

AUSTRALIAN TEST PROCEDURES FOR DETERMINATION OF  
COMPATIBILITY AND STABILITY OF MILITARY EXPLOSIVES

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ABSTRACT

Australian procedures for investigation of compatibility and stability of military explosives have been derived from those employed in other countries, particularly UK. Recently there has been a deal of interest in other tests which might supplement and complement these and provide a more rapid indication of compatibility. In this paper the tests used are described and are illustrated by recent investigations which have been undertaken.

1. INTRODUCTION

Most explosive stores for Australian military use are either foreign weapons bought overseas and imported in a ready to use condition or are weapons of foreign design manufactured in Australia under licence. A lesser number of munitions are local in both design and manufacture. It has usually been a requirement that materials, both explosive and inert, which are specified in designs manufactured under licence should meet the original foreign specification. It is however recognised that Australian raw materials, particularly gun propellants, and a number of commercial sealants and coatings, are not necessarily identical to those used overseas. It is then mandatory to ensure that any permitted deviation from specification does not compromise either serviceability or performance. This results in a continual

requirement to investigate the compatibility of a fairly wide range of inert compositions with formulations from across the complete spectrum of energetic materials.

In these investigations techniques which are basically common to most explosives laboratories have been employed. The most frequently used tool has been the vacuum stability test. This is still the basic method used for high explosives, propellants and gunpowder and is complemented by a repeat examination after accelerated storage. Contrary to US practices, vacuum stability testing is never used in investigations involving primary explosives. Thermal analysis, particularly differential scanning calorimetry (DSC), is used routinely to detect cases of gross incompatibility. More subtle interactions are being monitored using DSC to measure changes in

kinetic parameters during thermal decomposition.

## 2. HIGH EXPLOSIVES

As mentioned above vacuum stability is the basic test used. A 5g sample (5% inert 95% HE) is tested over 40 hours at 100°C. An increase in gas volume of 4 cm<sup>3</sup> or more relative to 5g of HE tested alone is judged a fail. As a supplementary test, pellets of HE in contact with inert are placed on accelerated storage under conditions of both high and low relative humidity. On completion of storage, explosive is removed from the contact surfaces by microtome and subjected to vacuum stability testing (40 hours at 120°C).

Performance of this test has been in accordance with UK Chemical Inspectorate Laboratory Method M240/61(1). Some of the modifications to this method which have recently been developed in UK (2) are being adopted. Similar heating baths have been commissioned and the traditional mercury manometers are being replaced by pressure transducers. Control, logging and processing of output from these will be by a microcomputer currently being designed and constructed.

At the moment no correlation between the volume of gas evolved in vacuum stability tests and changes in kinetic parameters as measured by DSC is attempted. As a result changes in kinetic parameters have not yet been used to help in quantitative assessments of suitability or otherwise of various combinations. Data is being collected for systems which yield a moderate volume of gas when examined in the vacuum stability test. It is hoped that this data may be used to establish criteria by which quantitative

compatibility judgements can be made solely on the basis of thermal analysis results.

## 3. PROPELLANTS

Over the last six years the UK silvered vessel test for propellant stability and compatibility has been abandoned in favour of an extended vacuum stability test. The silvered vessel test had been found to have a number of inherent shortcomings not the least of which was the 500 hour test period. Also although it was possible to arrange in relative order those samples in which some degree of fuming or self heating was detected no ordering could be applied to samples which passed test. While the procedure which has been adopted offers advantages over silvered vessel testing it does not provide an ideal solution. It has been pointed out by many people that the fundamental premise of vacuum stability testing - incompatibility will result in the formation of non condensable gases - may not always be valid. In a number of instances significant decreases in gas volume have been obtained.

