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Construction of an adiabatic detonation calorimeter and determination of the heat of detonation for 1,5-diazido-3-nitrozapentane (DIANP) ☆

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Abstract

A new adiabatic detonation calorimeter has been designed. The maximum amount of sample tested is 50 g and the precision of measurement is near 0.3%. The pellet can be detonated in vacuum or under nitrogen. The value of the heat of detonation of 1,5-diazido-3-nitrozapentane tested by this device is 3538 J g^{-1}.

Keywords: Adiabatic detonation calorimeter; Diazidonitrozapentane; Heat of detonation

1. Introduction

The heat of detonation is a very important parameter for evaluating the energies of explosives and relating directly with their power capacities. Therefore it is important to determine the heat of detonation of explosives by experiment. In the 1960s, Lebedev et al. [1] reported that they had designed an isothermal detonation calorimeter, which was placed in a wooden barrel in order to reduce the effect of the external environment; this instrument had a testing precision of 1%. Another isothermal detonation calorimeter designed by Ornellas et al. [2] has a jacket and shows a testing precision of 0.1%. However, the temperature of the isothermal

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detonation calorimeter needs to be measured often and the data so obtained need to be subjected to quite complex calculations. For these reasons the adiabatic detonation calorimeter is now used.

In this paper a new adiabatic detonation calorimeter which we have constructed is introduced and the test results are reported.

2. Experimental

2.1. Materials

1,5-Diazido-3-nitrozapentane (DIANP) was obtained from industrial sources. The purified sample was purified by ourselves in the laboratory.

2.2. Apparatus

The design of the large adiabatic detonation calorimeter is shown in Fig. 1 and consists of three parts. The bomb of the calorimeter is made of alloy steel, has an external diameter of 280 mm, is 400 mm high, an internal volume of 5.8 l and mass of 130 kg. It can withstand the power of 50 g explosive detonated with shell. To reduce damage to the bomb wall it is lined with stainless steel, which can be replaced, and a 10 mm stainless steel buffer pad is placed in the bottom of the bomb. The bomb is put in the centre of the bucket with a clearance of 20 mm from the wall of the bucket.

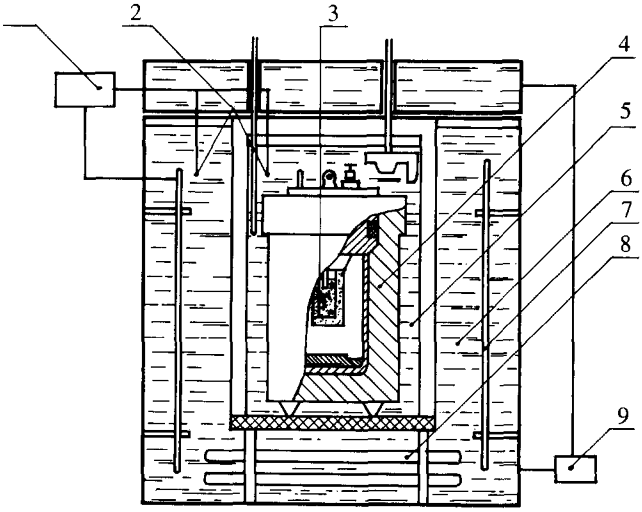

Fig. 1. Adiabatic detonation calorimeter: 1, temperature controller; 2, platinum resistance; 3, sample; 4, bomb; 5, bucket; 6, jacket; 7, heating plate; 8, cooling coil; 9, pump.

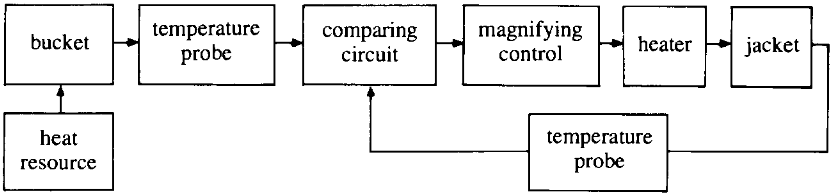

Fig. 2. Graphic representation of the temperature controller.

The calorimeter fundamentally consists of the bucket and the jacket. The bucket, with an internal diameter of 320 mm, contains 14 kg of distilled water, and a small pump is connected to circulate the water and distribute the heat evenly throughout. The temperature of the water is recorded by a Hewlett Packard HP 2804A quartz thermometer, the resolution of which is 0.0001 K. The jacket and the lid of the jacket (in which there is about 50 kg of distilled water containing some electrolyte) are lined. There are a heating plate and a cooling coil in the jacket.

The automatic temperature controller designed by ourselves is used to follow the temperature change of the calorimeter bucket closely. When the explosive is detonated, the water temperature of the bucket increases sharply within 10 min. Hence only a temperature controller with a large power capacity can satisfy the requirement of increasing the water temperature rapidly. In the temperature controller the platinum resistance and proportional temperature controlling circuit are adopted as the measuring temperature component and the temperature controlling system, respectively. A model of the controlling system of the temperature controller is shown schematically in Fig. 2. The most important characteristics of the temperature controller are as follows. I, The precision of temperature control at the balance point is 0.001 K. II, Stability of the balance point: the temperature change in the bucket is less than 0.003 K per 15 min. III, The maximum heating rate is 0.6 K min^{-1}. IV, The maximum heating electric current is 50 A.

2.3. Procedure

The heat equivalent of the instrument was obtained by burning the calorimetric reference material (standard benzoic acid) under an oxygen pressure of 30 bar in a bomb. It was ignited using a metal wire and a cotton thread. The heat equivalent of the instrument is an average value of five determinations, which is about 127 000 J K^{-1} with an error of $\sigma < 0.3\%$. The heat of detonation of the detonator was obtained by running a blank experiment with 20 detonators under the same conditions as for the explosive test.

