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Procurement Executive Ministry of Defence

Explosives Research and Development Establishment – Rocket Propulsion Establishment

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*Explosives Research
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Technical Report
No. 144

COUNTED IN

Heat Evolved in the Propellant/Nitrogen
Dioxide Reaction and its Variation with
Time of Ageing of the Propellant:
Measurement of Effective
Stabiliser Content of Propellants

D. G. Davies

December 1973

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EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT

Technical Report No 144

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Heat Evolved in the Propellant/Nitrogen Dioxide
Reaction and its Variation with Time of Ageing
of the Propellant: Measurement of Effective
Stabiliser Content of Propellants

by

D G Davies

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SUMMARY

The reaction between nitrogen dioxide and colloidal propellant stabilisers is exothermic, and an apparatus based on the heat of the nitrogen dioxide-propellant reaction is used in Holland for surveillance control of gun propellants. A similar apparatus has been constructed and the heat of this reaction compared in propellants stabilised with carbamate, diphenylamine, picrite, 2-NDPA, p-NMA and resorcinol. The observed heat from the nitrogen dioxide-aged propellant reaction, and the actual stabiliser content of the samples, are compared as these propellants are aged.

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CONTENTS

	<u>Page No</u>
1 INTRODUCTION	5
2 DESCRIPTION OF THE APPARATUS	5
3 EXPERIMENTAL PROCEDURE	6
4 RESULTS AND DISCUSSION	7
4.1 General Features of the Method, Reproducibility	7
4.2 Products of the Stabiliser/Nitrogen Dioxide Reaction	8
4.3 Unaged Propellants - Temperature Effect of the Propellant/Nitrogen Dioxide Reaction	10
4.4 Aged Propellants - Effect of Degree of Ageing on the Temperature Effect of the Propellant/Nitrogen Dioxide Reaction	11
4.5 The Stabiliser/Nitrogen Dioxide Reaction as a Method of Stabiliser Analysis	13
5 ACKNOWLEDGEMENTS	14
6 REFERENCES	15
Tables 1 - 4	16
Figures 1 - 7	

1 INTRODUCTION

Small amounts of stabilising compounds are incorporated into colloidal propellants in order to react with one or more of the initial nitric ester decomposition products (nitrogen dioxide, nitrous acid and nitric acid), and thus reduce the overall autocatalytic effect of these on the nitric ester decomposition. The rate of consumption of the stabiliser in a propellant at an elevated temperature is usually determined at intervals, by extraction of a propellant sample, separation of the ingredients and chemical analysis. Hence the safe life of the propellant at ambient temperatures may be predicted.

The reaction between nitrogen dioxide and the stabiliser in a propellant is exothermic, and an apparatus based on this fact has been developed in the Netherlands for surveillance control of the stabiliser content of carbamate- and diphenylamine-stabilised propellants.^{1,2} In this test, excess nitrogen dioxide is passed over a sample of propellant, and the residual stabilising compounds rapidly react with the gas. The heat generated in the reaction is correlated to the effective residual stabiliser content of the propellant. The major advantage of this method is reported^{1,2} to be that a complete analysis in duplicate takes only about one hour.

In this work, an apparatus, similar to that of the Dutch workers, has been used to investigate comparatively the heat generated in the nitrogen dioxide/propellant reaction using as substrates simple propellants stabilised with a single stabiliser, the stabilisers being carbamate, diphenylamine, 2-nitrodiphenylamine, p-NMA, resorcinol and picrite. It was thought unlikely that this would be a suitable stabiliser analysis method for complex rocket propellants, but the variation in the heat effects would provide valuable support information for other experiments on the overall heat of the exothermal decomposition reactions of ageing propellants.

2 DESCRIPTION OF THE APPARATUS

The glass apparatus, shown diagrammatically in Figure 1, consists of a helix (capacity 250 ml) connected at one end via a three-way valve to a

reaction vessel, and at the other end to dry nitrogen and nitrogen dioxide supplies. The helix and reaction vessel are thermostatted at 30°C in a water bath. Gas entering the reaction vessel, which is partially heat insulated, passes up through a glass sintered disc and then through the ground propellant sample. Any temperature changes occurring in the propellant are monitored by five thermocouples, sheathed in stainless steel, which project into the propellant sample. The "cold" junctions of the thermocouples are in a second empty reaction vessel in the thermostatic water bath, and the thermocouple outputs are connected to a variable range recorder and a circuit capable of supplying backing-off emfs.

3 EXPERIMENTAL PROCEDURE

After optimising conditions it was found necessary to rigidly adhere to the same procedure with no short-cutting of flushing times, etc, but if this was done reproducibility was quite good. The optimised procedure was as follows (see also Figure 1):

- i The apparatus was flushed with nitrogen.
- ii A sample (1 g) of the finely ground propellant was transferred into the reaction vessel and the lid containing the thermocouple system was replaced.
- iii The reaction vessel was flushed with nitrogen (100 ml/min) for 2 minutes, tap C was turned to vent the nitrogen to atmosphere and then tap A was turned.
- iv Tap B was turned to fill the helix as far as tap C with nitrogen dioxide. Taps B and C were then closed to retain the nitrogen dioxide in the helix, and the nitrogen dioxide was allowed to equilibrate to the temperature of the water bath for 10 minutes.
- v Taps A and C were then turned and the 'plug' of nitrogen dioxide was pushed through the reaction vessel by the nitrogen flow (100 ml/min). The temperature changes in the reaction vessel due to both reaction of nitrogen dioxide with stabiliser and also adsorption heat effects were recorded.

- vi When the temperature in the vessel returned to normal the nitrogen flow was increased to 200 ml/min for 7 minutes to flush nitrogen dioxide from the system, and to desorb it from the propellant.
- vii At the end of this time the procedure was repeated from iii to vi. The heat effects observed during this second run are due to adsorption of nitrogen dioxide on the propellant.
- viii To check that complete reaction between stabiliser and nitrogen dioxide occurred during the first cycle with any particular propellant, items iii to vi may be repeated for a third time when the heat effect observed should be identical to that of the second cycle.

