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Technical Report
No. 189

Compatibility and Stability Testing of Solid Propellants by Heat Flow Calorimetry

Part 2. Further Developments (U)

D. G. Davies
M. G. Farey

September 1974

PD 19395

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6a Technical Report No 189 7 -

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September 1974

3 Compatibility and Stability Testing of Solid Propellants by Heat Flow Calorimetry (U)

Part 2: Further Developments (U)

6U

by

4 D.G.Davies, et al
M G Farey

SUMMARY

The effects of variations in test procedure have been examined and a standard method of testing and interpretation is proposed using the Silvered Vessel test propellant (F527/327) as substrate.

A preliminary investigation into the use of other propellant compositions as substrate is described.

Further copies of this technical report can be obtained from Defence Research Information Centre, Station Square House, St Mary Cray, Orpington, Kent. BR5 3RE

CONTENTS

1 INTRODUCTION

A preliminary report on the use of heat flow calorimetry for compatibility testing of colloidal propellants has been published,[1] in which the feasibility of the method was evaluated, by measuring the effect of the addition of a number of compatibility test materials (plastics, rubbers, etc) on the heat generation of a double-base propellant.

In order to ascertain the most suitable compatibility test procedure, the effect on the heat generation of a double-base propellant caused by variations in test procedure (sample mass, volume of available oxygen, etc), or caused by the addition of extra stabilisers, inert material, or moisture, has now been investigated. From the results obtained, a standard method for future test procedure, conditions, and interpretation of the results is proposed for this propellant. One potential advantage of heat flow calorimetry is that any propellant composition may be used as substrate for this test, and not one standard reference propellant, as is used in the present Silvered Vessel compatibility test. The heat generation of a number of different propellants has therefore been measured and the effect of the addition to these of a few selected compatibility test materials have been measured from temperatures of 80°C to as low as 40°C.

2 EFFECT OF VARIABLES IN TEST PROCEDURE ON THE HEAT GENERATION OF A DOUBLE-BASE PROPELLANT

The measured rate of heat generation of a propellant sample is dependent on other factors besides temperature. Since the propellant is in a sealed sample vessel the physical and chemical changes in the propellant with time are also influenced by the volume of free space and the amount of available oxygen and moisture sealed into the vessel. The effects of variation in these experimental conditions and also the time for the sample to equilibrate with the temperature of the calorimeter have been measured.

2.1 Time to Reach Thermal Equilibrium after Insertion of Sample into Calorimeter

The normal procedure for measuring the heat generation of a sample is to place the sample vessel, which has been at ambient temperature, into the calorimeter which already contains the reference material at the elevated test temperature. Therefore, for a time after insertion in the apparatus, the sample and reference are out of thermal balance, and the output of the apparatus does not correspond exactly with the heat generation of the sample in spite of the compensation system. Information on the typical error during this initial period and the time taken before accurate and reproducible readings are produced, was obtained by inserting into the calorimeter a sample vessel filled with cold inert material (glass beads). The differential output of the two heat flow discs monitored by the calorimeter, and the temperature inside it, were recorded until steady readings were obtained. The results, shown for typical calorimeters in Table 1, indicate that although the calorimeter is at the correct temperature within 12 hours after insertion of a sample, it is one to two days before measurements of heat generations from the apparatus are completely reliable. Poor reproducibility in this period will also occur due to unavoidable variations in the insertion procedure. Practically, as far as a compatibility test is concerned, it is necessary therefore for both the heat generation of the propellant alone, and the heat generation of the propellant/test material mix to be measured in the same calorimeter using the same reference vessel, in order that a meaningful comparison of results may be made in the 0 - 2 day period, though these will still be subject to some error.

2.2 Effect of Sample Mass and Volume of Available Oxygen in the Sample Container

If the propellant is packed into the sample vessel in air, a concentration of oxygen will be sealed into the container and this will be consumed during the test. As this oxygen is used up, a change in the rate of heat generation of the propellant due to a decrease in oxygen-nitric oxide and other oxidation reactions occurs, therefore different volumes of available oxygen in the vessel may cause the propellant to give different rates of heat generation as a function of time. As oxygen is consumed, and other propellant degradation gases are evolved, changes in pressure in the sample vessel will develop dependent on the volume of free space relative to the mass of the propellant. These pressure changes have been observed to affect the rate of the propellant degradation reactions for some propellants,[2] although no explanation was offered.

The heat generation from sample vessels (60 cm^3 capacity) containing 30, 40 or 50 g (the maximum that could be conceivably used) of F527/327 double-base propellant (Table 2) sealed in air were measured for about 16 days at 80°C using the same calorimeter and reference vessel in all cases. The graphs obtained are shown in Figure 1. As expected the graphs differed in the period 0 - 2 days due to the different thermal masses of the samples as they attained thermal equilibrium with their surroundings. When the residual oxygen in the vessel was used up the rate of heat generation was observed to drop sharply and give a minimum. Obviously this occurred first in the sample with least available oxygen (50 g propellant) and last in the sample with most available oxygen (30 g propellant). One day after this minimum had occurred in all the samples however, there was no significant difference between the rate of heat generation per unit mass between the three samples. When the mass of reference material was changed, the graph was observed to differ in the first two days only.

The effect of less available oxygen in the sample vessel, as would occur by the addition of extra material for a compatibility test, was investigated by two methods - (a) by adding inert material to propellant in the sample vessel and (b) by flushing the vessel with nitrogen before sealing. The effect of the 2.5 or 5% addition of inert glass beads (density 2.8 g/cm^3) to the propellant was, as expected, to progressively move the minima due to loss of oxygen in the vessel to a shorter ageing time (Figure 2). The shift and the differences between these results were however extremely small. The effect on the heat generation of F527/327 propellant at 80°C of flushing the sample vessel with nitrogen before sealing is shown in Figure 3. After temperature equilibration no minimum was observed in the trace, and after 5 days ageing of a 50 g propellant sample there was no significant difference between the rate of heat generation of a sample sealed under air and one sealed under nitrogen. The rates at which oxygen reacts with double-base propellants and the nature of the gaseous products have been shown to be very dependent upon the propellant composition, different stabilisers and ballistic modifiers having pronounced effects upon the reactions involved.[3] The effects of oxygen on heat generation would therefore be expected to be influenced by propellant composition.

Comparisons of heat generated at $80^{o}C$ by a N-methyl-4-nitroaniline stabilised propellant F528/214 (Table 2) packed in air and nitrogen are shown in Figure 4. During the first two days the nitrogen filled sample evolved more heat than that packed in air, which is in contrast to the behaviour of F527/327 (2-nitrodiphenylamine stabilised) under similar conditions. During the middle period of the test a maximum produced by the air packed sample was increased in magnitude and displaced on the time scale by the sample flushed with nitrogen. From these results it can be seen that there is no regular advantage to be gained by flushing with nitrogen.

2.3 Effect of Water Content of Sample Vessel

Some materials which require testing for compatibility, such as rubbers, can absorb relatively large amounts of water if left in a humid atmosphere for some time before testing. The effect any additional moisture would have on the heat generation of F527/327 propellant was therefore evaluated.

The heat generation of two samples of propellant and 5% neoprene have previously been compared,[1] where one neoprene was dried over phosphorus pentoxide, the other having absorbed 5% water (the rubber under normal laboratory conditions contained ca 1% moisture). No detectable difference was observed between the heat generation of the two samples. The heat generation of normal F527/327 propellant (ca 0.2% water content) is shown in Figure 5, together with that for samples containing 2 and 5% additional distilled water. Whilst these higher rates of heat generation from the wet samples confirm the expected incompatibility of water, they also indicate that moisture levels of a few per cent in a test material would have little effect on the compatibility of that material, since the extra water introduced with 5% of test material would increase the water content of the mixture with the propellant by only approximately 0.1 - 0.2%.

2.4 Proposed Test Procedure for Compatibility Tests on Colloidal Propellants

2.4.1 Time and Temperature

The thermal decomposition reactions of nitrate esters are found to have an apparent activation energy of about 140 - 170 kJ/mole, in which case an accelerated ageing test lasting only one or two days at 80°C would be equivalent to ten years storage at 25°C.[3] In many cases however, these are not the only or even the principal reactions which take place during normal storage of colloidal propellants. In particular, when a propellant is mixed with a compatibility test material other reactions of lower activation energy may take place. For an activation energy of 84 kJ/mole, more typical of the majority of normal chemical reactions, an accelerated ageing test lasting 19 days at 80°C is necessary to be equivalent to ten years storage at 25°C.[4] Conventionally tests lasting 20 - 30 days at 80°C have, of experimental necessity, been used in the past for compatibility testing, and the evidence of these calculations is that this is the most suitable time scale and temperature for routine heat flow calorimetry testing also. Though heat flow calorimetry testing can concievably be performed at lower temperatures, a disproportionately long test time would be required for routine testing in order to guarantee adequate Service life.

2.4.2 Sample Mass and Gaseous Environment

In order for some oxygen to be present in the sample vessel throughout the time scale of the test, a very low mass of propellant would have to be used in the vessel (the output from this would be more difficult to measure) and a continuously varying atmosphere of depleting oxygen content would be present above the propellant. This continuously varying atmosphere was also found unsuitable for temperature dependence calculations in some earlier work[5] and is therefore not favoured. The procedure of packing the vessel with a large mass of propellant under a nitrogen atmosphere is one possible method, but is time consuming if done effectively enough to guarantee reproducibility and as has been shown earlier the effect differs with propellant composition. The second method therefore, and the one normally employed, was to pack the maximum mass of propellant (for ease of measuring the heat evolved) into the vessel in an air atmosphere (for reproducibility). This method produced depletion of oxygen in less than 5 days at 80°C and thereafter comparison between samples could be made for a long period, from 5 - 20 days in an oxygen-free atmosphere. With F527/327, and most propellants, this time scale 5 - 20 days also corresponds to a relatively more linear variation in rate of heat generation with time.