The charge (about 25–50 g) must be pressed into a pellet with a diameter of 25 mm and is weighed with an uncertainty of 0.1 mg. The pellet was put into a porcelain shell with a wall thickness of 8 mm and internal diameter of 25 mm. For liquid explosives, it was put into a thick wall porcelain shell or glass container and suspended under the lid of the bomb. The tests were run with no oxygen. The bomb was placed in the calorimeter by means of a crane and 14 kg of distilled water was added and weighed with an uncertainty of 1 g at 25°C. The lid of the calorimeter was replaced. After 1 h the automatic temperature controller was switched on and its balance knob was adjusted until the temperature change of water in the bucket was less than 0.003 K within 15 min. The temperature of the bucket T_1 was then recorded. The charge was detonated and the change of temperature in the bucket was observed. When the temperature change became less than 0.003 K per 15 min the temperature of the bucket T_2 was recorded. The bomb was then removed and the pressure inside it was measured. The heat of detonation of the charge could then be calculated according to the mass of sample, the heat equivalent of the instrument, the heat of detonation of the detonator and the values of T_1 and T_2.

3. Results and discussion

To assess the reliability of the results obtained with this instrument, we measured the heat of detonation and the products of detonation of pentaerythritol (PETN); these are shown in Table 1 where they are compared with the results reported by Ornellas et al. [2]. It is shown in Table 1 that the heat of detonation agrees well with the value in the literature, and the products are also similar to those in the literature. The results obtained verify the reliability of our device.

1,5-Diazido-3-nitrozapentane (DIANP) is a liquid explosive. Evacuating the bomb will result in evaporation of the liquid, or if the sample is spilled from the shell the sensitivity of detonation is reduced, even to the point where no detonation will take place. Therefore for non-metallic liquid explosives the method of compression in nitrogen was used. Nitrogen was compressed into the bomb up to a pressure 1.5 MPa, and the pressure was then released (once compressed nitrogen). The compression and release were repeated (twice compressed nitrogen). Finally the

Table 1
Heat of detonation and the products of detonation of pentaerythritol (PETN)

		This work	Ornellas [2] [a]
Diameter of pellet/mm		25	12.7
Density/g cm^{-3}		1.68	1.73–1.74
Material of shell		Porcelain	Gold
Thickness of shell/mm		8	12.7
Heat of detonation $\Delta H_{det} H_2O(l)/(cal\ g^{-1})$ [b]		1453	1487
Products of detonation (v/v)	CO_2	46.56	44.78
	CO	16.19	22.32
	N_2	31.45	26.42
	H_2	5.70	5.94
	H_2O	Not considered	Not considered
	NH_3		0.5
	CH_4	0.0137	0.04

[a] The values of the products of detonation have been changed into volume percentage from mol values (without considering the volume of water). [b] One calorie is 4.1868 joules.

Table 2
Influence of different methods on the heat of detonation

Explosive	Density/(g cm^{-3})	Heat of detonation/(J g^{-1})		
		Once compressed	Twice compressed	In vacuo
TNT	1.59	4502	4410	4402
PETN	1.69	6322	6196	6192
HMX	1.81		6075	6021

valve was closed and the test was conducted. The two methods gave two sets of values for the high explosives trinitrotoluene (TNT), PETN and cyclotetramethylenetetranitramine (HMX). All results are listed in Table 2.

After the once compressed nitrogen experiment, the residual oxygen in the bomb (known through calculation to be 1.4%) which reacts with the unoxidized detonation products generates a molar heat of 1.9 kJ, resulting in heat increase for the sample of 76 J g^{-1}. However after the twice compressed nitrogen experiment, there is only 0.09% oxygen in the bomb and its influence can be neglected. The results shown in Table 2 confirm this. Hence, for liquid explosives, the method of evacuation could be substituted by the twice compressed nitrogen method.

The values of the heat of detonation of DIANP obtained by the twice compressed nitrogen method and the compressed argon method are listed in Table 3 and we can see that the results obtained by both methods are almost identical. Hence, for non-metallic liquid explosives, nitrogen is an inert gas, as is argon, and can be used instead of the evacuation procedure when measuring the heat of detonation.

Table 3
The heat of detonation of purified DIANP

Method	Heat of detonation ΔH_{det}/(J g^{-1})		
	Expt. 1	Expt. 2	Average
Twice compressed nitrogen	3561	3435	3498
Compressed argon	3458	3545	3502

Table 4
Heat of detonation of different industral samples of DIANP

Lot number	Heat of detonation/ΔH_{det}/(J g^{-1})		
	Expt. 1	Expt. 2	Average
001	3527	3571	3549
002	3566	3514	3540
003	3479	3573	3526

In Table 4, the heats of detonation of different industrial samples of DIANP are listed. The values obtained for the different lots of DIANP are similar.

4. Conclusions

(1) The adiabatic detonation calorimeter constructed has a testing precision of 0.3% and can be used to test 50 g of high energy explosive with shell. The results obtained agree well with values in the literature.

(2) The compressed nitrogen method can be used to test non-metallic liquid explosives.

(3) The heat of detonation of DIANP has been measured as 3538 J g^{-1}.

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References

[1] Yu.A. Lebedev, G.G. Lipanin, V.I. Pepekin and A.Ya. Apin, Vzryvnoe Delo, Nauchno-Tekhn. Gorn. Obsch., 52(9) (1963) 80–90 (in Russian).
[2] D.L. Ornellas, J.C. Carpenter and S.R. Gunn, Rev. Sci. Instrum., 37 (1966) 907–912.