4 RESULTS AND DISCUSSION

4.1 General Features of the Method, Reproducibility

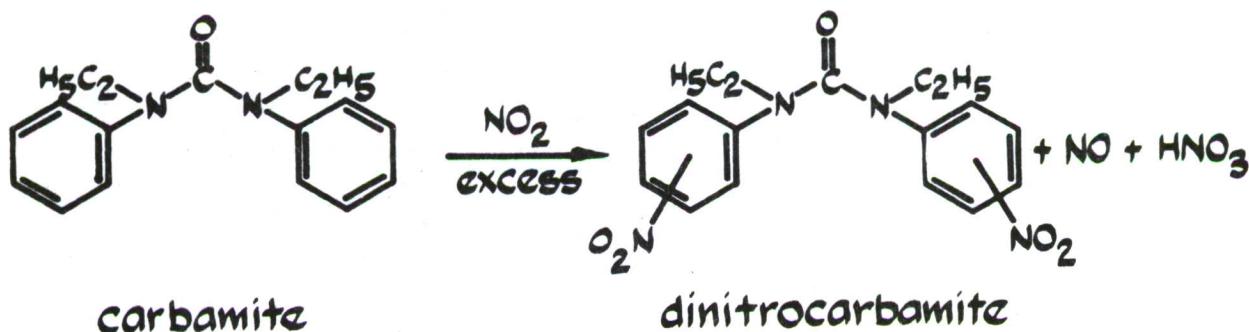
A typical trace of temperature recorded versus time is shown in Figure 2 for the reaction between nitrogen dioxide and unaged SC cordite (contains 9% carbamate). The higher graph is due to heat of the reaction of nitrogen dioxide with carbamate plus heat of adsorption of nitrogen dioxide on the propellant. The lower graph is the second cycle and is due to heat of adsorption effects only. (As it has been shown that at 30°C nitrogen dioxide reacts 20 000 times faster with carbamate than with nitrocellulose,¹ reaction between the nitric esters and nitrogen dioxide in the few minutes we are concerned with is therefore neglected.) The difference between the maxima (Figure 2), ΔT was recorded. Where the ΔT value was small compared to the maximum temperature recorded, a backing-off voltage was applied to counterbalance the temperature of adsorption effect, so that the ΔT value could be expanded onto the most sensitive range of the recorder.

Replicate results shown in Table 1 indicate the reproducibility of the method. Each propellant sample was usually run in triplicate or quadruplicate and a mean value of ΔT was taken. No attempt was made to calibrate the apparatus and so any linear comparison between different propellants, should not be made. It should be noted that not only the nominal stabiliser itself will react exothermally with nitrogen dioxide in the reaction vessel. Usually

the degradation products of the stabiliser which have been formed in a propellant during ageing will also react, and so the variation in ΔT of samples as a propellant is aged, may not be directly related to the percentage of nominal stabiliser in the samples. The variation will depend on the heats of the reactions of the individual stabiliser degradation products with nitrogen dioxide and their rate of formation. Effective stabiliser is therefore monitored by this method, a summation of the residual initial stabiliser plus those degradation products still capable of rapid reaction with nitrogen dioxide.

4.2 Products of the Stabiliser/Nitrogen Dioxide Reaction

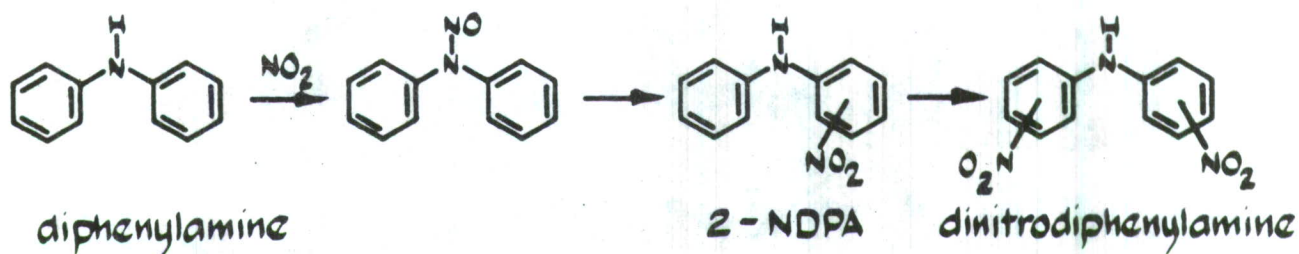
The mechanisms of these reactions have been reviewed in detail elsewhere.³ All the stabilisers considered here react extremely rapidly with excess nitrogen dioxide at ambient temperatures. Carbamate reacts to give 4,4'-, 2,4'-, and 2,2'-dinitrocarbamate ($4,4' \approx 2,4' > 2,2'$) as the major products and also nitric oxide and nitric acid.⁴⁻¹⁰ The overall heat of the reaction has been measured to be 60 kcal/mol.*^{2,8}



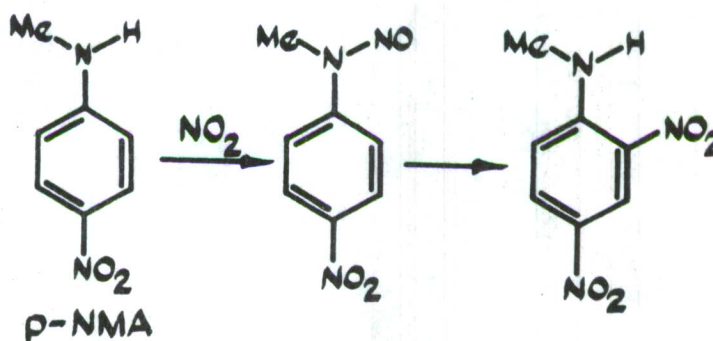
Diphenylamine forms dinitrodiphenylamines as the major products, probably via a mono-nitrodiphenylamine intermediate.^{6, 10-12} The most abundant isomer

*1 kcal/mol = 4.184 kJ/mol

formed is 4,4'-dinitrodiphenylamine. Similarly 2-nitrodiphenylamine gives a dinitrodiphenylamine product.^{7,8,10,11}

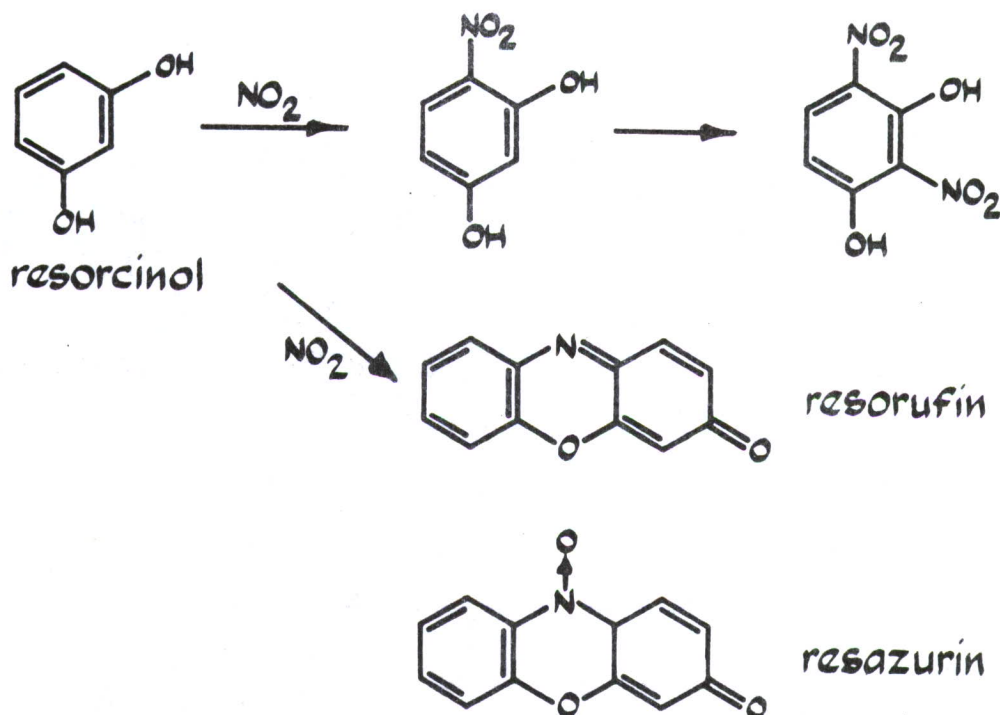


p-NMA reacts to give N-nitroso p-NMA very rapidly, and then at a slower rate forms mainly the C-nitro derivative, N-methyl 2,4-dinitroaniline.^{11,13}



A further stage in the reaction, a trinitro derivative, can slowly be formed but usually only under extreme conditions at an elevated temperature.

