Journal of Hazardous Materials, 12 (1985) 27—41
Elsevier Science Publishers, B.V., Amsterdam — Printed in The Netherlands

EVALUATION OF EXPLOSIVE PROPERTIES OF ORGANIC PEROXIDES WITH A MODIFIED Mk III BALLISTIC MORTAR

TADAO YOSHIDA*

Department of Reaction Chemistry, Faculty of Engineering, University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113 (Japan)

KOTARO MURANAGA

Hodogaya Factory, The Japan Carlit Co., Ltd., 1625 Bukko-cho, Hodogaya-ku, Yokohama-shi 240 (Japan)

TATEHIRO MATSUNAGA and MASAMITSU TAMURA

Department of Reaction Chemistry, Faculty of Engineering, University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113 (Japan)

(Received September 13, 1984; accepted December 12, 1984)

Summary

Ballistic mortar tests using the Mk III F mortar have been carried out to examine a procedure for assessing the explosive hazard of organic peroxides. The explosive properties of an organic peroxide can be evaluated from two series of experiments, for propagation and power of explosion, and for shock sensitivity. Among seven organic peroxides tested, tert-butyl peroxybenzoate and dibenzoyl peroxide have shown high shock sensitivity and explosive powers of 40 and 25% of TNT, respectively. Di-tert-butyl peroxide showed medium sensitivity and the power of 30% of TNT. Cumyl hydroperoxide, 80% in cumene, dibenzoyl peroxide, 75% with water, dicumyl peroxide and dilauroyl peroxide did not propagate explosion.

Introduction

Organic peroxides are important commercial products which are used as catalysts for polymerization and bleaching agents. This group of compounds has a functional group —O—O— which is relatively unstable and decomposes at moderate temperatures to give active radicals acting as polymerization initiators etc. On the other hand, some organic peroxides may undergo accidental explosion or violent decomposition, owing to their unstable nature. Explosion accidents caused by organic peroxides have actually been experienced [1, 2].

In order to prevent accidents with organic peroxides, hazard evaluation techniques have been devised by several groups [3—7, 21]. Here we describe

*To whom all correspondence should be addressed.

the results of our work on the evaluation of the explosiveness of organic peroxides using a modified Mk III ballistic mortar. Recently, a compact and less expensive ballastic mortar, the Mk III D, was developed by Turner [8] of the Royal Armament Research and Development Establishment (RARDE) in the U.K. A replica of the mortar was installed at the Safety Engineering Laboratory of Tokyo University and used extensively to assess the explosion hazards of many unstable materials including explosives [9], oxidizing agent compositions [11], pyrotechnic compositions [15], organic peroxides [12] and other hazardous materials [14]. Furthermore, standardization [10] of procedures, structural revisions [17, 18] and an examination of properties of the Mk III mortar [16] were performed.

Through our experiences, we have come to believe that we should use a new procedure to assess the explosive hazard of unstable materials. Here we describe this new procedure and its application to organic peroxides.

2. Experimental

2.1. Materials

Organic peroxides used in this study are commercial products supplied by the Nippon Oil & Fats Co. Ltd. Detonators, TNT and PETN are commercial products made in Japan.

2.2 Apparatus

The Mk III F ballistic mortar used here is a modification of the Mk III D. The outline (a) and cross-section (b) of the mortar are shown in Fig. 1. The structural characteristic of the mortar is that it has two dead spaces which are useful for preventing damage to the mortar by the explosion of charge. A 2 kg projectile shown in Fig. 2 was used in this study in order to make the ballistic mortar test applicable to the explosion propagation of organic

