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Evaluation of systems for use in DSC measurements on energetic materials

David E.G. Jones *, Rainer A. Augsten

*Canadian Explosives Research Laboratory, Natural Resources Canada
555 Booth Street, Ottawa, ON K1A 0G1, Canada*

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Abstract

Tray and tube holders for microampoules containing energetic material were fabricated for use in DSC measurements. Comparison with earlier, independent studies using an aluminum tube holder demonstrated that equivalent performance was obtained. While the system with the tray holder provided statistically improved results over those obtained using the tube holder, it clearly afforded less protection for the DSC cell in the event of a microampoule rupture. Results obtained for a number of energetic materials including *t*-butyl peroxide and the explosives, tetryl, nitrocellulose and pentaerythritol tetranitrate are specifically described. In addition, explosion tests conducted using 2,4,6-trinitrotoluene enabled the elucidation of a qualitative ranking scale for potential damage to the DSC cell based on the deformation of the tray holder.

Keywords: Systems; DSC; Energetic material

1. Introduction

Use of glass microampoules in place of hermetic pans for DSC studies on energetic materials is well established. The pioneer work in this area was done by Whiting et al. [1]. They used a horizontal holder constructed of aluminum and evaluated the system performance by testing baseline stability, thermal response, high temperature performance, reproducibility and the effect of sample size using standard reference materials. Repeatability studies were also undertaken using standards and the decomposition of 2,2'-azobis (2-methylpropionitrile) (AIBN) in a methylene chloride solution. The

* Corresponding author.

statistically significant improvement over earlier results obtained for Diazald (N-methyl-N-nitroso-p-toluenesulfonamide), using a larger volume ampoule [2], was clearly demonstrated.

In this work, the aim was to evaluate the effect of different kinds of systems on the quality of the DSC results. The microampoule holders in these systems were constructed of silver, which is thermally more conductive but also denser, than aluminum. In addition, the work focuses on and compares the quality of DSC results obtained for highly energetic materials in the condensed phase. These materials are normally characterized by decomposition energies in excess of 2 kJ g^{-1} , accompanied by significant gas production. Specifically, results obtained for various explosives are outlined and also some of the pitfalls commonly encountered in thermal analysis studies on explosives are described. Our primary concern is to produce reliable results while protecting the DSC cell from damage which may occur as a result of the violent events possible from these materials.

2. Experimental

2.1. Materials

Diazald and *t*-butyl peroxide were purchased from the Aldrich Chemical Co. These chemicals were claimed to be at least 98 mol% pure and were used without further purification. The source and purity of tetryl was as before [3]. Nitrocellulose (NC) was supplied by Expro Chemical products. The sample contained approximately 70% water and was dried by storing it in a desiccator over freshly activated silica gel for one week. NC was used without any further purification. TNT (trinitrotoluene) in pill form was obtained from ICI Explosives Ltd and was used, after crushing and sieving to a 100 mesh, to conduct explosion tests on the microampoule. Pentaerythritol tetranitrate (PETN) was used as supplied by ICI Explosives Ltd.

2.2. Sample Preparation

DSC measurements were conducted on standards and on energetic material loaded into sealed glass microampoules, similar to those described in the literature [6]. A glass capillary of $1.0 \pm 0.1 \text{ mm}$ ID and wall thickness $0.35 \pm 0.05 \text{ mm}$ (Kimble Products Inc.) was sealed at one end. Solid samples were loaded by compacting them into the sealed capillary and liquids were loaded by slow injection from a syringe with its tip at the sealed end. In both cases, the inner walls were carefully cleaned after loading.

The microampoules containing sample were constructed by sealing the capillary with an oxyacetylene flame, while maintaining the end containing the sample at about 233 K by means of a specially designed holder cooled by a thermoelectric device. The microampoule could be manufactured in any size from 5 to 10 mm in length (limited by the size of the thermocouple platform in the DSC cell chamber).

For all the measurements done in this study, an effort was made to make the microampoules of uniform length and therefore of uniform volume, namely about 5 mm³.

After sealing, each microampoule was examined under a microscope for any sign of a flawed seal or of premature decomposition of sample. The microampoule was reweighed to check for sample loss and/or decomposition. Consistency of results obtained for analysis of standard and energetic material provides additional evidence for the integrity of the sealing technique.

The systems used in this study are shown in Table 1. The silver tray was made from a disc approximately 8 mm in diameter. Prior to punching out the disc, silver foil of known thickness was annealed so that the disc could easily be wrapped around a microampoule and flattened on the side in contact with the sample or reference platform in the DSC cell. For explosion tests, some additional experiments were done using foil of thickness 0.25 mm, both annealed and unannealed.

2.3. DSC measurements

A DSC 910-TA 2100 System (TA Instruments) with an oxygen-free purge gas at a flow rate of 50 cm³ min⁻¹ was used for all measurements.

In the system evaluation measurements, the procedure described in the earlier work [1] was closely followed. In general, calibration for temperature and heat flow was carried out under a standard condition, such as heating rate or mass, and other measurements were referred to that calibration.

For all measurements on energetic materials, the instrument was calibrated for temperature and heat flow using an appropriate pair selected from the standards gallium, biphenyl, indium, tin, zinc, and lead. Calibration was checked by running another standard with an intermediate melting temperature and was performed at each heating rate used with the appropriate system in Table 1. Sample sizes for the energetic materials tested were generally 1 mg or less. An additional protective plate was inserted in the DSC cell for the purpose of conducting the explosion tests, which were performed using up to 1.4 mg of TNT.

Table 1
Identification of systems used in the study

Number	Description	Mass/mg
1	Hermetic Al pan	57 (includes lid)
2	Microampoule wrapped in 0.13 mm Ag tray (Fig. 1a)	74
3	Microampoule wrapped in 0.20 mm Ag tray	118
4	Microampoule inserted in Ag tube (Fig. 1b)	590

3. Results and discussion

3.1. System evaluation

The thermal responses, at different heating rates β , for the various systems of Table 1 are compared in Table 2. The thicker Ag tray was used for the additional strength that it might provide in a catastrophic event. Surprisingly, the thermal resistance R_0 for system 4 is significantly better than that for system 3, although it is clearly more heating rate dependent. As expected, R_0 for the hermetic pan is an order of magnitude better than for either system 3 or 4, and the thermal time constant t_c increases. These results are similar to those found by Whiting et al. [1]. Fig. 2a shows that $T_f - T_i$ is heating rate dependent and also that this dependency increases with the mass of the holder, while from Fig. 2b, t_{lag} (the time from onset to peak) appears to decrease with increase in heating rate and system 4 is the least responsive, as expected. The results shown in Table 2 suggest that ΔH for system 3 decreases with increasing heating rate and is independent of β for both systems 1 and 4, although more scatter in the results is apparent for the latter system. Table 2 shows that the extrapolated onset temperature is dependent on β and this dependency is more pronounced for the more massive system.