The products from the resorcinol/nitrogen dioxide reaction are complex, postulated as due to the reaction of resorcinol reaction products with resorcinol itself.¹⁴ Not only are nitroresorcinol and dinitroresorcinol formed, but also dimeric products resorufin and resazurin and a percentage of unidentified polymeric material.



4.3 Unaged Propellants - Temperature Effect of the Propellant/ Nitrogen Dioxide Reaction

The compositions of the propellants examined are given in Table 2, and the temperature effects of the reaction of these propellants with nitrogen dioxide are shown in Table 3.

A simple double-base propellant stabilised with 9% carbamate (SC) gave a temperature effect $\Delta T = 10.38^{\circ}\text{C}$ in the apparatus, a similar propellant stabilised with 3.5% carbamate (HSC) gave $\Delta T = 4.56^{\circ}\text{C}$, not a direct relationship because of heat losses from the apparatus. The heat of the adsorption of

nitrogen dioxide onto the two propellants was found however to be almost identical. If these ΔT values (10.38°C at 9% carbamite, 4.56°C at 3.5% carbamite) are used for an approximate calibration, the replicate results on samples of SC cordite (Table 1) may be interpreted in terms of stabiliser content as 9.1, 9.1, 9.3, 8.9, 8.3 and 9.3% carbamite content.

When a propellant containing nitroguanidine (picrite) was used in the apparatus, the temperature recorded rose very rapidly and the propellant inflamed. This is due to the nitrogen dioxide also reacting exothermally with the high percentage of picrite in the propellant (N) and thus causing the temperature of the propellant to rise beyond its ignition temperature. This has since also been reported by Dutch workers.¹⁵ Although ignition of the propellant might be prevented by dilution with some inert material, this was not thought worthwhile as the experiment would be looking for changes due to small variations in stabiliser (initially 7%) in the presence of a large percentage of reacting material (55% picrite).

Measurements on the other propellants, containing stabiliser contents in the range 0.5 to 2% were more difficult, as the temperature effect T due to the stabiliser/nitrogen dioxide reaction is now a small value in comparison with the large nitrogen dioxide/propellant adsorption temperature effect of about 10°C which is simultaneously recorded. Using F527/327 propellant as an example however, by electrically backing-off the adsorption thermocouple output and expanding ΔT onto the most sensitive recorder range, replicates gave values of ΔT in the range 1.14 to 1.24°C , with a mean of 1.21°C .

4.4 Aged Propellants - Effect of Degree of Ageing on the Temperature Effect of the Propellant/Nitrogen Dioxide Reaction

All the propellants shown in Table 2, with the exception of N and HSC, were ground and then aged in a dry air atmosphere at 80°C . Samples were removed at appropriate time intervals for conventional stabiliser analysis and also for measurement of the nitrogen dioxide/propellant temperature effect ΔT . The conventional stabiliser analysis results on these propellants are shown in Table 4. Figures 3 - 7 show the variation in ΔT with time of ageing for each propellant. It is acknowledged that some variation in the identity

of the stabiliser degradation products produced occurs by ageing at 80°C rather than at temperatures closer to ambient.

The decrease in the temperature effect ΔT was relatively linear with time of ageing for the carbamate stabilised propellant (SC) in the range 9 to 5% actual stabiliser content, and also linear with respect to the actual stabiliser content (Figure 3). As the carbamate is consumed in the ageing propellant at 80°C, the main identified derivatives formed are 4-nitrocarbamite and N-ethyl N-nitrosoaniline.³ The decrease in ΔT is due to the difference in the heat of reaction of these products with nitrogen dioxide relative to the heat of the carbamate/nitrogen dioxide reaction.

When the diphenylamine stabilised propellant (FNH) is aged at 80°C the temperature effect ΔT of samples is observed to decrease very rapidly with time of ageing initially (0 to ca 9 days), and then a change of slope occurs in the graph and ΔT decreases much slower and relatively linearly from 9 to 28 days (Figure 4). The observed change of slope at 9 days probably coincides with complete conversion of diphenylamine to its primary degradation product N-nitrosodiphenylamine in the aged propellant, and the latter part of the graph, corresponds to the consumption of this product, also an efficient stabiliser. The change of slope may be explained both by the fact that diphenylamine will be consumed at a more rapid rate in the propellant than N-nitrosodiphenylamine due to direct nitric ester-diphenylamine reactions, and also because the heat of the nitrogen dioxide/diphenylamine reaction (20 kcal/mol) is much greater than that of the nitrogen dioxide/N-nitrosodiphenylamine reaction (7 kcal/mol).² It is difficult to differentiate diphenylamine and N-nitrosodiphenylamine in a mixture of both in a propellant, so the analytical results superimposed on Figure 4 are, as is usual, the summation of both compounds.

2-Nitrodiphenylamine stabiliser in F527/327 propellant will degrade on ageing to dinitrodiphenylamine, and not to any further nitrated derivatives until the concentration of 2-nitrodiphenylamine is very low. 2-Nitrodiphenylamine reacts rapidly with nitrogen dioxide in this experiment, dinitrodiphenylamine does not. As also ΔT is a small value in this case compared to the overall temperature maxima and thus less susceptible to errors due to heat loss

from the apparatus, the decrease in ΔT on ageing (Figure 5) would be expected to be linearly related to the actual 2-nitrodiphenylamine content of the propellant and this was observed to be so.

The decrease in ΔT of the propellant/nitrogen dioxide reaction of the p-NMA (F488/2268) and resorcinol (F488/2269) stabilised propellants are shown in Figures 6 and 7 respectively, and are on a curve versus ageing time similar to that obtained for the 2-nitrodiphenylamine stabilised propellant. Although to be less expected than in the case of 2-nitrodiphenylamine they also show an approximate linear relationship between the ΔT and the actual nominal stabiliser content of the samples.