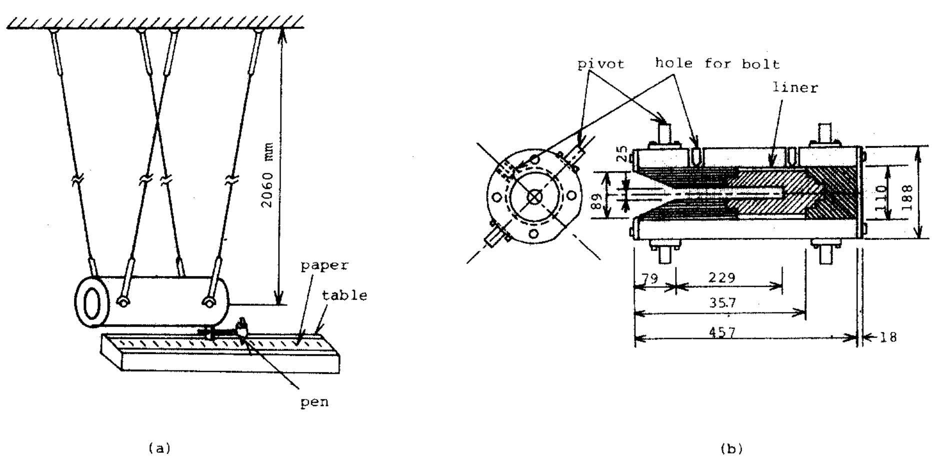

Fig. 1. Outline (a) and cross-section (b) of the Mk III F ballistic mortar.

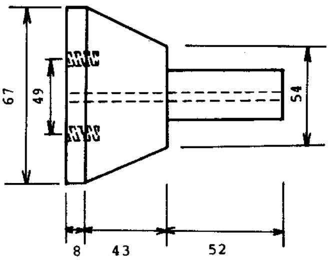

Fig. 2. Cross-section of a 2 kg projectile for the Mk III ballistic mortar.

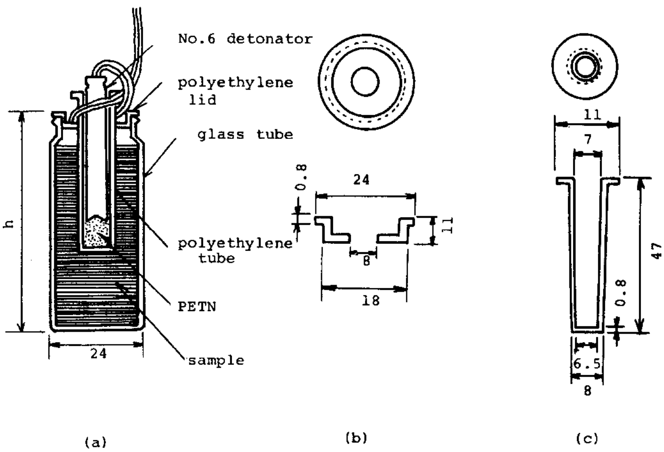

Fig. 3. Cross-sections of sample vessel (a), polyethylene lid (b) and polyethylene tube (c).

TABLE 1

The length of glass tube corresponding to sample weight

Sample weight (g)	Length of glass tube (mm)
5	50
10	50
15	80
20	110
25	140
30	170

peroxides. The projectile is made of steel and fitted in front of the mortar. There is an axial hole for passing the leg wires of the detonator.

The cross-section of the sample vessel assembly is shown in Fig. 3(a). A glass sample tube, and polyethylene lid and inner tube, are used for their compatibility with organic peroxides. Figures 3(b) and (c) show the shape and size of the polyethylene lid and inner tube, respectively. Table 1 lists the sizes of the glass sample tubes.

3. Procedure

3.1 Test for propagation and power of explosion

(1) For solid samples, $5n$ g (n = 1—6) of the organic peroxide to be tested is weighed out accurately and placed in the glass sample tube. For liquid samples, $5n$ g (n = 1—6) of the organic peroxide to be tested is poured into the glass sample tube and weighed out accurately.

(2) A lid is put on the filled glass tube.

(3) About 0.6 g of PETN powder is weighed out accurately and placed in the inner polyethylene tube.

(4) The filled inner tube is inserted into the sample tube through the hole of the polyethylene lid.

(5) A No. 6 electric detonator is inserted into the fixed inner tube and the leg wires of the detonator are wound around the top of the inner tube in order to fasten the detonator to the sample vessel assembly (Fig. 3a).

(6) To record the mortar recoil, a strip of recording paper is fastened to the table.

(7) The sample vessel assembly is inserted into the ballistic mortar bore and rammed firmly by means of a ramrod.

(8) The leg wires of the electric detonator are threaded through the small axial hole of the projectile and the projectile is inserted into the hole of the mortar.

(9) A pen is inserted into the hole of the pen holder and a reference line is marked on the recording paper when the fully loaded mortar is at rest.

(10) The detonator leg wires are connected to the firing circuit.

(11) The charge is fired, and the pendulum swing is taken from the result recorded on the recording paper.

