 10.6 days at 80 °C, less than 4 J g^{-1} of total heat was detected.

However, Compositions B and C produce significant quantities of heat, to the extent that it is so great that these samples fail the STANAG 4582 criteria for propellants and would not be considered safe to store for a further ten years at 25 °C [11].

As well as the quantities of heat from Compositions B and C compared with Composition A, the shape of the HFC traces are significantly different. The heat evolved from the Compositions B and C samples begins to rise rapidly, passes through a peak then decays. For Composition B this then quickly rises through another peak maximum whereas for Composition C, the experiment was terminated during the increase in heat generation.

When combined with the HPLC results, it would appear that the first rise in heat flow coincides with significant stabilizer depletion. It was found that for all three compositions, the pNMA stabilizer depleted in preference to the 2-NDPA which was to be expected [12]. For Composition A, at the end of the HFC test, 50 % pNMA and 87 % 2-NDPA remained. For Composition B, just 4 % pNMA survived and for Composition C there was 3 % pNMA. Neither Composition B nor Composition C contained any 2-NDPA at the end of the experiment.

The main difference between Compositions A and B is that Composition B does not contain any AP. It is unlikely that the nitramine content has an influence on the degradation of the propellant as such compounds decompose at temperatures above 200 °C [13]. It therefore appears probable that AP has a stabilizing effect when it is present as in Composition A. Its absence in Compositions B and C allows these materials to degrade rapidly at 80 °C.

When removed from the HFC, the Composition B were visibly degraded, softened and slumped, while the Composition C samples had formed a viscous liquid.

This indicates that the PEG binder had undergone significant chain scission in Compositions B and C. It is likely that acid hydrolysis of the NG had occurred, catalyzed by the breakdown products of PEG such as formic acid and water [14,15]. To confirm this, Composition A and Composition C samples were subjected to NMR and acidity testing to quantify the level of 1,2-Dinitroglycerine (1,2-DNG) arising from the acid hydrolysis of NG, the amount of soluble PEG and the change in pH levels compared to unaged material (Table 2).

Table 2 indicates that virtually all PEG in Composition C was uncross-linked and therefore soluble whereas for Composition A, very little could be extracted even after aging. Also, a greater degree of acid hydrolysis of NG to produce 1,2-DNG had occurred in Composition C compared with Composition A. Furthermore, Composition C showed a much larger fall in the pH.

The samples which had been subjected to the longest periods of aging were analyzed using GPC to quantify the length of the PEG chains (Table 3). It should be noted that the maximum molecular mass that the columns had been optimized to identify was 4000 Da. There were peaks associated with molecular weights of >4000 Da in many samples but as the columns had been developed for identifying polymers which were smaller than this, the molecular mass for such samples are reported as >4000 Da as specific values cannot be assigned.

Meanwhile, at the other end of the range, all of the samples, contained polymer chains with molecular weights of less than 400 Da. Only the largest molecular mass polymers have been used to compare samples. Finally, for this research, the molecular weight at the peak maximum (Mp) was used for comparison purposes as this gave a good indication of depolymerization. For many of the samples, the

Table 3. GPC results for Compositions A, B and C.

Composition	Aging time [d]	Molecular weight of longest polymer chain [Mp]	Molecular weight of second longest polymer chain [Mp]
A	25	1238[a]	< 400
B	21	>4000	2694[c]
C	11	>4000	3194[c]

a) Denotes broad peak, range of molecular masses are present but the length of the longest chain in this range is provided.

peaks were very broad indicating a wide range of molecular weights. Therefore the other parameters used to describe the data, such as the weight molecular mass value (Mw), varied depending on the method of data processing making comparisons difficult.

For Compositions B and C, the molecular mass of the longest chain of PEG in the unaged material was greater than 4000 Da. However, for Composition C, there was a peak due to a slightly lower molecular weight of around 3200 Da which suggests that a very small degree of depolymerization has taken place.

There was a similar result for Composition B. Simple chain scission has occurred but not the entire polymer has depolymerized into fragments with lower molecular weights.

The data for Composition A indicates that the entire PEG has depolymerized and broken down into fragments with much lower molecular weights than the original main chains.

To investigate further, PEG with a molecular weight of 4000 was heated both with and without the AP and other filler particles which comprise Composition A. The results are shown in Table 4.

When PEG was aged in isolation, there was no change in terms of the molecular weight. Additionally, Al and nitramine do not have an effect. However, with AP, the Mp value of the longest polymer species fell to approximately 1100 Da which is a significant difference.

