

AGEING STUDIES ON A COMPOSITE PROPELLANT
CONTAINING A NEW ORGANO-COPPER BURN
RATE CATALYST

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B.L. HAMSHERE AND P. RAWSON

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Ageing Studies on a Composite Propellant Containing a New Organo-Copper Burn Rate Catalyst

B.L. Hamshire and P. Rawson

MRL Technical Note
MRL-TN-627

Abstract

The effect of a new organo-copper burn rate catalyst on the ageing properties of an HTPB/AP based composite propellant has been studied and compared with a similar propellant containing copper chromite. The degradation of the propellant surface under conditions of thermo-oxidation was studied by monitoring oxygen uptake using a gravimetric technique. The oxidative induction times for the reaction were determined for a series of elevated temperatures and the data used to predict the storage life of the propellant at ambient temperatures using an Arrhenius plot. It was shown that the storage life of a copper chromite propellant was approximately 7.5 years at 20°C while the corresponding organo-copper propellant had a storage life of approximately 49 years.

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Contents

1. INTRODUCTION	5
2. EXPERIMENTAL	6
2.1 <i>Materials and Equipment</i>	6
2.2 <i>Sample Preparation</i>	7
2.3 <i>Gravimetric Methods</i>	8
2.3.1 <i>Thermogravimetric Analyser</i>	8
2.3.2 <i>Manual Weighing</i>	8
3. RESULTS AND DISCUSSION	9
3.1 <i>Thermogravimetric Analyser</i>	9
3.2 <i>Manual Weighing</i>	11
3.3 <i>Comparison of Manual and Automatic Weighing Data</i>	16
3.4 <i>Surface Hardening</i>	17
3.5 <i>Prediction of Ambient Storage Lifetimes</i>	19
4. CONCLUSIONS AND RECOMMENDATIONS	20
5. ACKNOWLEDGEMENTS	21
6. REFERENCES	21

Ageing Studies on a Composite Propellant Containing a New Organo-Copper Burn Rate Catalyst

1. Introduction

Copper compounds, such as copper oxide and copper chromite, find use as burn rate catalysts in some composite propellant formulations due to their ability to produce an enhanced burning rate while also promoting a very low burn rate pressure exponent over an extended pressure range [1]. However, these burn rate catalysts have a deleterious effect on the ageing properties of hydroxyl terminated polybutadiene, HTPB, based composite propellants.

The mode of action of this process is to catalyse the thermo-oxidative degradation of polybutadiene type binder materials. During the process free radical attack at double bond sites in the polymeric backbone cleaves the double bond permitting cross-linking to occur with an accompanying absorption of oxygen. This occurs after the depletion of the antioxidant and is manifested by a rapid increase in the surface hardness of the propellant. Because the hard layer is very susceptible to cracking and could result in motor failure if undetected, an understanding of the process and the rate at which it occurs is essential.

As these catalysts are essential in certain applications, an investigation was undertaken to identify copper compounds which were effective burn rate catalysts but substantially less effective in promoting the degradation of HTPB. Monash University, under a research contract [2], synthesised a range of potential copper catalysts. Screening these compounds for their affect on the burning and ageing properties of HTPB propellants showed that a compound consisting of dinuclear copper (II) complexed with compartmental tridentate ligands had promising properties. Initial testing [3] showed the catalyst was nearly as effective as copper chromite in enhancing the burn rate, but far superior in terms of the propellant's ability to resist mechanical property change during ageing. More precise data on the relative rates of propellant degradation were required and that work is the subject of this report.

The object of this extended investigation is to compare the surface ageing processes for propellants containing copper chromite and the new organo-copper compound. The propellant binder is protected initially by the antioxidant. Once the antioxidant has been consumed or reduced to a very low level, the binder cross-linking reaction occurs accompanied by the onset of oxygen uptake. Thus, a measure of the time to onset of oxygen uptake at various temperatures can be used to compare the relative rates of the initial ageing process of propellants containing copper compounds. The process of oxygen absorption can be measured experimentally by volumetric, manometric and gravimetric methods. In this work the gravimetric technique is employed.

2. Experimental

Previous experience [4] involving the accelerated ageing of composite propellants containing copper chromite had shown that, for samples exposed to an oxygen containing atmosphere, the surface of the propellant degraded to a greater extent than the bulk. This was observed, after a certain inhibition time, as a rapid increase in surface hardness of the propellant. The inhibition time was noted to be dependent on the temperature at which the accelerated ageing trial was conducted. For instance, at 60°C it took approximately 10 weeks for the rapid surface hardening phenomenon to occur. This duration was seen as impractically long to enable a full range of laboratory tests to be conducted and posed many problems in developing the experimental technique. For these reasons it was proposed that the initial experiments be performed at 100°C to enable the experimental method to be established before conducting additional runs at lower temperatures.

