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LARGE SCALE CALORIMETER EXPERIMENTATION

SANL 712-004

Jacob Sandoval

January, February, March 1970

ATOMIC ENERGY COMMISSION
UNITED STATES OF AMERICA

DEVELOPMENT DIVISION

MASTER

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ABSTRACT

A number of PETN charges and one confined TNT charge were fired, and the product gases were analyzed by mass spectroscopy (at both LRL and Pantex; agreement was good).

The investigation into the feasibility of large scale calorimeter experimentation on the Pantex detonation sphere continues; conclusions are not yet drawn.

DISCUSSION

During the first quarter of 1970, five unconfined PETN charges and one heavily confined TNT charge were tested in the four-foot detonation sphere.

The first heavily confined HE was detonated in the confinement sphere in March. The TNT charge (1-inch dia x 10-inch long; 197.99 g; ρ 1.545) was confined in a copper cylinder (11-1/2 inches long, 1-inch ID, 3-inches OD; 10,806 g) whose open end was capped with a copper plug (1-inch long x 1-inch dia). The assembly was lowered into and suspended centrally within the sphere. Detonation of the TNT was initiated by means of the detonator alone and no booster was required.

The valve on the sphere, used in pressurizing and evacuating the vessel, was forced open by the shock wave; consequently, the detonation products were lost and no temperature rise was measured.

Examination of the sphere after the detonation suggested the need for protecting the lid against damage from shrapnel and debris. Protective plates for the sphere lid have been ordered.

In the PETN experiments the density of the HE was maintained at approximately the same level; however, the sizes and masses of these charges were varied (see Table I). The detonation products were characterized and analyzed and the heats of detonation were measured for the series of tests. Additionally, the energy equivalent of the system was calibrated. The energy equivalent of the sphere, taken as the average of five calibrations, was established as 222,000 ± 7000 cal/°C (see Table II).

Table I

PETN Charges Tested in PDS-5 Through PDS-10

PDS	Mass (g)	Size (in)	Density (g/cc)
5	212.2230	L = 4; dia = 1.5	1.7690
6	252.2953	1-1/2 x 1-1/2 x 4	1.7660
7	174.1900	1-1/2 x 1-1/2 x 2-2/3	1.7668
8	99.4907	1 x 1 x 3-1/2	1.7692
9	48.7354	1 x 1 x 1-3/4	1.7727
10	103.3649	1 x 1 x 3-1/2	1.7675

Table II

Energy Equivalent of the Detonation Sphere

Test	PETN (g)	Q (cal)	ΔT (°C)	$Q/\Delta T = E_1$ (cal/°C)
PDS-5*	212.4733	298,525	1.47	200,352
6	252.5505	354,830	1.65	215,050
7	174.4356	245,080	1.074	228,190
8	99.4907	139,464	- Lost -	-
9	48.7354	69,102	0.303	228,060
10	103.3649	145,580	0.678	214,530

**PDS-5 not included in calculations. Temperature rise questionable due to poor instrument response.*

The gaseous products from all detonations of PETN (50 - 250 g) have been evaluated (mass spectroscopy) at both the LRL and Pantex installations. The analyses of the gaseous components are found to be in good agreement with product composition established from small charge (PETN, 25 g) investigations conducted by LRL in their calorimeter (see Table III).

The test conditions and the detonation results for the series of PETN tests are tabulated below:

PDS-5

January 19, 1970

Charge Data

HE: PETN	=	212.2230 g (ρ = 1.769) Lot 73057Y3103
Detonator: PETN	=	0.2503 g (XI - 81 - 1)
PETN Total	=	212.4733 g = 0.6721 moles

Volume (water-free gas)

Volume Sphere	=	947.7 L
Pressure	=	118.3 mm
Temperature	=	22.56°C
n = PV/RT	=	5.0267 moles

Product Transfer

Transfer promoted @ 20 mm pressure through trapping line.
Time = $\sim$ 5-1/2 hours