A qualitative comparison of the nature of the thermogram peaks for the different systems is shown in Fig. 3. These results were obtained using the DSC in an uncalibrated condition. Clearly, the hermetic pan of system 1 gives the most desirable peak shape. But, since the hermetic pan cannot, in general, withstand the pressures reached in the decomposition of an energetic material, it can only be used with very small sample sizes and/or a vented lid. The uncertainties inherent in the selection of an appropriately small sample size in order to avoid venting of a hermetically sealed aluminum pan, in the first instance, and in the energy accompanying decomposition (as a result of mass loss), in the second, make the use of the sealed system of the microampoule attractive. Hence, it is important to consider the nature of the peaks for systems 2 to 4. There is no apparent difference in the results for systems 2 and 3,

Table 2
Thermal response of 10.0 mg In samples in different DSC systems

System	$\beta/^\circ\text{C min}^{-1}$	Temperature/ $^\circ\text{C}$		$R_0/^\circ\text{C mW}^{-1}$	t_c/s	$\Delta H/\text{J g}^{-1}$
		T_{onset}	T_{peak}			
1	5	156.36	157.03	0.0351	7.0	28.48
	10	156.68	157.54	0.0359	7.0	28.47
	15	157.15	158.11	0.0323	6.7	28.31
3	5	155.70	158.26	0.250	9.0	28.46
	10	156.65	160.05	0.259	9.3	28.18
	15	157.36	161.30	0.261	9.0	27.91
4	5	155.22	156.97	0.180	16.0	28.19
	10	156.61	158.91	0.193	16.0	28.35
	15	157.43	160.23	0.222	16.0	28.07

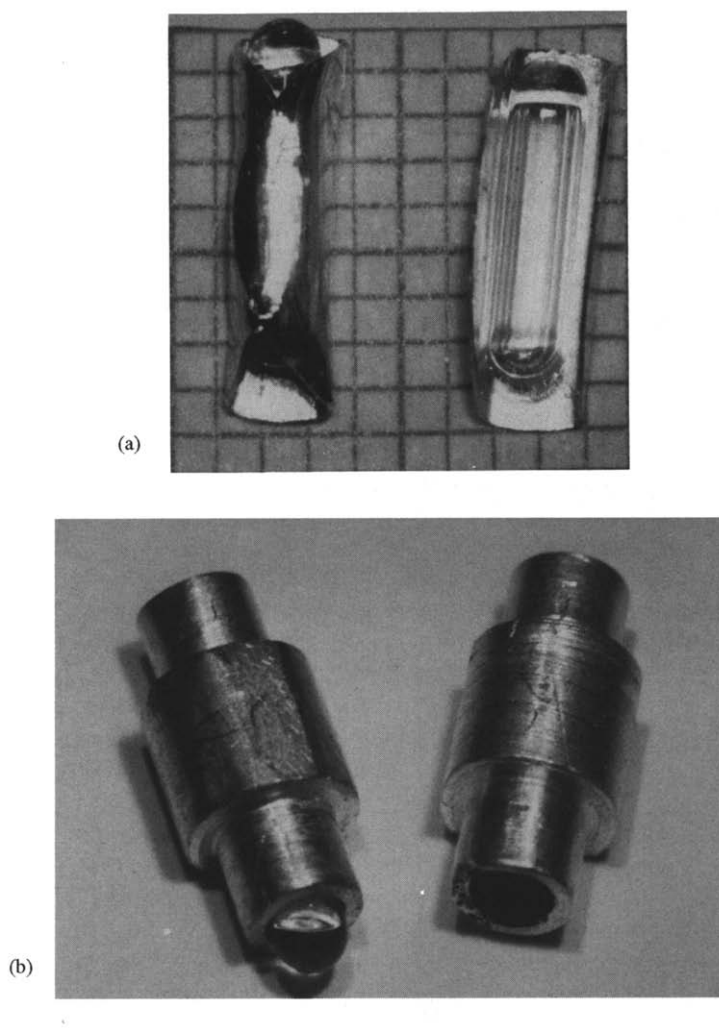


Fig. 1(a) Microampoule in tray and (b) tube holder.

although they give peaks which are less satisfactory than that for system 1, as illustrated both by the slope of the leading edge ($1/R_0$) and the decay portion of the thermograms. For system 4, the situation is somewhat worse, as apparent from the displaced onset temperature and the additional broadening of the decay portion of the thermogram trace. Use of He as the carrier gas improves both the location of the onset temperature and the sharpness of the peak, at the expense, however, of the sensitivity. Table 3 provides a comparison of various parameters using N_2 and He as the carrier gases for system 4. In Table 4, results of repeatability studies for systems 2 to 4 are summarized.

