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Part II. (3) Build-Up of Detonation of Lead Azide in Various Media

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(3) BUILD-UP OF DETONATION OF LEAD AZIDE IN VARIOUS MEDIA

By A. R. Ubbelohde and P. Woodward

(*Report first issued by the Armament Research Department*, *September* 1945)

Two mechanisms have been proposed for the build-up of detonation by solid explosives:

(*a*) In the *self-heating* mechanism, when *heat* is evolved during thermal decomposition of the explosive faster than it can be conducted away, the temperature of the mass and the consequent rate of decomposition rise more and more. Ultimately the whole mass deflagrates more or less violently. The mathematical condition for self-heating has been formulated, but experiments show that a further condition is required for transition from deflagration to detonation, which has not yet been formulated mathematically.

(*b*) In the *mass-flow* mechanism, when the *gas* evolved during chemical decomposition of the explosive becomes comparable with the molecular mass flow required for stable detonation in the explosive, thermal decomposition changes into detonation.

To test these mechanisms measurements of delay to detonation were made with loose masses of lead azide, both Service and dextrinated, ranging from 10 to 200 mg. using previously described apparatus.

The azides were wetted with measured volumes of liquids with various boiling points, including: water, benzene, quinoline, diethylene glycol, glycerol, dibutyl phthalate, benzyl benzoate, nujol, tricresyl phosphate, and the effect on the detonation was observed. Mixtures of benzene with nujol and with dibutyl phthalate were also investigated.

Comparative measurements were made on the deflagration of cyclonite in the same apparatus, both dry and with added liquids.

The effect of liquids on the detonation of lead azide when heated was found to belong broadly to one of two classes:

(i) For liquids with the boiling points considerably below the temperature at which the test was being carried out, *detonation* followed after a longer delay than in the absence of liquid. There was evidence that the liquid first evaporated, and then normal detonation of the azide grains took place in the vapour phase thus formed.

This behaviour was shown by the following liquids:

liquid	b.p. (°C)	azide	range of testing temperatures (°C)
benzene	80	dextrinated	270–340
		Service	325–350
water	100	dextrinated	270–340
		Service	310–370
quinoline	238	dextrinated	300–350
diethylene glycol	244	dextrinated	260–350
		Service	320–360

A noteworthy feature was that the threshold detonation temperature was lower when the grains of azide were surrounded by various vapours in place of air (see also tables 22 and 24).

azide	vapour	lowering of threshold temperature compared with air
10 mg. dextrin	benzene	23°
	water	30°
	diethylene glycol	22°
	dibutyl phthalate	(raised 5°)
10 mg. Service	benzene	no effect
	water	(raised 3°)
	diethylene glycol	5°
	dibutyl phthalate	6°

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(ii) For liquids with boiling points considerably above the temperature of test, *no detonation* was observed. However, under certain circumstances a new phenomenon was observed, in that the lead azide '*deflagrated*' in a manner closely resembling the behaviour of the (self-heating) deflagration of an explosive such as cyclonite. This is quite different from the sharp detonation obtained with loose azide in air, when the masses are small.

When the boiling point was in the neighbourhood of the testing temperature, or with mixtures of liquids with boiling points above and below the testing temperature, both classes of behaviour were observed, according to the conditions of test.

Further, the temperature coefficient of the induction period for azide wetted with these intermediate liquids suggested that detonation occurred after the liquid had been displaced by nitrogen produced by thermal decomposition of some of the lead azide.

From the experimental results, it is concluded that

(*a*) With the masses used, lead azide will *detonate* only when the grains are surrounded by gas or vapour.

(*b*) Lead azide can *deflagrate* by a self-heating mechanism even under conditions where it will not detonate, e.g. when wetted by a liquid of very high boiling point such as tricresyl phosphate.

These conclusions support the view that the 'normal' mechanism of detonation of lead azide is controlled not by self-heating but by some process such as mass flow. When this normal mechanism fails to operate explosion may still occur by self-heating.