The test which has been adopted employs a longer heating period at lower temperatures than that of the US MIL-STD-286B Method 403.1.2. Samples (4.75g propellant, 0.25g inert) are heated at 90°C (single base propellants) or 80°C over a period of 168 hours. Regular readings are taken throughout the test period. Under these conditions gas volumes in the range 1.0 to 7.0 cm<sup>3</sup> have been obtained for various propellant samples in normal condition (Figures (1 and 2)). Examples of compatibility examinations are shown in Figures (3, 4 and 5). Figure (3) shows the test applied to the compatibility of Australian

manufactured M7 propellant with Silastic 734 RTV silicone rubber cured for 24 hours (minimum recommended) and for 7 days. This combination easily passes test. In Figure (4) it can be seen that although there has been some reaction between NACO propellant and Loctite 312 adhesive (uncured) this is not excessive. In contrast Figure (5) shows results from examination of MNF 2P/S propellant (16.5% nitrocellulose, 21% nitroglycerine, 55% nitroguanidine, 7.5% carbamate, 2%  $K_2SO_4$  then added) with a commercially supplied phenol formaldehyde based resin. If this resin is insufficiently cured there is extensive reaction with a considerable volume of gas generated. Further curing of the resin reduces interaction until the combination passes examination.

This test is supplemented by a study of deterioration of propellant samples after accelerated storage. Tests usually performed are extended vacuum stability, analysis of stabilizer content and thermal analysis. Results obtained from examination of NH 055 propellant (86% NC (13.2%N), 10% DNT, 3% DBT 1% diphenylamine) with candidate moth proofing agents for felt in ammunition boxes are typical (Table (1), Figure (6)). In these results and in thermal analysis examination (ignition temperature, activation energy) differences were minor and all three combinations were acceptable.

#### 4. PRIMARY EXPLOSIVES AND PYROTECHNICS

Analysis and functioning tests after accelerated storage are complemented by thermal analysis studies. Functioning tests include:

temperature of ignition  
ignition delay

ball and disc test of sensitivity  
stab sensitivity  
for primary explosives and,  
temperature of ignition  
electric spark test  
lead tube burning rate  
Rotter impact test

for pyrotechnic delay compositions. Analysis and performance evaluation after accelerated storage is a lengthy process and in the past has been the only means of investigation available for these classes of explosives. It is hoped that the rapid results available from thermal analysis will be of most use with these compositions.

As in other laboratories mercuric 5 nitrotetrazole has been of interest as a replacement for lead azide in stab sensitive compositions. Two such compositions (Table (2)) have been prepared and compatibility examined. Compositions were pressed into 2.3 gr copper detonator cups onto inert lead oxide at 35,000 psi. Stab sensitivity before and after accelerated storage at 75°C is shown in Table (3). Both compositions were examined by DSC. Exothermic decompositions were recorded at 150°C, 200°C and 290°C for tetracene, mercuric 5 nitrotetrazole and monobasic lead styphnate respectively. When MNA was examined there was no evidence of any further reactivity but when MNB was heated an additional fourth exotherm was recorded (Figure (7)). This commenced at 170°C and continued till ignition of mercuric 5 nitrotetrazole and was initially attributed to interaction between antimony sulphide and mercuric 5 nitrotetrazole. In order to confirm this explanation the following compositions were prepared and tested under similar conditions:

tetracene - Hg 5 nitrotetrazole 1 - 4  
Pb styphnate - Hg 5 nitrotetrazole 2 - 1

Ba(NO<sub>3</sub>)<sub>2</sub> - Hg 5 nitrotetrazole 1 - 1

CaSi<sub>2</sub> - Hg 5 nitrotetrazole 1 - 1

Sb<sub>2</sub>S<sub>3</sub> - Hg 5 nitrotetrazole 1 - 1

With the exception of antimony sulphide - mercuric 5 nitrotetrazole none of these mixtures showed any evidence of incompatibility. In the case of antimony sulphide - mercuric 5 nitrotetrazole there did appear to be some slight lowering of the ignition temperature of the mercuric 5 nitrotetrazole but there was no evidence of an extra exotherm. However if a small amount of tetracene was added an additional exotherm was recorded. This commenced after decomposition of the tetracene and continued until ignition of the mercuric 5 nitrotetrazole.

It would thus appear that there is reactivity between antimony sulphide and mercuric 5 nitrotetrazole and that the reaction is aided by the presence of liquid decomposition products of tetracene. It is currently postulated that these decomposition products act as a solvent.