In addition, the peaks for the AP containing samples were broad, indicating a range of molecular weights of PEG

and therefore the degradation of the polymer. The results imply that in the presence of AP, PEG no longer undergoes simple chain scission but bonds break within the main polymer chain and lower molecular weight fragments form. These results suggest that AP facilitates the degradation of PEG.

The presence of oxygen degrades PEG and ultimately it will depolymerize but it is clear from the results in Table 3 and Table 4 that this process is accelerated in the presence of AP.

The data from the HFC, HPLC, NMR and acidity testing indicated that Composition A degraded at a slower overall rate than Compositions B and C. In addition, very little soluble PEG could be extracted from Composition A after aging for 10.6 days. That which was soluble had depolymerized. In 2002, Celina et al. [16] reported that in mixtures with hydroxyl-terminated polybutadiene (HTPB), AP might act as a limited oxygen inhibitor and so slow down the oxidation of the polymer. It is possible that this is the case in Composition A. Once any PEG becomes uncross-linked, the presence of AP causes it to depolymerize. It has been found that even when Composition A has been aged for 25 days without a humidifying agent, the amount of soluble PEG is only about 1%. Therefore, it appears that AP helps to slow the degradation of Composition A relative to Compositions B and C.

3.2 Aging of Composition B and C in Contact with Inorganic Compounds

The results of aging of Composition B in contact with the different inorganic compounds are in Table 5. It should be noted that the highest standard deviation for the NG results was 0.4% whilst for the stabilizer concentrations it was 0.006%.

Table 5 illustrates that NaCl accelerated the decomposition of Composition B. The samples, which were aged in contact with this salt, were withdrawn from aging after just three days. This was because there were signs that the propellant had degraded with a significant color change in the composition and evidence of liquefaction. When Composition B was analyzed after aging there was virtually no sta-

Table 4. GPC results for PEG mixtures.

Sample	Molecular weight of longest polymer Chain [Mp]
Unaged PEG (initial state)	> 4000
Aged PEG	> 4000
Aged PEG/AP	1140[a]
Aged PEG/Al	> 4000
Aged PEG/nitramine	> 4000
Aged PEG/nitramine/Al/AP	1132[a]

a) Denotes broad peak, range of molecular masses are present but the length of the longest chain in this range is provided.

Table 5. HPLC results for Composition B samples aged in contact with inorganic salts.

Compound added to Composition B	Aging period [d]	% NG remaining	% 2-NDPA remaining	% pNMA remaining
None (Composition B only)	11	93.6	None	4.0
Resorcinol	3	80.7	82.7	95.7
AP (damp)	11	80.0	None	3.6
Calco 2246	3	78.4	72.0	10.7
AP (dry)	11	73.4	None	< 0.001
KClO$_4$	11	69.6	None	2.0
Calco 2246	10	62.0	78.7	9.6
NaCl	4	52.6	None	2.5
KClO$_4$	11	69.6	None	2.0
Ammonium carbonate	3	None	None	None

Table 6. Concentration of soluble PEG in selected samples.

Compound added to Composition B	% soluble PEG
NaCl	4.44
KClO$_4$	5.74
AP (damp)	1.62

bilizer remaining in these samples. In addition, there were significant quantities of soluble PEG (Table 6) indicating that this polymer had decomposed and the cross-links between the chains had broken.

In terms of stabilizer depletion, the addition of 'damp' AP did not appear to have a significant effect when aged in contact with Composition B. There was slightly less pNMA remaining compared to Composition B aged in isolation, as well as a lower concentration of NG in the aged samples. This may be because the NG adsorbed onto the AP which was then largely removed from the surface of Composition B before analysis. Table 6 does indicate that Composition B aged in contact with damp AP has a lot less soluble PEG compared with material aged in contact with NaCl or KClO$_4$. The results also suggest that when the AP is dried, there is a greater degree of NG and stabilizer decomposition. This may be because water can form a layer on the surface of the antioxidant inhibiting the decomposition of the AP [3].

Table 5 also demonstrates that KClO$_4$ resulted in slightly greater levels of stabilizer and NG degradation than when AP was present. Table 6 shows that this compound enhanced the decomposition of PEG compared to AP. It suggests that the ammonium component of AP might have an important role in the interaction of AP with NG.

With respect to the Calco 2246 and resorcinol results, two of the resorcinol and one of the Calco 2246 samples were withdrawn from aging after three days because the propellant looked degraded. However, it was decided to leave one sample containing Calco 2246 on aging for a further seven days. The results show that the samples containing Calco 2246 and resorcinol contained a significant amount of stabilizer although there was less NG present. Calco 2246 appears to have a protective effect on the stabilizer as after ten days, there was significantly more 2-NDPA and pNMA present than in the sample aged in isolation. The data indicated that Calco 2246 might involve different decomposition pathways for 2-NDPA and pNMA as very little of these stabilizer degradation products were detected.