2.1 Materials and Equipment

HTPB	hydroxyl terminated polybutadiene Atochem R45M Lot 607145
DDI	dimeryl diisocyanate General Mills 1410 Lot 4F10835
AO2246	2,2'methylene-bis-(4-methyl-6-tert.-butyl-phenol) Lowinox 22M46, Chemische Werke Lowi
AP	ammonium perchlorate Kerr-McGee 200 µm, rotary rounded, used as received nom. 16 µm produced by KEK milling 400 µm as received material
Copper Chromite	Harshaw, Cu0202P (nominal composition 82% CuO, 17% Cr ₂ O ₃)

Organo-Cu Compound	Monash University - contains 2 Cu atoms/molecule within a tridentate compartmental ligand
Mixer	Baker-Perkins 1 pint, planetary action thermostatted to 60°C vacuum capability - 10 torr or better
Gravimetric Equipment	Perkin Elmer TGS-2 - thermogravimetric analyser consisting of Analyser unit, Heater control unit and Balance control unit; Nicolet 4094 Digital Storage Oscilloscope Amplifier, x 20
Oven	small air circulating oven capable of maintaining temperatures to 100°C ± 1°C
Balance	Sartorius five decimal place electronic balance

2.2 Sample Preparation

Four propellant formulations were processed as 500 g batches in the Baker-Perkins mixer, cast into moulds measuring 125 × 87 × 25 mm and cured for seven days at 60°C. As copper chromite is a more effective burn rate enhancer than the organo-copper compound two formulations containing the latter were manufactured: one on an equivalent weight basis to the copper chromite and the other with an amount to produce an equivalent burn rate to the copper chromite composition. The fourth formulation contained no burn rate catalysts and was included to give base line data. The basic formulation was as follows:

17% HTPB, cured with DDI, using a 1:1 NCO/OH equivalence ratio.
Antioxidant, AO 2246 was added at 1% by mass of the binder.
83% AP, a bimodal blend of 200 µm and 16 µm particle sizes, ratio 71:29

The burn rate catalyst was added at the expense of the AP, 0.5% by mass for the copper chromite and 0.5% and 1.0% by mass for the organo-copper compound.

For the thermogravimetric analyser (TGA) method, only small samples of propellant could be accommodated in the sample pan. Thus, the test samples were cut from the parent block of propellant as thin slivers having a total mass of approximately 30 to 40 mg. As, after the initial weighing, there was no further handling of the sample there was little concern that the large surface area to volume ratio would result in the progressive loss of dislodged AP crystals.

For the manual weighing method the samples were machined from the parent block of propellant as 10 × 7 × 1.9 mm slabs.

2.3 Gravimetric Test Methods

Two gravimetric techniques were employed, the first, an automated thermogravimetric analyser and the second, a manual weighing technique.

2.3.1 Thermogravimetric Analyser

Approximately 30 to 40 mg of propellant slivers were accurately weighed into the aluminium sample pan and placed in the stirrup of the microbalance. The mass of the propellant was electronically suppressed with the balance control unit. The pen of the millivolt recorder monitoring the change in mass of the sample was positioned to one side of the chart to enable maximum use of the chart to be made without scale change.

The furnace temperature was controlled at 100°C by the Heater Control Unit and checked by monitoring the thermocouple output. A 160°C/minute heating rate was used to raise the furnace temperature to its control setting. The apparatus was trickle purged with dry air using a bubbler bottle on the outlet to observe the flow, approximately one bubble every five seconds.

As there was a considerable delay between starting each run and the time at which the weight gain reaction occurred it became the practice not to engage the chart drive of the weight monitoring recorder until shortly before the reaction was anticipated. This was a practice which improved with experience but which was still unable to ensure trouble-free collection of data. Each test was allowed to run until the weight gain reaction had stopped or fallen to a very low rate.

Due to difficulties arising from the collection of data with this technique the weight monitoring chart recorder was replaced by a Nicolet digital storage oscilloscope. As the maximum output voltage of the electrobalance was 10 mV and only a small portion of this range was used, it was necessary to amplify the voltage prior to inputting to the Nicolet which had a minimum full scale voltage range of - 100 mV to + 100 mV. The Nicolet was set to acquire data every 10 seconds; it had sufficient capacity to store data for a run in excess of 24 hours and could be down loaded and reset each day.

2.3.2 Manual Weighing

1.4 to 1.9 g of propellant was accurately weighed into an aluminium foil pan. With the increased handling required for this test propellant slab measuring approximately 10 × 7 × 1.9 mm were considered more appropriate in minimising possible weight loss due to dislodged AP crystals. Four pans of propellant were placed on small trays and loaded into a pyrex tube fitted with a stopper containing a 6 mm diameter orifice. It was necessary to protect the propellant, by means of a pyrex tube, from the turbulent atmosphere of the air circulating oven to prevent enhanced oxidation of the samples. The tubes were stored in an oven controlled at either 80, 90 or 100°C. Three samples of each propellant were tested. As data could not be collected during the weekend the starting times of the three tests were staggered by one day to ensure that the weight increasing reaction would be monitored for at least one of the samples.

3. Results and Discussion

Considerable work over the last 25 years has been conducted into examining the effect and mechanism of thermal oxidative degradation of polymeric materials [5-12]. Much of this work has been directed at improving their high temperature and long term ageing properties and has led to the development of many novel techniques for testing polymer resistance to oxidising atmospheres at elevated temperature. Many polyolefins undergo thermal oxidation by a process of hydrocarbon free radical formation, often catalysed by the presence of transition metal compounds, which then forms a peroxy radical and subsequently a hydroperoxide. This enables the process to be studied by volumetric techniques by measuring the volume of oxygen absorbed with a manometer [5], infra red spectroscopy by, for example, monitoring the growth of carbonyl absorption as degradation products such as acids, aldehydes and ketones are formed [6], thermal analysis using such techniques as differential thermal analysis and differential scanning calorimetry [9], chemical analysis by measuring, for example, the change in concentration of antioxidant [4] and gravimetric methods by measuring weight gain or loss.