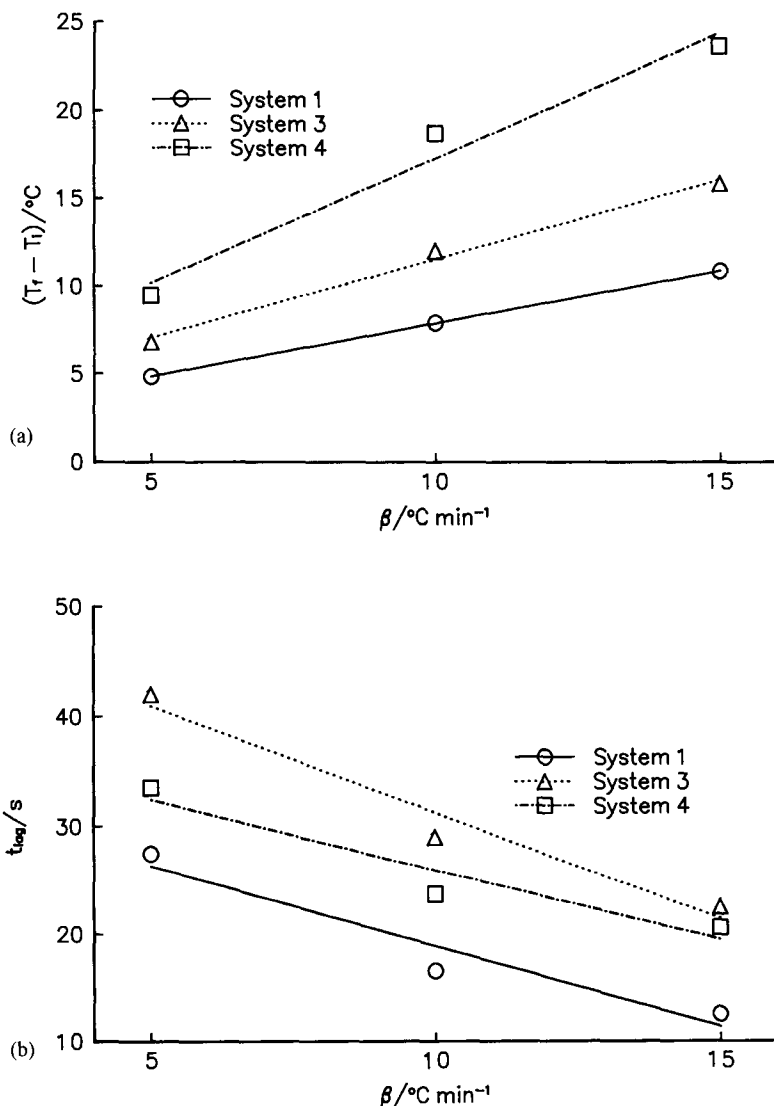


Fig. 2a,b. Comparison of performance of systems 1,3 and 4 at 5,10 and 15°C min⁻¹; Data obtained in the experiments yielding the results summarized in Table 2.

The method used is similar to that used by Whiting et al. [1] and produces results of comparable statistical significance. Table 5 presents data obtained for a sample size study done using system 2. The standard deviations obtained for the onset temperature and ΔH are similar to those obtained by Whiting et al. [1]. Figs. 4a-4d illustrate the effect of sample size on performance for systems 2 to 4. The results in Table 6 compare the high temperature performance of systems 2 and 3 with those for system 1.

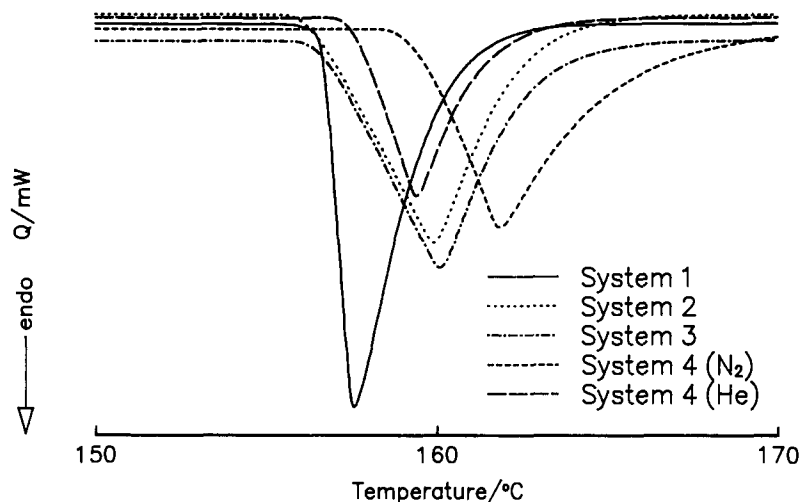


Fig. 3. Comparison of DSC curves of Indium for systems 1–4.

3.2. Constant volume measurements

A decomposition study in a microampoule is conducted under constant volume conditions, i.e. the pressure continuously varies with temperature, initially as a result of sample volatility and later because of decomposition. The latter pressure may be dynamic and abrupt for energetic materials. The pressure limits of the microampoule in a static environment have been established [1]. Indeed, critical conditions can be measured in a DSC [4], as illustrated by our results for mesitylene in Fig. 5. However, there are no published results describing the effect of the extreme dynamic loading conditions generated by an explosive in a microampoule. In our laboratory, we have had a number of microampoule ruptures but many were attributed to a weakness or fissure in the seal. In other cases, the microampoule was shattered so that evidence was

Table 3
Comparison of indium results using different purge gases for system 4

Parameter	N ₂	He
$T_{\text{onset}}/^{\circ}\text{C}$	159.49	157.65
$T_{\text{peak}}/^{\circ}\text{C}$	161.81	159.41
Q/mW	-9.81	-10.1
$R_0/^{\circ}\text{C mW}^{-1}$	-0.231	-0.172
t_{lag}/s	23.7	17.5
$t_{\text{decay}}^{\text{a}}/\text{s}$	81.5	38.0
t_{c}/s	17.0	8.2
$(t_{\text{f}} - t_{\text{i}})/\text{s}$	105.0	55.5

^a Peak to base.

Table 4
Repeatability study using 10.0 mg In samples at 5°C min⁻¹

System	$T_{\text{onset}}/^{\circ}\text{C}$	$\Delta H/\text{J g}^{-1}$	$R_0/^{\circ}\text{C mW}^{-1} t_{\text{lag}}/\text{s}$	$t_{\text{decay}}/\text{s}$
2	156.64 ± 0.20	27.94 ± 0.62	0.244	42
3	156.38 ± 0.10	28.28 ± 0.11	0.249	30
4	156.72 ± 0.15	28.24 ± 0.31	0.147	30

Uncertainties are standard deviations based on 7 tests.

destroyed, occasionally causing damage to the platen of the DSC cell. In a series of these measurements, such as required for an investigation of the dynamics of decomposition, the volume of the microampoule is maintained constant [6] in order to avoid the effect of an additional variable.

Under constant volume conditions, the energy measured is ΔU , where

$$\Delta H = \Delta U + V\Delta p$$

For decomposition energies of the magnitude observed for energetic materials, the term $V\Delta p$ is negligibly small, so that

$$\Delta H = \Delta U$$

This approximation may not be valid, however, for solid–solid phase transition energies measured under these conditions. Thus, the decomposition energy obtained from measurements in the DSC using a microampoule is equivalent to ΔH .