Introduction

Earlier work (parts II (1) and II (2)) has shown that there are at least two possible mechanisms by which explosives can reach detonation on heating. The first of these, so-called 'self-heating', has been shown *not* to explain the detonation of small masses of initiator. It has been suggested (part I) that these detonate as a result of the build-up of shock waves from the gas evolved from individual grains of initiator.

However, it was found by Hawkes & Winkler (1945) that the induction period of dextrinated lead azide approximates to that of Service azide when it is wetted with dibutyl phthalate. The authors explained this by suggesting that self-heating in the case of dextrinated azide heated in air is reduced by the improved thermal conductivity of the liquid medium. In view of the results quoted in the first paragraph, some doubt was felt about this explanation, and since the change from a gaseous to a liquid medium would be of critical importance in any shock wave build-up between the particles of lead azide, further experiments with other liquids appeared desirable.

The effect of adding various low-boiling and high-boiling liquids to Service and dextrinated azides before heating has been investigated; mixtures of liquids have also been tested in some cases, for reasons explained in the text.

Experimental methods

Lead azide was weighed into no. 8 Briska aluminium detonator sheaths using a paper balance (cf. part III (2)), and drops of the liquid under investigation were added from a small glass dropping tube, allowing one drop of liquid for every 10 mg. of initiator. These samples were then plunged into a furnace at constant temperature, and the time to detonation (or other effect) measured with a stopwatch.

Among the liquids used were benzene, water, diethylene glycol, glycerol, quinoline, dibutyl phthalate, 'nujol' (medicinal paraffin) and tricresyl phosphate; mixtures were also used. The sensitiveness of the samples to heat was tested as described previously (part II (1)

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and (2)), except that the heating bath in its more recent form contained *two* holes for molten solder, connected by a channel, such that surplus metal from one hole, on inserting a tube, can flow into the other.

EXPERIMENTAL RESULTS

Two classes of behaviour are observed on heating lead azide after wetting it by liquid, depending on whether the boiling point of the liquid is (*a*) lower, or (*b*) higher than the temperature at which the test is carried out.

In table 21 is given a list of liquids used with their respective boiling points.

TABLE 21

liquid	boiling point (°C)	liquid	boiling point (°C)
benzene	80	dibutyl phthalate	320
water	100	benzyl benzoate	323
quinoline	238	nujol	360
diethylene glycol	244	butyl glycol phthalate	370
glycerol	290	tricresyl phosphate	385

Low-boiling liquids

As might be expected, if the induction period is very much longer at the boiling point of the liquid than at the bath temperature, the first effect of adding a liquid of fairly low boiling point to lead azide is to lengthen the induction period, on account of cooling by vaporization.