3.1 Thermogravimetric Analyser

As the uptake of oxygen by the sample during the test was accompanied by a corresponding weight increase, the use of a thermogravimetric analyser operating in the isothermal mode appeared to offer an excellent means of performing both accelerated ageing and continuously recording the mass of the sample. Limited success was achieved running the propellants containing 0.5% copper chromite and 0.5% and 1.0% organo-copper compound at 100°C before major equipment malfunction prevented any further testing. A successful run with the propellant containing no burn rate catalyst was not achieved.

The percentage weight increases of the three propellant samples are shown in Figure 1, recorded as a function of the time exposed to the test temperature. The data are characterised by a period of time over which no significant weight change occurs followed by a period of rapid weight gain. The time until the weight increasing reaction commences is referred to as the oxidative induction time, OIT, and corresponds to the time during which the antioxidant in the propellant binder is exhausted. The weight increasing reaction for the copper chromite propellant occurs after a shorter OIT than the two organo-copper propellants. The organo-copper propellant with the higher concentration of catalyst appears to undergo a shorter induction time. The difference in times is small, about 0.3 days. Although this may have been due to small changes in the test conditions, e.g. temperature and gas flow in the TGA, or experimental error, the trend was repeated for the OITs obtained from the manual weighing technique at 80 and 100°C (Table 2). This suggests that OITs are concentration dependant; the more copper compound present the shorter the OIT (the 90°C results exhibit the same OITs for both concentrations). The OITs determined from the data in Figure 1 are presented in Table 1.

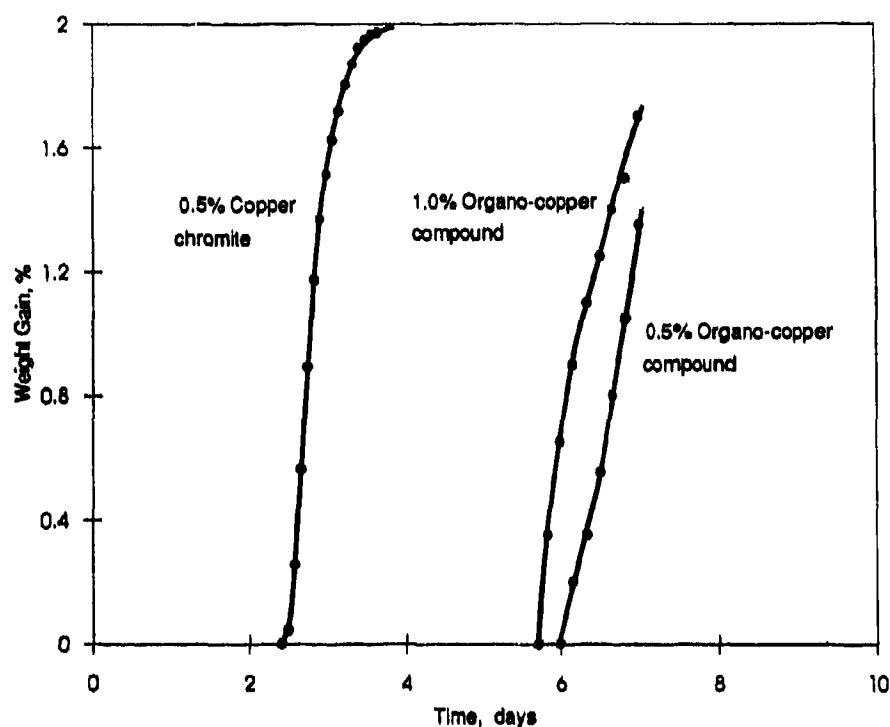


Figure 1: Oxygen uptake curves for propellants at 100°C. Determined using a thermogravimetric analyser.

Table 1: Oxidative induction times from TGA data (100°C)

Propellant	Time to Onset of Wt Gain (day)
With 0.5% Copper Chromite	2.42
With 0.5% Organo-Copper	6.00
With 1.0% Organo-Copper	5.70

All of the runs were terminated prior to the completion of the weight gain reaction due to recorder problems. Consequently, the extent of the propellant weight gain for the complete reaction could not be determined, although the copper chromite results appear to be levelling off at approximately 2%.

3.2 Manual Weighing

After the malfunction of the TGA, the manual weighing technique was adopted in an attempt to gather further data on the oxygen uptake process at additional temperatures. Although it suffered from the lack of a means for automatically recording mass changes, the method was simple and fairly reliable.

The weight changes, exhibited by each of the four propellants, viz no catalyst, 0.5% copper chromite, 0.5% organo-copper and 1.0% organo-copper, when tested at 80, 90, and 100°C are presented in Figures 2 to 4 respectively. Examination of the weight gain curves shows the presence of either of the burn rate catalysts is necessary for the propellant to undergo the weight gain reaction during the period of the test. As would be intuitively expected, the higher the test temperature the more rapid is the degradation process, as evidenced by the shorter OITs. It is also apparent that the presence of copper chromite in the propellant promotes a more rapid onset of the weight gain reaction than the organo-copper compound and causes the reaction to be completed in a shorter time. Apart from the 90°C results the data suggests the OITs are concentration dependant. However, as the effect is small it may be that the concentration of catalyst has reached a saturation level. This effect has been noted by other workers, see Figure 7 in reference 10. The data from Figures 2 to 4 are summarised in Table 2.