4.5 The Stabiliser/Nitrogen Dioxide Reaction as a Method of Stabiliser Analysis

The method was designed by Dutch workers² for surveillance control of gun propellants, and it appears in this work that simple carbamate- and diphenylamine-containing gun propellants may readily be examined by this means. A complete analysis time of about $1\frac{1}{2}$ hours was found to give reproducible results. As is to be expected accuracy is not as good as by other analytical methods in use. Although differences between, for example, initial stabiliser content and half initial stabiliser content could be readily measured, as required for surveillance control, accuracy would not normally be good enough for some other uses, for example for stabiliser consumption trials where losses of stabiliser are more moderate and akin to Service Storage conditions. The inability to apply the method to propellants containing picrite appears however a significant disadvantage.

Rocket propellants normally contain smaller percentages of stabiliser than gun propellants, and so the temperature effect of the stabiliser/nitrogen dioxide reaction is small and less accurately measured because it is in addition to a large propellant/nitrogen dioxide adsorption effect. Also the percentage decrease in the temperature effect ΔT is small in the propellants stabilised with 2-NDPA, p-NMA or resorcinol even when the stabiliser content has dropped to a small fraction of its nominal initial value. Of more importance is the fact that rocket propellants are more complex and usually contain ballistic modifiers as additives, many of which may be expected to

react exothermally with nitrogen dioxide and contribute to the heat effect. The additive may also be consumed partially during the ageing of the propellant. Thus the variation in the temperature effect due to stabiliser consumption may be lost in the background of other reactions. If, as is common, the propellant contains two stabilisers, a separate chemical analysis for one of the stabilisers would have to be performed, in addition to determining the temperature effect due to both stabilisers in this apparatus. In contrast therefore to non-picrite containing gun propellants, the possibility of useful application of this method to stabiliser analysis of rocket propellants appears minimal.

5 ACKNOWLEDGEMENTS

The determination of the stabiliser content of these propellant samples by conventional analysis by P G Maher and E F Pembridge is gratefully acknowledged.

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TABLE 1Replicate Results for the Nitrogen Dioxide-Unaged SC Cordite Reaction

Run No	Temperature Effects, °C		
	T _{max} (Reaction + Adsorption)	T _{max} (Adsorption)	ΔT
1	20.30	9.80	10.50
2	20.23	9.73	10.50
3	20.00	9.23	10.77
4	19.61	9.30	10.31
5	18.53	8.84	9.69
6	21.07	10.38	10.69

Mean = 10.38°C

Standard Deviation ± 0.15°C

TABLE 2Nominal Propellant Compositions

Propellant	NC	NG	Picrite	Carbamite	DPA	2-NDPA	p-NMA	Resorcinol	Other
SC	49.5	41.5		9.0					
HSC	49.5	47.0		3.5					
FNH	84.0				1.0				DNT 10.0) DBP 5.0)
N	19.0	18.7	55.0	7.3					
F527/327	37.3	56.1				0.5			DBP 6
F488/2268	45.0	45.0					2.0		TA 8
F488/2269	45.5	45.5						1.0	TA 8

TABLE 3

Mean Temperature Effects of the
Unaged Propellant - Nitrogen Dioxide Reaction

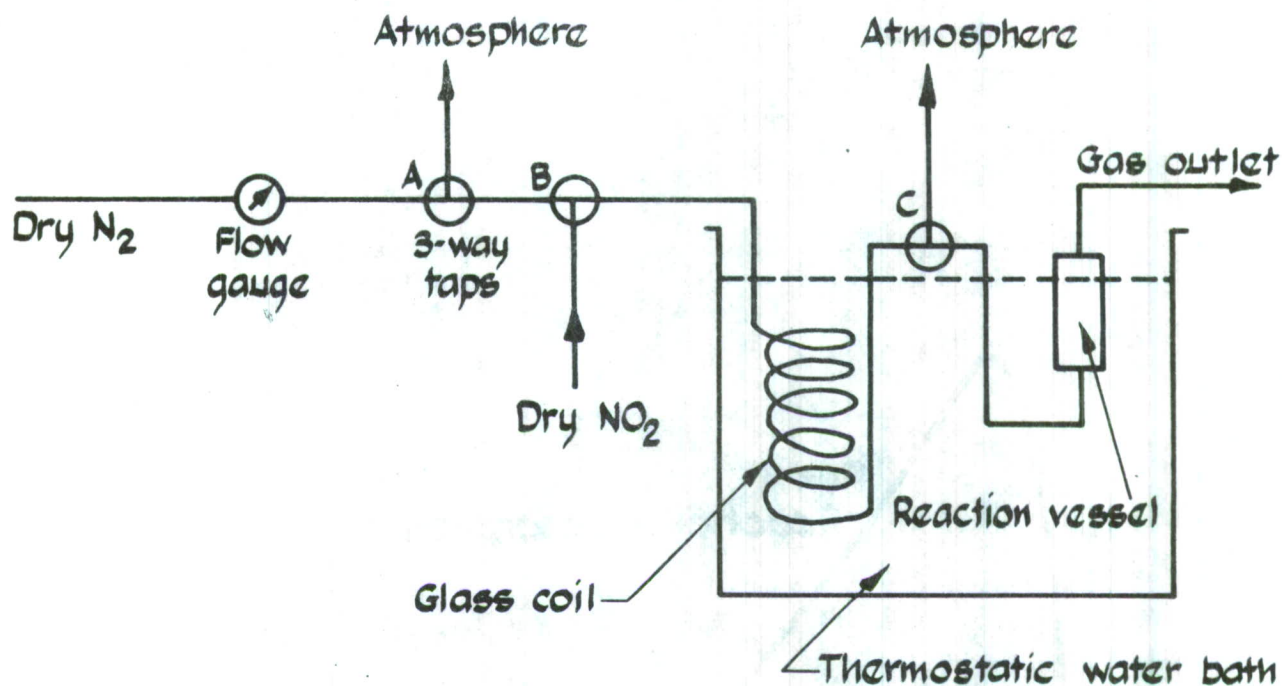
Propellant	Stabiliser	Temperature Effect ΔT (°C)
SC	Carbamite 9%	10.38
HSC	" 3.5%	4.56
N	Carbamite + Picrite	Inflamed
FNH	Diphenylamine 0.9%	0.95 ^a
F527/327	2-NDPA 0.5%	1.20
F488/2268	p-NMA 1.7%	2.03
F488/2269	Resorcinol 0.5%	1.65

a - used different particle size to other samples

TABLE 4Stabiliser Analyses of Propellants on Storage at 80°C

Propellant	Time (days)	% Stabiliser
SC	0	9.01
"	8	8.14
"	16	7.41
"	24	6.59
"	43	4.79
"	78	0.07
FNH	0	0.90*
"	3	0.76*
"	10	0.51*
"	21	0.24*
F527/327	0	0.50
"	4	0.28
"	6	0.20
"	8	0.16
"	16	0.02
F488/2268	0	1.79
"	2	1.22
"	4	0.69
"	16	0.06
F488/2269	0	0.50
"	3	0.35
"	9	0.21
"	16	0.12