#### 5. CONCLUSIONS

Compatibility test procedures adopted in Australia are similar to those employed in explosives laboratories elsewhere. In spite of their disadvantages vacuum stability methods are the most commonly used tests and in instrumented form are likely to be used for a considerable period. It is hoped that this introduction of an instrumented system will make it easier to undertake a regular analysis of gaseous reaction products. Such analyses might help to counter one of the most important disadvantages of vacuum stability testing.

It is intended that use of thermal analysis will be extended, particularly in the areas

of primary explosives and pyrotechnics where there is currently no capability to provide rapid assessments of compatibility.

Functioning tests and study of deterioration after accelerated storage will continue to be employed to complement the shorter term tests.

#### 6. REFERENCES

1. United Kingdom Chemical Inspectorate Laboratory Method M240/61.
2. T.A. Rudram and J.W. Taylor MQAD Report 228 (1975).

TABLE 1.

STABILIZER CONTENT OF NH 055 PROPELLANT AFTER ACCELERATED STORAGE  
WITH CANDIDATE MOTH PROOFING AGENTS AND WITH DINITRO $\alpha$ -NAPHTHOL

	Total Stabilizer (%)	TLC Analysis (%) *		
		DPA	NNODPA	2NDPA
NH 055 Control Ambient	0.72	0.64	0.07	0.03
NH 055 Blank	0.69	0.50	0.16	0.10
NH 055/Mitin FF	0.69	0.51	0.15	0.10
NH 055/Eulan W	0.69	0.49	0.18	0.10
NH 055/DAN (current material)	0.70	0.51	0.16	0.09

\* Total stabilizer as normally determined by steam distillation/gravimetric analysis is equivalent to DPA + 0.75 (2NDPA + NNODPA) where 2NDPA = 2 nitrodiphenylamine and NNODPA - nitrosodiphenylamine.

TABLE 2.

STAB SENSITIVE COMPOSITIONS INCORPORATING MERCURIC 5 NITROTETRAZOLE

	MNA	MNB
Tetracene	5	5
Mercuric 5 nitrotetrazole	20	20
Monobasic lead styphnate	40	40
Antimony Sulphide		15
Calcium Silicide	15	
Barium Nitrate	20	20

TABLE 3.

STAB SENSITIVITY OF MERCURIC 5 NITROTETRAZOLE COMPOSITIONS

Storage History	Sensitivity (mJ)		
	NOL 130	MNA	MNB
Original	5.7	5.8	5.3
5 weeks at 70°C	4.9	5.3	5.4
10 weeks at 70°C	6.0	7.0	6.6
Control 5 weeks	4.9	5.6	5.8
Control 10 weeks	5.3	5.7	5.3

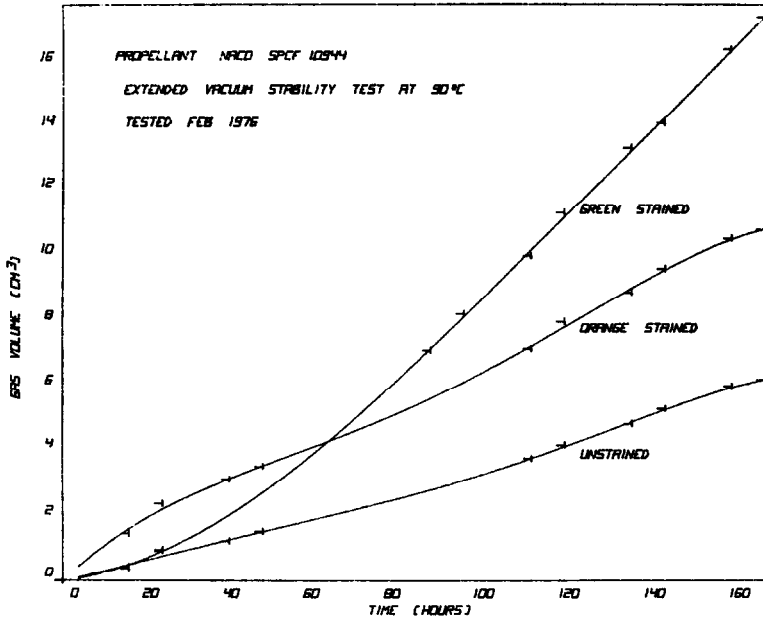


FIGURE 1. EXTENDED VACUUM STABILITY TEST FOR NACO PROPELLANT.