Table 2: Summary of Oxygen Uptake Data Obtained from Manual Weighing Method

Propellant	Oxidative Induction Time (days)			End of Reaction (days)			Max % Wt Gain		
	Temp °C			Temp °C			Temp °C		
	80	90	100	80	90	100	80	90	100
No Catalyst	-	-	-	-	-	-	0	0.05	-0.05
0.5% Copper Chromite	17.7	7.7	3.5	20.0	9.3	4.0	0.79	0.73	0.60
0.5% Organo-Copper	30.5	12.7	7.5	36.4	15.8	11.1	1.03	0.82	0.90
1.0% Organo-Copper	30.1	12.7	7.0	35.4	15.9	10.1	1.00	0.82	0.85

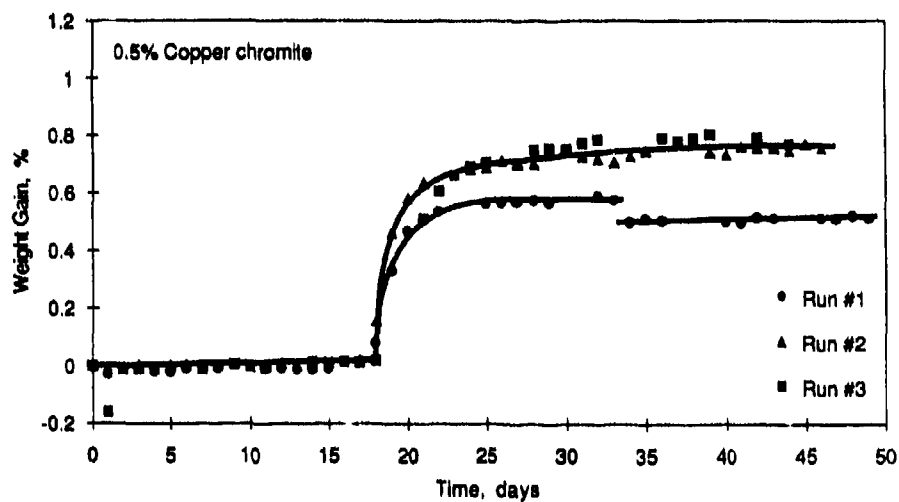
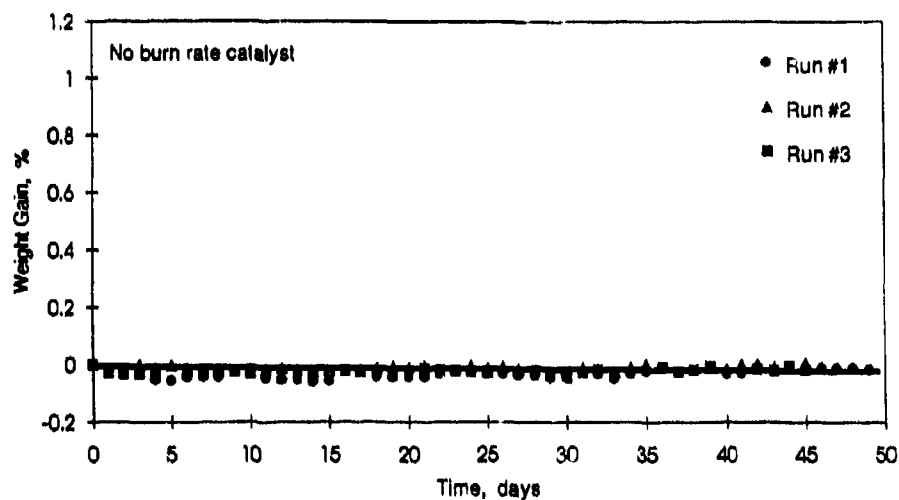


Figure 2: Oxygen uptake curves for propellants at 80°C. Determined using manual weighing technique.