2.4.3 Proportion of Propellant to Test Material

The proportion of test material relative to propellant has been kept low for compatibility tests for the following reasons. If a large proportion of material is added to propellant in the sample vessel, it may affect the rate of heat generation solely because it has displaced a large volume of available oxygen from the vessel and not because of chemical reactions. Also, interest in this test is primarily in the reactions occurring in the propellant, not in the test material. By using a low proportion of test material the contribution to the total heat generated from degradation reactions of the material, reaction of acids on the material, heat of absorption of nitroglycerine, etc is kept to a minimum; addition of 5% material to the propellant has therefore been favoured.

Figure 6 shows how increasing the quantity of material under test, in this case natural rubber, increases the quantity of heat generated by the test propellant F527/327 at 80°C; it is therefore important that a standard addition of test material is used when testing compatibility by this method.

3 EFFECT OF THE ADDITION OF STABILISING MATERIALS ON THE HEAT GENERATION OF A PROPELLANT

3.1 Effect of Addition of Stabilisers to Unstabilised Double-Base Propellant

The heat generation of two plasticised but unstabilised nitrocellulose/ nitroglycerine mixes was initially measured over 8 days at 80°C. Propellant compositions are shown in Table 2. The rate of heat generation of F488/2368 plasticised with triacetin rose extremely rapidly and the propellant fumed and was removed after 4 days at 80°C. In contrast the otherwise identical F488/2369 mix, but plasticised with dibutyl phthalate, was reasonably stable for greater than 8 days. This difference is probably due to hydrolysis of triacetin to diacetin in the F488/2368 propellant, with liberation of acetic acid which would accelerate the nitric ester decomposition.

The effect on the heat generation of the dibutyl phthalate plasticised propellant F488/2369 of the addition of 2% and 5% stabilising compounds was then determined over 7 days at 80°C. "Propellant-grade" samples of 2-NDPA, p-NMA and resorcinol were obtained from the propellant manufacture section for this purpose. Results obtained are shown in Figure 7.

The addition of 2-NDPA to unstabilised propellant was observed to produce a slightly higher rate of heat generation in the period 1 - 3 days until a minimum in the trace occurred, presumably associated with complete oxygen consumption in the system. Thereafter the sample produced much lower rates of heat generation and showed good stabilising action in that the rate of heat generation was not rapidly increasing as was that of the unstabilised propellant.

The addition of resorcinol produced significantly higher rates of heat generation initially, and the minimum in the trace corresponding to oxygen depletion appeared sooner, these effects are no doubt associated with oxidation reactions of resorcinol. Thereafter the resorcinol stabilised propellants continued to have higher heat generations than both unstabilised material and 2-NDPA stabilised propellant. Whilst some of this significant effect may be due to more rapid reaction of resorcinol with degradation products and acids than 2-NDPA and a higher heat of reaction, also side-reactions where resorcinol is reacting with its own degradation products may be contributing to this effect.[5]

The addition of p-NMA increased the rate of heat generation of the propellant by an unexpected degree. As p-NMA is reputed to have no direct reaction with propellant ingredients or its own degradation products,[5] more rapid reaction with propellant degradation products, and higher heats of this reaction would not appear sufficient to explain this effect. Repeating these experiments with recrystallised p-NMA did not significantly alter the heat generation shown in Figure 7. No explanation can be offered for this behaviour at present.

3.2 Effect of Addition of Extra Stabilisers to a 2-NDPA Stabilised Double-Base Propellant

In order to study the effects of stabilisers over a longer time scale than in the previous sections, and also to examine the effect of the addition of a stabilising material in a compatibility test, the effect of the addition of ca 2 and 5% of 2-NDPA, p-NMA and resorcinol respectively on the heat generation of F527/327 was determined by mixing the stabilisers and propellant by hand in the vessel and heating for 20 days at 80°C. This substrate propellant F527/327 is stabilised with 0.5% 2-NDPA.

As also noted in experiments on the unstabilised propellant, the addition of extra 2-NDPA was observed to decrease the rate of heat generation of the propellant throughout the 20 days at 80°C, and the time of ageing corresponding to oxygen depletion (minimum in the trace) also increased. The addition of 4% stabiliser had a greater effect than 2% stabiliser (Figure 8).

The addition of p-NMA increased the rate of heat generation of the propellant throughout the 20 days at 80°C, as shown in Figure 9. Though an increase in rate of heat generation may be due to increased rate or heat of reaction between p-NMA and nitric ester decomposition products, these increases were higher than expected and are being further investigated. The increase however was not as high as that obtained by contact between the propellant and an incompatible material (see later sections).

The addition of resorcinol to F527/327 increased the rate of heat generation very significantly over 20 days at 80°C (Figure 10), the amount of extra heat evolved being such that resorcinol would have been deemed incompatible by this method for use in this propellant, because of the excessive extra reactions occurring. This extra heat evolved probably comes from the side-reactions of the resorcinol stabiliser where it reacts with its own decomposition products.[6]

4 COMPATIBILITY TESTING OF COLLOIDAL PROPELLANTS USING A STANDARD DOUBLE-BASE PROPELLANT AS SUBSTRATE

4.1 Tests at 80°C

The rates of heat generation in 30 compatibility tests on mixtures of F527/327 propellant (50 g) and test material (2.5 g), packed into the sample vessel in air atmosphere, have been measured over about 20 days at 80°C. These results are collated in Table 3 and presented as a ratio of the rate of heat generation of the propellant/test material mix to the rate of heat generation of the propellant alone at various times of storage. The test materials in

Table 3 are also arranged in sections, determined by the percentage effect the material has on the time to fuming in the Silvered Vessel compatibility test. If these results are studied the following points emerge for compatibility testing by Heat Flow Calorimetry using F527/327 as substrate at 80°C:

(i) Materials which were incompatible or showed a high degree of reaction with the propellant (effect on Silvered Vessel fume time greater than 33%) gave higher ratio in the heat flow calorimetry over the 20 day test as a whole.

(ii) Compatible materials (effect on Silvered Vessel -33 to +33%) had no heat flow calorimetry ratio of greater than about 2 - 2.1 at any time during the 2 - 20 day period.

(iii) Incompatible materials had heat flow calorimetry ratios of at least 2.0 at some time during the 2 - 20 day period. This high ratio may be present throughout (eg Tretobond) or may develop after the test has been in progress for some days (eg Rigid PVC), illustrating the need to extend the test to about 20 days. (Only one exception has been noted to this conclusion, Araldite 901 hardener, and this was a borderline material by both compatibility tests.)

(iv) Some materials compatible by previous Silvered Vessel test have high reaction rates with the propellant in the first 2 days at 80°C (eg ethyl cellulose sample and Tufnol), an effect the Silvered Vessel test does miss. Unfortunately these do occur in the 0 - 2 day period when results may have some error due to the calorimeter warm-up time. These high readings can be and were confirmed however by a compatibility test at a lower temperature when these high reaction rates extend over a longer time scale.

4.2 Tests at Temperatures Lower than 80°C

Routine compatibility testing at temperatures lower than 80°C would have to last correspondingly longer periods of time in order to correspond with an adequate life-time at ambient storage temperatures. But tests at lower temperatures have certain applications, eg a test at 80°C may give high rates of reaction in the first 2 days of test when results are subject to some error, or give erroneous results if the material itself is decomposing at these temperatures.

Using F527/327 propellant, the test could be adequately performed at temperatures down to 60°C. A normal compatible material (Delrin acetal resin) which produces heat flow calorimetry ratios of 0.94 to 1.31 over 20 days at 80°C, also produced ratios regarded as normal and compatible over 7 days at 60°C. Two test materials which gave very high ratios in the 0 - 2 day period at 80°C, which then decreased at longer ageing times, were confirmed to have a high rate of reaction with the propellant by tests at 60°C. These high heat flow calorimetry ratios then continued well after the 0 - 2 day warm-up period. These results are shown in Table 4.

By the use of other propellant compositions as substrate which have higher rates of heat generation, compatibility tests could be successfully done at lower temperatures. Results are shown in Table 4 for the p-NMA stabilised propellant F528/214 at 50°C, and for SC, a high carbamite content propellant, at 40°C.

4.3 Conclusions

Compatibility testing of materials with colloidal propellant can conveniently be performed by heat flow calorimetry at 80°C using a standard double-base propellant as substrate. Though an indication of a material's compatibility is usually obtained within 7 days, it is necessary to continue the accelerated ageing for about 20 days in order to ensure adequate Service life. Judgement of results for compatibility will be similar to those indicated in Section 4.1. With this propellant it may in future be more economic in terms of instrument usage to measure the heat generation of the sample from, for example, 0 - 7 days, and then unless the test material is already indicated as incompatible, transfer the sample to an oven at 80°C between 7 and 15 days and then reinsert in the calorimeter for further readings in the period 15 - 20 days ageing.

If any test material produces high rates of heat generation in the first two days of ageing only, or gives results indicating that the material itself has decomposed at 80°C, then a compatibility test may be also performed at 60°C over a longer period of time.

5 COMPATIBILITY TESTING USING OTHER COLLOIDAL PROPELLANTS AS SUBSTRATE

Due to the sensitivity of the heat flow calorimetry technique it has the potential advantage of being applicable to any propellant composition. Consequently, tests could be conducted on mixtures of the test material with Service propellants instead of with a special standard one such as F527/327. Figures 11 and 12 show the heat generation over 20 days at 80°C of a number of single, double-base and composite modified propellants, these results were obtained using 50 - 60 g of sample packed into the vessel in air. As expected differences were obtained in the overall heat generated by these propellant compositions but, more importantly, large variations were obtained in the manner in which their rates of heat generation varied with time. Somce propellants (EJC90 and NCP/302/47) gave rates of heat generation which increased steadily during the tests, ATN(D28)/19 produced an initially steady increase up to 15 days, this then increased very rapidly and culminated in the bursting disc of the sample vessel rupturing near the end of the 20 day test period. Other propellants (BDI(D1)/4 and FNH) produced rates of heat generation which rose quite sharply to a maximum and then slowly decreased. The effects of propellant composition on heat generation properties are being investigated using simple compositions specially formulated for this purpose, but as yet no explanations for the varying behaviour of the propellants shown in Figures 11 and 12 can be advanced.

