

THERMAL CHARACTERISTICS OF DESENSITIZING WAXES FOR EXPLOSIVE COMPOSITIONS*

JOEL HARRIS

Picatinny Arsenal, Dover, N.J. (U.S.A.)

ABSTRACT

Waxes are added to explosive compositions to provide an explosive binder and lubricant for press-loaded explosives and to desensitize both press-loaded and cast-loaded explosives. The most significant thermal criterion of a good desensitizer is its ability to absorb large quantities of heat at above environmental temperatures. The enthalpy of each wax investigated for explosive incorporation was determined from room temperature to liquefaction by means of a Perkin-Elmer DSC-2. Correlation of enthalpy and desensitization was accomplished by relating the results of a velocity projectile impact test on explosives containing the investigated waxes.

Melt and solidification temperatures are also determined from DSC thermograms. The temperature at which a wax ceases to absorb large amounts of heat is the temperature at which liquefaction takes place. The temperature at which large quantities of heat are released during a cooling cycle is the solidification temperature. Use of wax having too low a liquefaction temperature results in excess exudation from a loaded high explosive charge; too high a liquefaction temperature inhibits incorporation of the wax into the molten explosive.

INTRODUCTION

Waxes have been used in cast 60/40 RDX/TNT (Composition B) since 1940. The primary purpose for the wax is the desensitization of the RDX part of the explosive. Most waxes used in Composition B are derived from petroleum sources. The waxes are mixtures of a variety of hydrocarbon materials. They are classified broadly into two categories, paraffin and microcrystalline waxes although large quantities of each may be found in each other.

Paraffin waxes are straight chain-saturated hydrocarbon with few branched chains. In the pure form they range from $C_{18}H_{38}$ melting point 28 °C to $C_{32}H_{66}$ melting point 71 °C. Their density is approximately 0.7 g ml^{-1} . On solidifying paraffins crystallize as relatively large plates or needles.

Microcrystalline waxes are long-chain hydrocarbons in which the branches

*Presented at the 6th North American Thermal Analysis Society Conference, Princeton, N.J., June 20-23, 1976.

consist of a variety of other hydrocarbons such as olefins, paraffins, and aromatics. They are characterized by higher molecular weights and higher viscosity than paraffin waxes. They crystallize in microcrystalline structure.

The mechanism as to how waxes desensitize is a subject of much controversy. It is apparent that there are several additive mechanisms of desensitization some of which are³:

1. The absorption of heat to prevent hot spot propagation.
2. The wax provides a less intimate contact of the particles of explosives with one another.
3. Burning wax releases hydrogen atoms which function as a slowing barrier.

The object of this investigation was to determine heat content and melt and solidification temperatures of commercial waxes by differential scanning calorimetry (DSC). Those commercial waxes which were tested either met or approached the requirements of MIL-W-20553 (ref. 2). Blending of waxes was also attempted.

EXPERIMENTAL PROCEDURE

A Perkin-Elmer DSC-2 of temperature range -40 to 725°C was calibrated for temperature and heat of fusion with indium, melting point 156.60°C , heat of fusion 6.79 cal g^{-1} . The temperature was further calibrated with water of 0°C , naphthalene of 80.5°C , and lead of 327.47°C . The heat of fusion was checked against stearic acid at 69.4°C and lead at 327.47°C . The calibration was accomplished at a heating rate of $10^{\circ}\text{C min}^{-1}$ in an argon atmosphere of flow-rate 20 ml min^{-1} . Heat content, liquefaction and solidification temperatures of the waxes listed in Table 1 were obtained with samples of wax of 6–9 mg at a range sensitivity (Y axis) of 5 mcal sec^{-1} . The heat of fusion was calculated by obtaining the area under the curve of each endotherm and comparing its value in square inches with the value obtained for the standard indium⁴.