(12) Before each shot, both the mortar bore and the projectile are cleaned to remove any foreign matter.

(13) Sometimes an unexpected larger swing is obtained when firing a charge showing complete explosion after an experiment with a charge showing incomplete explosion. In this case the residue in the mortar bore should be removed by firing 5 g of PETN.

3.2 Test for standard explosive, TNT

(1) n g (n = 1—10) of TNT powder is weighed out accurately and placed in the glass sample tube of 15 ml.

(2) A lid is put on the blank or filled tube.

(3) The blank inner tube is inserted into the sample tube through the hole in the lid.

From then onwards the procedure is the same as from 3.1 (5) to (12).

3.3 Test for the power of detonators and PETN boosters

(1) A lid is put on the blank glass tube of 15 ml.

(2) $0.1n$ g (n = 1—6) of PETN powder is weighed out accurately and placed in the polyethylene inner tube.

(3) The blank or filled inner tube is inserted into the blank sample tube through the hole in the polyethylene lid.

(4) For the power of electric detonators, an electric detonator of up to No. 6 is inserted into the blank inner tube. For the power of PETN boosters, a No. 6 detonator is inserted into the filled inner tube.

(5) The leg wires of the detonator are wound around the top of the inner tube in order to fasten the detonator to the sample vessel assembly (Fig. 3a).

From then onwards the procedure is the same as from 3.1 (6) to (12).

3.4 Test for shock sensitivity

(1) For solid samples, 10 g of the organic peroxide to be tested is weighed out accurately and placed in a commercial 15 ml glass sample tube. If the apparent density of the sample is too low to put 10 g into the tube, less than 10 g may be weighed accurately. For liquid samples, 10 g of the organic peroxide to be tested is poured into a commercial 15 ml sample tube and weighed out accurately.

(2) The filled glass tube is put on the lid.

(3) $0.1n$ g (n = 1—6) of PETN is weighed accurately and placed in the polyethylene inner tube.

(4) The inner tube is inserted into the sample through the hole in the lid.

From then onwards the procedure is the same as from 3.1 (5) to (12).

4. Data treatment

4.1 The power of explosion of electric detonators and PETN boosters

The length of swing of the mortar (d_i) is plotted against the weight of PETN (W_{PETN}) on a section paper, and a coefficient a is determined statistically from eqn. (1)

$$d_i = d_0 + aW_{PETN} \quad (1)$$

where d_0 is the length of swing with a No. 6 electric detonator only.

The PETN equivalence of detonators is calculated from the comparison of the lengths of swing with detonators and with PETN boosters.

4.2 The power of explosion of TNT

The length of swing with TNT (d_{TNT}) is plotted against the weight of TNT (W_{TNT}) on a section paper, and a coefficient b is determined statistically from eqn. (2)

$$d_{TNT} = d_0 + bW_{TNT} \quad (2)$$

4.3 The propagation and power of explosion of an organic peroxide

(1) The difference of the length of swing with an organic peroxide charge and with that of 0.6 g PETN ($d - d_i$) is plotted against the weight of the organic peroxide sample (W_s) on a section paper.

(2) If $(d - d_i)$ does not increase monotonically with W_s, that is, the increase stops halfway or $(d - d_i)$ has a maximum, the explosion of the peroxide does not propagate in this tester.

(3) When $(d - d_i)$ increases monotonically with W_s, coefficients c and e are determined statistically from eqn. (3)

$$d - d_i = cW_s + eW_s^{1/3} \tag{3}$$

(4) $(d - d_i)_{10}$ corresponding to $W_s = 10$ g is estimated using the fixed eqn. (3).

(5) $(d_{TNT} - d_0)_{10}$ corresponding to $W_s = 10$ g is estimated using the fixed eqn. (2).

(6) The explosive power of an organic peroxide (*EP*) is calculated from eqn. (4):

$$EP = \frac{(d - d_i)_{10}}{(d_{TNT} - d_0)_{10}} \times 100\% \tag{4}$$

4.4 The shock sensitivity of organic peroxides

(1) The length of swing with 0.1n g (n = 1—6) PETN plus a No. 6 electric detonator is given by the symbol d_i. The length of swing with 10 g or n g ($n \leqslant$ 10) samples and the primer (a No. 6 detonator + PETN) is given by the symbol d_{10} or d_n, respectively, $(d_{10} - d_i)$ or $(d_n - d_i)$ is then plotted against the weight of PETN (W_{PETN}).

