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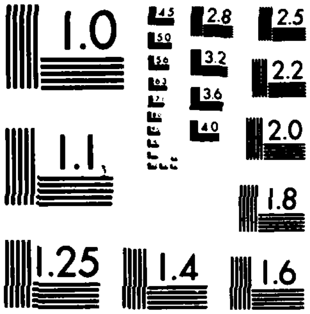

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SPECIFIC HEAT OF OCTAHYDRO - 1,3,5,7 - TETRANITRO - 1,3,5,7 - TETRAZOCINE (HMX)

A Special Report to AFOSR

F49620-81-K-0011

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by

L.G. Koshigoe, R.L. Shoemaker and R.E. Taylor

Janaury 1983

School of Mechanical Engineering

Purdue University, West Lafayette, Indiana

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ABSTRACT

The specific heat of octahydro - 1,3,5,7 - tetrenitro - 1,3,5,7 - tetrazocine (HMX) has been studied with a Differential Scanning Calorimeter from 315-486°K. Measurements were made on both small pieces of single crystals of HMX and on a powdered blend. In both cases, the specific heats of the β and δ phases, and also of partially decomposed HMX were determined. The results show that the specific heat values for the two phases are nearly the same. Differences in the specific heat values for the single crystals, powdered, and partially decomposed samples are small, and all lie within a band of 6%. The values for the single crystals are the lowest; for the powdered blend, intermediate; and for the partially decomposed, highest. In the case of the single crystals versus the powder, the values are within the combined experimental error of 3%.

INTRODUCTION

Knowledge of the specific heats of rocket propellants, especially at higher temperatures where significant decomposition occurs, is a necessity for understanding various physical processes which occur in the combustion of these propellants. In addition, the specific heats may reveal certain characteristics of the propellants which are important for their use. When specific heat is combined with other thermophysical properties (conductivity or diffusivity) and densities of the propellants, then the nature of heat conduction in the propellants is essentially characterized.

As a step towards the goal of understanding the nature of heat transfer in a particular propellant, namely octahydro - 1,3,5,7 - tetranitro - 1,3,5, 7, - tetrazocine (HMX), the specific heat was determined by the use of a Perkin-Elmer Model DSC II Differential Scanning Calorimeter (DSC). Both small pieces of single crystals and a powdered blend of HMX were studied.

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* Specific heat refers to that at constant pressure.

Previous determinations of the specific heat of HMX were made by the use of an ice calorimeter (1) and a Perkin-Elmer Model DSC-1B Differential Scanning Calorimeter (2), but no determinations were made past the β to δ phase transformation. In this study results were obtained for both the β and δ phases. From accurate specific heat values over a reasonable temperature range, values for the non-decomposed material can be safely extrapolated to higher temperatures. This will give usable values into the combustion temperature range, where the greatest interest lies.

In addition, the material present at the combustion temperatures consists of HMX and probably decomposition products (3), so it is important to know what dependence the combination (rather than pure HMX) has on specific heat. In this study efforts were made to determine the effects on specific heat due to decomposition products intermixed in the HMX lattice.

Specific heat results were also obtained for a sapphire standard immediately following the HMX runs, to make checks on the experimental error. The sapphire used was approximately the same size as the HMX samples to insure comparable errors. Results for sapphire typically fell with 1.5% of the NBS values.

Determinations of the temperature at which HMX transforms from β to δ, and the temperature interval over which the transformation occurs were also made.

EQUIPMENT

A Perkin-Elmer Model DSC II Differential Scanning Calorimeter was used for the specific heat determinations (see Figure 1). Slight modifications were made to the sample enclosure by using a Flow-Through Cover (Perkin-Elmer accessory 319-002), which decreases the amount of contamination to the enclosure due to decomposition or vaporization of the sample. Nitrogen gas was continuously allowed to pass through the enclosure and cover, thus purging out most evolving gases from the sample.

The DSC measures the differential power required to keep a sample's temperature rising at a chosen rate when compared to that of a reference. When a standard such as sapphire is measured in addition to the sample, then the calculation of specific heat is greatly simplified. The defining equation for specific heat at constant pressure is $C_p = \frac{1}{M}\frac{dq}{dt}$ where M denotes the sample mass, dq the differential heat absorbed, and dt the differential temperature change. Given known specific heat values for the standard material (sapphire), and from measurements of the standard's and sample's masses and differential power requirements obtained by raising their temperatures at the same rate as that of the reference, the specific heat of the sample may then be calculated from

$$C_{psam} = C_{pstd} \frac{M_{std}}{M_{sam}} \cdot \frac{\left(\frac{dq}{dt}\right)_{sam}}{\left(\frac{dq}{dt}\right)_{std}} .$$

All measurements of HMX were made using sapphire as the standard.

Data retrieval and analysis were completely controlled by a digital data acquisition system (PDP 11/34A, see Figure 2). This minimized errors caused by human interaction and also significantly reduced total time spent for data analysis.

SAMPLE PREPRATION

Little sample preparation was required prior to the DSC measurements. Relatively large single crystals of HMX were remotely cleaved, until small enough pieces were obtained to fit into the sample pans (diameter = 5mm, thickness = 1mm). It was found that the sample needed to be as massive as possible to obtain good repeatability. Typical sample masses were about 17 mg. The single crystals were grown as described by Boggs, Price, Zurn, Derr and Dibble (4), and as stated in their paper were believed to have been of very high purity. Their electrochemical analysis showed no evidence of RDX (hexahydro - 1,3,5, - trinitro - s - triazine) to an accuracy of < 0.1%.

Powdered samples consisted of a blend of various particle sizes (see Table 1) and are listed to be 99.7% pure HMX. In this case samples of about 19 mg. were typical.

In both the single crystals and powdered blend samples, the DSC pan lids were crimped onto the sample pans using a DSC crimping press.