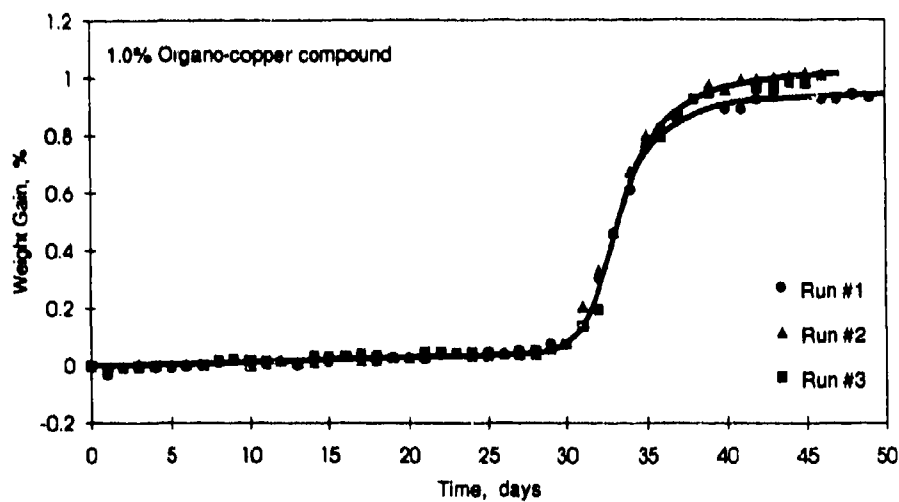
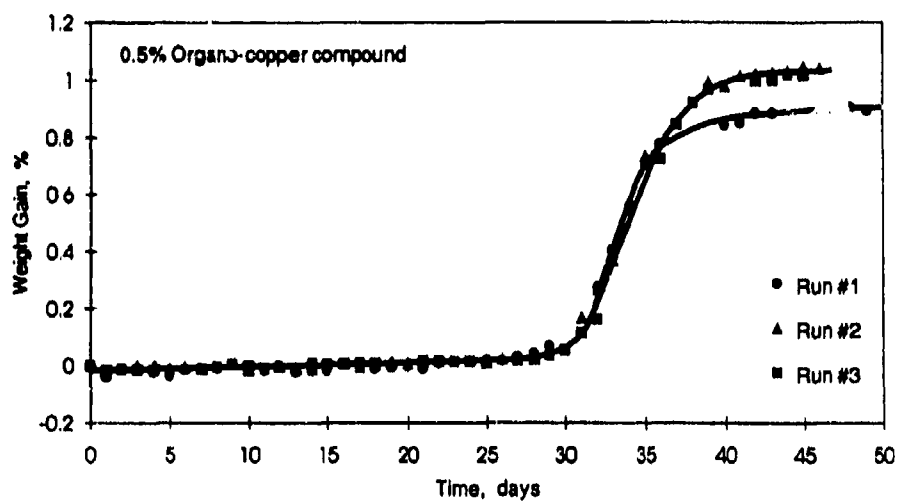


Figure 2 (Contd): Oxygen uptake curves for propellants at 80°C. Determined using manual weighing technique.

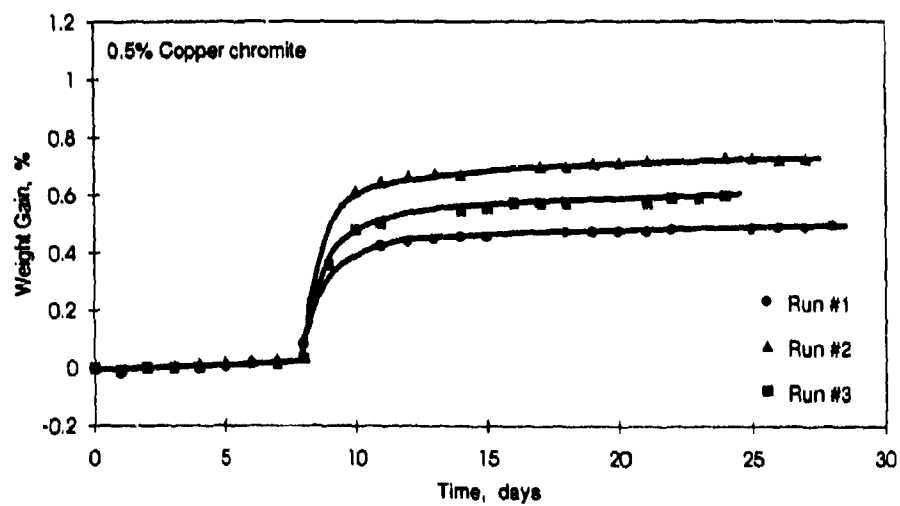
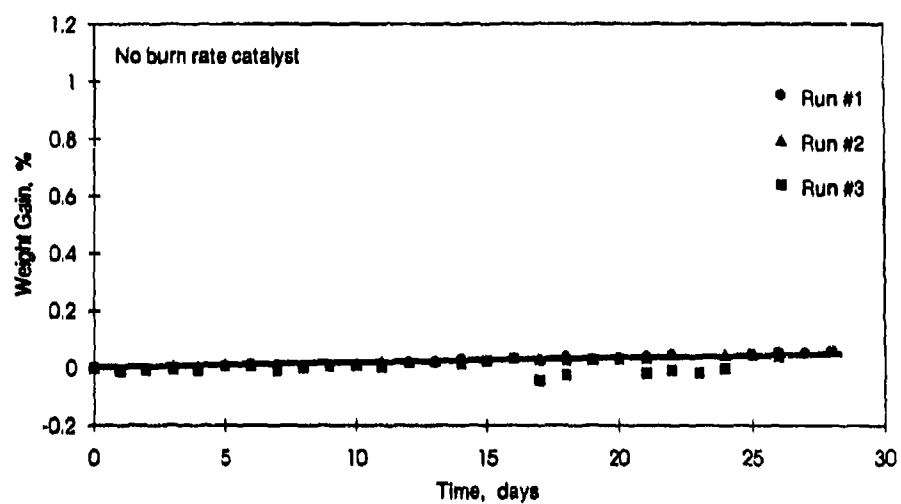


Figure 3: Oxygen uptake curves for propellants at 90°C. Determined using manual weighing technique.