(2) The shock sensitivity of the sample is high if $(d_{10} - d_i)$ or $(d_n - d_i)$ is large and nearly independent of the weight of the booster PETN.

(3) The shock sensitivity of the sample is medium if $(d_{10} - d_i)$ or $(d_n - d_i)$ increases with W_{PETN} at first and becomes nearly independent of W_{PETN} later, and furthermore explosion propagates by the initation with a No. 6 detonator and 0.6 g PETN.

(4) The sample which does not propagate explosion by initiation with a No. 6 detonator and 0.6 g PETN may be judged as non-explosive, although the detailed properties of this sample are unable to be made clear using this method only.

5. Results and discussion

The lengths of swing obtained in this study are listed in Tables 2 and 3.

5.1 The explosive power of detonators and PETN boosters

Nos 0, 1, 2 and 6 electric detonators made by Nippon Kayaku Co. Ltd. were used. A No. 6 electric detonator is the most popular detonator and the quality is well controlled. PETN powder maufactured by the same company was used. The plot of d_i versus W_{PETN} is shown in Fig. 4.

TABLE 2

The length of swing (d) together with the weight of PETN booster (W_{PETN})

Material	Length of swing (mm); ( ): accurate weight of PETN (g)							
air[a]	7 (0.0)		8 (0.203)	8 (0.339)	10 (0.432)	11 (0.557)		14 (0.829)
air[b]	28 (0.0)	35 (0.1005)	43 (0.2642)	49 (0.3573)	56 (0.4481)	53 (0.5028)	62 (0.6782)	
Benzoyl peroxide, 8 g[a]	32 (0.0)	33 (0.1107)	35 (0.2175)		39 (0.4259)	41 (0.5212)	42 (0.6210)	
Benzoyl peroxide, 8 g[b]	105 (0.0)	110 (0.1177)	111 (0.2124)		119 (0.4008)		126 (0.6105)	
Benzoyl peroxide, 75% with water, 10 g[b]	49 (0.0)	84 (0.1056)	82 (0.2055)	92 (0.3299)	123 (0.4001)	134 (0.5015)	136 (0.6069)	
Lauroyl peroxide, 6 g[b]	36 (0.0)	52 (0.1211)	54 (0.2057)	64 (0.3440)	72 (0.4050)	74 (0.5045)	73 (0.6141)	
Dicumyl peroxide, 7 g[b]	30 (0.0)	42 (0.1094)	47 (0.2119)	54 (0.3099)	60 (0.4080)	78 (0.5012)	98 (0.5990)	
Tert-butyl peroxybenzoate, 10 g[b]	150 (0.0)	155 (0.1136)	159 (0.2116)		165 (0.4014)			
Di-tert-butyl peroxide, 10 g[b]	45 (0.0)	76 (0.1254)	157 (0.1971)	172 (0.3986)		182 (0.5345)	187 (0.6371)	
Cumyl hydroperoxide, 80% in cumene, 10 g	26 (0.0)	37 (0.1149)	57 (0.2213)		87 (0.4236)	89 (0.5052)	92 (0.6500)	

[a]No projectile.
[b]2.0 kg projectile.

TABLE 3

The length of swing (d) together with the weight of organic peroxide (W_s)

Material	Length of swing (mm); ( ): accurate weight of PETN (g)									
	2 g	3 g	5 g	7 g	8 g	10 g	15 g	20 g	25 g	30 g
Benzoyl peroxide[b]			97 (0.6014)		119 (0.6008)	148 (0.6038)	175 (0.6042)	211 (0.6258)	243 (0.6105)	277 (0.6227)
Benzoyl peroxide[a]			30 (0.6068)			49 (0.6210)	64 (0.6134)	82 (0.6220)	99 (0.6109)	118 (0.6149)
Benzoyl peroxide, 75% with water[b]			106 (0.6232)			136 (0.6062)	142 (0.6275)	136 (0.6186)	131 (0.6195)	166 (0.6095)
Dicumyl peroxide[b]		82 (0.6137)	98 (0.6140)	98 (0.5990)			84 (0.6166)	100 (0.6089)	88 (0.6012)	92 (0.6029)
Tert-butyl peroxy-benzoate[b]			135 (0.6221)				233 (0.6171)	276 (0.6182)	312 (0.6197)	347 (0.6000)
Di-tert-butyl peroxide[b]			132 (0.600)			187 (0.6371)	217 (0.6000)	153 (0.6322)	308 (0.6060)	225 (0.6235)
Cumyl hydroper-oxide 80% in cumene[b]	75 (0.6408)		103 (0.6383)			92 (0.6500)	91 (0.6058)	73 (0.5935)	60 (0.6028)	81 (0.6334)
Lauroyl peroxide[b]		84 (0.6065)				78 (0.6132)	78 (0.6326)	79 (0.6096)	85 (0.6151)	97 (0.6024)