Partially decomposed samples were prepared by heating a single crystal or powdered blend sample up to approximately 510°K in a furnace, and maintaining it at that temperature until sufficient decomposition was apparent. If a sample had lost one-eighth or greater of its mass, it was considered to have undergone sufficient decomposition to assume that a significant portion of the remaining material consisted of decomposition products. Checks were made on mass losses every few minutes. It was assumed that the mass loss was caused by decomposition and volatilization of gaseous products, and volatilization of higher vapor species. However, it was not known how much of the remaining material consisted of HMX and how much was decomposition products. It was also assumed that a sufficient amount of decomposition products were retained in the lattice to claim the sample was actually a mixture of HMX and a significant amount of decomposition products. Evidence for the validity of this assumption will be given later.

RESULTS AND DISCUSSION

Specific heat results are shown in Figure 3 and Table 2 for the single crystal and powdered blend HMX samples at a heating rate of 5°K/min. These results are for the β phase only and were obtained over a range of 315-445°K.

Differences in the single crystal and powdered HMX specific heats were small. The maximum deviation between the two was approximately 3%. The powder results tended to be biased slightly upwards from that of the single crystal. The difference is at the limit of the combined experimental error, and could be in part attributed to small amounts of impurities within the powdered sample's lattices (at most 0.3% impure, see Table 1). This effect would be small though, since the impurities probably consist of molecules of similar atomic weights as those present in the HMX molecule. Usually the major impurity in HMX is RDX (5), which is chemically very similar to HMX (6). This gives further strength to the argument for only a small impurity cause.

Also given in Table 2 are results for runs made on single crystal and powdered HMX using heating rates other than 5°K/min (i.e. 2.5°K/min, and 10°K/min). The different rates allowed for a check on whether uniform heating of the samples was maintained. If no biasing in results were observed over a factor of four variation in heating rates, then it could be assumed that specific heat results were insensitive to any existing temperature gradients. Several samples were tested at the varying rates, and some were tested more than once, to observe the effects in specific heat values with repeated heatings. No apparent change was seen with repeated heatings.

In addition to the specific heat results obtained for the β phase in this work, the results of Velicky, Lenchitz, and Beach (1) using an ice calorimeter and those of Wilcox (2) using a DSC 1B are shown in Figure 3. Differences in all cases fell within a band of approximately 4% of each other, which is very good given the inherent experimental and calculational errors in each case. The average slope of the specific heat versus temperature curves for references (1) and (2) are less than that obtained here, but confidence exists in the present results due to good repeatability for varying heating rates, and also due to good sapphire results obtained immediately following the HMX runs.

Delta phase results obtained beyond the phase transformation are shown in Figure 4 and are given in Table 3 for both the single crystal and the powdered blend samples. Also shown in Figure 4 are the β phase results for comparison. The temperature range over which δ phase data past the transformation could be obtained was small due to appreciable exothermic effects accompanying sample decomposition. It was found that accurate specific heat values in the δ phase (beyond the transformation) could only be obtained from 472 to 486°K.

Since the δ phase results could only be obtained over a very short temperature range above the phase transformation, other techniques were sought for extending this range. It was apparent that after a sample had been taken below the transformation temperature, the reverse transformation (i.e. δ to β) occurred very slowly. In fact it could take hours or weeks to completely transform back to β phase, depending on the sample's purity and crystallinity (3). Taking advantage of this fact allowed for δ phase results to be obtained at temperatures below the transformation temperature, thus extending the range of valid δ phase specific heat data. Sample temperatures were normally lowered to 400°K immediately following completion of the phase transformation, and then the samples were run again to determine their specific heats from 410-485°K. See Figure 5 for comparisons of [illegible] differential power absorbed during a run through the transformation, and then a run immediately following cooling to 400°K. As seen, there is only a small difference between the amplitudes (thus implying equivalent specific heats). Also it is apparent that no transformation peak occurred in the second run, thus implying the sample remained in the δ phase over the entire temperature range for this run. Results for the δ phase could not be obtained at a temperature lower than approximately 400°K due to partial transformation back to the β phase during subsequent runs. Also shown in Figure 5 is the empty pan reference run.

Figure 6 shows specific heat results for both the β and δ phases, where the δ phase data includes the runs made immediately following the transformation, and those made over the extended temperature range of 410-485°K. Also, the data for the extended temperature range is given in Table 3. As seen, the β and δ phase specific heats appear to be in agreement, as are the δ phase runs made after the transformation and over the extended range. Specific heat is relatively insensitive to changes in crystal structure, and in fact, is also fairly insensitive to changes in concentrations of similar materials. Essentially no changes in concentrations are taking place during the phase transformation. Concentration changes exist due to decomposition and evolution of the gaseous products, but these are undetectable at the transformation temperature. Therefore the specific heat values for the two phases would be expected to be in good agreement.

The specific heats of samples which had undergone significant decomposition in a separate oven were measured from 315 to 445°K. No results could be obtained beyond this range due to excessive decomposition occurring during the run. Since specific heat data could not be obtained beyond this range due to large decomposition effects, it is believed that sufficient decomposition products had been retained in the sample lattice and were catalyzing further decomposition (7). No β to δ phase transformation was observed for the partially decomposed samples unless several days had past. For the powder, the transformation was small, and in the case of the single crystal, it was almost non-existent, as seen by the use of the DSC. This gives additional evidence that the samples contained significant portions of decomposition products. Thus, the values obtained from these runs could be assumed to be for a mixture of HMX and decomposition products. Both single crystal and powdered HMX were measured in this way, but only at the 5°K/min heating rate, since sufficient evidence had already been obtained to show that any temperature gradients were negligibly small for determinations of specific heat.

Figure 7 compares the results for the partially decomposed single crystal and powdered samples to those for the parent materials. Data for these results are given in Table 4. The specific heats of the partially decomposed samples tend to be shifted upwards by a few percent. Since significant portions of the samples had decomposed, and since large portions of the decomposed material had vaporized, the change in chemical makeup of the remaining material could cause this increase in specific heat. Note that the partially decomposed powder results tended to be slightly above those of the partially decomposed single crystal. The maximum deviation between the single crystal and decomposed single crystals was 5%, whereas for the powder versus the decomposed powder, it was 4%. While the difference is within the combined experimental error, the average increase appears to be slightly greater for the crystal than for the powder, and this may be attributed to decomposition products being retained in the crystal lattice to a much greater extent than in the case of the powder, due to its larger particle size (7).