DSC analysis was conducted from -20°C to 100°C at $10^{\circ}\text{C min}^{-1}$. By initiating the analysis below room temperature, a zero energy line can be established before the wax begins to absorb large amounts of energy. In order to control the crystallization process of the wax, the melted wax is cooled at $10^{\circ}\text{C min}^{-1}$. The sample is then reheated after a ten-minute delay. A reheat of the recrystallized wax at $10^{\circ}\text{C min}^{-1}$ creates a more uniform test condition than do samples which were only heated. The results obtained from the recrystallized wax DSC curve show only minor variation from the original heat DSC curve.

Liquefaction or melt temperatures in Table 1 were determined from DSC curves by obtaining the temperature at the beginning of the curve's straight line return from an endothermic peak to the baseline of the curve during a heating cycle. The DSC melt temperatures represent points where large amounts of heat cease to be absorbed since melting has taken place. In many cases, especially with slow heating rates of $2\frac{1}{2}^{\circ}\text{C min}^{-1}$, the DSC melt temperatures are in close agreement with those determined by visual observation (Table 1).

TABLE 1

THERMAL WAX ANALYSIS

Heating or cooling rate, 10 °C min⁻¹ unless notated; flow-rate, 20 ml min⁻¹ of argon.

Weight (mg)	Wax		Temp. range (°C)	Area (in. ²)	Liquefaction temp. (°C)	Solidification temp. (°C)	Heat content cal. g ⁻¹
(1)							
7.3	Sunoco 8810	heat	23-93	7.86	77		
7.3		cool	77-30	7.50		75	
7.3		reheat	27-91	7.80	78		
average				7.80	77	75	45.0
(2)							
8.3	Petrolite ES670	heat	46-90	7.06	63, 80		
8.3		cool	80-7	7.20		78	
8.3		reheat	28-91	7.14	61, 80		
average				7.10	62, 80	78	36.0
(3)							
7.7	Indramic 170C	heat	31-78	7.30	61, 83		
7.7		cool	79-13	6.94		75, 54	
7.7		reheat	15-86	7.39	59, 82		
average				7.34	60, 82	75, 54	40.4
(4)							
8.2	Petrolite ES672	heat	32-98	8.52	?		
		cool	92-11	8.30		90	
		reheat	26-98	8.45	89		
average				8.50	89	90	44.0
(5)							
8.0	Castor wax NFM	heat	36-91	5.49	88		
		cool	66-37	5.20		65	
8.0		reheat	46-98	5.40	87		
8.0		reheat	47-90	5.45	86		
average			5.45	87	65	29.0	
(6)							
7.6	Standard wax of Knoxville 123	heat	24-99	7.8	70		
7.6		cool	69-19	7.7		65	
7.6		reheat		7.9	69		
average				7.80	69	65	43.5
(7)							
8.5	Indramic 3000	heat	29-91	6.5	81		
		cool	19-3	6.5		76	
		reheat	19-90	6.7	80		
average				6.60	81		33.0

(Continued on p. 128)

TABLE 1 (continued)

THERMAL WAX ANALYSIS

Heating or cooling rate, $10^{\circ}\text{C min}^{-1}$ unless notated; flow-rate, 20 ml min^{-1} of argon.

Weight (mg)	Wax		Temp. range ($^{\circ}\text{C}$)	Area (in.^2)	Lique- fication temp. ($^{\circ}\text{C}$)	Solidi- fication temp. ($^{\circ}\text{C}$)	Heat content cal. g^{-1}
(8)							
7.6	Indramic X	heat	31-89	6.5	76		
7.6		cool	76-17	6.4		74	
7.6		reheat	17-87	6.8	72, 77		
average				6.50			36.0
(9)							
9.0	Ross refined Candellila	heat	31-78	7.38	68		
9.0		cool	66-17	7.11		62	
9.0		reheat	28-76	7.05	68		
average				7.30	68	62	34.0
(10)							
8.0	Western Mekon	heat	31-97	9.85	93		
8.0		cool	96-21	9.75		95	
8.0		reheat	27-98	10.00	89		
average				9.90	91	95	52.0
(11)							
8.0	Petrolite Ultraflex	heat	17-77	6.20	69		
8.0		cool	62-13	6.00		60	
8.0		reheat	21-75	5.85	65		
average				6.00	64	60	32.0
(12)							
8.3	25% Indramic 170C/X-75%	heat	27-82	7.60	62, 84		
8.3		cool	77-8	7.47		76	
8.3		reheat	20-88	7.70	61, 84		
average				7.60	61, 84	76	39.0
(13)							
7.1	75% Indramic 170C/X-25%	heat	30-89	7.40	63, 68		
7.1		cool	79-13	6.90		77, 55	
7.1		reheat	27-90	7.20	60, 85		
average				7.30	62, 85	77, 55	43.5
(14)							
7.6	Sunoco 985	heat	46-95	7.50	85		
7.6		cool	81-30	7.76		79	
7.6		reheat	55-92	6.93	83		
7.6		recool	81-49	7.34		80	
average				7.50	84	80	42.0