[a] No projectile.
[b] 2 kg projectile.

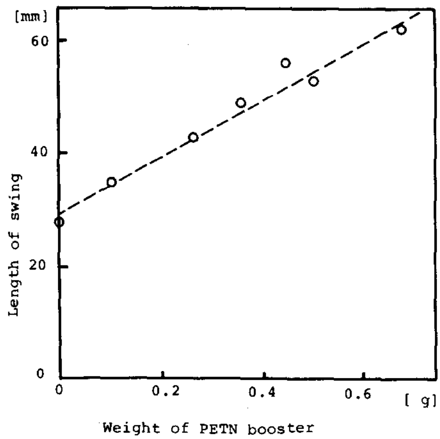

Fig. 4. Variation of length of swing with PETN booster.

TABLE 4

The length of swing with several detonators and with 8 g BPO along with PETN equivalence of detonators

No. of detonator	Charge weight in detonator (g)		d_i (mm)	d_s (mm)	$d_s - d_i$ (mm)	PETN equivalence (g)
	DDNP	PETN				
0	0.2	0.0	9	94	85	0.18
1	0.2	0.1	16	93	77	0.32
2	0.2	0.2	21	93	72	0.42
6	0.2	0.45	30	105	75	0.60

The following parameters were obtained by the least square analysis:

$d_i = 30 + 50.3\ W_{PETN}$

$n = 7,\ r = 0.985$

$d = 30$ mm

$a = 50.3$ mm/g

$a = 50.3$ mm/g means that explosion of one gram PETN gives 50.3 mm length of swing. Thereforem the PETN equivalence of a No. 6 detonator is calculated as $d_0/a = 30/50.3 = 0.60$ g.

From the experimental results with other detonators, the PETN equivalences of 4 types of detonators were obtained as listed in Table 4.

5.2 The explosive power of standard explosive, TNT

Powdered TNT manufactured by Chugoku Kayaku Co. Ltd. was used. The plot of d_{TNT} against W_{PETN} is shown in Fig. 5. The following parameters were obtained by the least square analysis:

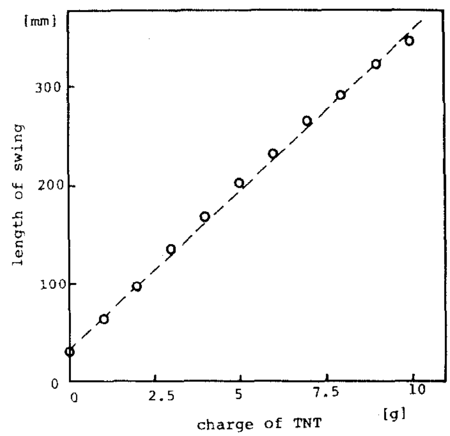

Fig. 5. Variation of length of swing with charge of TNT.