Although the β to δ phase transformation energy was not determined in the present work, the temperature of initiation of the transformation, and the interval over which it occurs upon heating was investigated. Much smaller samples were prepared for this test (5 mg.) to insure that there would be little chance for temperature gradients to develop. Indium and lead samples were run through their melts to verify the accuracy of the DSC temperature measurements. The melt temperatures for the two are 429.78°K and 600.65°K, respectively. From these melts, an error of 1 to 2°K too low was found. Since HMX transforms at a temperature between these two melts, and assuming the temperature gradients were negligible, the observed values for HMX should have been accurate to within 2°K.

In all cases, regardless of rate and sample mass, the start of the transformation occurred at approximately 453°K and 460°K for the single crystal and powdered HMX, respectively. The difference between these temperatures could be attributed to the fact that smaller particles in the β phase are more stable toward the transformation (7). Note that the observed transition temperatures are higher than those found by Karpowicz and Brill (3) and by Teetsov and McCrone (8) at 433°K and 430 ±1°K, respectively. Different sample cells and configurations were used in those cases, than in this work. On the other hand, similar results were reported by Hall (9) with the use of a Perkin-Elmer Model DSC-1 Differential Scanning Calorimeter. He obtained a transition temperature of 460±3°K at a rate of 8°K/min, and the length of the transformation occurred over an interval of 16°K.

Unlike the specific heat results, the β to δ phase transformation temperature range did show variations with heating rate. The greater the heating rate, the longer the temperature range over which the transformation took place. For example, ranges at rates of 2.5, 5, and 10°K/min were typically 8, 16, and 20°K long, respectively. This was true for both the single crystal and powdered HMX. Also, the range was dependent on sample mass (although the above comparisions were made on equivalent masses), and decreased with decreasing sample mass. For the runs made on the small 5 mg. samples at a rate of 2.5°K.min, a range of 3-4°K was observed.

In addition to the normal β to δ phase transformation endotherm, extra endotherms were observed at other temperatures in some cases. These additional endotherms were sometimes seen during a first run of a sample, but were most often seen after repeated heatings. They typically occurred between 490-500°K, although on occasion they were observed at lower temperatures. The causes could be due to : (1) Non-uniform heating because of poor thermal contact between portions of the samples and the pans they were contained in or because of an increase in intergranular resistance after transformation due to a decrease in sample density; (2) Creation of nucleation sites after partial decomposition or by retention of δ-HMX in the lattice from previous runs, thus causing a transformation to occur at lower temperatures; or (3) an additional polymorphic conversion other than β to δ. Cady and Smith (5) observed that sometimes α-HMX is formed along with β-HMX during the β to δ phase transformation, and then α would undergo conversion to δ upon additional heating. Probably the first and second possibilities were responsible for the conversions in samples which had already been heated, but the third could explain the endotherms in virgin samples.

CONCLUSIONS AND SUMMARY

Specific heat results for the β phase of HMX have been obtained for both single crystals and a powdered blend from 315 to 445°K. The powdered blend samples had specific heats which were biased upwards from that of the single crystals, with a maximum deviation of about 3%. This might be due to impurities, but is at the limit of the combined experimental error. Literature values (1) and (2) for the specific heat of HMX are in good agreement with those found in the present work, and differ at most by about 4% (from either the single crystals or powdered blend results).

Heating rates were changed by a factor of four to check on any specific heat dependence with rate. No differences were found, thus implying temperature gradients were negligibly small.

In addition to the β phase results, determinations of the specific heat of HMX were also made in the δ phase. Again, measurements were made on both single crystal and powdered blend HMX. Data beyond the normal transformation temperature (i.e. β→δ transition) were obtained from 472 to 486°K. Also, due to the sample's hysteresis for the reverse transformation (i.e. δ→β) after cooling below the transition temperature, results could be obtained in the δ phase over an extended range of 410 to 485°K. Differences in results for the two ranges were within the experimental error.

Comparison between the β and δ phase specific heat values show the two have similar dependences on temperature and can be assumed to be the same within ±1%. Thus, specific heat as a function of temperature for HMX in both the β and δ phases is known from 315 to 486°K within an experimental uncertainty of ±1.5%.

Samples which had undergone partial decomposition and contained significant portions of decomposition products were also studied. Values of the specific heat for these samples were found to be slightly larger, with the partially decomposed single crystal deviating from the undecomposed material by about 5% and the powdered blend deviating from the undecomposed material by 4%. This upwards biasing effect could be caused by differences in specific heats between the pure HMX and its decomposition products. Also, as seen, the biasing effect would be expected to be small since the decomposition products retained in the HMX lattice would most likely be of a similar nature to that of pure HMX. Note that the greater amount of biasing seen for the single crystals than for the powdered blend can be explained by increased retention of decomposition products in the lattice of the single crystal (7), but not enough evidence exists for this to make definite conclusions. From these results, it may be concluded that the specific heat of any combination of HMX and its decomposition products existing during combustion will have a similar specific heat to that of the undecomposed material.

The temperature of initiation of the transformation of HMX from the β to δ phase was found to occur at 453°K for the single crystals and 460°K for the powdered blend. These results are higher than those seen by others (3) and (8) using different equipment and techniques, but similar to that found by Hall (9) using a DSC 1.

From the specific heat values obtained for both the β and δ phases of HMX, and from determinations of their corresponding thermal diffusivities, values for the thermal conductivity may then be calculated (given the density of the material). Studies are underway to determine the thermal diffusivity of HMX in both the β and δ phases, and to determine the effects of partial decomposition. Note that this information is important since partially decomposed HMX is the existing form at the combustion temperature (3).