* Figures are Diphenylamine plus N-nitroso-diphenylamine



SCHEMATIC FLOW DIAGRAM OF APPARATUS

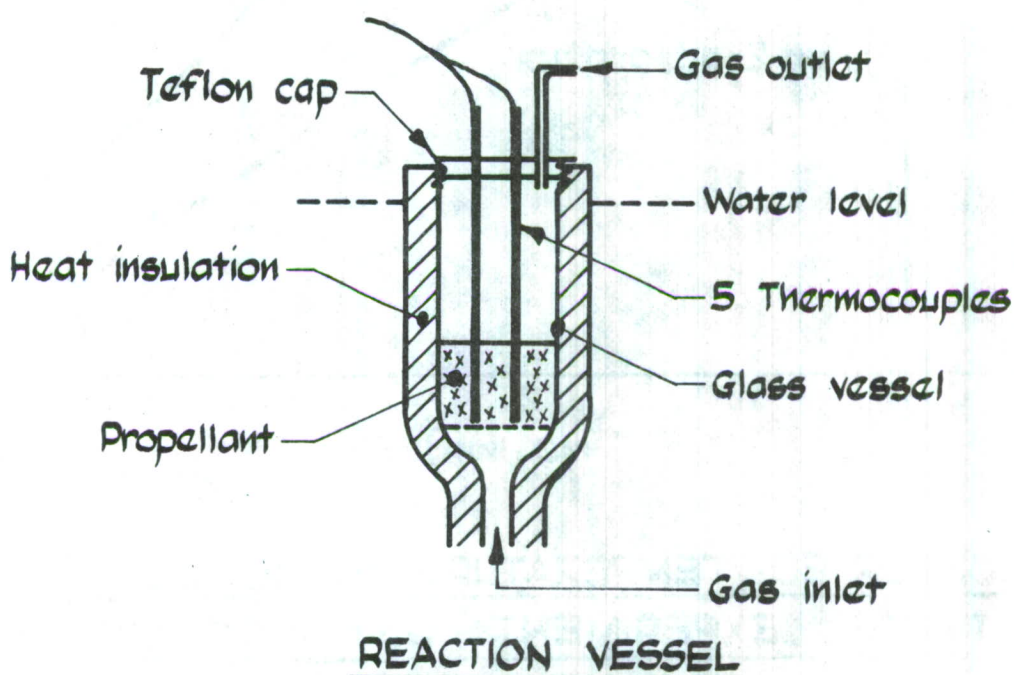


FIG. 1

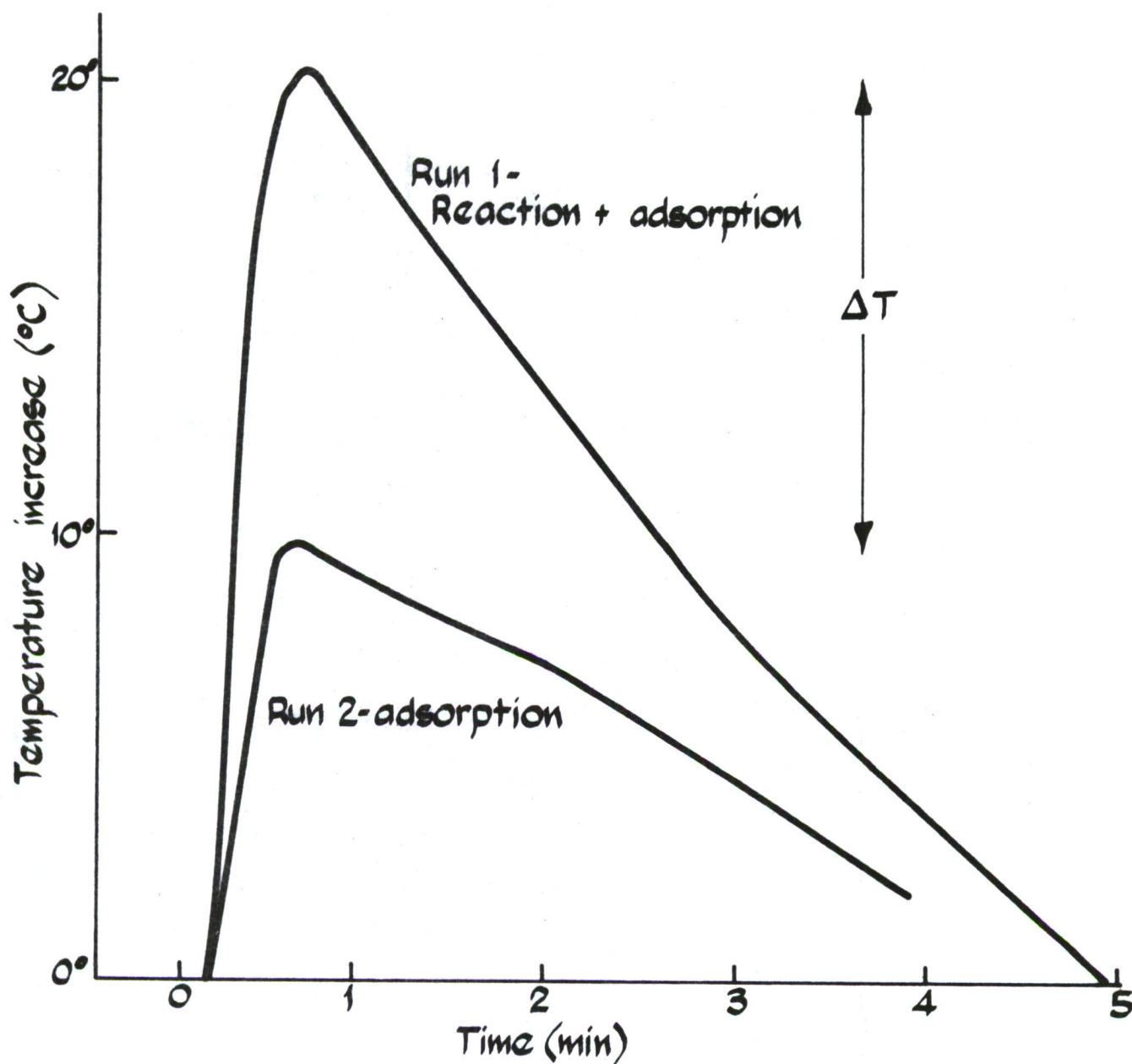


FIG.2 OBSERVED TEMPERATURE INCREASES IN A
TYPICAL EXPERIMENT USING SC CORDITE
(9% CARBAMITE)