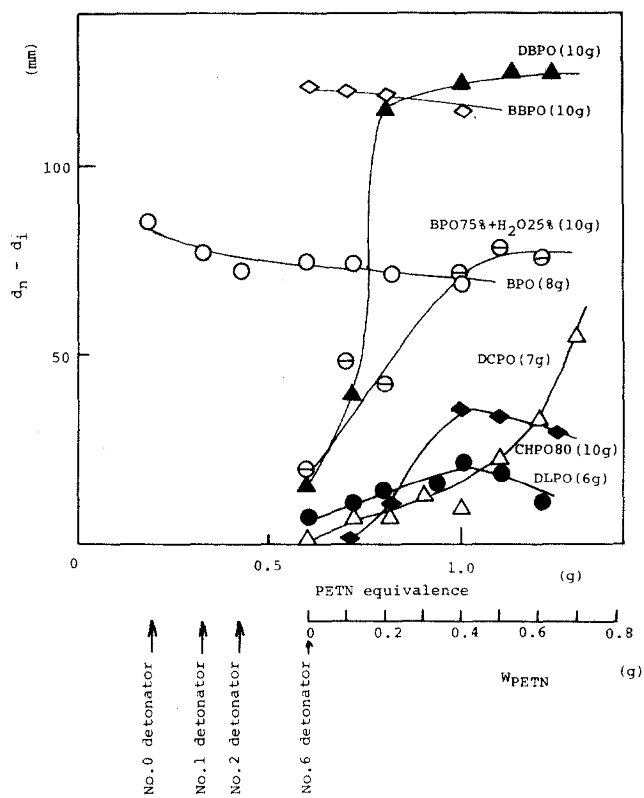

Fig. 6. Plots of $(d_n - d_0)$ vs. PETN equivalence; BPO: dibenzoyl peroxide, BPOB: tert-butyl peroxybenzoate, DBPO: di-tert-butyl peroxide, LPO: lauroyl peroxide, DCPO: dicumyl peroxide, BPO75% + H_2O25%: dibenzoyl peroxide, 75% with water, CHPO80: cumyl hydroperoxide, 80% in cumene.

$d_{TNT} = 30 + 32.7\ W_{TNT}$
$n = 11, \quad r = 0.9995$
$d_0 = 30$
$b = 32.7$
$(d_{TNT} - d_0)_{10} = 318$ mm

5.3 Shock sensitivity of organic peroxides

The samples used were the common organic peroxides discussed in the UN seminar on the test method for organic peroxides at PML-TNO, The Netherlands, on 9—11 October 1984. These samples were supplied by the Nippon Oil & Fats Co. Ltd.

The plots of $(d_n - d_i)$ against W_{PETN} and PETN equivalence are shown in Fig. 6. We can see three patterns in these plots. Tert-butyl peroxybenzoate (BPOB) and dibenzoyl peroxide (BPO) are examples of the first pattern; $(d_n - d_i)$ values of these peroxides are large and nearly independent of either PETN equivalence or W_{PETN}. The peroxides of this pattern may be highly shock-sensitive. Di-tert-butyl peroxide (DBPO), dibenzoyl peroxide, 75% with water (BPO75 + H_2O25), and dilauroyl peroxide (DLPO) are examples of the second pattern; $(d_n - d_i)$ values of these peroxides at first increase with PETN equivalence or W_{PETN}, but these increases stop halfway. The peroxides of this pattern may be of medium shock sensitivity or no shock sensitivity. If an organic peroxide of this pattern can propagate explosion, this peroxide has medium shock sensitivity; di-tert-butyl peroxide is an example. If an organic peroxide cannot propagate explosion, this peroxide is not shock-sensitive, because this material cannot propagate explosion even though an explosion could be initiated by shock. An example of the third pattern is dicumyl peroxide (DCPO); $(d_7 - d_i)$ of the peroxide increases over the complete range of W_{PETN} values used. The shock sensitivity of the peroxide may be low, but this peroxide may explode completely and propagate explosion if initiated by a stronger booster. At the moment, we expect that dicumyl peroxide will not propagate explosion by stronger shock initiation.

From these experiments, either 1.2 g PETN equivalence or No. 6 detonator plus 0.6 g PETN are sufficient to initiate most of an amount of 10 g or less organic peroxide, leading to complete explosion.

5.4 The propagation and power of explosion of organic peroxides

In order to know whether an organic peroxide propagates explosion or not, experiments with varied sample weight (W_s) have been carried out using enough booster to initiate the explosion of the peroxide completely. The plots of $(d - d_i)$ against W_s are shown in Fig. 7.

Values of $(d - d_i)$ of dibenzoyl peroxide, di-terty-butyl peroxide and tert-butyl peroxybenzoate increases monotonically with W_s. These peroxides can sustain their explosion in the ballistic mortar under defined confinement. The explosion power of dibenzoyl peroxide is the least amongst those of the three peroxides. The other two have similar explosive powers.