TABLE 1

• PURITY AND BLEND INFORMATION FOR HMX POWDERED BLEND SAMPLES

MATERIAL	HOLSTON LOT NUMBER	PARTICLE SIZE (μm)	PERCENT OCCUPYING BLEND	PERCENT HMX (PURITY)
HMX	5486	18.9	50	99.7
HMX	7016	6.0	30	99.7
HMX	7016	3.3	20	99.7

TABLE 2

SPECIFIC HEAT VALUES FOR SINGLE CRYSTAL AND POWDERED BLEND β-HMX AT VARIOUS HEATING RATES

		Cp (J/g°K) HMX-Single Crystal β Phase			Cp (J/g°K) HMX-Powdered Blend β Phase		
TEMP. (°K)	TEMP. (°C)	2.5°K/min	5°K/min	10°K/min	2.5°K/min	5°K/min	10°K/min
315	42		1.065	1.049	1.084	1.072	1.063
320	47	1.054	1.078	1.062	1.099	1.085	1.076
325	52	1.065	1.089	1.074	1.115	1.102	1.089
350	77	1.122	1.152	1.136	1.149	1.166	1.158
360	87	1.147	1.177	1.162	1.172	1.191	1.188
375	102	1.189	1.217	1.200		1.241	1.232
400	127	1.275	1.284	1.269		1.299	1.297
420	147		1.337	1.318		1.359	1.341
425	152		1.349	1.332		1.376	
445	172		1.399	1.384		1.423	

TABLE 3

SPECIFIC HEAT VALUES FOR SINGLE CRYSTAL AND POWDERED BLEND δ-HMX OBTAINED IMMEDIATELY FOLLOWING THE β to δ TRANSFORMATION AND ALSO OVER THE EXTENDED RANGE

		C_p (J/g°K) HMX-Single Crystal δ Phase		C_p (J/g°K) HMX-Powdered Blend δ Phase	
T(°K)	T(°C)	After Transition	Extended Range	After Transition	Extended Range
410	137		1.315		
415	142		1.330		1.347
425	147		1.362		1.381
450	177		1.411		1.439
472	199			1.461	
475	202		1.448		1.502
476	203			1.475	
477	204	1.443			
480	207	1.450		1.491	
483	210	1.458			
484	211			1.503	
485	212		1.470		1.524
486	213	1.465		1.506	

TABLE 4

SPECIFIC HEAT VALUES FOR PARTIALLY DECOMPOSED SINGLE CRYSTAL AND POWDERED BLEND HMX

T(°K)	T(°C)	C_p (J/g°K) Single Crystal Partially Decomposed	C_p (J/g°K) Powdered Blend Partially Decomposed
315	42	1.095	1.095
325	52	1.122	1.126
350	77	1.194	1.203
375	102	1.265	1.278
400.	127	1.323	1.347
425	152	1.401	1.410
445	172	1.440	1.423