Trap Condensation

H_2O + NH_3	=	41.1967 g
NH_3 (0.2% by wt.)	=	0.0824 g
H_2O	=	41.1143 g

Table III

Products of Detonation of PETN Unconfined Charges

	LRL*	Pantex
Sphere ID (in)	8.5	48.0
Charge diameter	1.27 cm 2.54 cm	Varied sizes, shapes
Charge length	11.40 cm 2.80 cm	Varied
Charge mass (g)	25	50 to 250
No. experiments	14	7
PRODUCTS		
(mole/mole PETN)		
CO_2	3.50 ± 0.04	3.30
CO	1.56 ± 0.08	1.59
N_2	2.00 ± 0.04	2.00
H_2	0.51 ± 0.04	0.50
H_2O	3.45 ± 0.04	3.47
NH_3	< 0.002	-

Ornellas, D. L., et al, Detonation Calorimeter and Results obtained with Pentaerythritol tetranitrate. The review of Scientific Instruments, Vol. 37, No. 7, 907-912, July 1966.

PDS-5 - Continued

Product Analysis

Pantex Mass Spectroscopy

Species	Mole, %	Moles	Mole/Mole PETN
CO_2	43.69	2.196	3.268
N_2	25.31	1.272	1.893
CO	23.17	1.165	1.733
H_2	7.79	0.392	0.583
H_2O	-	2.282	3.395
NH_3	-	0.0048	0.0072
NO	0.03	0.0015	0.002

(Sample contained 1.3% air)

LRL Mass Spectroscopy

CO_2	43.76	2.200	3.273
N_2	26.48	1.331	1.980
CO	22.57	1.135	1.688
H_2	7.19	0.361	0.538
H_2O	-	2.282	3.395
NH_3	-	0.0048	0.0072

(Sample contained 6.0% air)

Material Balance

		Pantex		LRL	
Element	Moles Fired	Moles Found	% Recovery	Moles Found	% Recovery
C	3.3605	3.361	100.0	3.335	99.2
H	5.377	5.362	99.7	5.300	98.6
N	2.688	2.550	94.9	2.667	99.2
O	8.065	7.841	97.2	7.817	96.9

PDS-5 - Continued

Recorder Conditions (L&N Amplifier 9835; HP Moseley Recorder 7100 B)

1/Amplification	=	2000
Recorder Sensitivity	=	1 V/full scale = 1000 μV (full scale on chart)
Absolute Temperature Reading	=	.536 of full scale (amplifier @ 2000; recorder @ 1)

Energy Calculations

ΔT	=	1.47°C
Heat of Detonation $C_{d_{PETN}}$	=	1405 cal/g
PETN Heat Output	=	298,525 cal
Equivalent of Sphere, E_i	=	200,352 cal/°C

PDS-6

February 4, 1970

Charge Data

HE: PETN	=	252.2953 g (ρ 1.766) Lot 99638Y2703
Detonator: PETN	=	0.2552 g (XI - 82 - 1)
PETN Total	=	252.5505 g = 0.7988 moles

Volume (water-free gas)

Volume Sphere	=	947.7 L
Pressure	=	138.9 mm
Temperature	=	22.40°C
n = PV/RT	=	6.1002 moles

Product Transfer

Transfer promoted @ 15 mm pressure through trapping line.
Time = ∿ 6 hours

Trap Condensation

$H_2O + NH_3$	=	49.5561 g
NH_3 (0.04% by wt.)	=	0.0198 g
H_2O	=	49.5363 g

PDS-6 - Continued

Product Analysis

Pantex Mass Spectroscopy

Species	Mole, %	Moles	Mole/Mole PETN
CO_2	31.03	1.893	2.370
N_2	13.39	0.8168	1.023
CO	51.40	3.136	3.925
H_2	2.08	0.1269	0.159
H_2O	(49.5363 g)	2.750	3.443
NH_3	(0.0198 g)	0.00116	0.0015
Acetone	2.08	0.1269	0.159

(Contained 87% air; corrected for air)