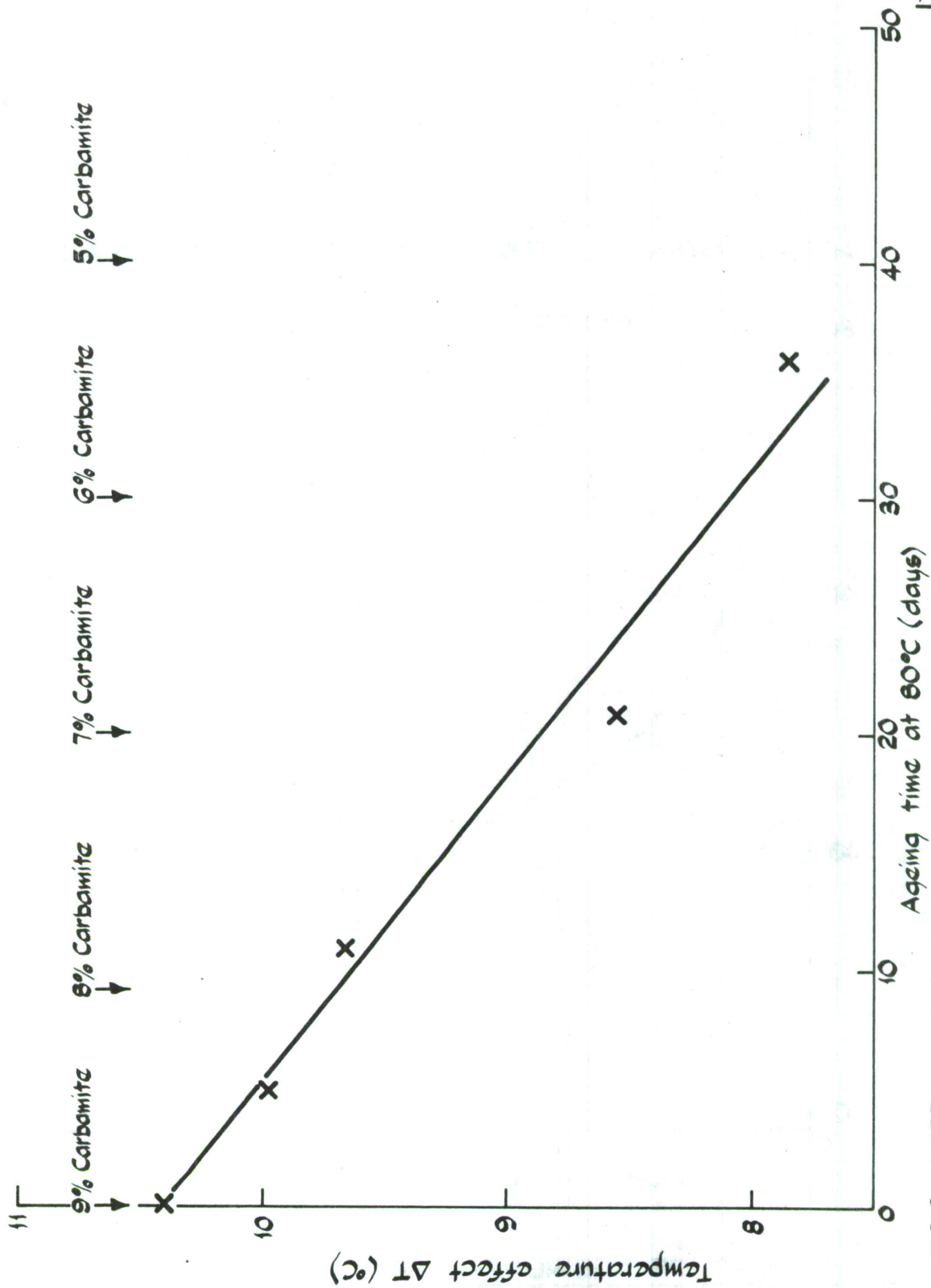


FIG. 3 EFFECT OF AGEING SC CORDITE ON THE TEMPERATURE EFFECT ΔT AND STABILISER CONTENT

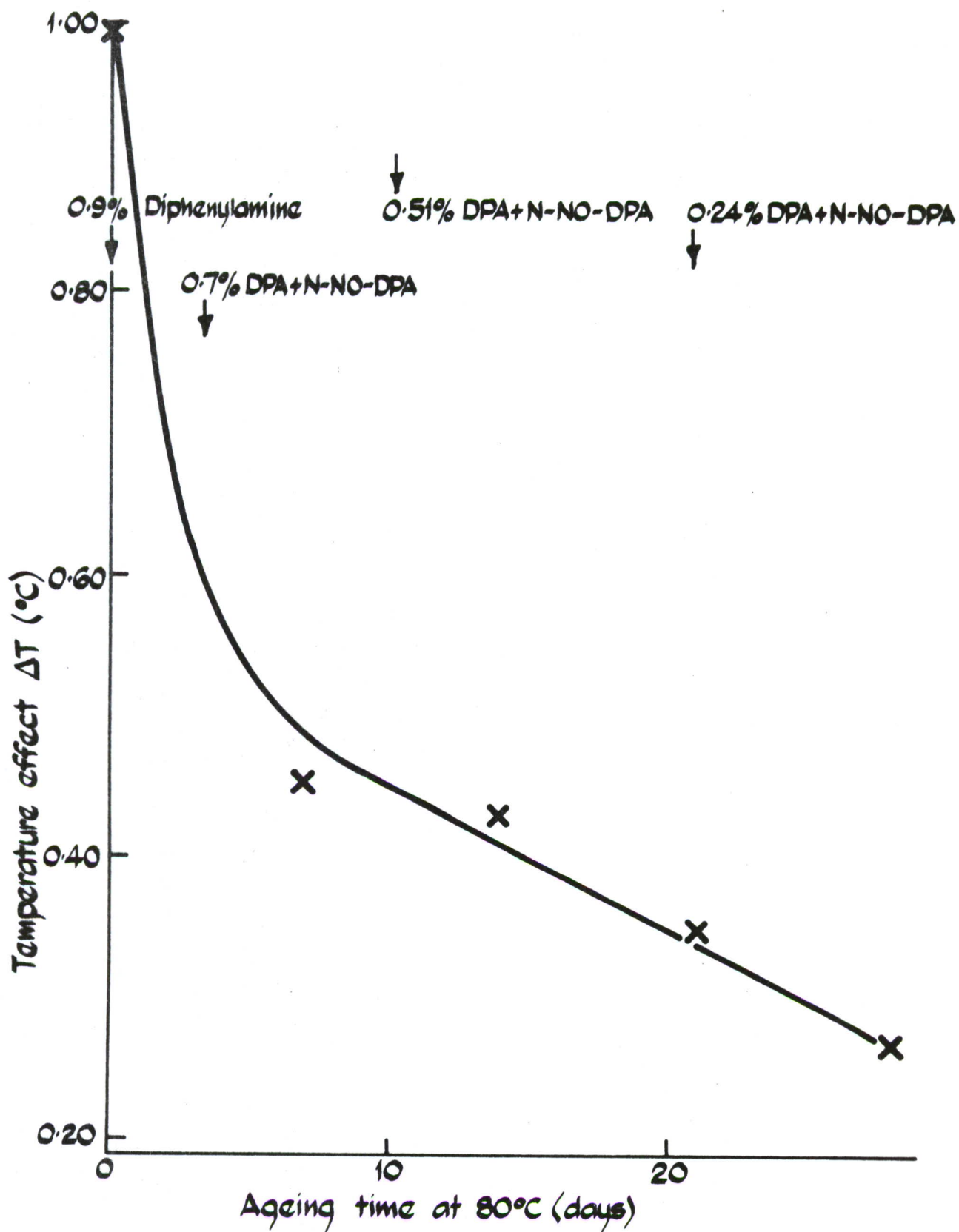


FIG.4 EFFECT OF AGEING FNH PROPELLANT ON THE TEMPERATURE EFFECT ΔT AND STABILISER CONTENT

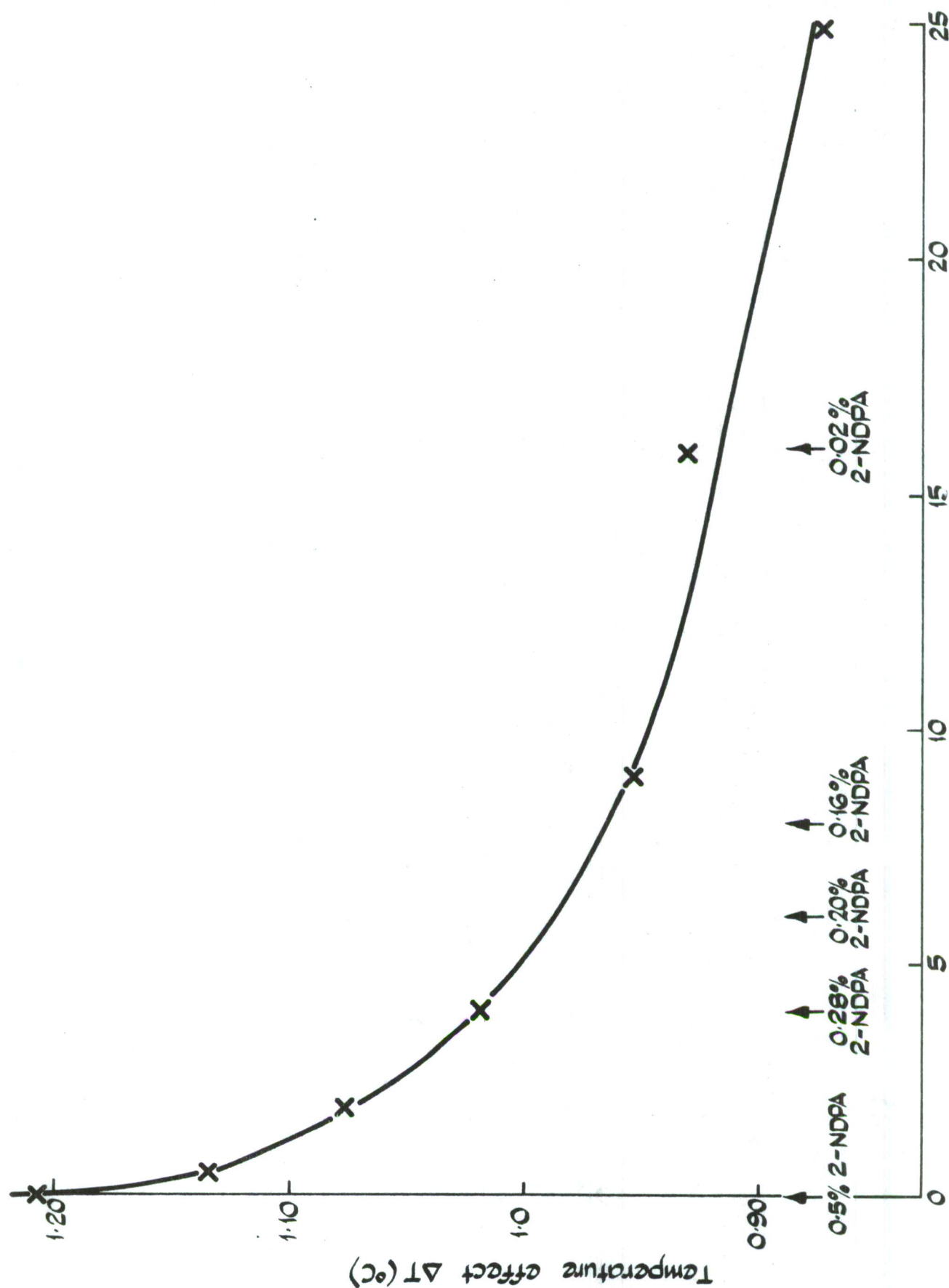


FIG. 5 EFFECT OF AGEING F527/327 PROPELLANT ON THE TEMPERATURE EFFECT ΔT AND STABILISER CONTENT

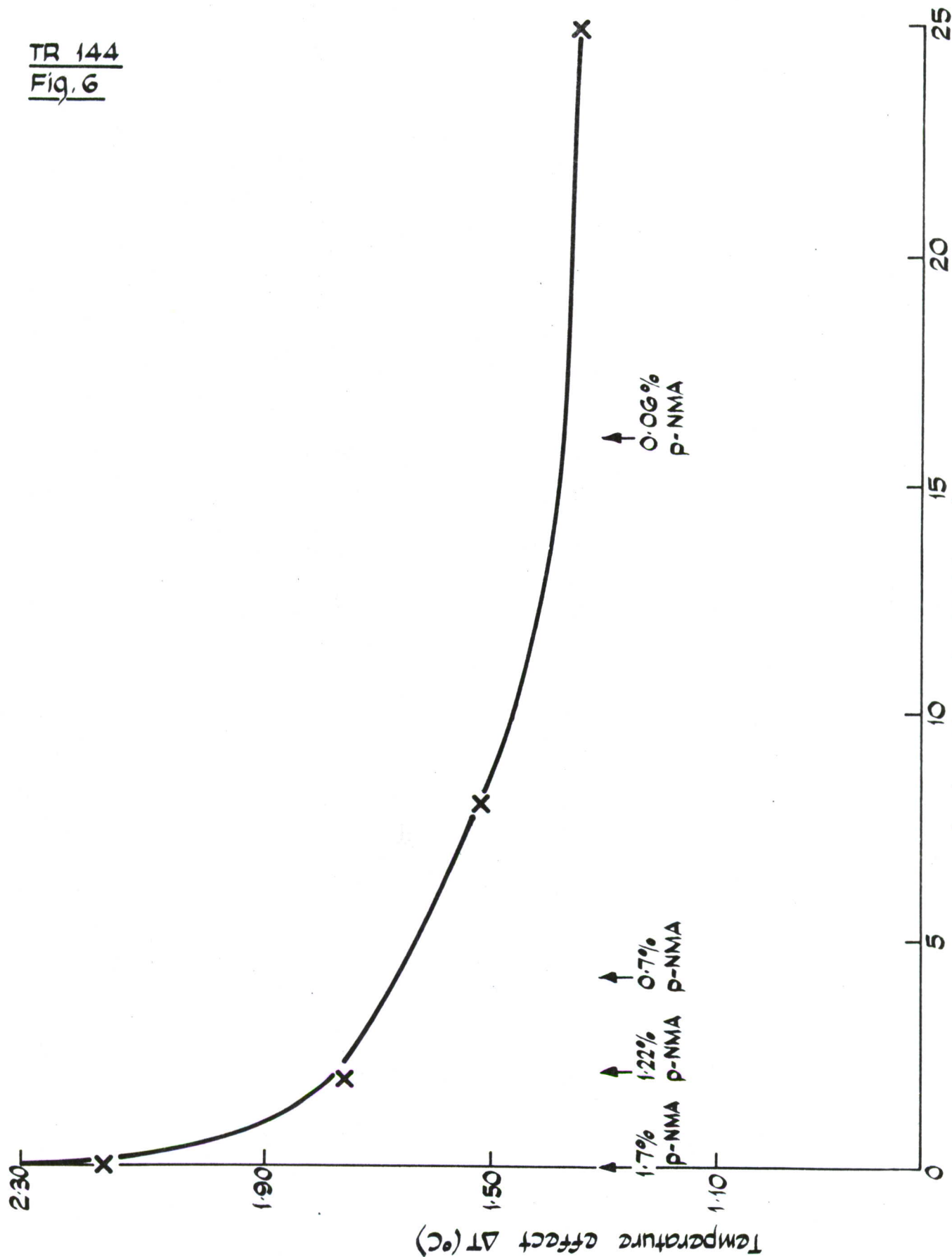


FIG. 6 EFFECT OF AGEING F488/22G8 PROPELLANT ON THE TEMPERATURE EFFECT ΔT AND STABILISER CONTENT