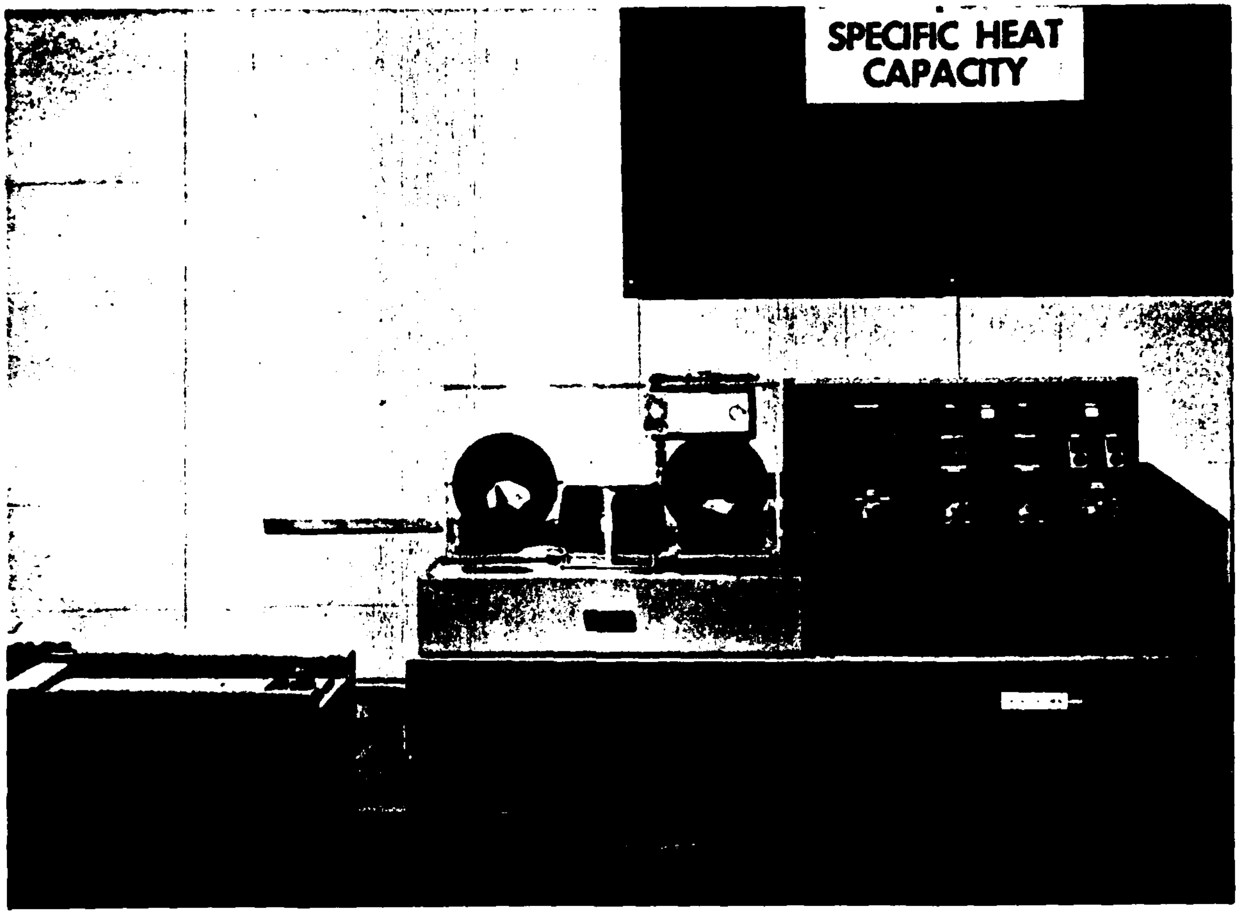

Figure 1. Perkin-Elmer Model DSC-2 Differential Scanning Calorimeter

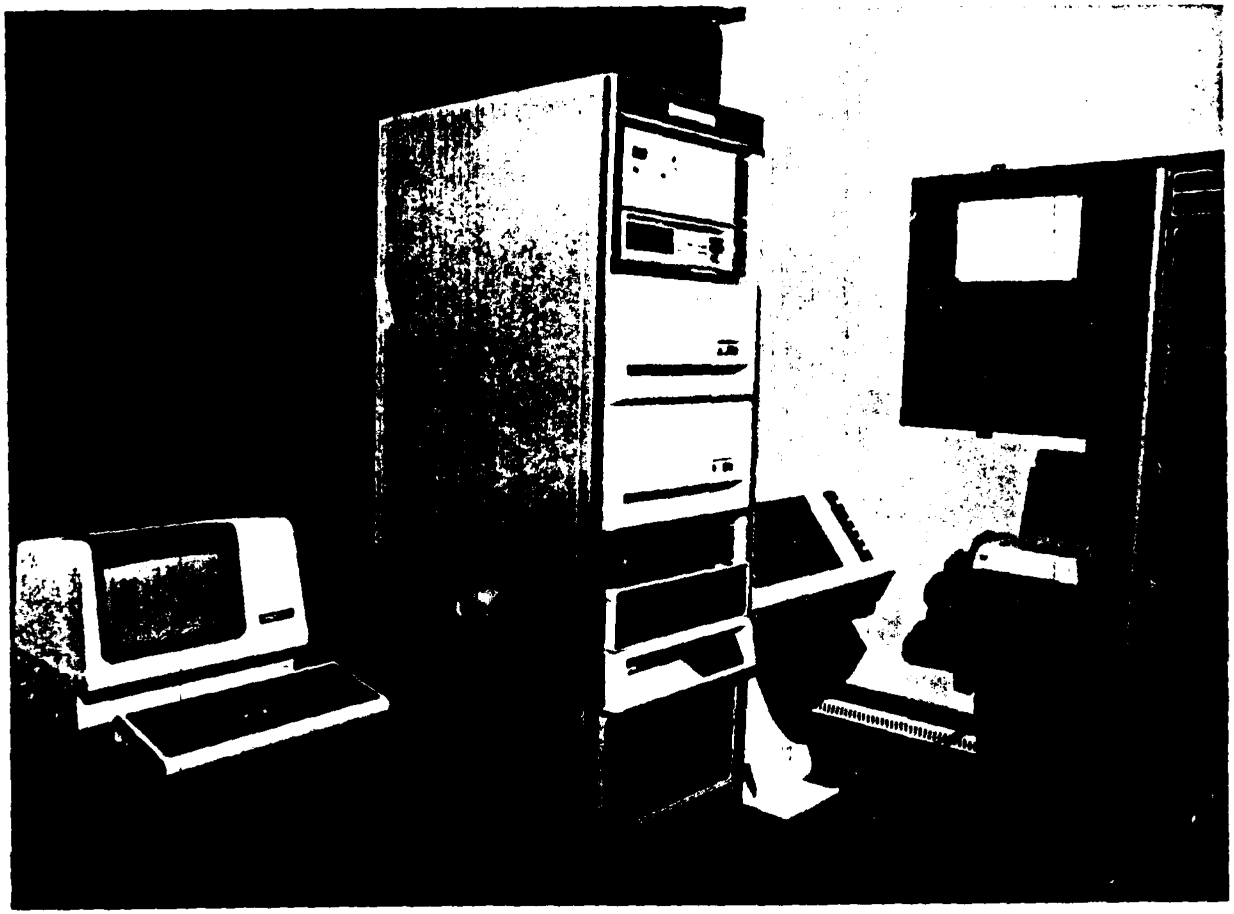