LRL Mass Spectroscopy

CO_2	27.28	1.664	2.083
N_2	18.08	1.103	1.381
CO	47.67	2.909	3.642
H_2	4.96	0.3026	0.379
H_2O	(49.5363 g)	2.750	3.443
NH_3	(0.0198 g)	0.00116	0.0015
Acetone	2.03	0.1238	0.155

(Contained 91% air; corrected for air)

Material Balance

		Pantex		LRL	
Element	Moles Fired	Moles Found	% Recovery	Moles Found	% Recovery
C	3.994	3.091	77.4	3.138	78.6
H	6.390	6.519	102.0	6.851	107.0
N	3.195	6.273	196.0	5.819	182.0
O	9.586	7.480	78.0	7.304	76.2

PDS-6 - Continued

Recorder Conditions (See PDS-5)

1/Amplification	=	2000
Recorder Sensitivity	=	0.050 V/full scale = 100 μV (full scale on chart)
Absolute Temperature Reading	=	.539 of full scale (amplifier @ 2000; recorder @ 1)

Energy Calculations

ΔT	=	1.650°C
$C_{d_{PETN}}$	=	1405 cal/g
Q per $C_{d_{PETN}}$	=	354,830 cal
Equivalent of Sphere, E_i	=	215,050 cal/°C

PDS-7

February 10, 1970

Charge Data

HE: PETN	=	174.1900 g (ρ 1.767) Lot 99638Y2703
Detonator: PETN	=	0.2456 g (XI - 82 - 2̇)
PETN Total	=	174.4356 g = 0.5517 moles

Volume (water-free gas)

Volume Sphere	=	947.7 L
Pressure	=	94.0 mm
Temperature	=	17.4°C
n = PV/RT	=	4.1381 moles

Product Transfer

Transfer promoted @ 15 mm pressure through trapping line.
Time = ∿ 9 hours

Trap Condensation

$H_2O + NH_3$	=	34.6065 g
NH_3(0.001% by wt.)	=	0.0003 g
H_2O	=	34.6062 g

PDS-7 - Continued

Product Analysis

Pantex Mass Spectroscopy

Species	Mole, %	Moles	Mole/Mole PETN
CO_2	44.73	1.851	3.355
N_2	26.99	1.117	2.024
CO	21.30	0.8814	1.598
H_2	6.97	0.2884	0.5228
H_2O	(34.6065 g)	1.9209	3.482
NH_3	-	-	-

(Sample contained 4.8% air)

LRL Mass Spectroscopy

CO_2	44.37	1.836	3.328
N_2	26.68	1.104	2.001
CO	22.47	0.9298	1.685
H_2	6.48	0.2681	0.486
H_2O	(34.6065 g)	1.9209	3.482
NH_3	-	-	-

(Sample contained 12.8% air)

Material Balance

		Pantex		LRL	
Element	Moles Fired	Moles Found	% Recovery	Moles Found	% Recovery
C	2.759	2.732	99.0	2.766	100.2
H	4.414	4.419	100.1	4.378	99.2
N	2.207	2.234	101.2	2.208	100.0
O	6.620	6.504	98.3	6.523	98.5

PDS-7 - Continued

Recorder Conditions (See PDS-5)

1/Amplification	=	2000
Recorder Sensitivity	=	0.050 V/full scale = 100 μV (full scale on chart)
Absolute Temperature Reading	=	.411 of full scale (amplifier @ 2000; recorder @ 1)

Energy Calculations

ΔT	=	1.074°C
$C_{d_{PETN}}$	=	1405 cal/g
Q per $C_{d_{PETN}}$	=	245,080 cal
Equivalent of Sphere, E_i	=	228,190 cal/°C

PDS-8

February 17, 1970

Charge Data

HE: PETN	=	99.4907 g (ρ = 1.769) Lot 99638Y2703
Detonator: PETN	=	0.2412 g (XI - 82 - $\dot{3}$)
PETN Total	=	99.7319 g = 0.3155 moles