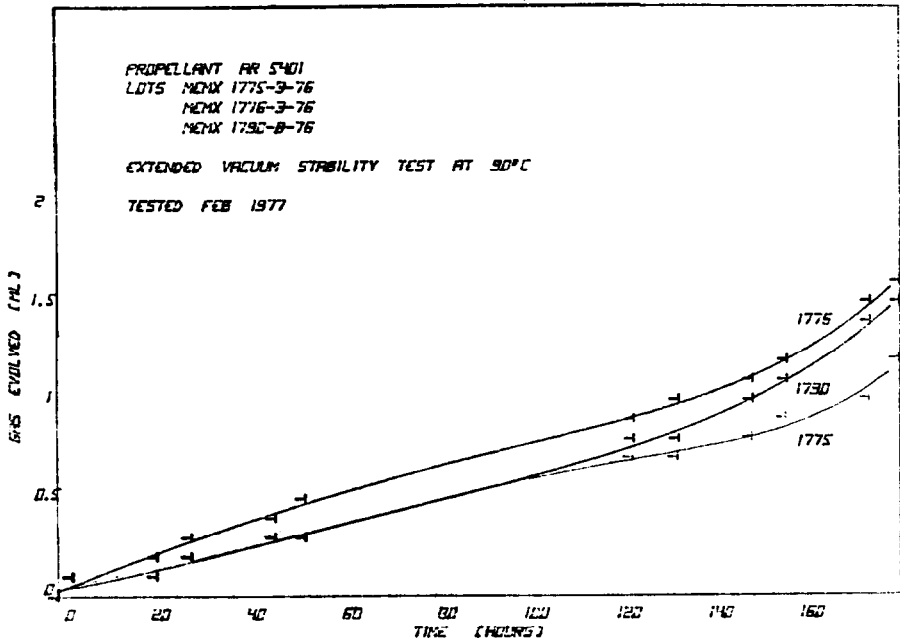


FIGURE 2. EXTENDED VACUUM STABILITY TEST FOR PROPELLANT AR5401.

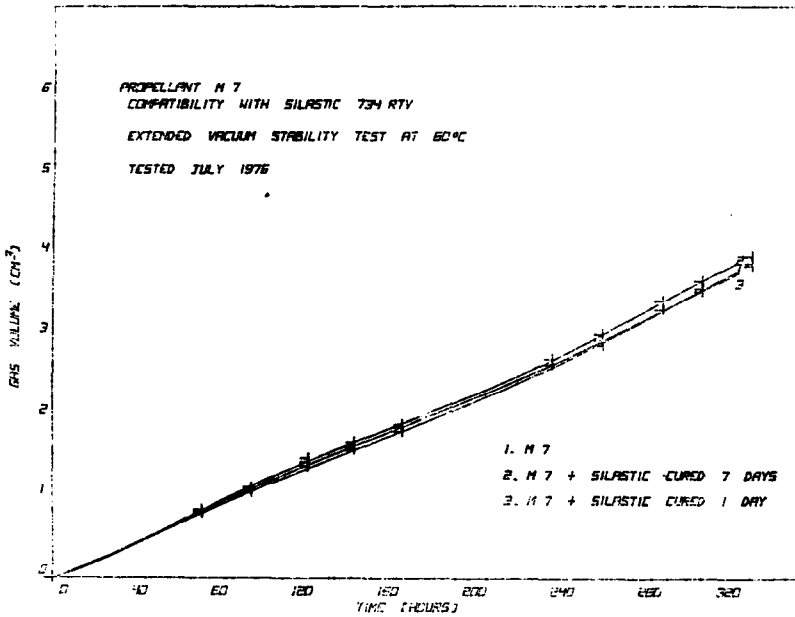


FIGURE 3. COMPATIBILITY OF PROPELLANT M7 WITH SILASTIC 734RTV SILICONE RUBBER.