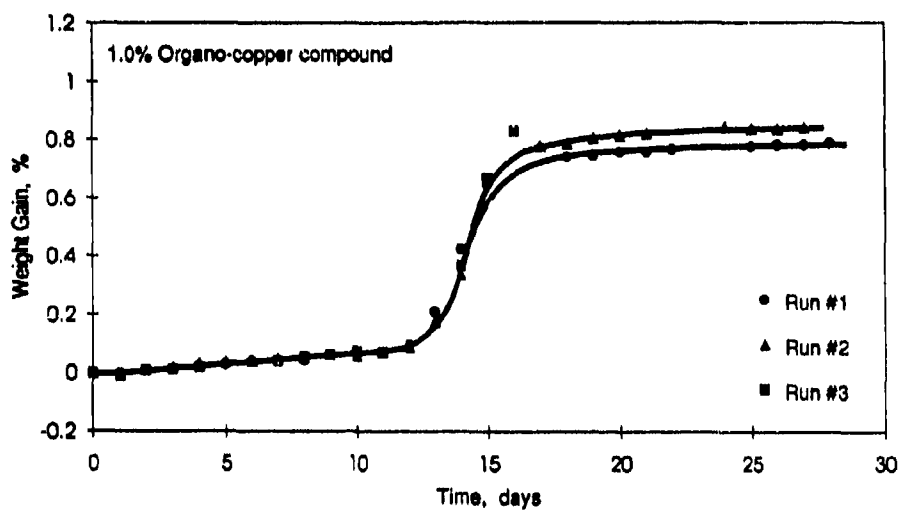
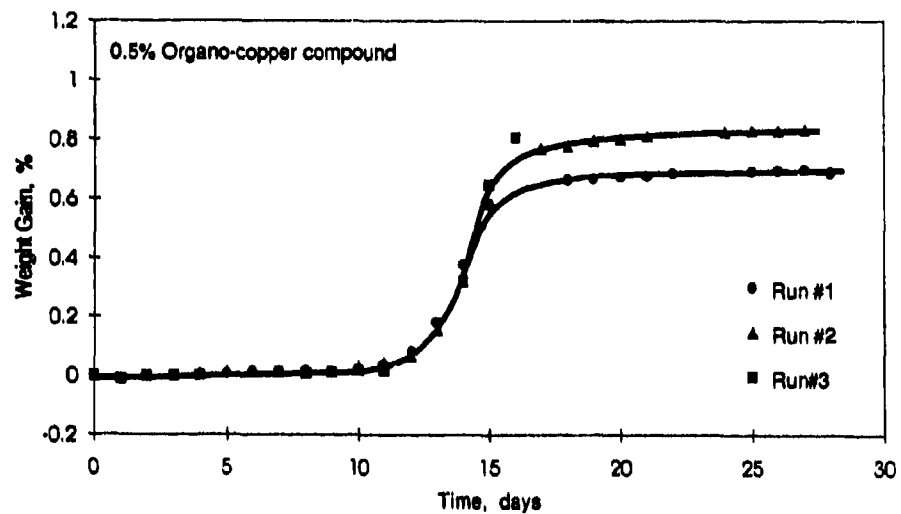


Figure 3 (Contd): Oxygen uptake curves for propellants at 90°C. Determined using manual weighing technique.

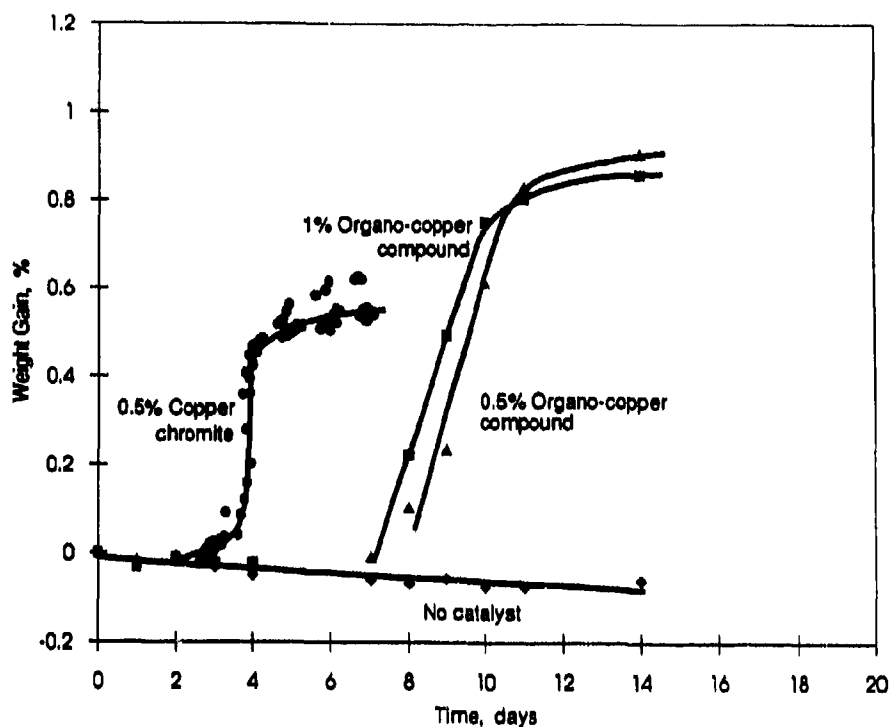


Figure 4: Oxygen uptake curves for propellants at 100°C. Determined using manual weighing technique.