Some preliminary results for compatibility tests conducted under the standard conditions between a few selected materials and a number of propellant compositions are shown in Table 5; several points of interest emerge from these:

(i) The degree of compatibility between a given material and different propellants, as expected, may vary. For example paraformaldehyde was shown to be relatively compatible with F527/327 propellant by heat flow calorimetry and Silvered Vessel Test. However the pressure of gaseous decomposition products caused the safety disc of a sample vessel containing a mixture of KU propellant and paraformaldehyde to burst after less than 15 days at 80°C.

(ii) Addition of test materials to propellant such as F528/214 and BDI(D1)/19 which have pronounced maximum rates of heat generation mid-way through the test, caused this maxima to appear either earlier or later as well as increasing the overall rate of heat generation.

These results whilst demonstrating the possibilities of using other propellants for compatibility testing by heat flow calorimetry, also show that it will be necessary to test many more materials with different propellant compositions before any conclusions may be formed as to a standardised method of interpretation.

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TABLE 1

Heat Evolution from Calorimeter after Insertion of Inert Sample at 25°C

Time (hours)	Differential Output (μV)	Temperature Inside Calorimeter (°C)
Before sample insertion		78.5
Insertion + 2		78.1
" + 5		78.3
" + 9		78.4
" +12		78.5
Calorimeter 4 @ 80°C		
Insertion + 6	- 1	
" +12	+ 0.5	
" +24	+ 0.5	
" +48	0	
" +72	0	
Calorimeter 3 @ 60°C		
Insertion + 6	+11	
" +12	+ 5	
" +24	+ 2	
" +48	0	
" +72	0	

TABLE 2

Propellant Composition

Propellant No	Nitro-cellulose %	Nitro-glycerine %	Triacetin %	Dibutyl phthalate %	p-NMA %	Resorcinol %	2-NDPA %
F488/2368	46	46	8	-	-	-	
F488/2369	46	46	-	8	-	-	
F488/2267	45.5	45.5	8	-	-	-	1
F488/2268	45	45	8	-	2	-	
F488/2269	45.5	45.5	8	-	-	0.44	
F527/327	36.4	57.1	-	6.0	-	-	0.5
F528/214	36.0	56.5	-	6.0	1.5	-	-

TABLE 3

Compatibility Tests at 80°C with F527/327 Propellant

Material	% Effect on Silvered Vessel Test Time	HFC Ratio = Rate of Heat Generation Propellant/Test Material / Rate of Heat Generation Propellant at Days								
		$\frac{1}{4}$	$\frac{1}{2}$	1	2	5	6	7	Maximum in 10 - 15	Maximum in 15 - 20
HMP 7102 Resin	-81	> 55	> 66	> 100	-	-	-	-	-	-
Chloroprene rubber	-61	7.6	4.7	1.34	1.52	-	1.89	2.15	-	2.83
Bostik Contact Adhesive	-63	3.3	2.19	1.80	-	-	-	-	2.63	5.3
Polystyrene Latex	-72	5.4	2.07	0.92	0.87	2.97	2.97	-	Burst seal	-
Araldite Hardener 901	-37	2.31	1.82	1.60	1.50	1.75	-	-	1.76	1.91
Vinylite VAGH	-37	1.15	1.16	1.29	1.84	2.54	-	-	3.4	3.7
Rigid PVC	-37	2.03	1.43	1.09	0.98	1.23	-	-	4.62	6.1
Zinc sulphide	- 5	6.0	3.8	2.52	1.72	1.71	-	-	1.45	1.34
Ethyl cellulose Mix 22	- 6	18.3	5.6	2.97	1.71	2.04	2.02	1.99	-	-
Ethyl cellulose Mix 1369	- 8	3.5	2.41	2.06	1.66	1.71	-	-	1.08	-
Flomat polyester resin	+ 3.9	1.42	1.27	1.29	1.27	1.41	1.39	1.60	-	-
Butyl rubber (BS 3227)	+ 6	2.61	2.63	2.00	1.08	-	-	-	1.84	1.82
Delrin acetal resin	15	1.31	1.06	1.01	0.94	0.95	-	-	1.00	1.08
Kemetal	1	1.96	1.59	1.24	0.95	0.90	-	-	1.51	-
Paraformaldehyde	15	- ve	- ve	0.57	1.09	-	-	-	1.90	-
Polypropylene white	0	1.40	1.12	1.11	1.10	1.04	-	-	1.05	-
Polythene PN 220	0	1.22	1.09	1.04	1.05	-	-	-	0.96	-
Alkathene 902/75	5	2.17	1.60	1.49	1.20	-	-	-	1.18	1.28
Ethyl cellulose Mix 71	5	10.2	3.3	2.19	1.74	1.47	1.45	1.42	1.31	-
DAX 505/45	2	3.5	1.91	1.60	1.53	0.98	-	-	1.16	-
DMC L3581 polyester resin	2	2.02	1.99	1.69	0.96	1.25	-	-	-	-
Coupled polystyrene	2	1.27	1.28	1.24	1.14	-	-	-	-	1.14
Fluorosint	16	2.06	1.27	1.32	1.31	1.28	-	-	1.10	-
B590 rubber	2	1.24	1.00	0.98	0.97	0.97	-	-	-	0.92
Cellulose acetate	29	0.90	1.06	1.07	1.08	1.38	-	-	-	1.06
Molecular sieve 5A	35	1.73	1.54	1.40	1.33	1.11	-	-	-	-
Tufnol	57	8.0	1.52	1.72	-	2.17	-	-	2.10	2.40
Natural rubber CS 3105	75	4.6	3.1	0.90	1.24	2.56	-	-	4.53	-
Redux 120 No 80/7034	60	2.75	2.42	2.38	2.06	1.40	-	-	-	-
Subaseal closures	66	2.12	2.33	2.32	1.64	1.61	1.88	-	4.30	-
Araldite Ay 103/Hy 956	169	>20	>20	> 20	> 20	> 20	-	-	19	-
Adipic dihydrazide	201	3.9	4.2	4.4	5.6	11.2	-	-	11.1	-
Tretobond AP 333	169	3.7	2.82	2.19	2.95	3.62	-	-	5.30	-

TABLE 4

Compatibility Tests at 40 - 60°C

Propellant	Temperature, °C	Test Material	HFC Ratio = $\frac{\text{Rate of Heat Generation Propellant/Test Material}}{\text{Rate of Heat Generation Propellant}}$ at Days								
			$\frac{1}{4}$	$\frac{1}{2}$	1	2	3	4	5	6	7
F527/327	60	Ethyl cellulose Mix 71	76	14	6.6	4.5	3.1	2.3	-	-	2.06
"	60	Delrin acetal resin	0.66	1.75	1.25	1.20	1.19	1.16	1.40	1.40	1.41
"	60	Natural rubber (CS 3105)	27	7.7	6.2	6.0	5.6	4.0	-	-	-
F528/214	50	Ethyl cellulose Mix 71	2.40	2.23	2.43	-	-	1.98	1.71	1.42	
SC	40	Vinylite VAGH	-	-	4.6	2.8	2.1	2.0	1.67	1.69	-
For comparison:											
F527/327	80	Ethyl cellulose Mix 71	10.2	3.3	2.2	1.7					
"	80	Delrin acetal	1.31	1.06	1.01	0.94					
"	80	Natural rubber (CS 3105)	4.6	3.1	0.92	1.24					
"	80	Vinylite VAGH	1.15	1.16	1.29	1.84					

TABLE 5

Compatibility Tests at 80°C Using Various Propellants as Substrate

Material	Propellant	HFC Ratio = $\frac{\text{Rate of Heat Generation Propellant/Test Material}}{\text{Rate of Heat Generation Propellant}}$ at Days										
		$\frac{1}{4}$	$\frac{1}{2}$	1	2	3	4	5	6	7	Maximum in 10 - 15	Maximum in 15 - 20
Araldite Hardener 901	F527/327	2.31	1.82	1.60	1.50	1.73	1.66	1.75	-	-	1.76	1.91
	F528/214	2.09	1.74	1.42	1.78	2.06	1.69	-	-	-	1.35	1.70
	SC	3.22	3.63	1.56	1.68	-	-	-	-	-	-	1.64
	N	5.03	4.06	2.65	1.36	1.21	1.25	-	-	-	1.88	1.88
	KU	2.63	2.25	1.92	1.53	1.32	1.23	1.32	1.52	1.61	1.49	1.20
Butyl rubber (BS 3227)	F527/327	2.61	2.43	2.00	1.08	1.73	-	-	-	-	1.84	1.82
	F528/214	1.16	1.01	0.82	1.00	1.14	1.12	-	-	-	1.23	1.10
	SC	3.84	1.41	1.16	1.14	0.93	-	-	-	-	0.73	0.80
	N	10.02	2.52	2.44	2.00	1.52	1.57	1.33	-	-	1.17	1.12
	KU	2.97	1.83	1.23	1.61	1.46	-	-	-	-	1.09	0.70
	BDI/D1/4	2.95	2.46	1.76	1.10	1.32	2.08	2.70	-	-	1.13	1.10
Natural rubber (CS 3105)	F527/327	4.6	3.1	0.92	1.24	2.56	-	-	-	-	4.53	> 4
	BDI/D1/4	2.66	1.97	1.05	1.10	1.16	1.16	-	-	-	1.95	2.44
	ATN/D28/19	1.72	1.58	0.84	0.82	0.79	0.77	0.76	0.74	-	0.77	0.94
Paraformaldehyde	F527/327	-ve	-ve	0.57	1.09	-	-	-	-	-	1.90	-
	F528/214	-ve	0.38	0.61	0.88	1.03	-	-	-	-	1.92	2.70
	NC	-ve	-ve	1.73	1.90	-	-	2.25	-	-	-	3.45
	KU	-ve	-ve	0.81	1.57	-	1.76	1.80	2.43	3.11	burst seal	-
	EJC 90	> 16	-	-	-	-	-	-	-	-	-	-
Delrin acetal resin	F527/327	1.31	1.06	1.01	0.94	0.95	-	-	-	-	1.00	1.08
	N	1.78	1.41	1.35	1.24	1.03	1.22	-	-	-	1.80	1.74
	BDI/D1/4	1.45	1.20	1.12	1.08	1.02	1.13	1.15	1.26	-	1.06	1.06
PVC	N	3.71	2.44	2.10	1.67	0.97	0.92	0.80	1.0	1.0	1.0	1.0
	BDI/D1/4	2.52	1.96	1.61	1.04	1.09	1.27	1.33	1.58	-	0.72	0.73