Calco 2246 is added to composite propellants as an antioxidant so this might have resulted in the slower decomposition of Composition B.

In the absence of an antioxidant the aging processes for Compositions B and C at elevated temperatures may be:

1. Atmospheric oxygen initiates chain scission of PEG.
2. The cross-links between the PEG chains break, the PEG end groups are oxidized, acidic compounds and water form.

Table 7. Relative amounts of 1,2-DNG and 1,3-DNG in aged Composition C samples.

Sample	1,2-DNG	1,3-DNG
Uncured Batch 1	3.6	1.5
Uncured Batch with AP	1.0	0.3
Cured Batch 2	5.2	2.4
Cured Batch 2 with AP	4.3	1.7
Cured Batch 3	5.1	2.4
Cured Batch 3 with AP	2.7	0.9

3. The pH of the propellant is lowered.
4. NG undergoes thermolysis and acid hydrolysis at a faster rate as the pH is lower.
5. Stabilizer consumption is increased.
6. NG hydrolysis and thermolysis leads to further acidic compounds such as nitrous and nitric acid.
7. The pH of the propellant decreases further and the chain scission of PEG is enhanced.
8. The decomposition of PEG and NG is further catalyzed leading to more rapid stabilizer depletion and loss of mechanical integrity.

It is possible that both Calco 2246 and AP do not accelerate the decomposition of Composition B compared with the other inorganic salts studied because these species act as oxygen inhibitors and slow the first step in this process. Even though AP is a known oxidizer, there is some evidence that it might act as an antioxidant in some propellants. However, the actual mechanisms for this are unclear [16].

Composition C was also aged in contact with AP. The samples were held at 80 °C for 11 days prior to analysis by NMR spectroscopy. The relative amounts of 1,2-DNG and 1,3-DNG were determined (Table 7). The amounts of the DNG where normalized by a factor with the NG level set to 1000.

Table 7 indicates that the addition of AP to the binder samples reduces the hydrolysis of NG to 1,2-DNG and 1,3-DNG. This could be because the AP reacts with any nitric acid that forms but experiments are required to study this in more detail. It is possible the extract did not contain such a high concentration of the hydrolysis products as the pure binder, because some of the mass was taken up by AP. However, as a whole the results from Compositions A, B and C suggest that it is worth exploring the effect of AP on NG further.

4 Conclusions

It has been shown that AP has a significant influence on the aging behavior of propellants containing both NG and PEG. AP, or a combination of the AP and stabilizers, hinders the atmospheric oxidation of PEG. As a result, the oxidation of PEG is limited. When oxidation does occur, a small

amount of PEG undergoes chain scission and depolymerizes. As the degree of PEG degradation is very small, the pH of the mixture does not fall substantially.

Simultaneously, NG decomposes. AP inhibits this process at low temperatures in the short term (80 °C or less) as it might react with the nitric acid so slowing the acid hydrolysis of NG. The overall result of these processes is that Composition A ages less rapidly than Compositions B and C at lower temperatures.

However, if AP containing propellants are heated at high temperatures (greater than 100 °C), as occurs in the ARC test, or for long periods at lower temperatures such as at 80 °C, then it is thought that perchloric acid accumulates. This either causes rapid acid hydrolysis of NG or there is direct reaction between the oxides of nitrogen and the products from the decomposition of NG. This leads to the fast degradation of Composition A and ultimately, the propellant suddenly ignites.

Symbols and Abbreviations

Al	aluminum
ARC	accelerating rate calorimetry.
Calco 2246	2-tert-butyl-6-[(3-tert-butyl-2-hydroxy-5-methylphenyl)methyl]-4-methylphenol
CMCDB	composite modified cast double base
Da	daltons
DBP	dibutyl phthalate
DNG	dinitroglycerine
1,2-DNG	1,2-dinitroglycerine
1,3-DNG	1,3-dinitroglycerine
HTPB	hydroxyl-terminated polybutadiene
IC	ion chromatography
HPLC	high performance liquid chromatography
Mp	molecular weight at height of peak
NC	nitrocellulose
2-NDPA	2-nitrodiphenylamine
NG	nitroglycerine
NMR	nuclear magnetic resonance
pNMA	*N*-methyl-4-nitroaniline
RDX	cyclo-1,3,5-trimethylene-2,4,6-trinitramine
TAM	thermal activity monitor
TTMS	tetrakis(trimethyl)silane

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