3.3. Constant pressure measurements

A number of studies have been carried out on energetic materials under constant pressure conditions [7]. An extensive list of results for such measurements has been published [8]. The latter compilation includes some energetic nitro compounds, as well as the explosives PETN, TNT and nitroguanidine. Our laboratory is now conducting

Table 5
Sample size study using 10.0 mg In at 5°C min⁻¹ using system 2

Mass/mg	$T_{\text{onset}}/^{\circ}\text{C}$	$T_{\text{peak}}/^{\circ}\text{C}$	$\Delta H/\text{J g}^{-1}$
0.57	156.90	157.65	28.14
1.07	156.97	157.94	27.91
2.58	157.10	158.45	28.61
5.06	156.62	158.50	28.01
7.52	156.28	158.44	27.83
10.05 ^a	156.30	158.45	28.17
12.50	156.50	159.15	27.81
15.02	156.76	159.31	28.16
average	156.68	158.49	28.08
S.D.	0.31	0.30	0.24
Ref. [1]	156.63	–	28.42

^a Calibrated.

some DSC studies on energetic material at constant pressures up to 7 MPa, using hermetic pans with laser pin hole lids.

3.4. Evaluation of results for energetic materials

Diazald

In an earlier study [2], Diazald was used in the evaluation of a large size ampoule (approximately 40 mm³). The results obtained for Diazald using systems 2,3 and 4 are

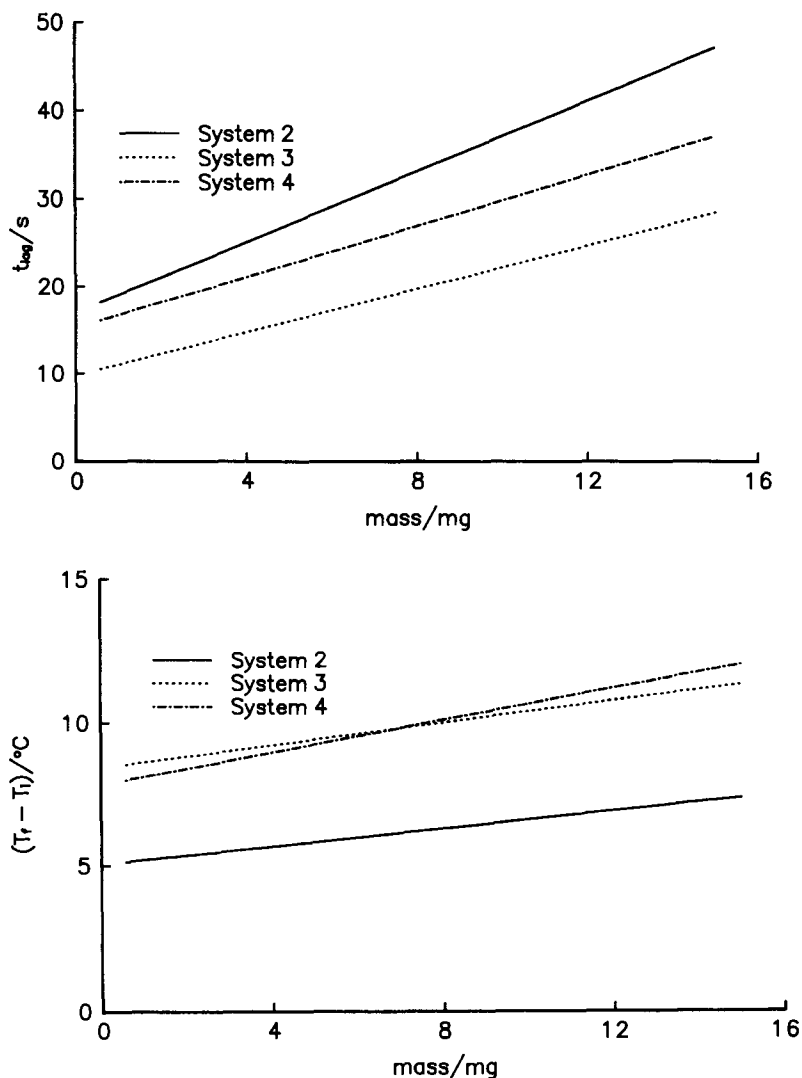


Fig. 4. a–d. Comparison of performance of systems 2–4 at 5°C min⁻¹ using different sample masses; data obtained in the experiments yielding the results summarized in Table 5.

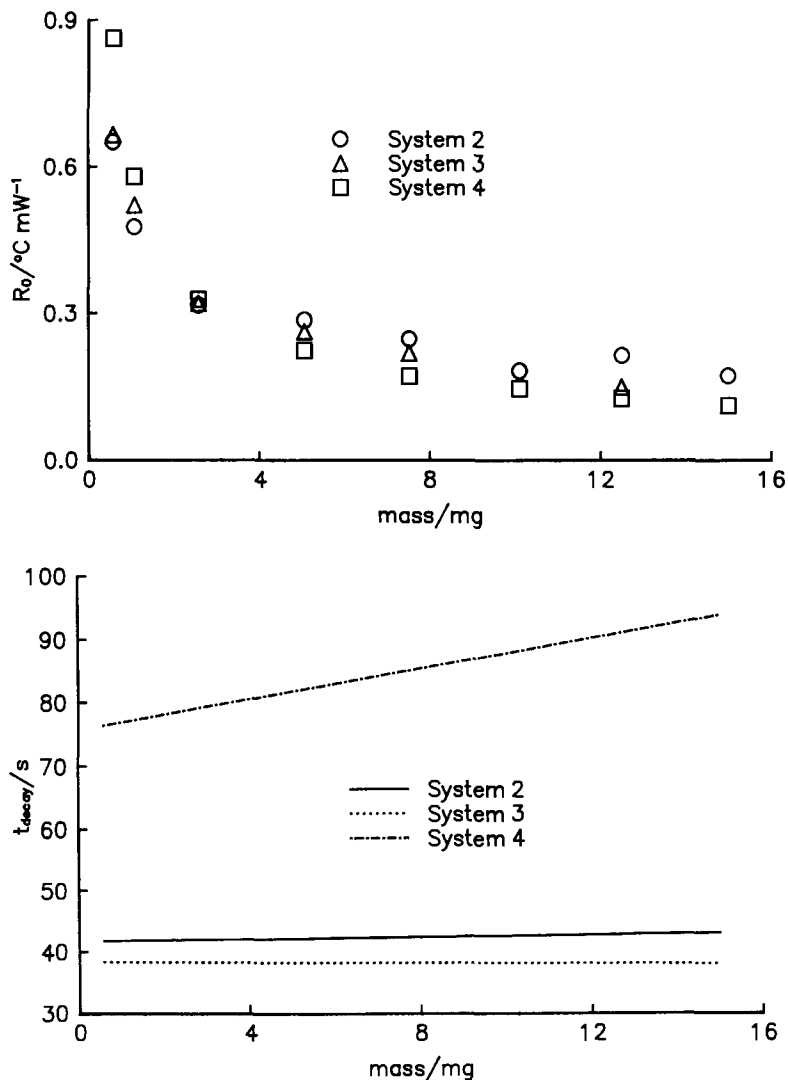


Fig. 4. (Continued).