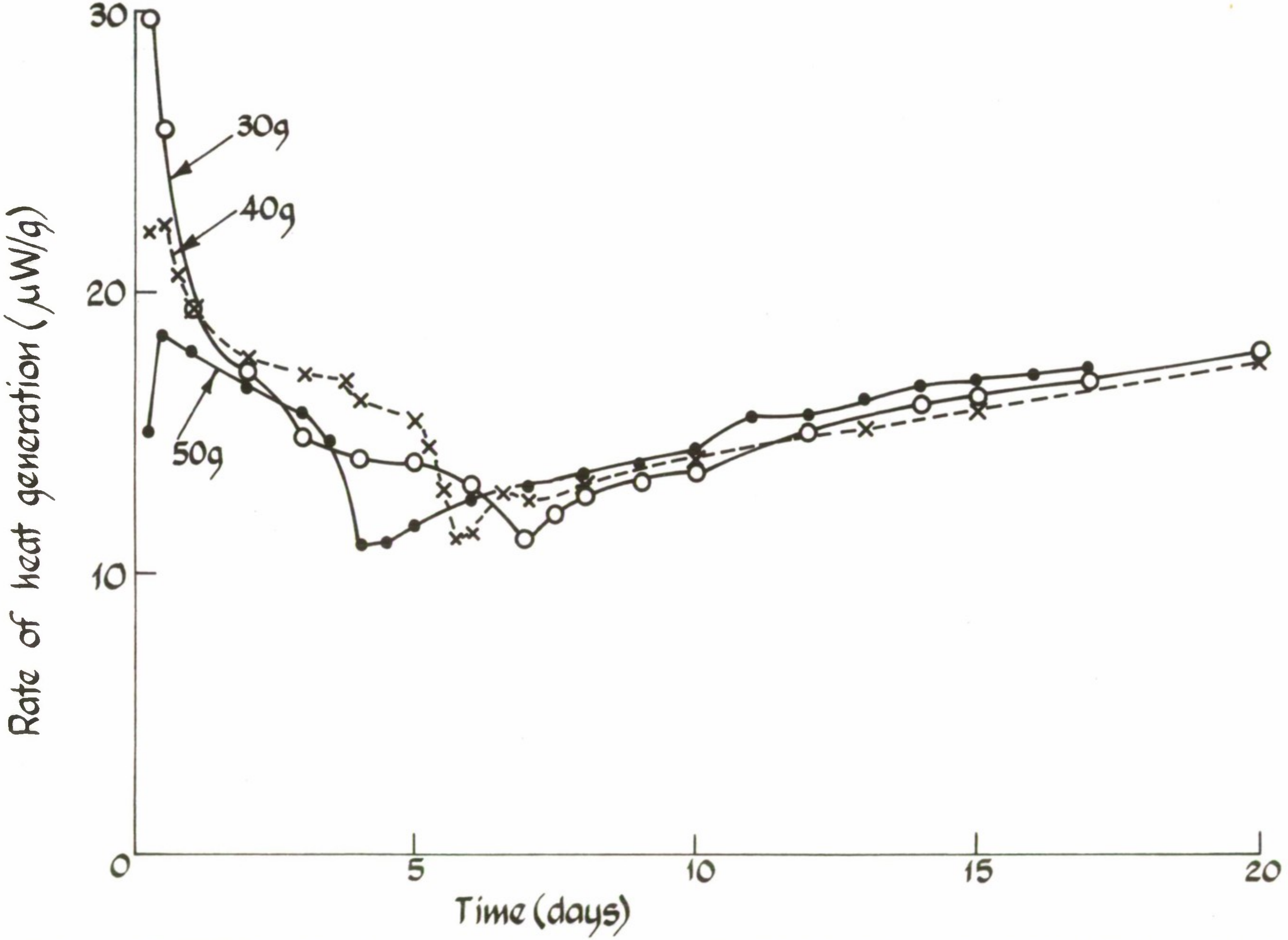

FIG. 1 EFFECT OF SAMPLE MASS ON HEAT GENERATION OF F527/327 AT 80°C

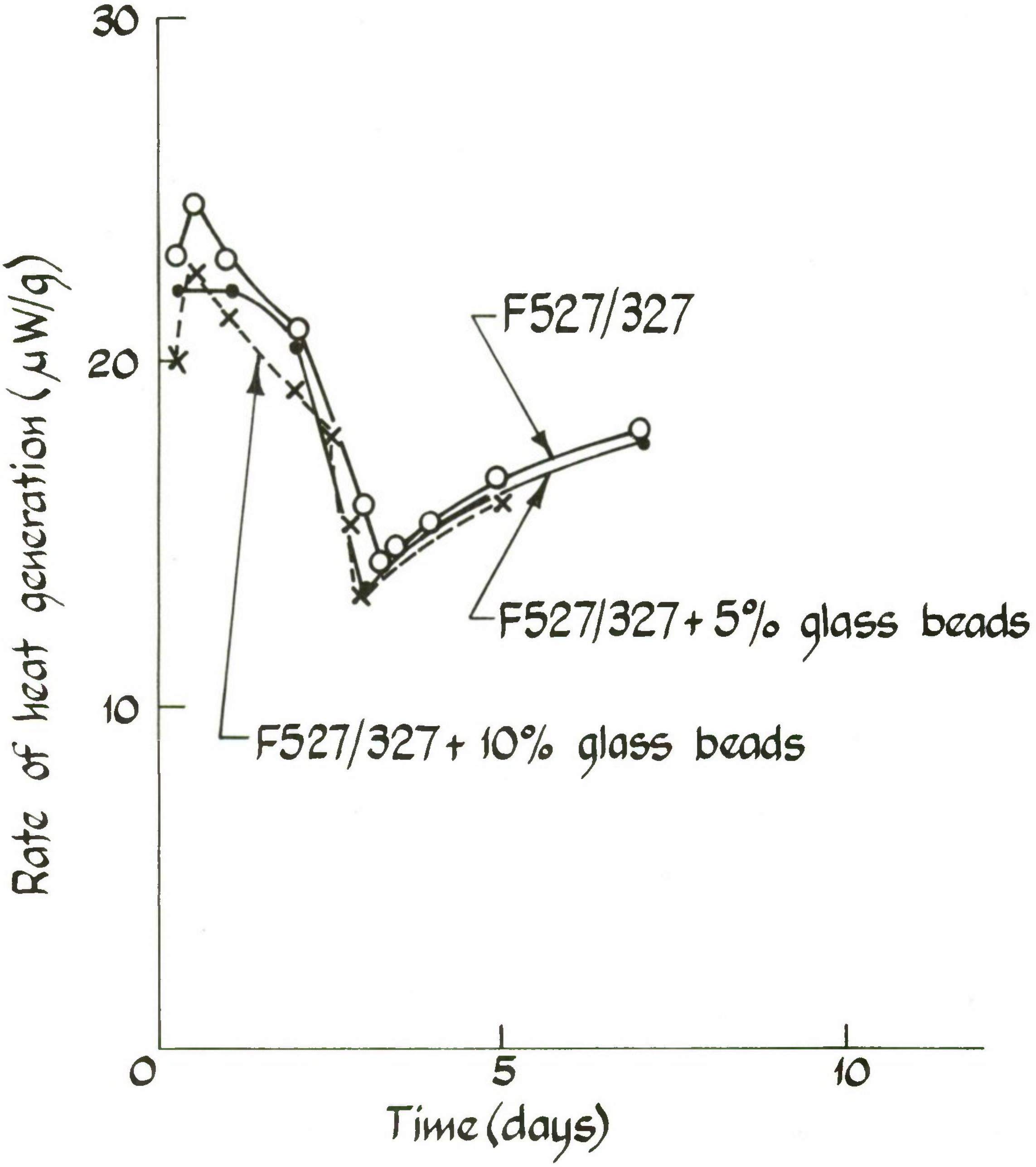

FIG. 2 EFFECT OF AVAILABLE OXYGEN ON HEAT GENERATION OF F527/327 AT 80°C

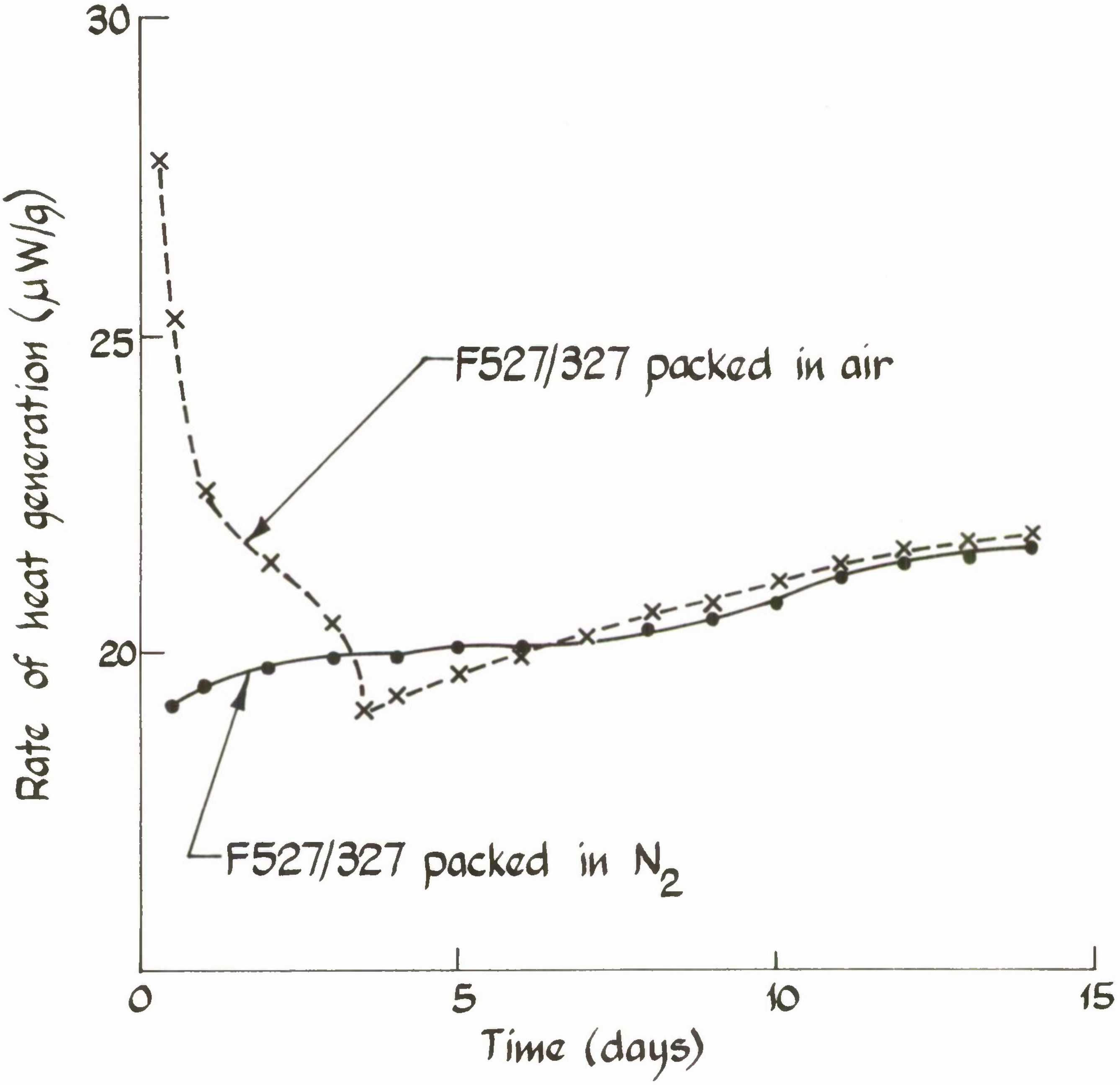

FIG. 3 EFFECT OF AVAILABLE OXYGEN ON HEAT GENERATION OF F527/327 AT 80°C

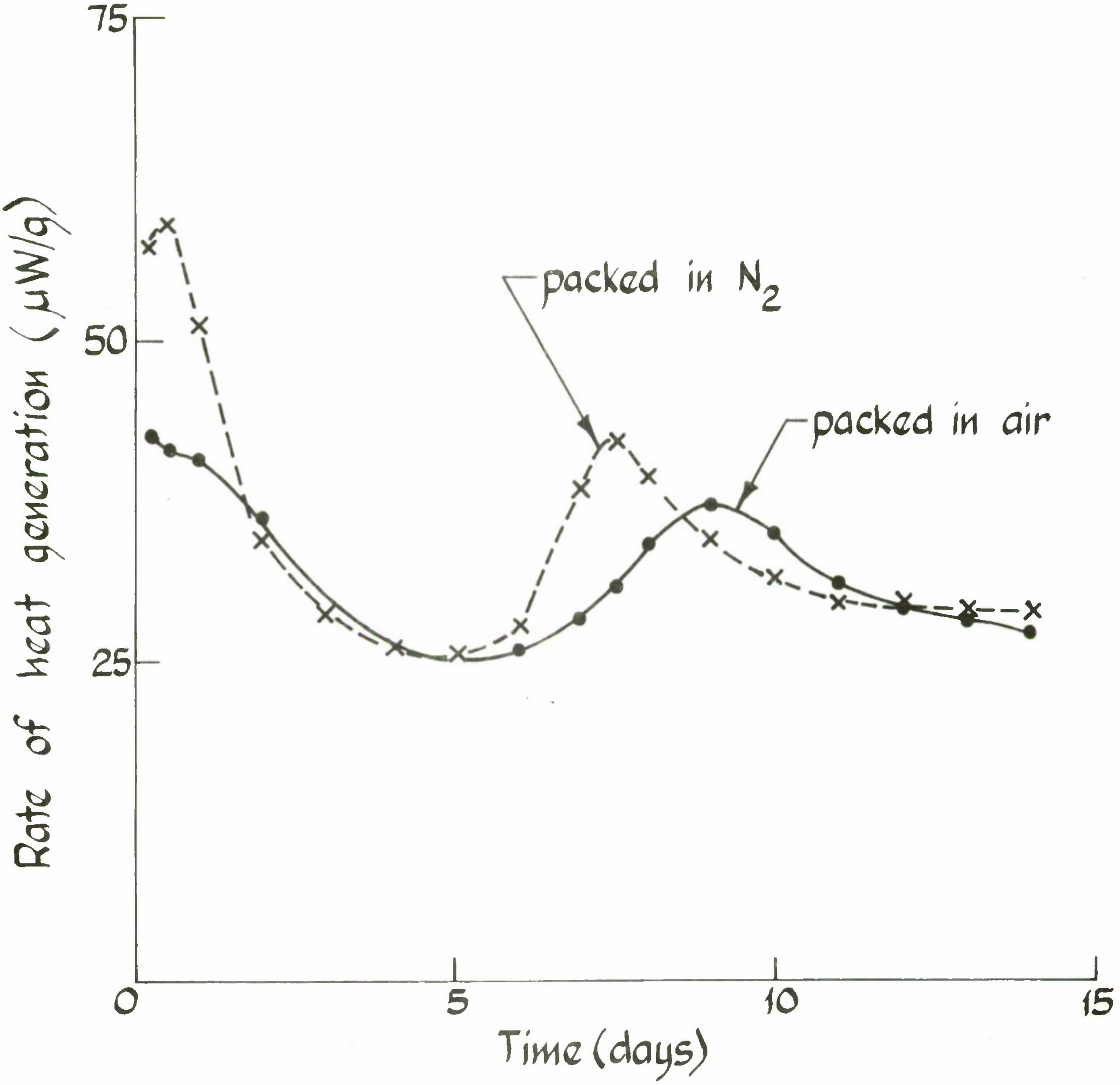

FIG. 4 EFFECT OF AVAILABLE OXYGEN ON HEAT GENERATION OF F528/214 AT 80°C

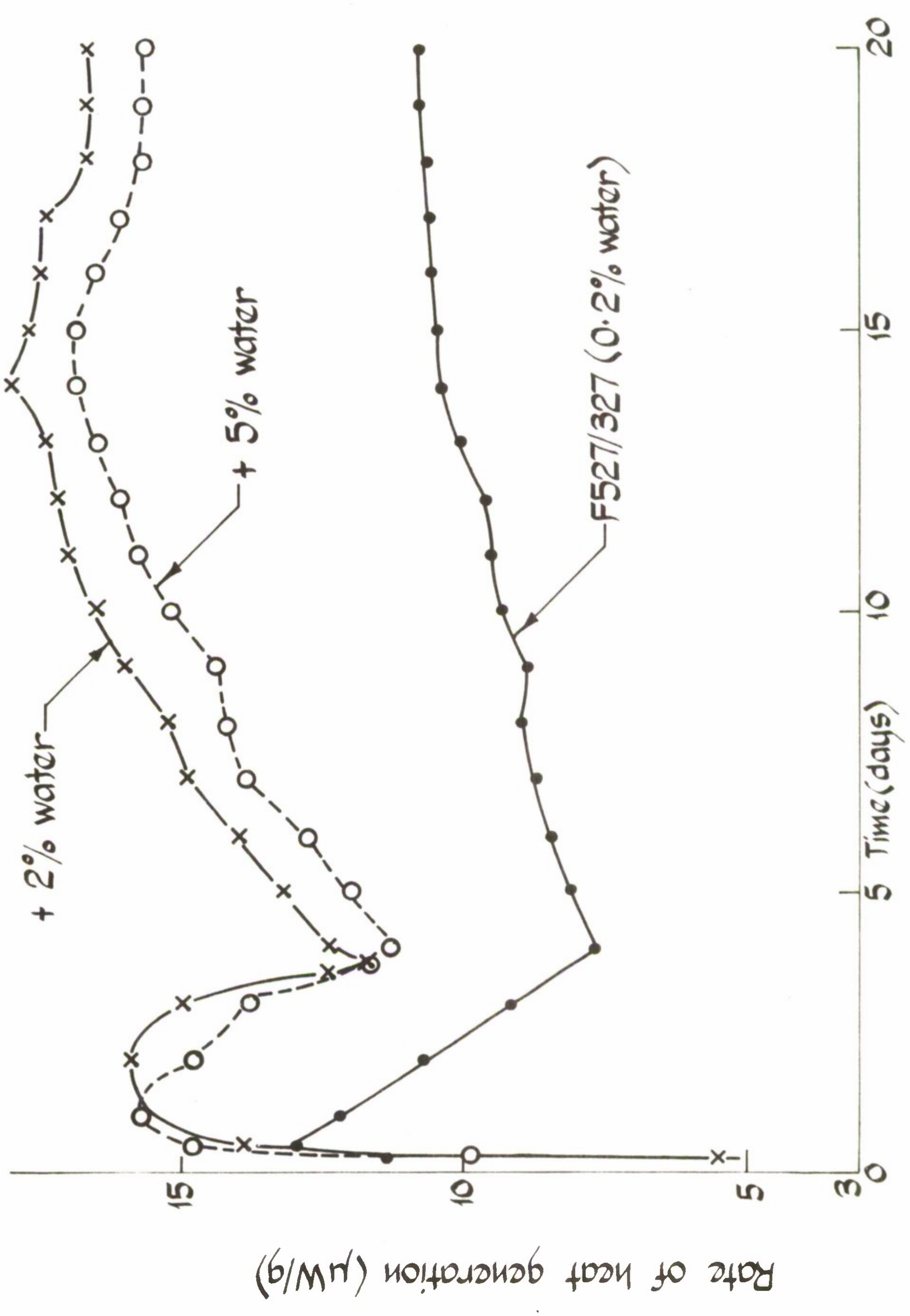

FIG.5 THE EFFECT OF WATER ON THE HEAT GENERATION OF F527/327 AT 80°C

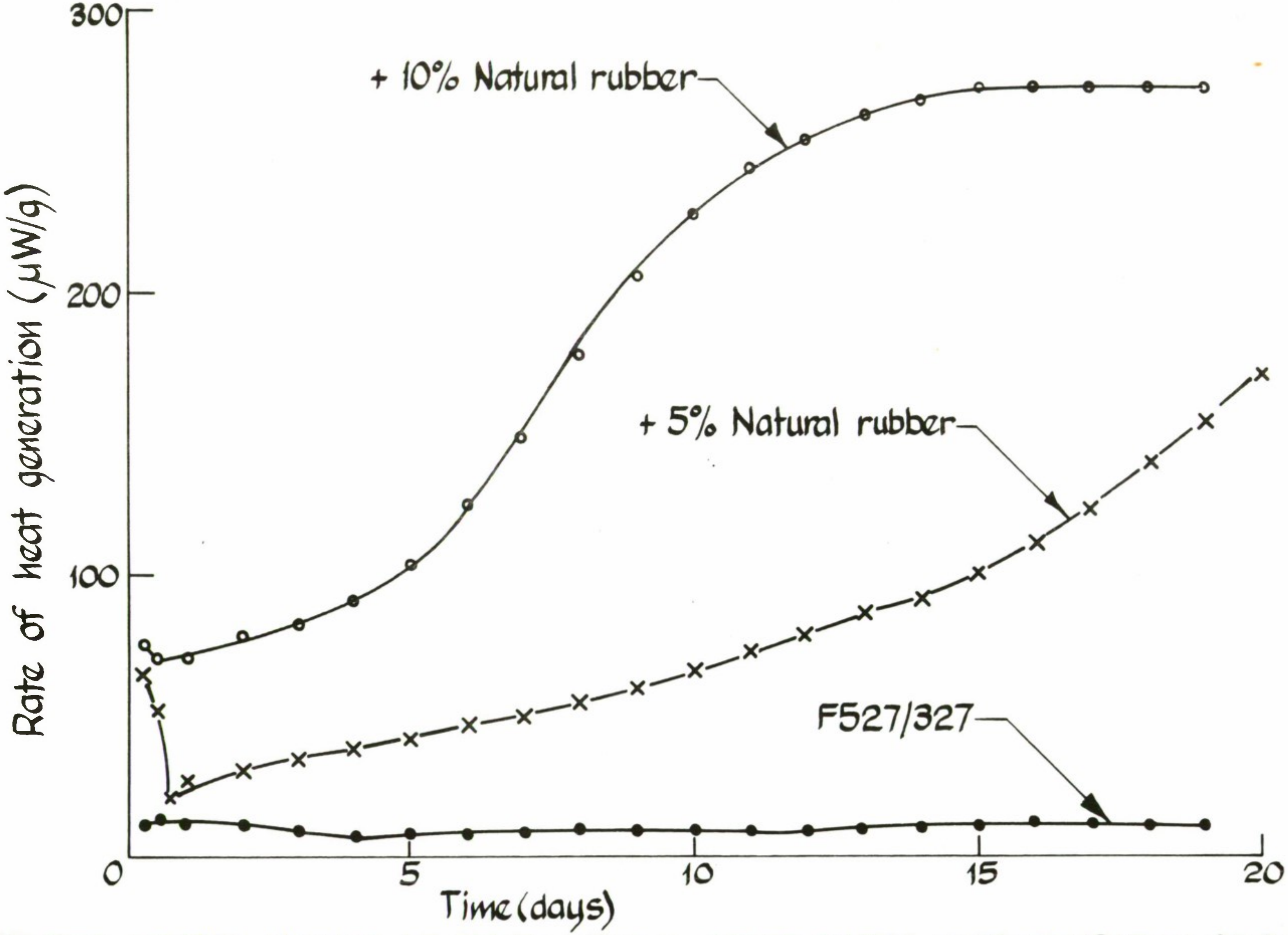