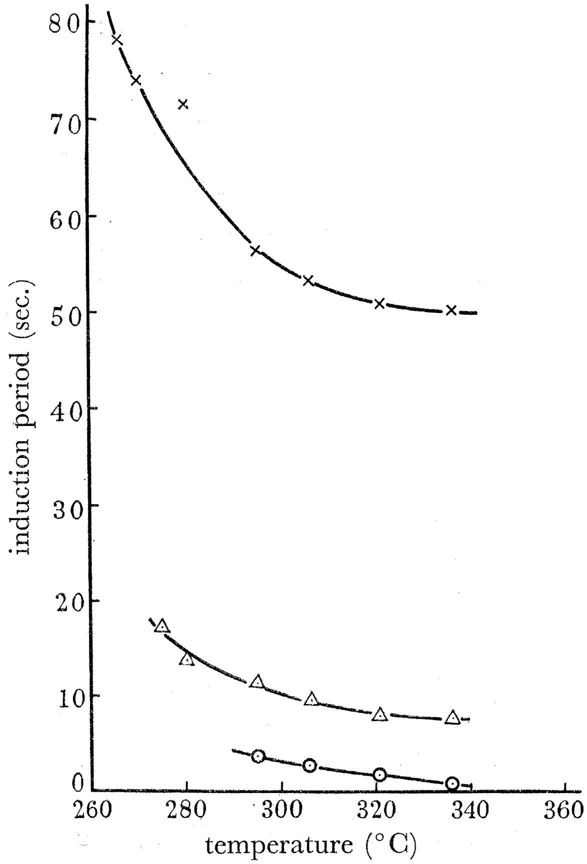

FIGURE 25. Effect of liquids on induction period of dextrinated lead azide. 10 mg. dextrinated lead azide. ⊙ Loose azide; × azide + 1 drop water; △ azide + 1 drop benzene.

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Assuming that no chemical reaction occurs between azide and liquid, the main effect will be that detonation is postponed while the liquid vaporizes, and then proceeds normally. A further point is that the grains are now surrounded by solvent vapour, plus a certain amount of nitrogen arising out of the thermal decomposition, in place of air.

If the increased induction period is due to vaporization only, and if the bath temperature is considerably higher than the boiling point of the added liquid, the time required for vaporization will lead to a more or less constant increment to the induction period, since the rate of vaporization will be approximately constant over the range of temperatures involved. Figure 25 shows a direct plot of induction period against temperature for dextrinated azide alone; also for dextrinated azide with benzene and with water. Figures are also given in table 22 showing the effect of diethylene glycol and quinoline. The corresponding failure temperatures are also indicated. It will be seen that in all cases, the lengthening of the induction period is approximately constant over the range of temperature for which detonation occurs.

TABLE 22. EFFECT OF VARIOUS LIQUIDS ON THE INDUCTION PERIOD OF DEXTRINATED LEAD AZIDE, AND THE CORRESPONDING THRESHOLDS OF DETONATION

substance (10 mg.)	bath temperature (°C)	induction period (sec.)	failure temperature below which no detonation is observed (°C)
dextrin azide alone	336	0·7	295
	321	1·6	
	306	2·6	
	295	3·5	
dextrinated azide + benzene (0·1 c.c.)	321	7·8	272
	306	9·3	
	295	11·4	
	280	13·6	
	275	17·2	
dextrinated azide + water (0·1 c.c.)	338	50·2	265
	321	50·9	
	306	53·4	
	295	56·5	
	280	71·3	
	270	74·0	
	266	78·0	
dextrinated azide + diethylene glycol (0·1 c.c.)	350	4·6	273
	336	6·1	
	306	7·0	
	295	6·0	
	280	7·2	
dextrinated azide + quinoline (0·1 c.c.)	350	7·0	not determined
	320	6·7	
	306	7·8	
dextrinated azide + dibutyl phthalate (0·1 c.c.)	321	4·2	300
	313	19·2	
	306	37·6	

The time taken for liquid to evaporate will depend mainly upon (*a*) its latent heat of vaporization, L; (*b*) its boiling point.

If we designate the average increment to the induction period for a given liquid as t, then we might expect t/L to be approximately constant if the boiling points of the liquids

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are of the same order, since the rate of heat input from the bath will be much the same. This condition is satisfied for water and benzene, as illustrated in table 23.

Similar experiments were carried out with Service azide, and results are shown in table 4. Again the effect is to produce an approximately constant lengthening of the induction period, presumably attributable to evaporation.