compared with those obtained previously in Table 7 and a sample thermogram is shown in Fig. 6. There is a significantly improved precision in the results for Diazald, and the uncertainties are comparable to the corresponding values obtained by Whiting et al. [1] in their study of a methylene chloride solution of AIBN in an ampoule of approximately the same size. The quality of the results is surprising in view of the expected increase in uncertainty in the heat flow arising from the use of neat material rather than a dilute solution.

The onset temperatures in Table 7 were obtained by extrapolating the leading edge of the exotherm peak at the point of inflection back to the baseline. These temperatures

Table 6
High temperature performance using 10.0 mg metal at 5°C min⁻¹

System	Tin			Lead		
	$T_{\text{onset}}/^{\circ}\text{C}$	$T_{\text{peak}}/^{\circ}\text{C}$	$\Delta H/\text{J g}^{-1}$	$T_{\text{onset}}/^{\circ}\text{C}$	$T_{\text{peak}}/^{\circ}\text{C}$	$\Delta H/\text{J g}^{-1}$
1	232.11	233.37	58.11	327.18	328.14	22.03
2	231.65	236.38	58.79	327.52	330.19	21.73
3	231.69	235.98	58.91	328.25	331.18	22.55
Ref. [1]	231.88	–	60.5	327.47	–	23.0

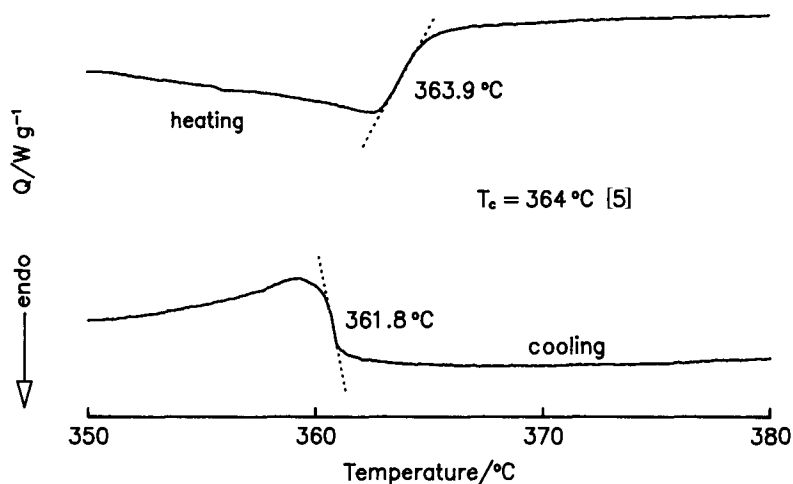


Fig. 5. DSC curve illustrating determination of critical temperature for mesitylene.

Table 7
DSC results for diazald in various systems^a

System	$T_{\text{onset}}/^{\circ}\text{C}$	$\Delta H/\text{J g}^{-1}$	$T/^{\circ}\text{C}$	$-\Delta H/\text{J g}^{-1}$
2	56.94 ± 0.10	108.3 ± 2.8	111.82 ± 0.17	1366 ± 26
3	55.17 ± 0.42	108.6 ± 2.6	111.83 ± 0.15	1345 ± 45
4	55.86 ± 0.33	108.1 ± 7.1	112.10 ± 0.29	1358 ± 63
Ref. [2] (40 mm ³ vessel)	–	100 ± 12	–	1210 ± 150

^a Uncertainties are standard deviations based on 9 measurements for ΔH and 7 measurements for T .

are clearly higher than the “true” onset temperature ($75 \pm 5^{\circ}\text{C}$), a phenomenon which has been previously discussed [9].

It has been found that the onset temperatures are sample-size dependent, but the ΔH values are not. Further, it is evident that system 2 provides the best results statistically.

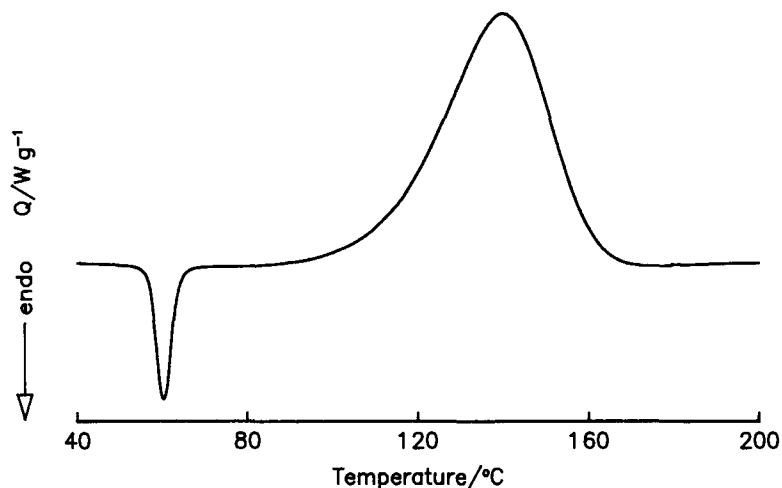


Fig. 6. DSC curve for Diazald at $5^{\circ}\text{C min}^{-1}$.