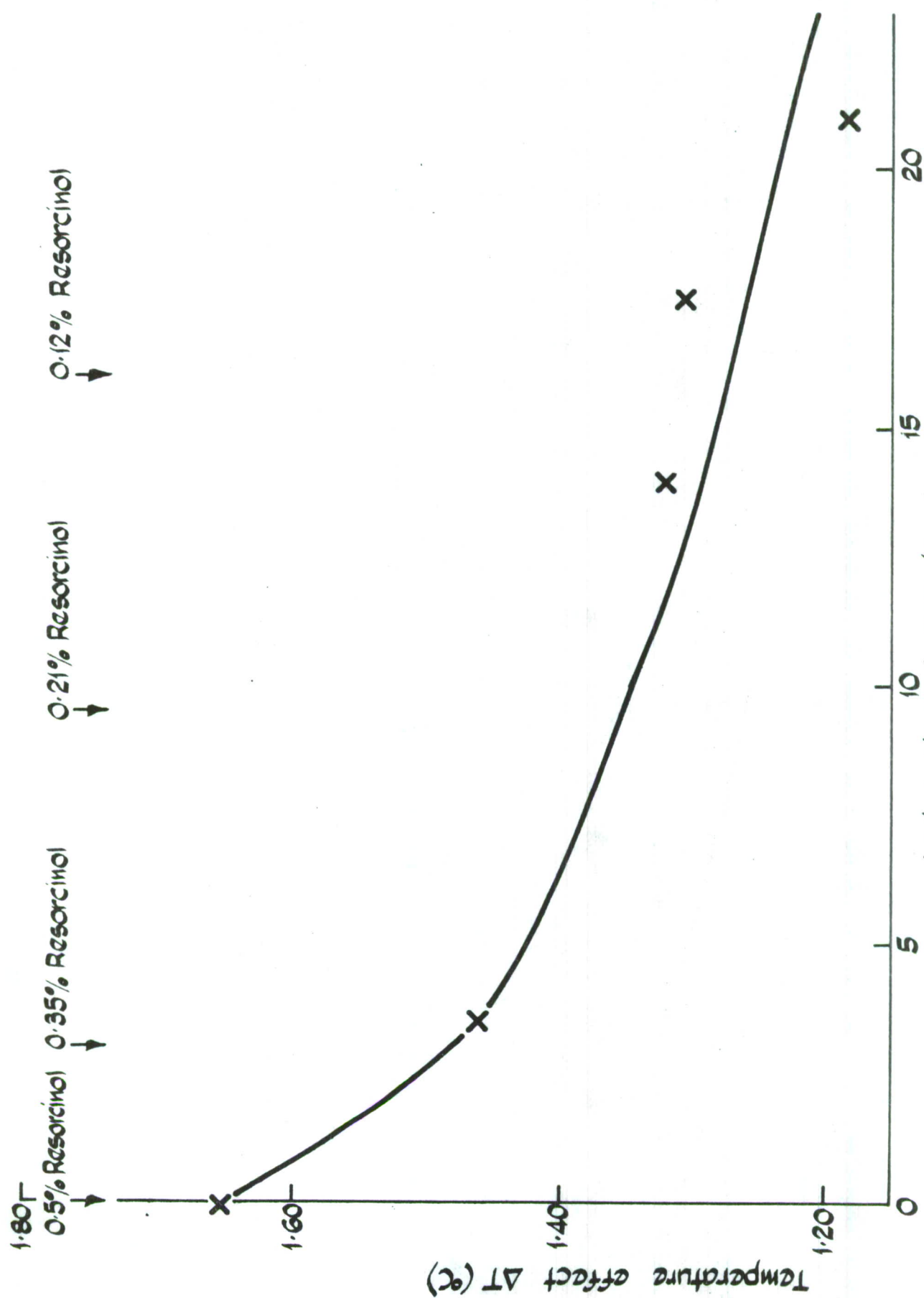


FIG. 7 EFFECT OF AGEING F488/22G9 ON THE TEMPERATURE EFFECT ΔT AND STABILISER CONTENT

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15. Distribution statement			
Descriptors (or keywords) Propellants, Stabilisers (agents), Nitrogen dioxide (TEST) continue on separate piece of paper if necessary			
Abstract The reaction between nitrogen dioxide and colloidal propellant stabilisers is exothermic, and an apparatus based on the heat of the nitrogen dioxide-propellant reaction is used in Holland for surveillance control of gun propellants. A similar apparatus has been constructed and the heat of this reaction compared in propellants stabilised with carbamite, diphenylamine, picrite, 2-NDPA, p-NMA and resorcinol. The observed heat from the nitrogen dioxide-aged propellant reaction, and the actual stabiliser content of the samples, are compared as these propellants are aged.			

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