TABLE 23

liquid	t (sec.)	L (cal./g.)	t/L
benzene	7	94·3	0·93
water	52	540·0	0·96

TABLE 24. EFFECT OF VARIOUS LIQUIDS ON THE INDUCTION PERIOD OF SERVICE AZIDE, AND THE CORRESPONDING THRESHOLDS OF DETONATION

substance (10 mg.)	bath temperature (° C)	induction period (sec.)	failure temperature below which no detonation is observed (° C)
Service azide alone	350	1·9	325
	340	4·2	
	330	7·8	
	326	14·4	
Service azide + benzene (0·1 c.c.)	350	5·2	325
	340	8·5	
	330	16·6	
	325	24·5	
Service azide + water (0·1 c.c.)	360	30·6	328
	340	60·0	
Service azide + diethylene glycol (0·1 c.c.)	360	7·9	320
	340	6·2	
	328	8·2	
Service azide + dibutyl phthalate (0·1 c.c.)	343	2·5	317
	336	4·6	
	328	11·3	
	323	22·7	
	319	39·7	

Further evidence for this interpretation of the displacement of the induction period curves when low-boiling liquids are added, is provided by experiments in which the amount of added liquid is progressively increased. This leads to a progressive rise in the increment of time required for vaporization as shown by the figures in table 25 obtained for 10 mg. of dextrinated lead azide with 0·1 c.c. of benzyl benzoate:

TABLE 25

temperature 320° C

amount of liquid added (c.c.)	induction period (sec.)
0·1	1·9, 2·1, 2·1, 2·0, 1·9
0·2	21, 22, 21, 22, 22
0·3	fail, 24 (feeble), 20 (feeble), fail, fail
0·4	fail, fail, fail, fail, 62 (*very* feeble)

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Eventually detonation fails to be established altogether owing to the gradual decomposition of the lead azide during vaporization of the liquid.

After vaporization is completed, since the individual grains of initiator are surrounded by vapour of the liquid used, instead of air, it might be expected that a change in threshold would occur. This is observed in practice, and in the case of dextrinated lead azide a *lowering* of as much as 30° C may be observed in certain cases (see tables 22 and 24).

High-boiling liquids on dextrinated lead azide

With dextrinated azide wetted by glycerol, nujol, or tricresyl phosphate, one obtains on heating either (*a*) complete failure to detonate, or (*b*) a peculiar crackling noise lasting a few seconds (or less), and occurring after an induction period which decreases as the bath temperature rises, or (*c*) a 'puff', which occurs after an induction period, is nearly noiseless, and in which a cloud of fume is suddenly shot out from the aluminium tube. None of these samples will detonate in a bunsen flame after such treatment, and visual inspection shows that the grains have changed to what appears to be metallic lead. In order to compare this effect with 'self-heating', samples (up to 30 mg.) of cyclonite were tested in a similar manner to that used for azide, over approximately the same temperature range, 240 to 310° C. The behaviour was analogous in all respects to that of lead azide wetted with a high-boiling liquid. Nothing corresponding with the normal detonation of lead azide was observed. Moreover, addition of nujol to cyclonite caused no difference in the behaviour other than a very slight decrease in violence of the puff.

It may be noted that in the self-heating mechanism of explosion the presence of inert liquid between the grains of explosive will have only a secondary effect, since it merely changes the thermal conductivity somewhat.

All the above evidence leads to the conclusion that dextrinated lead azide *will not detonate as long as any liquid medium remains*. One reason for this may be that if shock waves have to be built up, mass motion of decomposition products will be much impeded by a liquid medium. From the parallel behaviour with cyclonite, the 'puff' given by dextrinated azide wetted with a high-boiling liquid appears to correspond with explosion by self-heating of the azide. With lead azide, self-heating was shown to be a slower mechanism than the shock wave mechanism (part II (2)), and would therefore not occur unless the shock wave mechanism were suppressed, by liquid between the particles, which would not impede deflagration by self-heating, except by increasing the thermal conductivity.

To test this conclusion further, attempts were made to obtain a threshold temperature for any given mass of material at which the behaviour changes from 'crackles' to 'puffs', and to plot a curve showing $\log_{10} m$ against T which can be compared with the theoretical curve for self-heating as given by Frank-Kamenetsky (part II (2)).