Figure 2. Digital Data Acquisition System

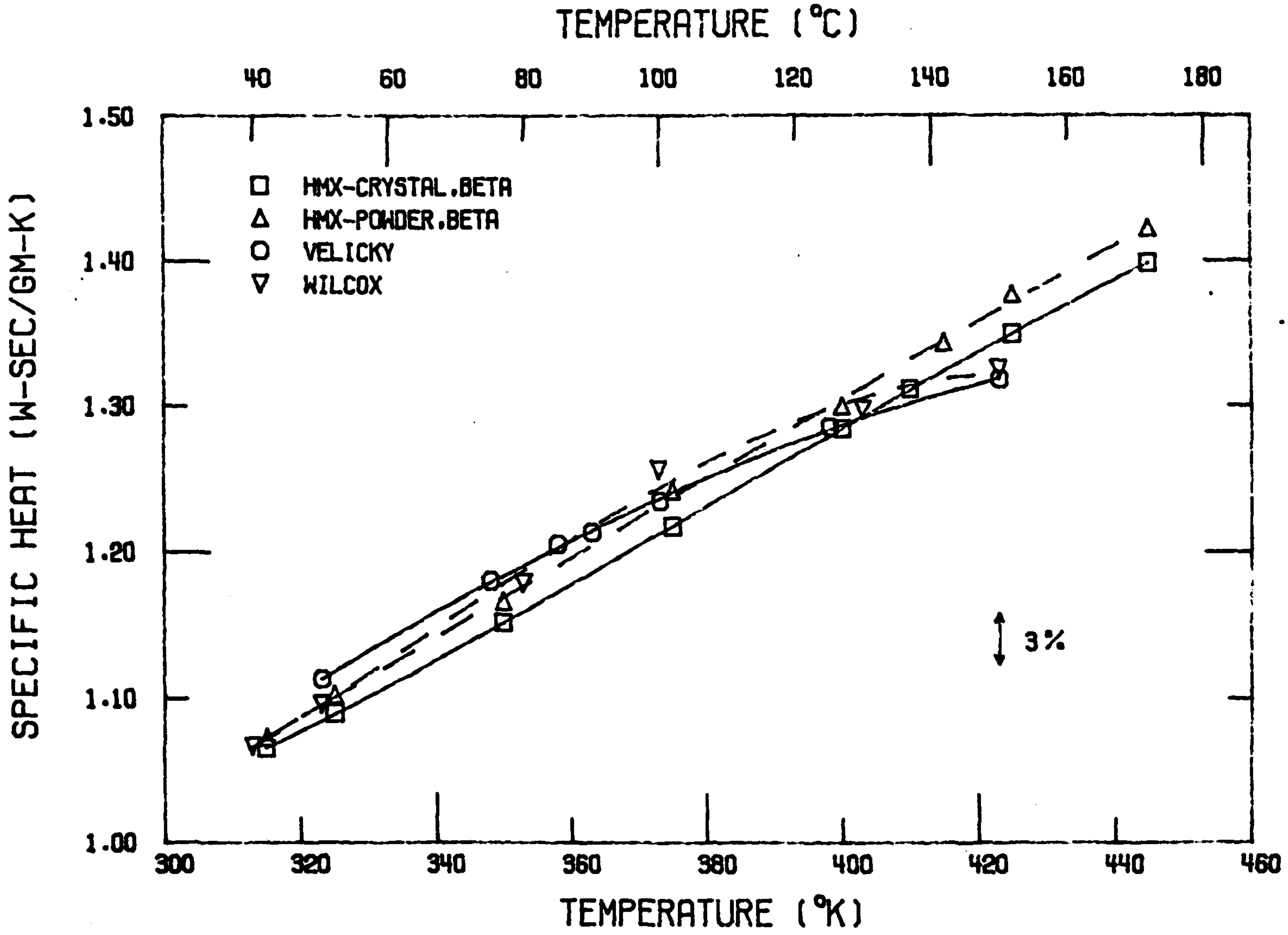

Figure 3. Specific Heat of Single Crystal and Powdered Blend β-HMX Against Literature Results