3.3 Comparison of Manual and Automatic Weighing Data

There is an obvious discrepancy in the data determined at 100°C for the TGA and manual weighing methods. The OITs determined using the TGA are consistently shorter than those from the manual weighing method and the weight increase experienced by the propellant is greater in the case of the TGA tests. The cause of this difference is believed to be due to the sample size ($10 \times 7 \times 1.9$ mm) used in the manual weighing tests. Due to the relatively large dimensions of these samples, atmospheric oxygen was unable to diffuse to the centre of the sample and thus degrade all of the antioxidant. Therefore, binder in this region would be unaffected and not show a weight gain and thus, result in a lower overall percentage weight gain for these samples. The reason for the shorter OITs when using the TGA method is thought to be the reverse of the above. As these samples were very thin (about 0.3 to 0.4 mm) oxygen diffusion throughout the samples would have been more rapid and the depletion of the antioxidant throughout the samples would have

been more rapid and the depletion of the antioxidant would have occurred faster. With hindsight, it would appear preferable to use more bulky test samples as the results should be more representative of mass propellant.

3.4 Surface Hardening

The onset of cross-linking, i.e. surface hardening, could also be detected, although with less precision, by measuring the surface hardness of the propellant. This has previously been undertaken for 0.5% copper chromite propellant aged at 60 and 70°C and for 1.0% organo-copper propellant aged at 60°C and the data is presented in Figure 5. The time required for the onset of the surface hardening reaction taken from the propellant hardness data in Figure 5 is given in Table 3. The degree of uncertainty in these values is probably of the order of 3 to 4 days as the hardness readings were only taken at weekly intervals.

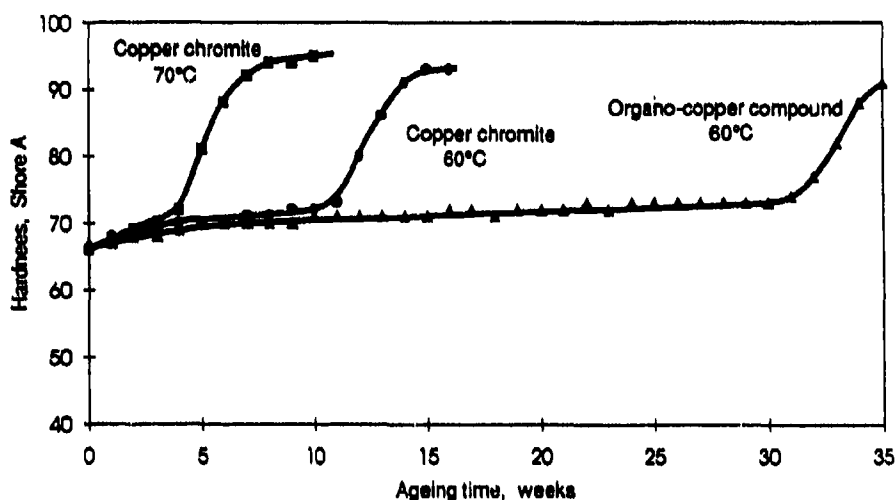


Figure 5: Surface hardness data for copper chromite and organo-copper compound as a function of time and temperature.

An Arrhenius plot (Fig. 6) was made of log inverse OIT against inverse absolute temperature for the propellants containing 0.5% copper chromite and 1.0% organo-copper compound using the data taken from Table 2 (80, 90 and 100°C) and Table 3 (60 and 70°C). The OIT corresponds to the time at which all of the surface antioxidant has been consumed (total antioxidant for thin sections) and the binder material starts to undergo a cross-linking reaction. Hence, the inverse of this time is proportional to the rate at which the antioxidant is consumed.

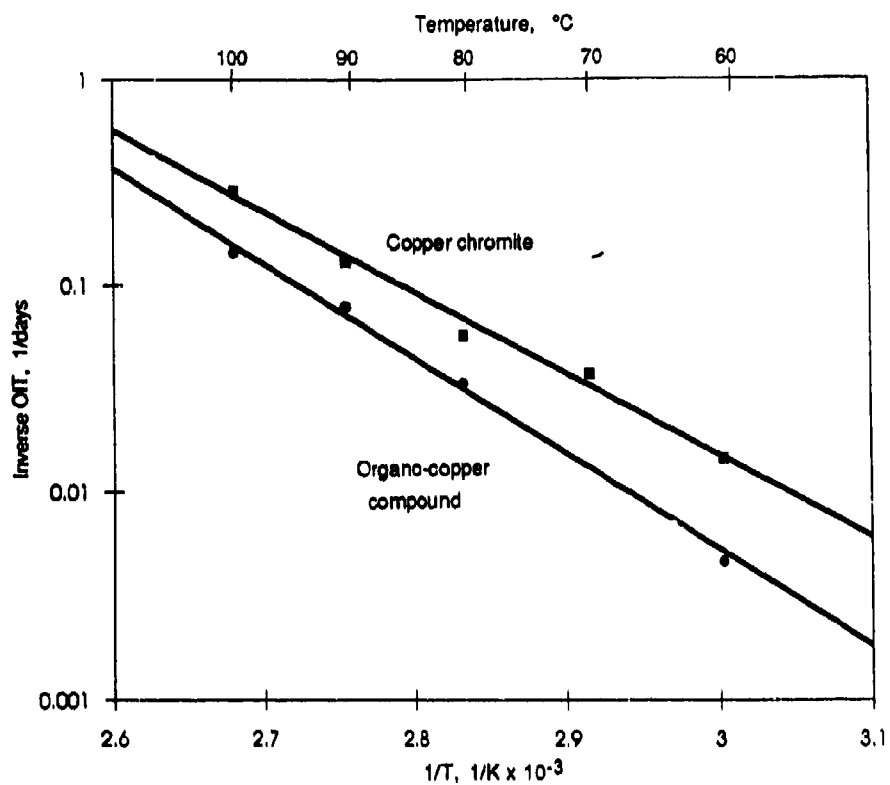


Figure 6: Plot of log inverse oxidative induction time versus inverse absolute temperature.