Table 26 gives the threshold temperatures observed for 'puffing off' for various masses of dextrinated lead azide when heated in nujol, allowing one drop of nujol to every 10 mg. of azide. These values are plotted in figure 26, and it will be seen that the values nearly coincide with the self-heating curve calculated for *Service azide*. Dextrinated and Service azides are structurally and chemically identical, and the liquid may possibly suppress any effect due to the dextrin itself. The chief physical difference between the two azides is particle size, Service azide comprising crystals $0{\cdot}01 \times 0{\cdot}04$ mm., while dextrinated azide contains crystallites of

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the order 10^{-4} to 10^{-5} cm. (part II (1)). The main fact is that, in presence of nujol, dextrinated lead azide behaves like any typical high explosive, and shows no real detonation until larger masses are involved. At 330° C, 100 mg. gave a violent 'puff' after a few seconds induction period, but with scarcely any noise, and no damage to the tube. At a similar temperature, 200 mg. gave a loud explosion, but the aluminium tube was quite undamaged. With cyclonite or other high explosive, much the same behaviour would be observed. On the other hand, with ordinary loose azide, the tube is completely shattered.

Table 26. Variation of 'self-heating' threshold temperature with mass for deflagration of dextrinated azide in nujol

mass (m) (mg.)	$\log_{10} m$	threshold temperature (°K)
10	1·0	625
20	1·3010	614
30	1·4771	606
50	1·6990	597

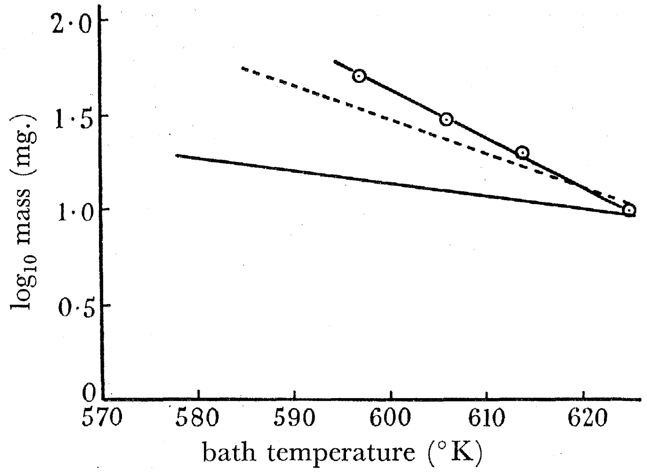

Figure 26. Threshold of self-heating for dextrinated azide in nujol. ⊙ Threshold curve (obs.); --- theoretical threshold curve for Service azide; —— theoretical threshold curve for dextrin azide.

Even though the presence of liquid prevents detonation, there is a further possibility to be considered. During decomposition by heat, azides evolve nitrogen, and if sufficient could be evolved to displace the liquid and surround the crystals with this gas, the shock wave mechanism might operate even though insufficient vaporization occurred. Experimental evidence for this displacement is more conveniently discussed in connexion with Service azide (see below). With dextrinated lead azide, the displacement of a high-boiling liquid by vapour from a low-boiling liquid was also investigated.

Mixtures of low-boiling and high-boiling liquids

To test the displacement of liquid around the initiator grains, by vapour, experiments were made to introduce into a high-boiling liquid a sufficient admixture of a low-boiling liquid to provide the necessary gaseous medium by its vaporization for the azide to detonate. It was anticipated that at a certain critical mixture both shock wave and self-heating

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mechanisms might occur in the same series of trials. Mixtures of benzene and nujol were used to wet dextrinated lead azide, and the anticipated result was obtained. The critical mixture contained 60 % benzene and 40 % nujol. A greater percentage of nujol caused all samples to give the self-heating 'crackle' or 'puff', below or above the threshold temperature for deflagration by self-heating. More benzene gave detonation in all cases. With the critical mixture *both* mechanisms were observed in the same series. Actual results are given in table 27.