FIG.6 EFFECT OF THE AMOUNT OF TEST MATERIAL ADDED ON THE HEAT GENERATION OF F527/327 AT

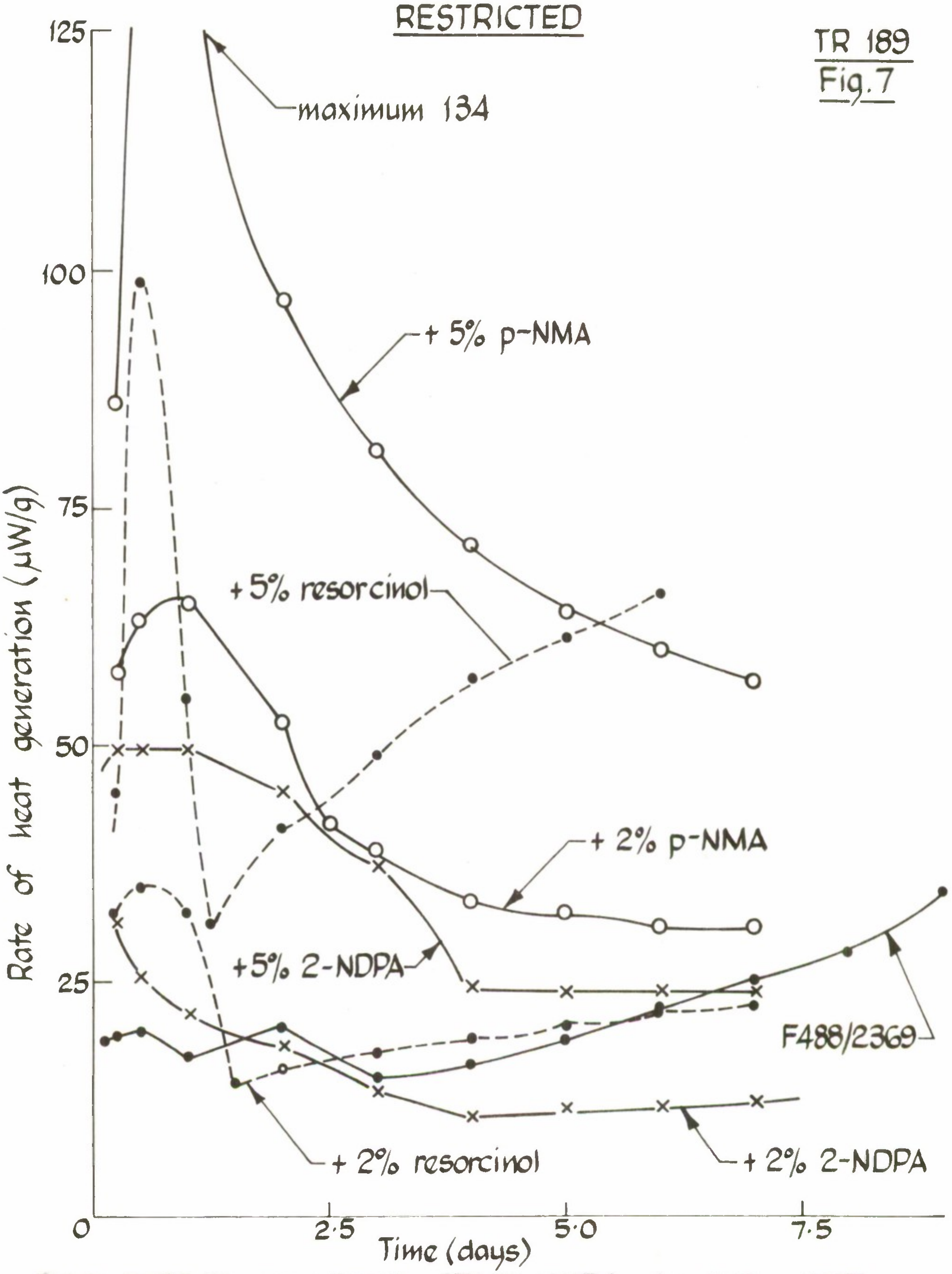

FIG.7 EFFECT OF ADDED STABILISERS ON THE HEAT GENERATION OF AN UNSTABILISED PROPELLANT AT 80°C

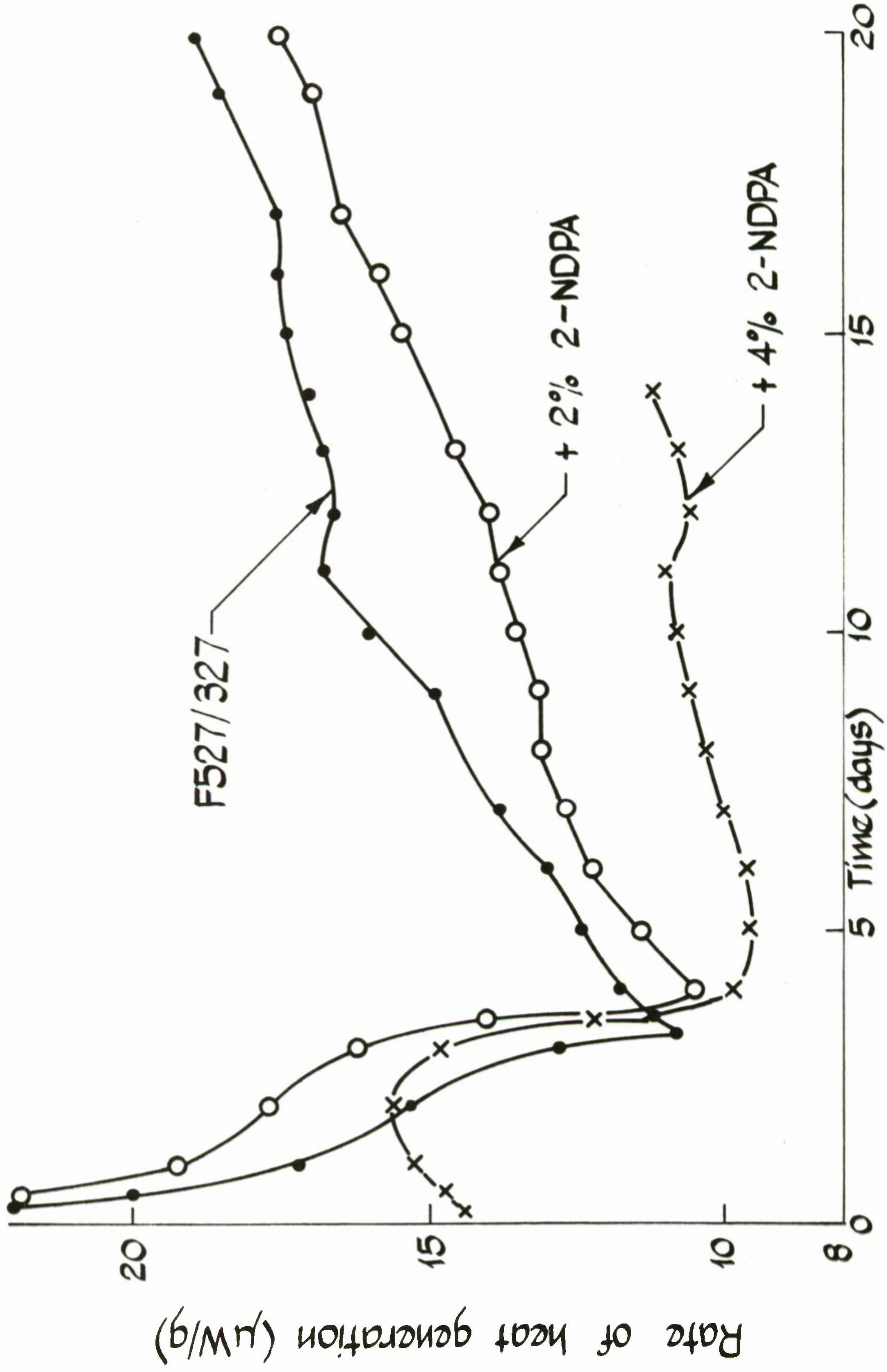

FIG. 8 THE EFFECT OF EXTRA ADDED STABILISER ON THE HEAT GENERATION OF A 2-NDPA STABILISED PROPELLANT AT 80°C

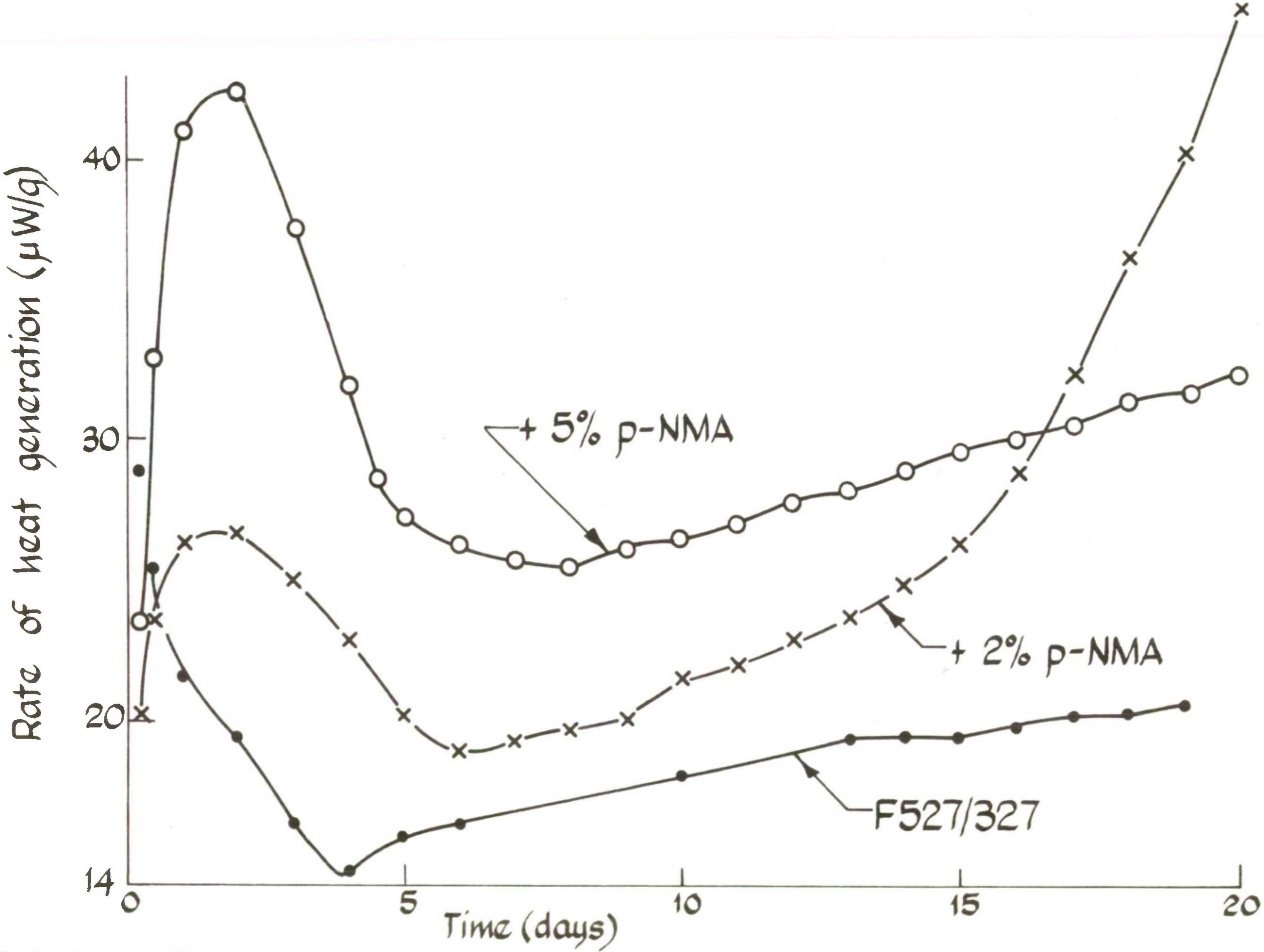

FIG.9 THE EFFECT OF EXTRA ADDED STABILISER ON THE HEAT GENERATION OF A 2-NDPA STABILISED PROPELLANT AT 80°C

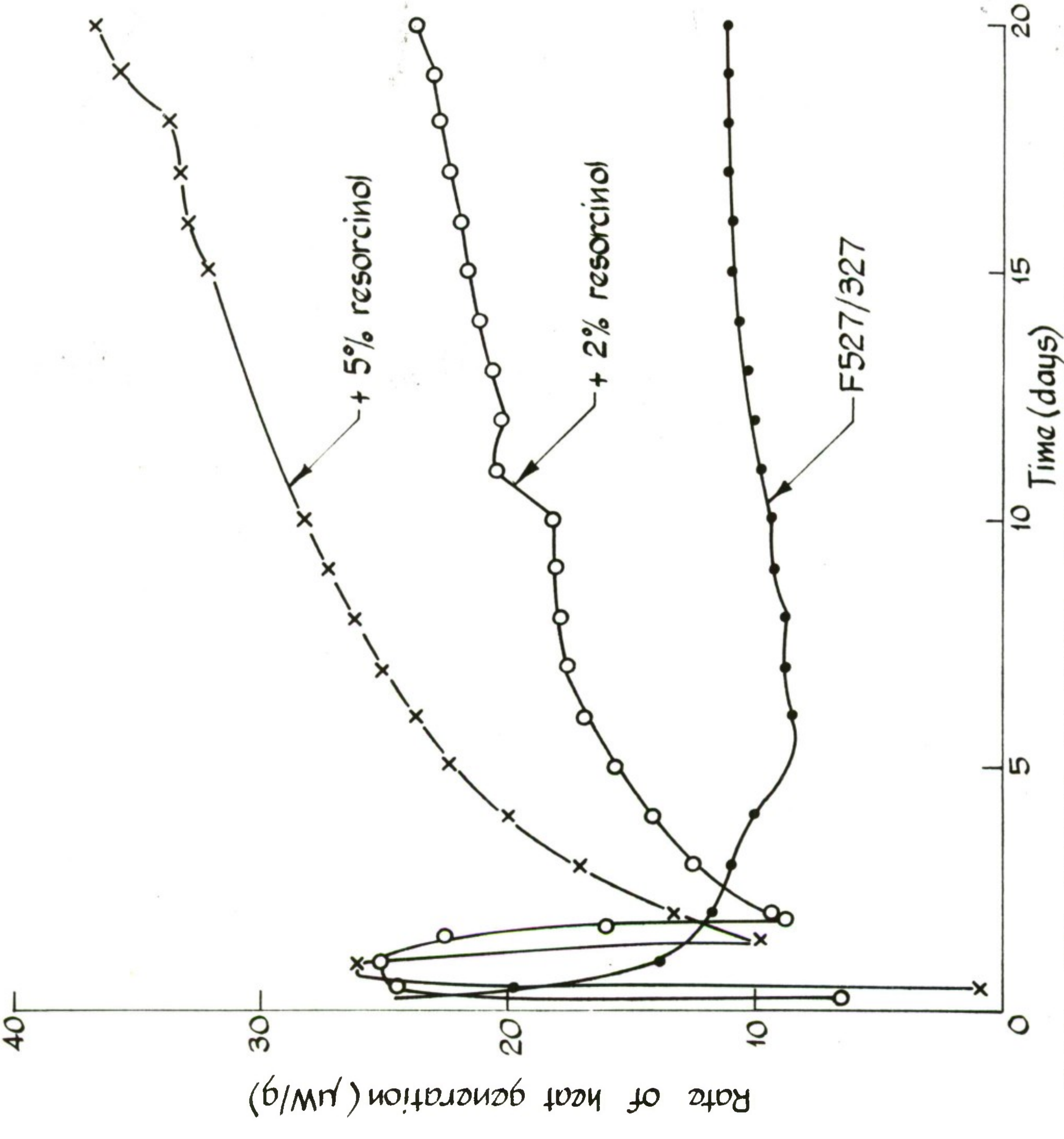

FIG. 10 THE EFFECT OF EXTRA ADDED STABILISER ON THE HEAT GENERATION OF A 2-NDPA STABILISED PROPELLANT AT 80°C