Table 3: Onset of a surface hardening from Shore A hardness measurements

Propellant	Onset of Hardening (Days)	
	60°C	70°C
With 0.5% Copper Chromite	70	27
With 1.0% Organo-Copper	220	-

Applying linear regression analysis to Figure 6 gave the following equations for the lines of best fit:

$$\text{Copper chromite: } \log 1/t = -3905.27(1/T) + 9.889893 \quad [R^2 = 0.988593]$$

$$\text{Organo-copper: } \log 1/t = -4723.90(1/T) + 11.86907 \quad [R^2 = 0.995359]$$

where t is the time in days and T is the absolute temperature, K.

3.5 Prediction of Ambient Storage Lifetimes

Using the above equations, predictions were made for the time required for each propellant to commence rapid surface hardening at more usually encountered temperatures. This was undertaken for 20, 25 and 30°C and the results are given in Table 4.

Table 4: Predicted Times for the onset of Surface Hardening

Temperature °C	Time to Onset of Surface Hardening (days)	
	Copper Chromite	Organo-Copper
20	2746	17925
25	1641	9615
30	997	5265

It can clearly be seen from these predictions that propellants containing the organo-copper compound have a far greater life at normal storage temperatures than copper chromite propellants, e.g. at 20°C the onset of hardening will occur after 2746 days (7.5 years) for copper chromite propellant or 17925 days (49.1 years) for organo-copper propellants.

To substantiate these predictions the hardness of propellant stored for varying times at ambient temperature in an explosives magazine was measured. All of the propellants were based on the composition HTPB/DDI/AP/copper chromite and contained the antioxidant AO2246 (at 1% by mass of the binder) although the amount of binder and other minor ingredients differed. The propellant was wrapped in aluminium foil and Kraft paper and stored in a wooden magazine box housed in a brick magazine (DSTO magazine area, Smithfield, SA). A record of the environmental conditions (temperature and humidity) during storage was not available. The surface hardness of the stored propellant is given in Table 5.

The information in Table 5 indicates the propellant underwent the surface hardening reaction between 1637 and 1867 days (4.5 and 5.1 years). Comparison with the data in Table 4 shows the predicted OIT at 25°C coincides quite closely with the shorter of the experimental data. These findings appear consistent, particularly when it is noted that an accurate knowledge of the thermal history of the stored propellant is not known.

Table 5: Surface Hardness of Propellants Containing Copper Chromite Stored at Ambient Conditions

Cast Number	Storage Time at Ambient Temperature (days)	Hardness (Shore A)
3955	2586	98
4150	2270	97
4403	1903	93
4434	1867	95
4550	1637	75

4. Conclusions and Recommendations

In terms of their ability to resist surface degradation caused by thermo-oxidation, HTPB based propellants containing the new organo-copper burn rate catalyst are far superior to those containing copper chromite. In the absence of either of the copper containing catalysts no surface degradation process is observed. This indicates an extremely long storage life for uncatalysed propellants at ambient temperatures.

The method of monitoring the oxygen uptake by means of a gravimetric technique is well suited to studying the surface degradation of these propellants. By suitable refinement and the addition of an automated weight recording device the technique could also be used to determine rate constants for the antioxidant consumption reaction and the surface hardening (cross-linking) reaction.

Measuring the weight increase as a function of time for a propellant containing copper chromite subjected to elevated temperature ageing conditions enables a prediction to be made for the onset of surface hardening, i.e. the OIT, at ambient temperature that is consistent with experimental data. For propellant containing copper chromite, stored at 20°C, a rapid deterioration of the surface properties will occur after approximately 7.5 years. For the propellant containing the new organo-copper compound it is predicted this time will be extended to approximately 49 years.

The method offers the possibility of determining the remaining storage life of propellants that contain copper compounds and are sensitive to the formation of a surface "crust". By taking a small sample of propellant from the surface of a propellant charge whose thermal history is not precisely known and measuring the OITs at various temperatures the remaining ambient temperature storage life could be estimated. It should be noted that although the onset of surface hardening may be a useful indicator of the end of a propellant's storage life other considerations

may have to be taken into account. For instance, a mechanical property of the propellant may be a critical limiting factor and have to be monitored closely to ensure it does not fall outside established safety limits.

5. Acknowledgements

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Copper catalysts

ABSTRACT

The effect of a new organo-copper burn rate catalyst on the ageing properties of an HTPB/AP based composite propellant has been studied and compared with a similar propellant containing copper chromite. The degradation of the propellant surface under conditions of thermo-oxidation was studied by monitoring oxygen uptake using a gravimetric technique. The oxidative induction times for the reaction were determined for a series of elevated temperatures and the data used to predict the storage life of the propellant at ambient temperatures using an Arrhenius plot. It was shown that the storage life of a copper chromite propellant was approximately 7.5 years at 20°C while the corresponding organo-copper propellant had a storage life of approximately 49 years.

Ageing Studies on a Composite Propellant Containing a New
Organo-Copper Burn Rate Catalyst

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