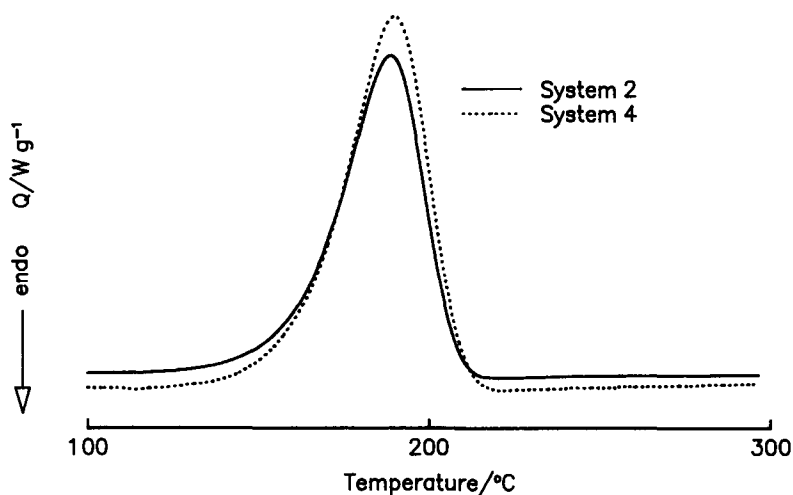


Fig. 7. DSC curves for *t*-butyl peroxide in systems 2 and 4 at $5^{\circ}\text{C min}^{-1}$.

t-Butyl peroxide

This material was chosen, firstly because it is a liquid, and secondly because it has been the subject of extensive kinetic studies; hence it has been selected as a standard test material for the ARC (accelerating rate calorimeter) [10]. Past studies were conducted in dilute solution, but the results presented here are for the neat material in systems 2 and 4. The thermograms for the two systems are compared in Fig. 7. ΔH , the “true” onset temperatures and the kinetic parameters are compared with published values [11] in Table 8. The uncertainty in ΔH is a standard deviation derived from 8 measurements at $10^{\circ}\text{C min}^{-1}$ with a 95% confidence limit of 0.05. ΔH was found to be

Table 8
DSC results for *t*-butyl peroxide

Parameter	Experimental		Literature ^a
$-\Delta H/\text{kJ g}^{-1}$	System 2	1.34 ± 0.07	1.39 ± 0.05
	System 4	1.25 ± 0.03	
T/C	System 2	125 ± 5	115 ± 5
	System 4	140 ± 5	
$E/\text{kJ mol}^{-1}$		163 ± 4	156 ± 4
$\ln(Z/\text{min}^{-1})$		42.57 ± 0.10	40.5 ± 1.0

^aRef. [5].

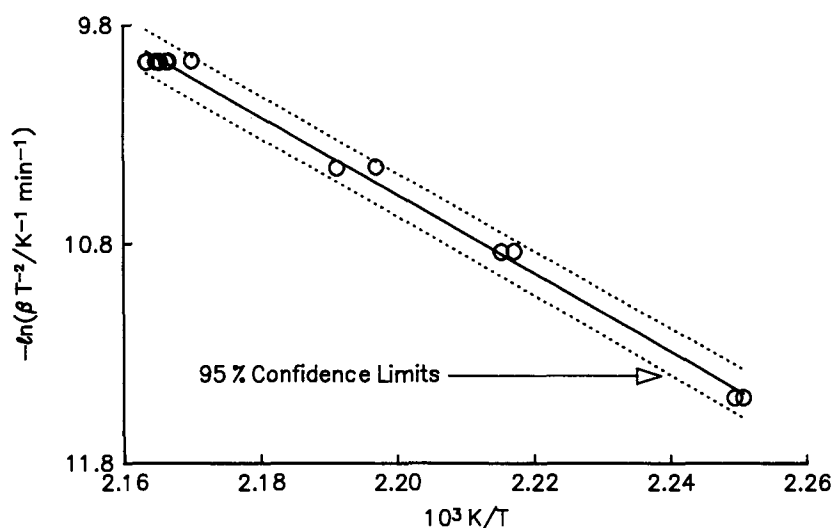


Fig. 8. Analysis of heating rate data for *t*-butyl peroxide according to ASTM E698.

independent of β over the range of $2\text{--}10^\circ\text{C min}^{-1}$, but the results for system 4 appear to be slightly low. The kinetic parameters were obtained following the procedure in ASTM E 698 [12] using system 2 and the results are plotted in Fig. 8. The uncertainties in E and $\ln(Z/\text{min}^{-1})$ were determined from the standard errors in the regression analysis.

Tetryl

Tetryl was investigated in order to illustrate the quality of results that can be obtained specifically for an explosive. These results were obtained using system 4 and $\beta = 10^\circ\text{C min}^{-1}$ and are shown in Fig. 9. As commonly observed for many energetic materials, decomposition proceeds from the liquid phase. Table 9 gives the peak temperatures and the ΔH values compared with earlier results, obtained using system 2 at 5°C min^{-1} [3]. The ΔH values for the exotherms in Table 9 were estimated by dropping a vertical line at the minimum value of the heat flow between the two peaks.

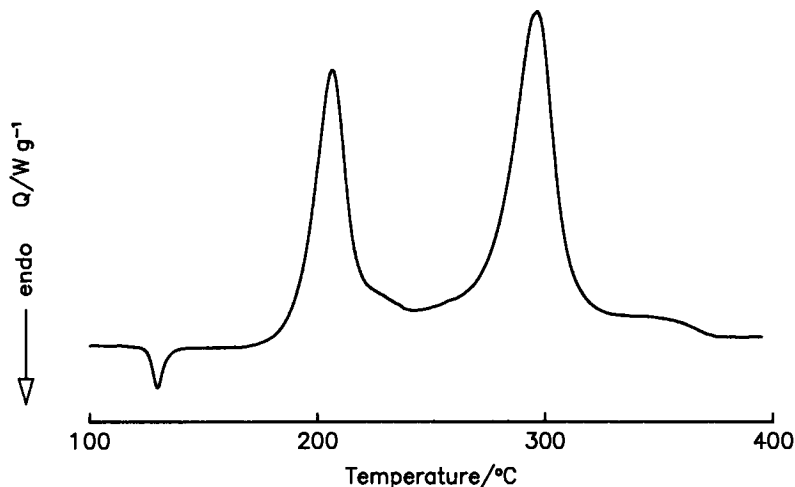


Fig. 9. DSC curve for tetryl at $5^{\circ}\text{C min}^{-1}$.

Uncertainties in T and ΔH are standard deviations based on the results of four experiments done in the DSC. The “true” onset temperature at $10^{\circ}\text{C min}^{-1}$ of exotherm 1 in the DSC is $170 \pm 5^{\circ}\text{C}$, as estimated by the first point of deflection from the baseline. The onset temperature determined from ARC measurements is $140 \pm 10^{\circ}\text{C}$ [13]. The “heat of detonation” for tetryl has been found to be density dependent with a maximum of about -4.9 kJ g^{-1} [14]. The sum of the energies of the exotherms 1 and 2 in Table 9 is in reasonable agreement with this value.