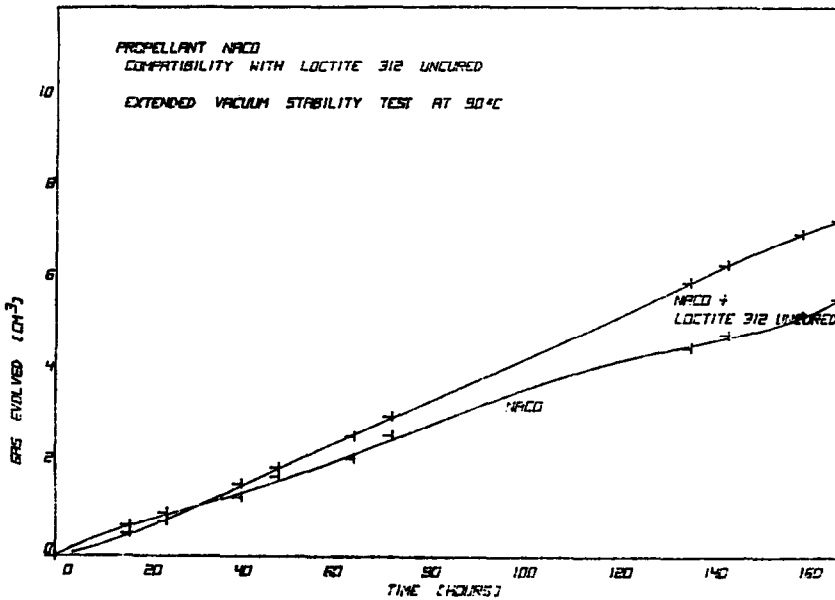


FIGURE 4. COMPATIBILITY OF NACO PROPELLANT WITH LOCTITE 312 (UNCURED).

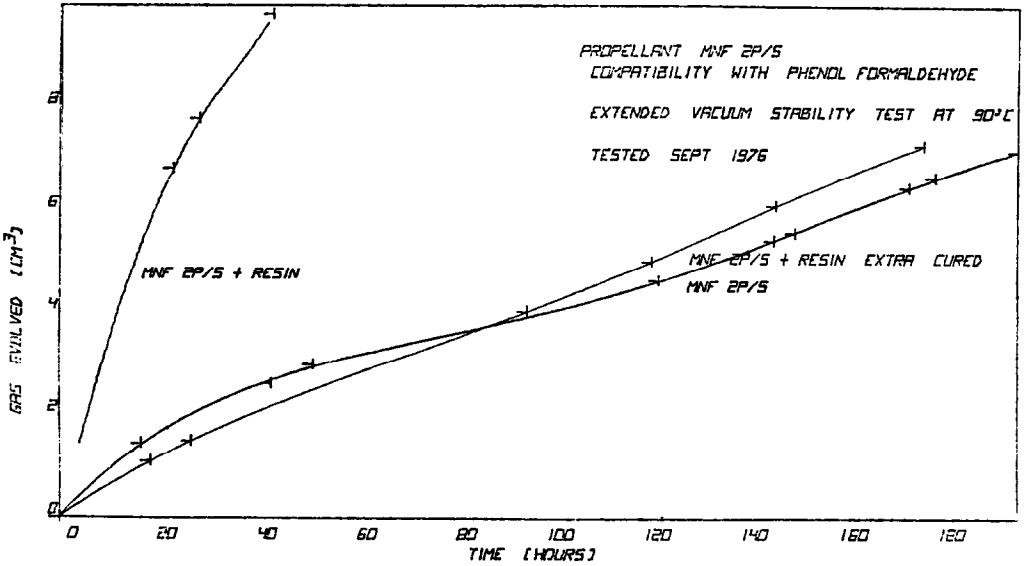


FIGURE 5. COMPATIBILITY OF PROPELLANT MNF 2P/S WITH PHENOL FORMALDEHYDE RESIN.