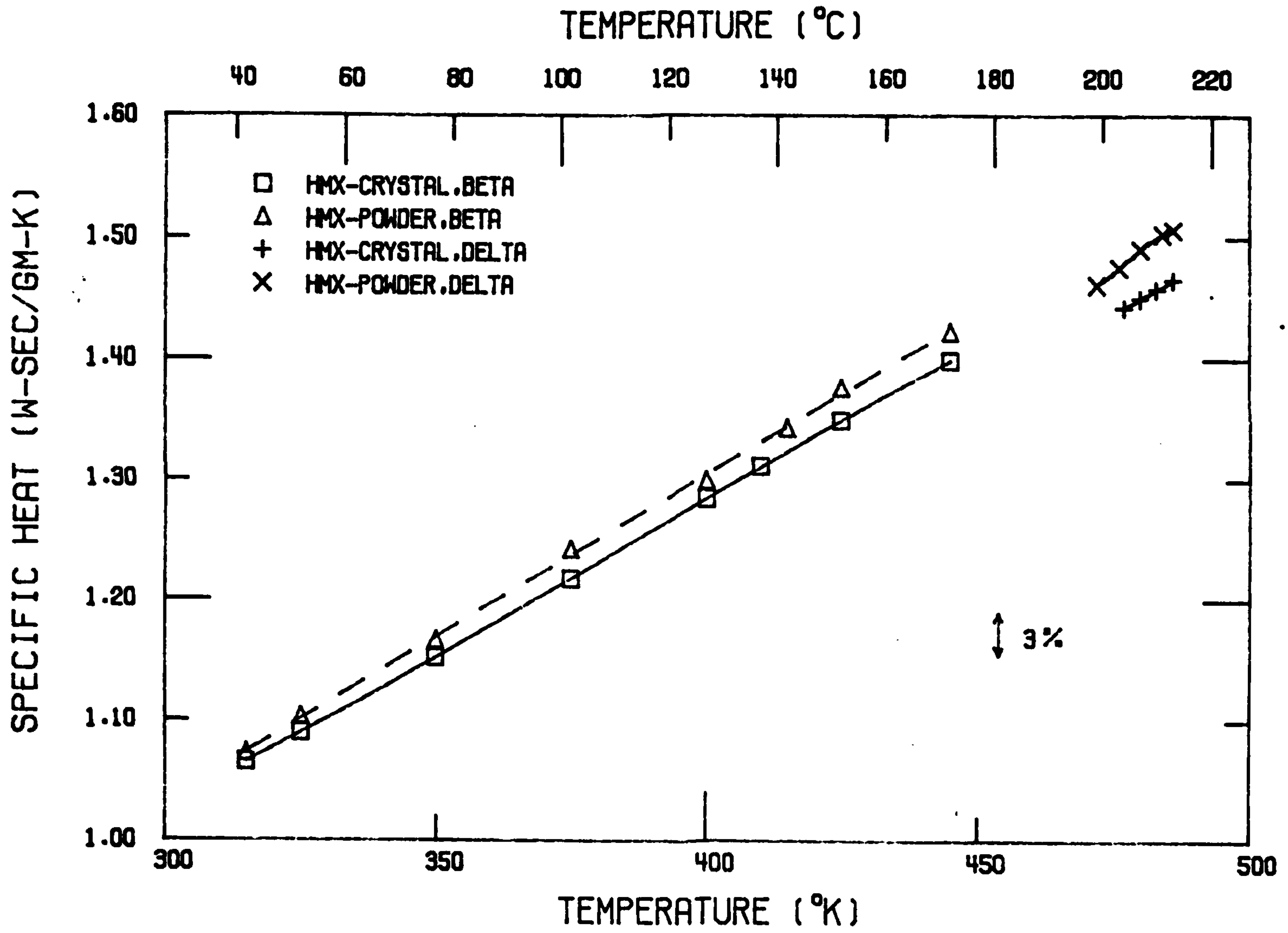

Figure 4. Specific Heat of Single Crystal and Bowdered Blend β and δ-HMX

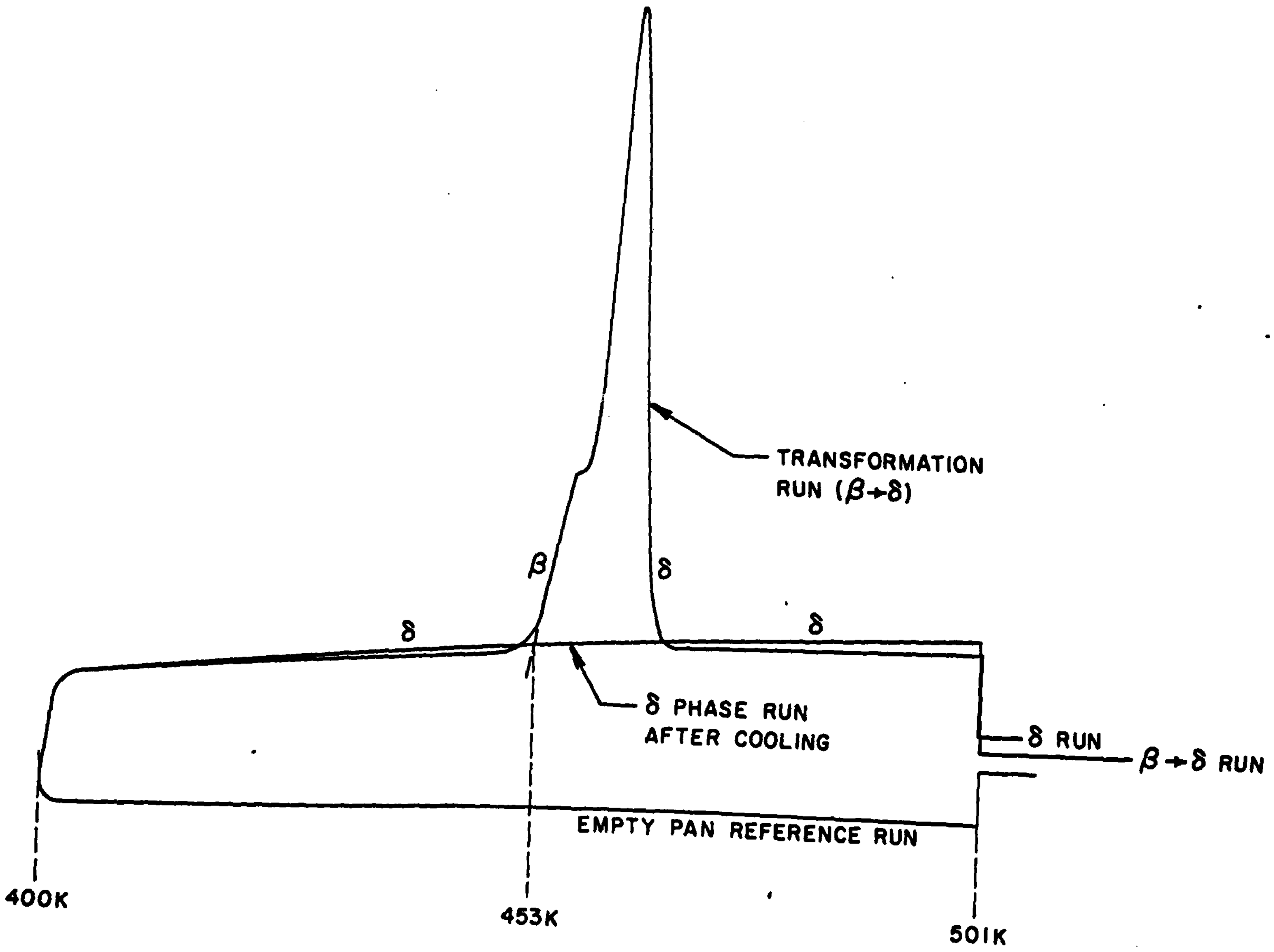

Figure 5. The DSC x-y Recorder Trace for Single Crystal HMX Through the β to δ Phase Transformation