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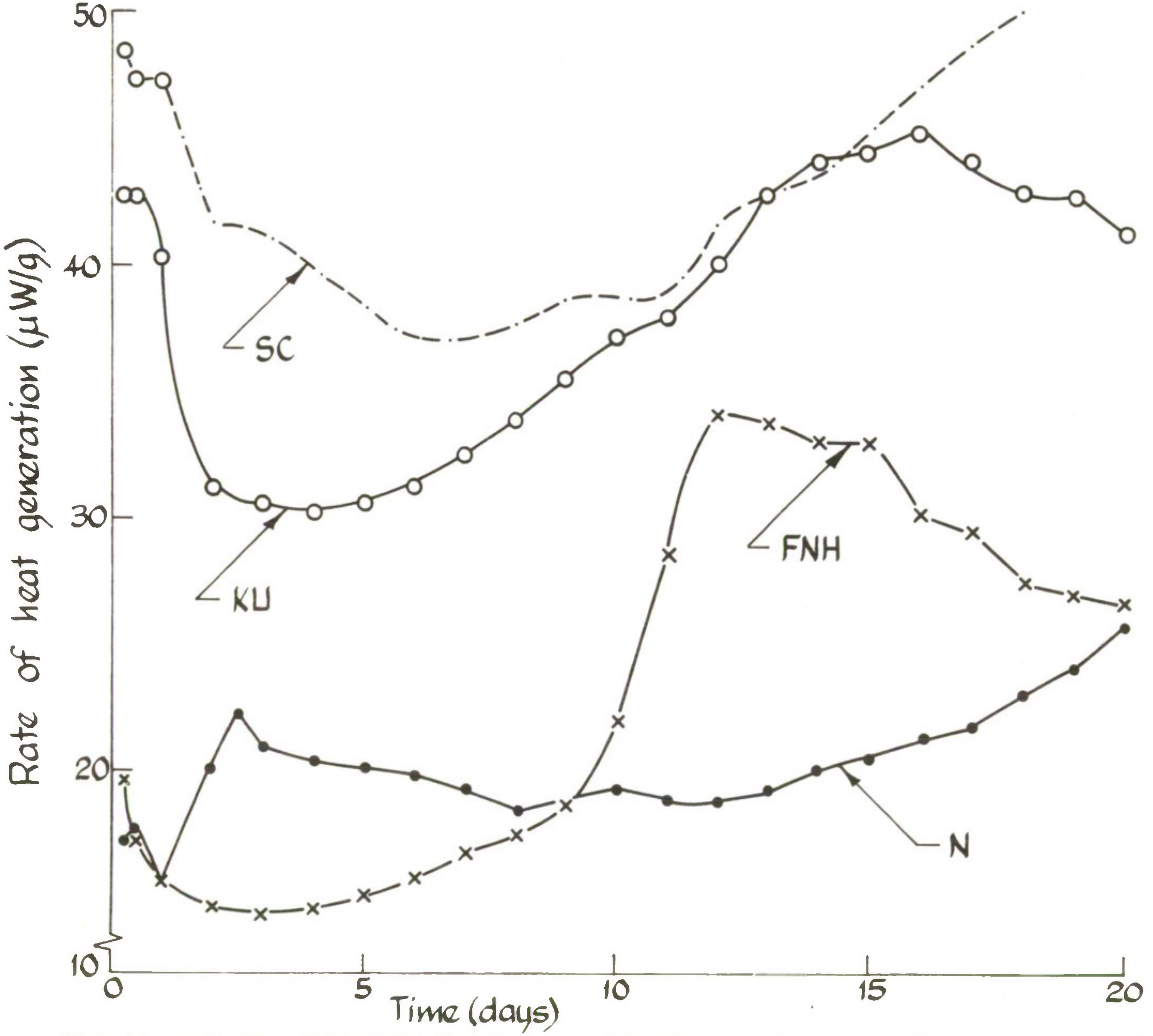

FIG. 11 HEAT GENERATION OF SOME SINGLE AND DOUBLE-BASE PROPELLANTS AT 80°C

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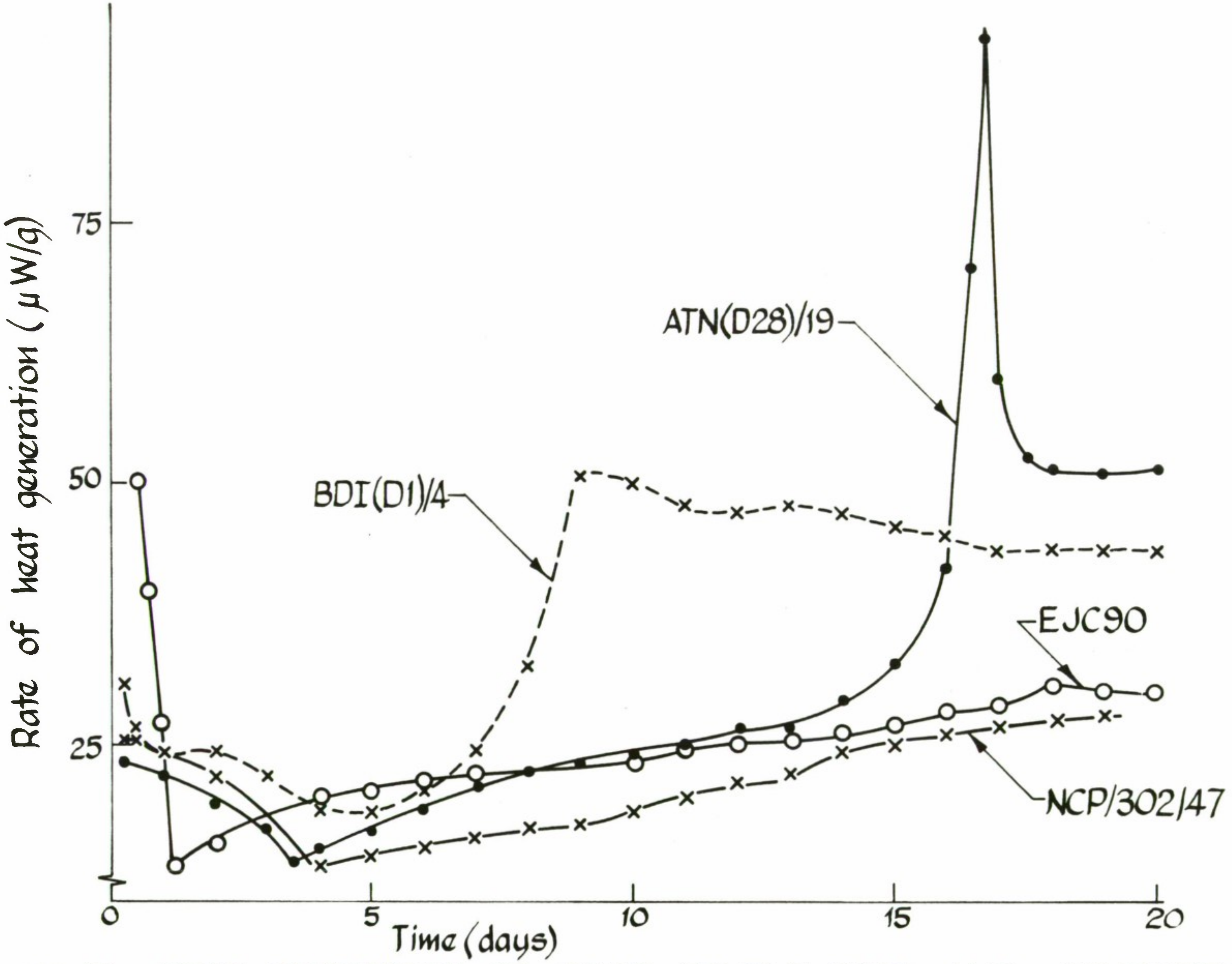

FIG. 12 HEAT GENERATION OF SOME DOUBLE-BASE AND COMPOSITE MODIFIED DOUBLE-BASE PROPELLANTS AT 80°C

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1. DRIC Reference (if known)	2. Originator's Reference ERDE TR 189	3. Agency Reference	4. Report Security Classification R

5. Originator's Code (if known)
445050

6. Originator (Corporate Author) Name and Location
Explosives Research and Development Establishment
Waltham Abbey
Essex

5a. Sponsoring Agency's Code (if known)

6a. Sponsoring Agency (Contract Authority) Name and Location

7. Title
COMPATIBILITY AND STABILITY TESTING OF SOLID PROPELLANTS BY HEAT FLOW CALORIMETRY. PART 2: FURTHER DEVELOPMENTS (U)

7a. Title in Foreign Language (in the case of translations)

7b. Presented at (for conference papers). Title, place and date of conference

8. Author 1, Surname, initials	9a Author 2	9b Authors 3, 4...	10. Date	pp	ref
Davies D G	Farey M G		9.1974	29	6
11. Contract Number	12. Period	13. Project	14. Other References		

15. Distribution statement

Descriptors (or keywords)

Propellant compatibility, Compatibility methods, Stability, Heat measurement

(TEST)

Abstract

The effects of variations in test procedure have been examined and a standard method of testing and interpretation is proposed using the Silvered Vessel test propellant (F527/327) as substrate.

A preliminary investigation into the use of other propellant compositions as substrate is described.

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Title: Compatibility and stability testing of solid propellents by heat flow calorimetry Pt 2 further developments
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