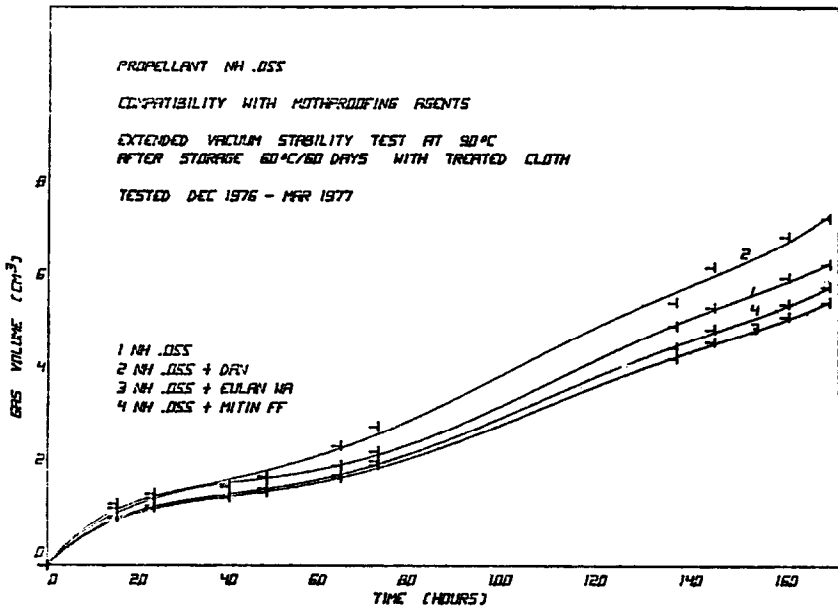


FIGURE 6. COMPATIBILITY OF PROPELLANT NH 055 WITH CANDIDATE MOTH PROOFING AGENTS.



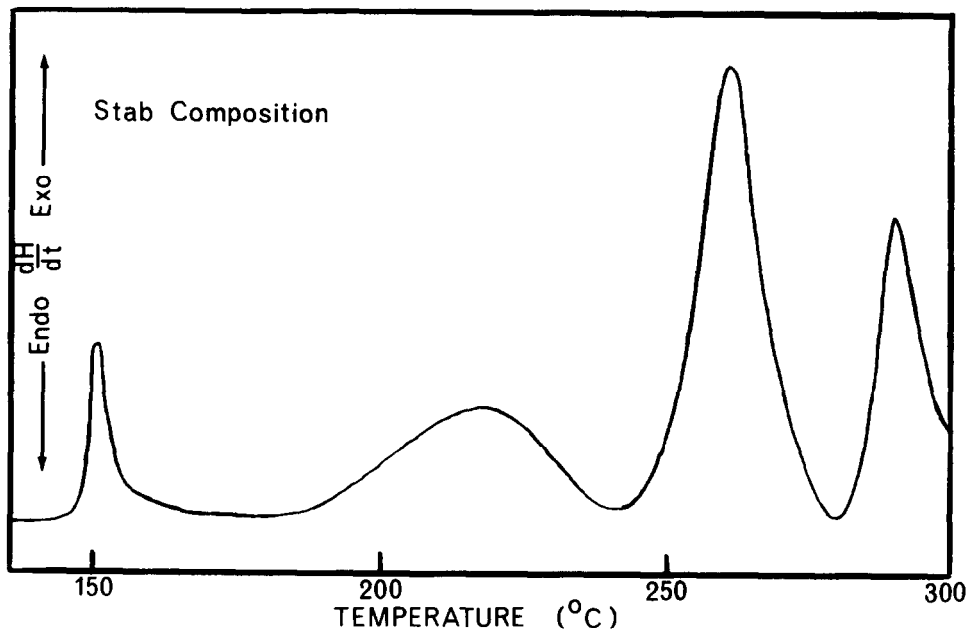


FIGURE 7. DSC CURVE OF STAB SENSITIVE COMPOSITION MNB. 2MG SAMPLE HEATED AT  $8\text{KMIN}^{-1}$  IN FLOWING DRIED HELIUM.