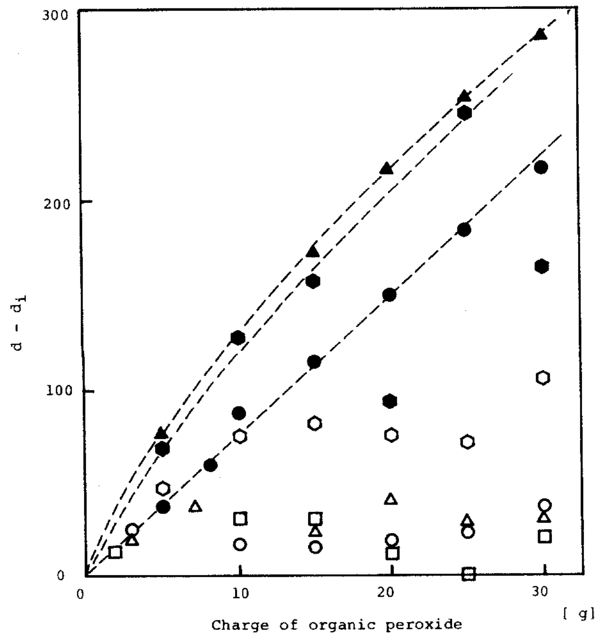

Fig. 7. Variation of length of swing with charge of organic peroxide: ●: di-benzoyl peroxide; ▲: tert-butyl peroxybenzoate; ⬢ : di-tert-butyl peroxide; ○: lauroyl peroxide; △: dicumyl peroxide; ⬡: dibenzoyl peroxide, 75% with water; □: cumyl hydroperoxide.

For dicumyl peroxide, cumyl hydroperoxide, 80% in cumene, and dilauryl peroxide, the increase of $(d - d_i)$ stops halfway. These peroxides may be regarded as organic peroxides of no propagation. In these cases, there are fairly large scatters of observed values. This may be due to the strong effect of physical conditions, because this apparent explosive power comes from non-self-substaining partial exothermic decomposition of the peroxide induced by the explosion of the initiating charge.

Dibenzoyl peroxide composition containing 25% water does not propagate explosion under this condition. However, the apparent explosive power is fairly large. It is an interesting subject whether a large mass of the composition propagates explosion when initiated by a much stronger shock.

As a test for evaluating the propagation of detonation, the BAM 50/60 tube test [19] is a well-known and reliable method for detonatable substances. However, for application to organic peroxides, two problems arise. One is fear of an accidental hazardous reaction between an organic peroxide and the steel tube. Another is that the explosion velocity of organic peroxide is rather low and that the steel tube will not be fragmented. For instance, dibenzoyl peroxide propagates explosion but the explosion does not fragmentate the 50/60 steel tube. The velocity of propagation of reaction wave was about 500 m/sec in 16/22 polyvinyl chloride tube [20]. Therefore, it is not easy to assess the explosion propagation at the moment.

6. Explosion hazard evaluation of individual organic peroxides

6.1 Tert-Butyl peroxybenzoate

Propagation of explosion: yes

$d - d_i = 5.4\ W_s + 23.1\ W_s^{1/2}$ ($r = 0.9999$)

$(d - d_i)_{10} = 127$ mm

$EP = 127/318 \times 100 = 40\%$ of TNT

Shock sensitivity: complete explosion with a No. 6 detonator

6.2 Cumyl hydroperoxide 80%, in cumene

Propagation of explosion: no

Explosive power: no

Shock sensitivity: no

6.3 Dibenzoyl peroxide

Propagation of explosion: yes

$d - d_i = 6.4\ W_s + 4.6\ W_s^{1/2}$ ($r = 0.9994$)

$(d - d_1)_{10} = 79$ mm

$EP = 79/318 \times 100 = 25\%$ of TNT

Shock sensitivity: high, complete explosion with a No. 0 detonator.

6.4 Dibenzoyl peroxide, 75% with water

Propagation of explosion: no

Explosive power: no

Shock sensitivity: no

6.5 Di-tert-butyl peroxide

Propagation of explosion: yes

$d - d_i = 6.0\ W_s + 19.1\ W_s^{1/2}$ ($r = 0.9995$)

$(d - d_i)_{10} = 121$ mm

$EP = 121/318 \times 100 = 38\%$ of TNT (calculation based on 5 points)

6.6 Dicumyl peroxide

Propagation of explosion: no

Explosive power: no

Shock sensitivity: no

6.7 Dilauroyl peroxide

Propagation of explosion: no

Explosive power: no

Shock sensitivity: no

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