Table 27. Effect of mixed liquids on dextrinated lead azide

75 % benzene 25 % nujol 321° C all real detonations with the following delays in sec.		66 % benzene 34 % nujol 321° C all real detonations	60 % benzene, 40 % nujol						50 % benzene 50 % nujol 321° C 'crackles' after delay (sec.)
			321° C			328° C			
			'crackles' after delay (sec.)	real detonations after delay	'puffs' after delay (sec.)	'crackles' after delay (sec.)	real detonations after delay	'puffs' after delay (sec.)	
5·6	5·2	4·1	about 10	3·2	none	none	2·1	about 5	about 5
1·6	6·0	3·6	„ 5	2·8	—	—	1·9	„ 5	„ 5
3·3	4·2	3·7	„ 6	2·6	—	—	2·2	„ 7	„ 6
1·9	7·1	2·4	„ 5	3·0	—	—	1·8	„ 8	„ 5
6·8	5·8	2·8	„ 5	2·6	—	—	1·9	„ 5	'puff' after about 8 sec.
—		3·2	„ 6	2·9	—	—	2·1	„ 6	
—		3·9	„ 5	3·0	—	—	2·3	„ 5	
—		8·5	in all cases duration of crackles about 10 sec.	—	—	—	2·4	—	

Results tabulated all occurred in a *single* series of trials.

High-boiling liquids on Service azide—evolution of nitrogen

In the presence of nujol, dextrinated lead azide never shows real detonation, at any rate in small quantities. However, Service azide shows a somewhat different behaviour. In the lower regions of temperature we again get complete failure to detonate, and this persists in regions where the azide in air would detonate normally. With 10 mg. Service azide, and one drop nujol, below 334° C, samples plunged into a furnace steadily emit fumes, and will not detonate in a flame afterwards, but no other effect can be noticed. At 334° C, however, crackles are obtained after an induction period of about 15 sec. (cf. dextrinated lead azide above), and this behaviour persists with rise of temperature (and consequently shorter induction period), up to about 337° C, *where it changes over to real detonation.* Nothing analogous to the 'puff' given by dextrinated azide is obtained here. This absence of deflagration by self-heating may be due to (*a*) different crystal size; (*b*) evolution of nitrogen by the crystals in sufficient quantity to allow the shock wave mechanism to operate; (*c*) absence of dextrin; (*d*) the fact that the region of detonation of Service azide is higher than that of dextrinated azide, and consequently nearer to the boiling point of nujol.

If (*d*) above is the correct explanation, then a higher-boiling liquid should give a possibility of deflagration by self-heating with Service azide.

This is in fact found with tricresyl phosphate, which boils at 385° C. The behaviour obtained was similar to that with nujol, except that between the region of 'crackles' and that of real detonation a temperature interval was found in which 'puffs' are given, exactly as in the case of dextrinated lead azide.

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If the induction periods for Service azide with and without nujol are plotted in the usual way ($\log_{10} t$ against $1/T$), it is seen that the resulting curves are parallel (figure 27), the induction period is increased by a constant *factor* instead of by a constant increment as with low-boiling liquids. Thus, in this case, the process causing the lengthening of the induction period must be characterized by an activation energy similar to that for the azide alone. This is additional evidence in favour of (*b*) above, as the rate of evolution of nitrogen will depend upon the rate of thermal decomposition of the azide itself.

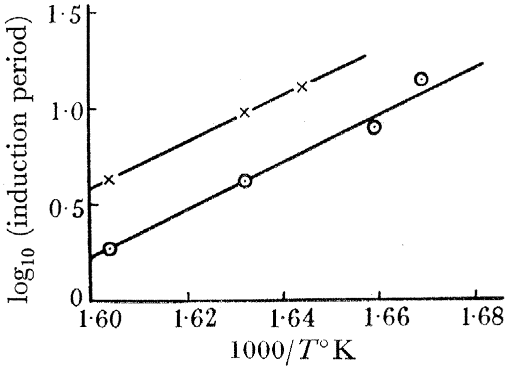

FIGURE 27. Effect of nujol on induction period of Service azide. 10 mg. Service azide. $E = 54{\cdot}8$ kcal./mol. ⊙ Loose Service azide crystals; × azide crystals + nujol.