Volume (water-free gas)

Volume Sphere	=	947.7 L
Pressure	=	65.0 mm
Temperature	=	22.64°C
n = PV/RT	=	2.2811 moles

Product Transfer

Transfer promoted @ 15 mm pressure through trapping line.
Time = ∿ 7 hours

Trap Condensation

$H_2O + NH_3$	=	19.6521 g
NH_3 (.001% by wt.)	=	-
H_2O	=	19.6521

PDS-8 - Continued

Product Analysis

Pantex Mass Spectroscopy

Species	Mole, %	Moles	Mole/Mole PETN
CO_2	45.30	1.033	3.276
N_2	27.31	0.6231	1.975
CO	20.79	0.4743	1.503
H_2	6.60	0.1506	0.477
H_2O	(19.6521 g)	1.0908	3.457
NH_3	-	-	-

(Contained 23.6% air; corrected for air)

LRL Mass Spectroscopy

CO_2	45.3	1.033	3.275
N_2	26.6	0.6068	1.923
CO	22.4	0.5110	1.620
H_2	6.70	0.1300	0.412
H_2O	(19.6521 g)	1.0908	3.457
NH_3	-	-	-

(Contained 4.7% air; corrected for air)

Material Balance

		Pantex		LRL	
Element	Moles Fired	Moles Found	% Recovery	Moles Found	% Recovery
C	1.578	1.507	95.5	1.544	97.8
H	2.524	2.483	98.4	2.442	96.7
N	1.262	1.246	98.7	1.214	96.2
O	3.786	3.631	95.9	3.668	96.9

PDS-8 - Continued

Recorder Conditions (See PDS-5)

1/Amplification	=	1000
Recorder Sensitivity	=	0.50 V/full scale = 1000 μV (full scale on chart)
Absolute Temperature Reading	=	.568 of full scale (amplifier @ 2000; recorder @ 0.5)

Energy Calculations

ΔT	=	lost
$C_{d_{PETN}}$	=	1405 cal/g
Q per $C_{d_{PETN}}$	=	139,464 cal
Equivalent of Sphere, E_i	=	-

PDS-9

February 18, 1970

Charge Data

HE: PETN	=	48.7354 g (ρ = 1.773) Lot 99638Y2703
Detonator: PETN	=	0.2513 g (XI - 83 - i)
PETN Total	=	49.1813 g = 0.1556 moles

Volume (water-free gas)

Volume Sphere	=	947.7 L
Pressure	=	32.6 mm
Temperature	=	22.51°C
n = PV/RT	=	1.6755 moles

Product Transfer

Transfer promoted @ 15 mm pressure through trapping line.
Time = ∿ 8 hours

Trap Condensation

$H_2O + NH_3$	=	9.7763 g
NH_3 (0.001% by wt.)	=	-
H_2O	=	9.7763 g

PDS-9 - Continued

Product Analysis

Pantex Mass Spectroscopy

Species	Mole, %	Moles	Mole/Mole PETN
CO_2	44.46	0.5037	3.252
N_2	27.57	0.3123	2.016
CO	20.62	0.2336	1.508
H_2	7.35	0.0833	0.538
H_2O	(9.7763 g)	0.5426	3.503
NH_3	-	-	-

(Contained 7.1% air; corrected for air)

LRL Mass Spectroscopy

CO_2	44.58	0.5050	3.260
N_2	27.21	0.3083	1.990
CO	22.29	0.2525	1.620
H_2	5.92	0.06707	0.433
H_2O	(9.7763 g)	0.5426	3.503
NH_3	-	-	-

(Contained 32.3% air; corrected for air)

Material Balance

		Pantex		LRL	
Element	Moles Fired	Moles Found	% Recovery	Moles Found	% Recovery
C	0.7780	0.7373	94.8	0.7575	97.4
H	1.245	1.2517	100.5	1.219	97.9
N	0.6224	0.6246	100.4	0.6166	99.1
O	1.867	1.784	95.5	1.805	96.7