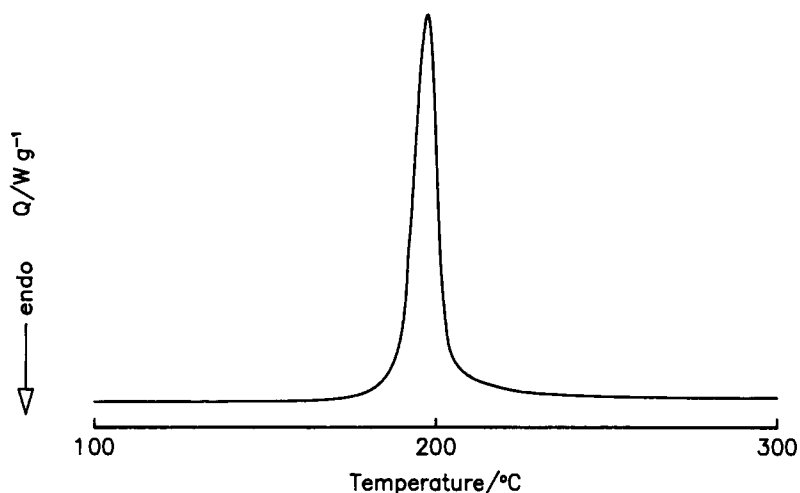
Nitrocellulose

The explosive nitrocellulose (NC) was investigated since it decomposes in the solid phase, a phenomenon that is rarely observed for energetic materials. The results give an indication of the effect of the particle-air gap on reproducibility. Furthermore, solid-phase models can be used to treat the dynamics of the decomposition process and this subject will be discussed elsewhere [15]. A thermogram showing the decomposition of NC at $5^{\circ}\text{C min}^{-1}$ using system 2 is depicted in Fig. 10 and Table 10 illustrates the reproducibility of DSC results for a set of 6 measurements.

Table 9
Peak temperatures and energetics for tetryl

	$T/^{\circ}\text{C}$		$\Delta H/\text{kJ g}^{-1}$		
	1 ^a	2 ^b	1 ^a	2 ^b	Literature
Fusion	$125 \pm 2^{\circ}$	$123 \pm 3^{\circ}$	0.078 ± 0.006	0.081 ± 0.002	0.080
Exotherm 1	197.5 ± 0.3	206.3 ± 0.5	-1.77 ± 0.03	-1.89 ± 0.05	–
Exotherm 2	283.7 ± 0.9	296.5 ± 0.3	-2.92 ± 0.01	-3.13 ± 0.24	–

^a System 2 at $5^{\circ}\text{C min}^{-1}$, Ref. [3]. ^b System 4 at $10^{\circ}\text{C min}^{-1}$. ^c Onset.

Fig. 10. DSC curve for nitrocellulose at $5^{\circ}\text{C min}^{-1}$.Table 10
DSC results for nitrocellulose using system 2

Trial	Mass/mg	$T_0/^{\circ}\text{C}$	$T_{\text{peak}}/^{\circ}\text{C}$	$-\Delta H/\text{kJ g}^{-1}$
1	0.64	188.84	196.82	4.18
2	0.64	189.80	198.30	3.94
3	0.66	189.28	196.53	4.07
4	0.82	190.31	197.32	3.66
5	0.71	188.48	196.01	4.15
6	0.71	188.24	196.00	4.14
Average	–	189.17	196.83	4.02
S.D.	–	0.82	0.88	0.20
95% Conf. Int.	–	0.86	0.92	0.21

PETN

Fig. 11 is shown to illustrate one of the common problems encountered when performing DSC experiments on energetic materials. If the mass of the sample is excessively large, the heat flow arising from decomposition may exceed that from the imposed heating rate, resulting in a leaning peak, as observed for the thermogram of the larger sample of PETN in Fig. 11. The thermogram for the sample of lower mass shows no apparent lean. However, in many cases, the attendant peak distortion is not readily apparent without expanding the temperature or time scale of the thermogram or checking the linearity of the temperature vs. time plot. Excessive heat flows caused by this phenomenon distort the correction term [12] used for thermal lag in the ASTM E 698 procedure used for determining kinetic parameters.

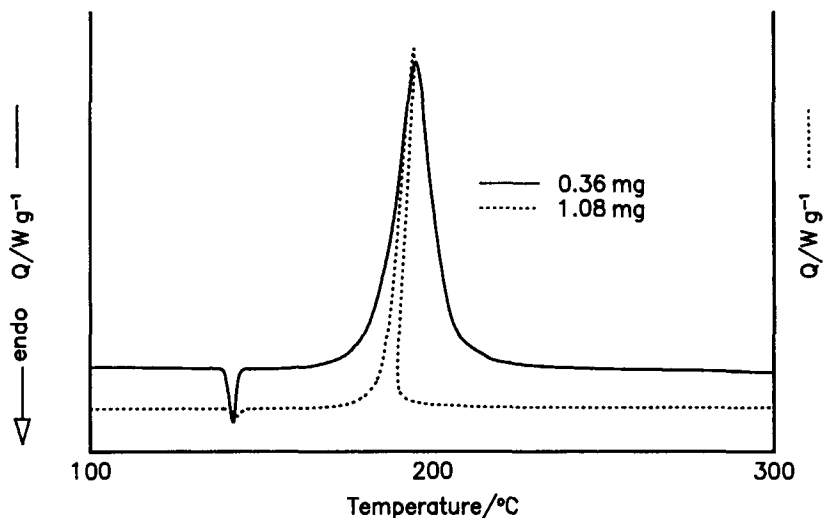


Fig. 11. DSC curve for PETN at $5^{\circ}\text{C min}^{-1}$ illustrating effect of excessive sample size.