The evolution of nitrogen from Service azide when heated under nujol can be demonstrated visually by heating 10 mg. of the azide under excess nujol in a glass ignition tube behind a suitable screen. Bubbles of gas were seen to form around the crystals, while the crystals themselves became somewhat discoloured. When a considerable 'cushion' of nitrogen had been formed, the sample detonated.

The effect of particle size was also investigated. With nujol, the effect of powdering to particles of size about 5×10^{-4} cm. was to raise the threshold at which 'crackles' change to real detonation, from 337 to 347° C. These particles are, however, still much larger than the crystallites of dextrinated lead azide. With tricresyl phosphate, powdered lead azide shows a broader interval of temperature within which deflagration is observed, though at still higher temperatures it detonates.

Effect of dibutyl phthalate

In the series of liquids so far discussed, dibutyl phthalate is unique in the sense that its boiling point at ordinary pressures (320° C) lies within the region of detonation for dextrinated azide, and below that for Service azide. With *Service azide* (figure 28), its effect is to increase the induction period, and also to increase somewhat the slope of the plot of $\log t$ against $1/T$. The threshold temperature is also lowered. With *dextrinated azide*, the increase in slope of the plot of $\log t$ against $1/T$ is much more marked, and indicates that the physico-chemical processes preceding detonation are being controlled by a new process. With both substances real detonation is observed.

Hawkes & Winkler (1945) suggested that when dextrin azide was wetted with butyl phthalate, the increased slope of the induction period curve was due to the mechanism of detonation changing over to that for Service azide, and ascribed this change to the increased thermal conductivity of liquid between the grains, compared with air.

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The behaviour of the various liquids described above shows that real detonation of the lead azide is highly unlikely unless the liquid is displaced in some way. Since the boiling point lies within the region of detonation, this displacement probably involves both vaporization and some displacement by nitrogen evolved from the azide, and on this view the

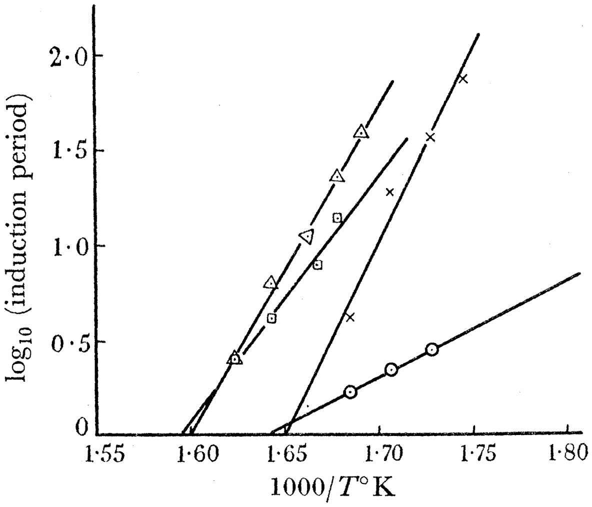

FIGURE 28. Effect of dibutyl phthalate on induction periods of Service and dextrinated azides. 10 mg. ⊙ Loose dextrinated lead azide ($E = 22·9$ kcal./mol.); × loose dextrinated lead azide + 1 drop dibutyl phthalate ($E = 93·7$ kcal./mol.); □ loose Service azide ($E = 59·0$ kcal./mol.); △ loose Service azide + 1 drop dibutyl phthalate ($E = 79·5$ kcal./mol.).