PDS-9 - Continued

Recorder Conditions (See PDS-5)

1/Amplification	=	2000
Recorder Sensitivity	=	0.020 V/full scale = 40 μV (full scale on chart)
Absolute Temperature Reading	=	.569 of full scale (amplifier @ 2000; recorder @ 1)

Energy Calculations

ΔT	=	0.303°C
$C_{d_{PETN}}$	=	1405 cal/g
Q per $C_{d_{PETN}}$	=	69,102 cal
Equivalent of Sphere, E_i	=	28,060 cal/°C

PDS-10

February 26, 1970

Charge Data

HE: PETN	=	103.3649 g (ρ 1.7615) Lot 99638Y2703
Detonator: PETN	=	0.2525 g (XI - 83 - $\dot{2}$)
PETN Total	=	103,6174 g = 0.3277 moles

Volume (water-free gas)

Volume Sphere	=	947.7 L
Pressure	=	66.4 mm
Temperature	=	22.23°C
n = PV/RT	=	2.3820 moles

Product Transfer

Transfer promoted @ 15 mm pressure through trapping line.
Time = ∿ 8 hours

Trap Condensation

$H_2O + NH_3$	=	20.9353 g
NH_3 (N.D.% by wt.)	=	-
H_2O	=	20.9353 g

PDS-10 - Continued

Product Analysis

Pantex Mass Spectroscopy

Species	Mole, %	Moles	Mole/Mole PETN
CO_2	44.69	1.065	3.248
N_2	31.43	0.749	2.285
CO	18.02	0.4292	1.310
H_2	5.85	0.1393	0.425
H_2O	(20.9353 g)	1.1620	3.546
NH_3	N.D.	-	-
Ar	0.06	-	-

(Contained 5.5% air; corrected for air)

LRL Mass Spectroscopy

CO_2	44.9	1.0702	3.265
N_2	26.9	0.6412	1.956
CO	22.5	0.5363	1.636
H_2	5.7	0.1358	0.414
H_2O	(20.9353 g)	1.1620	3.546
NH_3	N.D.	-	-

(Contained xylene 0.2%; toluene 8.2%)

Material Balance

		Pantex		LRL	
Element	Moles Fired	Moles Found	% Recovery	Moles Found	% Recovery
C	1.639	1.494	91.2	1.606	98.0
H	2.622	2.603	99.3	2.596	99.0
N	1.311	1.498	114.3	1.284	97.8
O	3.932	3.721	94.6	3.838	97.6

PDS-10 - Continued

Recorder Conditions (See PDS-5)

1/Amplification	=	1000
Recorder Sensitivity	=	0.050 V/full scale = 50 μV (full scale on chart)
Absolute Temperature Reading	=	.562 of full scale (amplifier @ 2000; recorder @ 1)

Energy Calculations

ΔT	=	0.678°C
$C_{d_{PETN}}$	=	1405 cal/g
Q per $C_{d_{PETN}}$	=	145,580 cal
Equivalent of Sphere, E_i	=	214,530 cal/°C

FUTURE WORK; COMMENTS; CONCLUSIONS

The detonation sphere has been relocated and is now housed in a temperature controlled room (Bldg. 11-20). The move from its former location was prompted by the extreme daily room temperature fluctuations.

The heats of detonation are presently measured by means of a thermocouple (iron-constantan) placed at one point of the sphere. This is inadequate since it is a single point determination. In order to promote greater reliability the use of a thermopile is being contemplated. The insulation of the sphere is also under consideration.

Examination of the sphere following the initiation of the heavily confined TNT charge revealed an indentation (1-inch x 1/4-inch) on the lid caused by shrapnel from the copper cylinder. This suggested the need for a protective plate. Serious damage could result should shrapnel strike vital regions on the lid. Four protective plates have been ordered.

Testing will be resumed after installation of the protective plates. Several tests are scheduled for the spring months. These will consist of solid and liquid HE involving confined and unconfined charges.