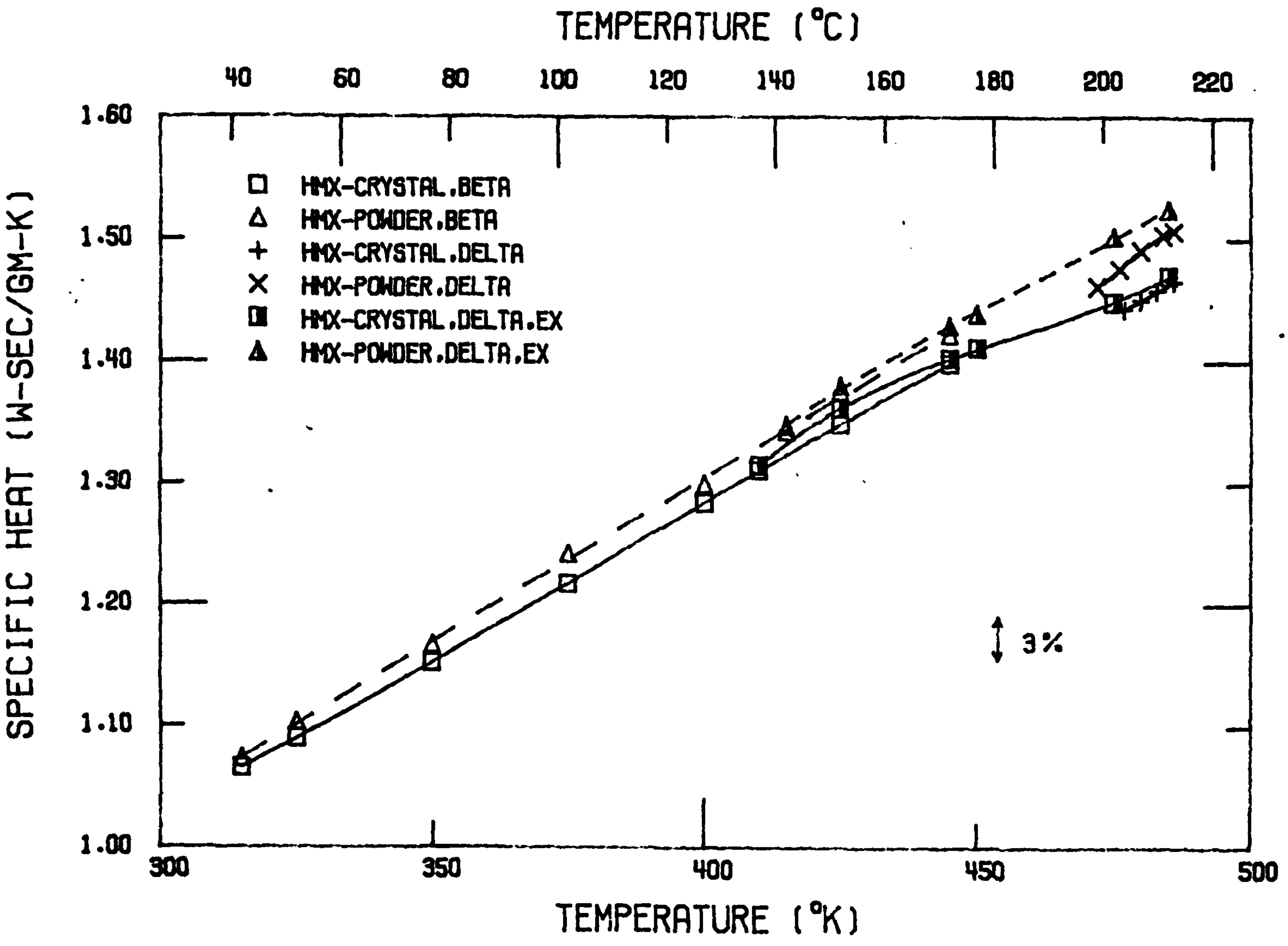

Figure 6. Specific Heat of Single Crystal and Powdered Blend β and δ-HMX Including the Extended δ Phase Run

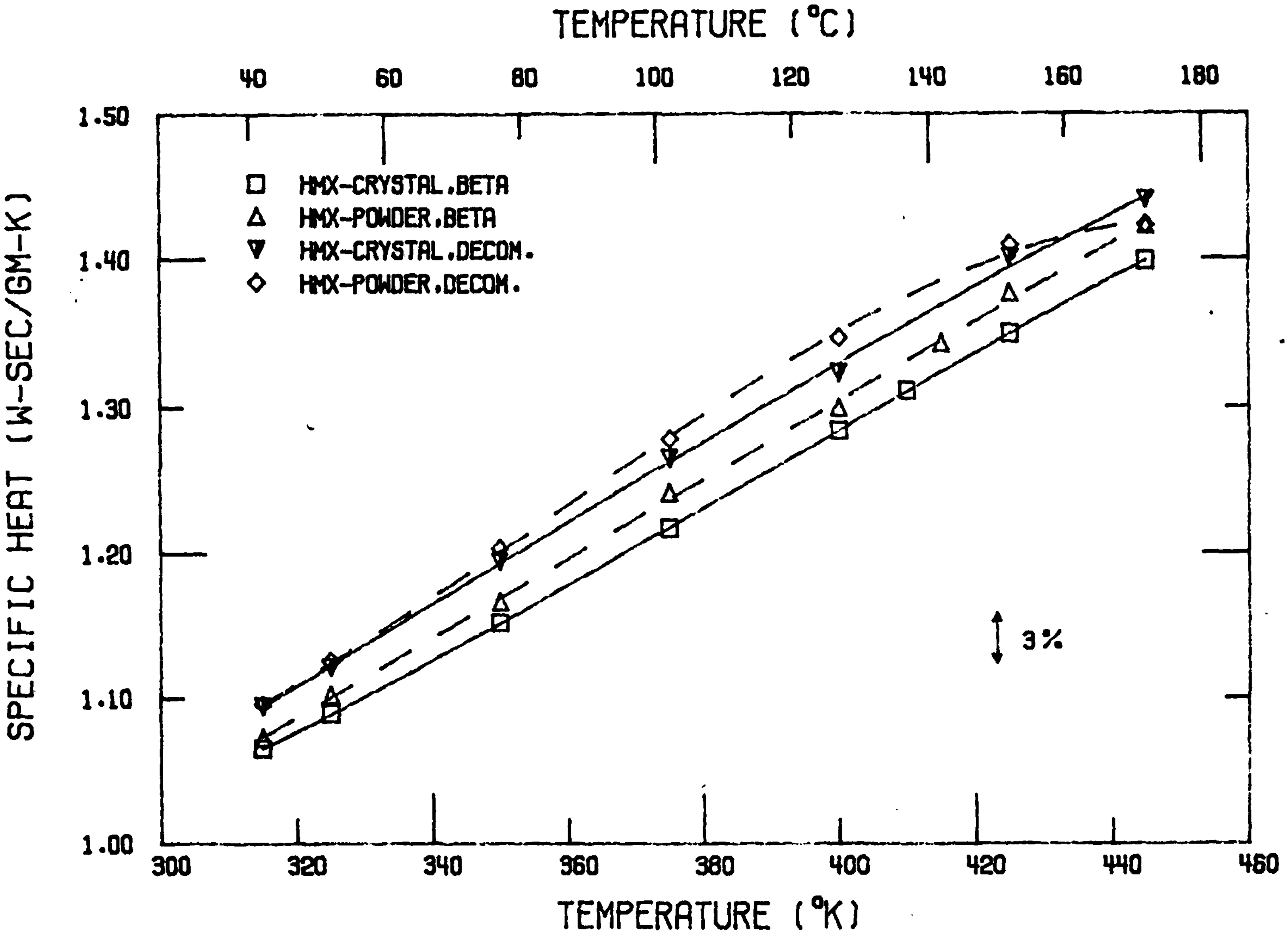

Figure 7. Specific Heat of Single Crystal and Powdered Blend β-HMX and Partially Decomposed HMX

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