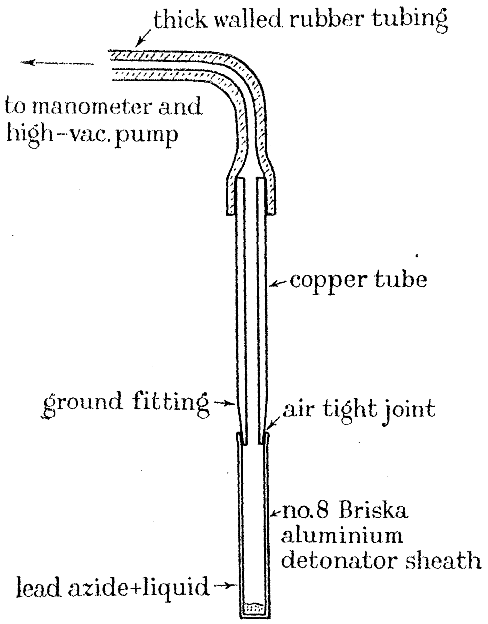

FIGURE 29. Apparatus used to investigate effect upon lead azide of liquids under reduced pressure.

high slope for dextrinated azide wetted with butyl phthalate (93·7 kcal./mol. in figure 28) means that both the evolution of nitrogen and the vaporization of butyl phthalate are controlled by considerable activation energies in the proximity of the boiling point.

To test this explanation, the boiling point of butyl phthalate was lowered to about 280° C by using it under reduced pressure. Figure 29 shows the simple apparatus used. The butyl

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phthalate was de-aerated by boiling with fragments of glass and cooling in vacuo before use. Figures given in table 28 show that under these conditions dibutyl phthalate behaves exactly like any other liquid whose boiling point lies below the detonation range, i.e. it first vaporizes and detonation then builds up in the vapour.

Table 28. Effect of reduced pressure on the induction period of dextrinated lead azide in dibutyl phthalate. (Boiling point about 280° C)

	313° C		321° C		335° C	
	8·0	7·2	9·0	8·0	7·9	10·3
	10·1	8·1	8·0	fail	9·2	8·5
	8·0	8·6	8·3	8·2	7·8	8·2
	7·8	7·6	6·5	fail	5·5	8·4
	5·0	8·8	8·3	8·6	6·5	9·2
mean		7·9		8·1		8·1

Comparative values at normal pressures extracted from table 22, are: 313° C −19·2 sec., 321° C −4·2 sec.

Discussion

The various experiments described make it clear that deflagration can be separated by experimental means from detonation, even with an initiator as prone to build up detonation as Service azide. Deflagration is a far milder form of energy release than detonation, even with cyclonite where the heat evolved per g. is some 3 or 4 times as large as for lead azide.

The results are of considerable interest in connexion with theoretical and practical problems. From the practical aspect, they support the view that separating grains of explosive by inert liquids which can absorb energy is one mode of desensitization, particularly when the liquids have a high boiling point.

The interesting new feature is that such phlegmatizers have far less effect on deflagration by self-heating, than on the build-up of real detonation. Build-up of real detonation is, apparently, much easier in a gaseous than in a liquid medium. The results probably explain why a low-boiling liquid such as water is not very effective in desensitizing Service azide.

This opens up the interesting practical possibility of phlegmatizing Service azide with a high-boiling liquid such as nujol or cresyl phosphate, for purposes of transport. When it is required to use the azide, the phlegmatizing liquid could be removed with some easily evaporated liquid with no solvent action on lead azide.

From the theoretical aspect, the results throw further light on the build-up of detonation, and emphasize its difference from deflagration by self-heating.

PART III

(1) THE GRIT SENSITIVENESS OF HIGH EXPLOSIVES

By J. L. Copp and A. R. Ubbelohde

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Risks in handling various high explosives are enhanced if grit is present. Quantitative measurements of the enhanced sensitiveness, made by means of the Rotter impact machine, have included tests on the high explosives:

T.N.T., picric acid, tetryl, cyclonite and waxed cyclonite, and penta-erythritol tetranitrate (P.E.T.N.).

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