Table 11

Correlation of microampoule failure and potential for damage to DSC cell based on tray deformation

System	Mass TNT/mg	$\beta/^{\circ}\text{C min}^{-1}$	Failure	Damage potential
2	0.977	10	No	—
2	1.004	10	No	—
2	1.006	10	No	—
2	1.012	10	Yes	High
2	1.028	10	No	—
2	0.976	10	No	—
2	0.986	10	No	—
2	1.104	10	Yes	Medium
3	0.949	10	No	—
3	1.016	10	Yes	Medium
3	1.025	10	No	—
3	1.140	10	No	—
3	1.156	10	No	—
3	1.168	10	Yes	Medium

3.5. Explosion tests

These tests were undertaken for the purpose of attempting to correlate the severity of deformation sustained by the silver tray of systems 2 and 3 and, in some cases, a 0.25 mm thick tray, with the potential for damage to the platen of the DSC cell. The force of an exploding ampoule causes the tray to open up and unfold to varying degrees. In the extreme case, the tray either inverts and strikes the platen or, is actually broken into pieces. Both of these situations pose the greatest potential for denting the platen.

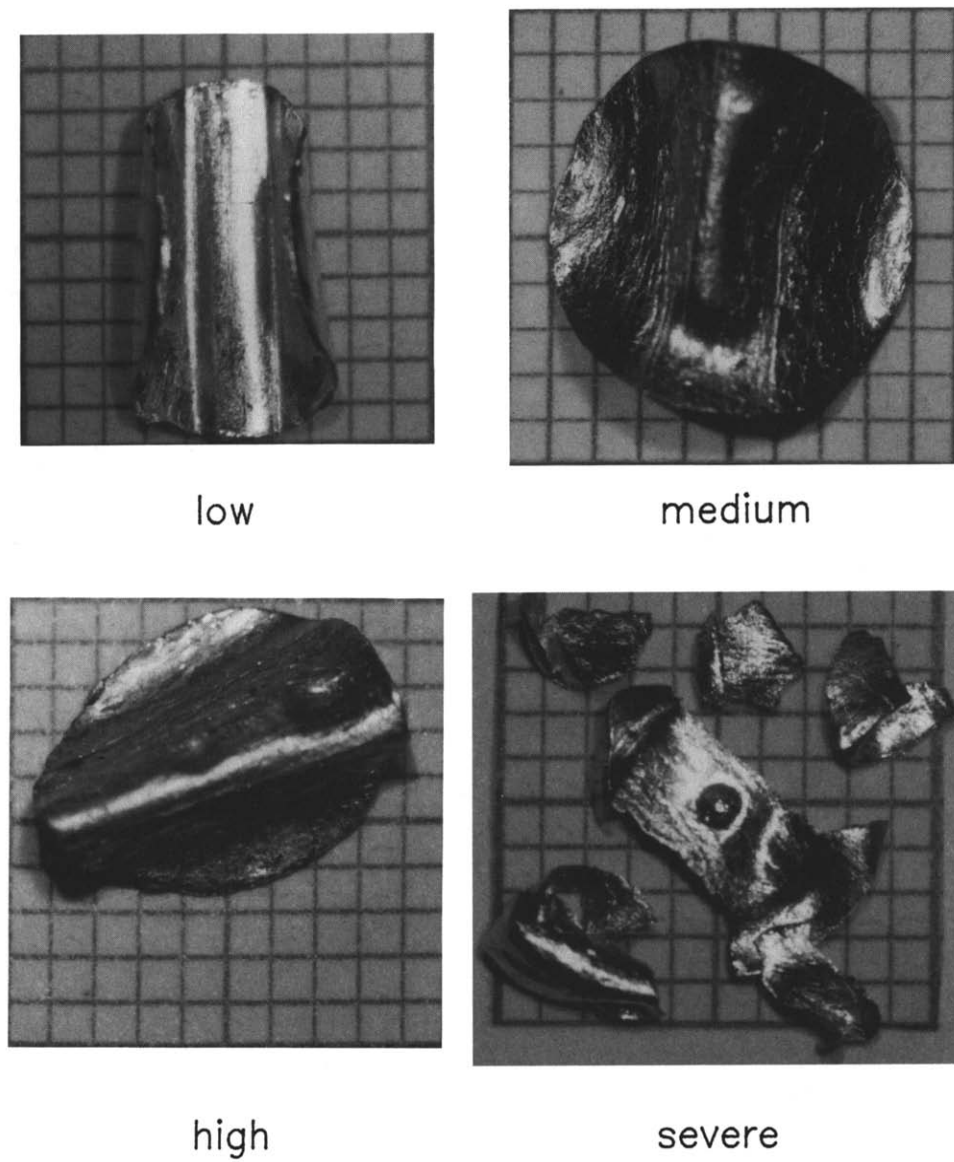


Fig. 12. Qualitative ranking scale for damage to DSC cell based on deformation of tray holder.

It seemed important therefore to explore the factors that determine the degree of unfolding which takes place. It has been found that one in ten ampoules, loaded with about 1 mg of TNT, will result in an ampoule explosion. For the purpose of these tests, the ampoules often contained slightly more than 1 mg. Fig. 12 shows a qualitative ranking scale for damage potential ranging from severe to low, based on the observed effect on the tray. An increase in the probability of explosion with increased heating rate

Table 12

Correlation of microampoule failure and potential for damage to DSC cell based on tray deformation using a 0.25 mm tray

Mass TNT/mg	$\beta/^\circ\text{C min}^{-1}$	Failure	Damage Potential
1.273	10	Yes	Medium
0.941	20	No	–
1.325	20	Yes	Severe
1.363	20	Yes	Severe
0.941	20	Yes	Low
0.960	20	Yes	Medium
1.074	20	Yes	Severe
1.038	20	Yes	High
1.134	20	Yes	Low

was clearly demonstrated. Tables 11 and 12 illustrate some of the results obtained in these tests.

4. Conclusions

It has been demonstrated that DSC studies using a glass microampoule sample holder can yield results of good quality. Experiments using both tray and tube type holders for the microampoule give comparable performance results to those reported earlier. In addition, these systems have provided both thermodynamic and kinetic data which agree with published results for known materials, even when the DSC measurements are carried out using the neat materials.

More importantly, the systems have been shown to give reproducible results for energetic materials, including explosives. It has been demonstrated that the maximum protection for the DSC cell is provided by the thicker tray or the tube, at the expense of some apparent loss in sensitivity. For energetic materials, it is critical to use the minimum sample size and a low heating rate. These factors are especially important when the thin tray is used. The latter generally provides results of the best statistical quality while, at the same time, the highest risk for potential damage to the DSC cell in the event of an ampoule explosion.

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