TABLE 1 (continued)

THERMAL WAX ANALYSIS

Heating or cooling rate, 10 °C min⁻¹ unless notated; flow-rate, 20 ml min⁻¹ of argon.

Weight (mg)	Wax		Temp. range (°C)	Area (in. ²)	Lique- fication temp. (°C)	Solidi- fication temp. (°C)	Heat content cal. g ⁻¹
(15)							
8.2	50% Indramic	heat	32-91	7.00	64, 84		
8.2	170C	cool	78-15	6.70		77, 53	
8.2	X-50%	reheat	24-89	6.93	61, 84		
average				7.00			36.0
(16)							
7.2	Indramic X-15%	heat	31-94	7.00	63, 86		
7.2	Indramic 170C	cool	80-14	6.50		80, 55	
7.2	85%	reheat	28-91	6.50	59, 86		
average				6.70			39.5
(17)							
7.9	Sunoco 1290	heat	21-93	8.10	70		
7.9		cool	76-24	7.20		74	
7.9		reheat	27-93	7.80	67, 73		
average				7.80		74	42.0
(18)							
7.9	Amoco (BLT -Eskar 65)	heat	27-82	8.00	76		
		cool	72-15	7.83		70	
		reheat	24-80	7.84	77		
average				7.90	77	70	42.0
(19)							
7.3	Bareco X715	heat	39-90	8.70	84		
7.3		cool	79-36	8.80		78	
7.3	Batch 110	reheat	30-87	8.60	84		
average				8.70	84	78	50.5
(20)							
7.2	Bareco X715	heat	33-89	8.20	83		
7.2	Batch 110	cool	86-30	8.40		79	
7.2		reheat	32-88	8.60	83		
average				8.40	83	79	50.0
(21)							
7.9	Bareco X715	heat	33-86	9.5	82		
7.9	74M0983	cool	80-27	9.2		77	
7.9		reheat	12-86	9.3	81		
average				9.3	82	77	51.0

(Continued on p. 130)

TABLE 1 (continued)

THERMAL WAX ANALYSIS

Heating or cooling rate, 10 °C min⁻¹ unless notated; flow-rate, 20 ml min⁻¹ of argon.

Weight (mg)	Wax		Temp. range (°C)	Area (in. ²)	Lique- fication temp. (°C)	Solidi- fication temp. (°C)	Heat content cal. g ⁻¹
(22)							
8.5	Bareco X715	heat	32-85	10.42	82		
8.5	74M0983	cool	78-15	10.86		77	
8.5		reheat	27-83	10.43	81		
average				10.5	82	77	52.0
(23)							
5.7	Knoxville 123	heat	22-89	10.3	69, 81		
2.5	Bareco X-715	cool	77-16	10.0		74	
		reheat	21-89	10.4			
		recool	78-14	10.1		74	
		reheat	18-89	10.4	71		
average				10.3	69, 81	74	53.0
(24)							
2.5	Knoxville 123	heat	18-86	11.0	No data		
6.2	Bareco X715	cool	78-15	10.1			
		reheat	70-90	10.5			
		recool	11-78	10.1			
		reheat	17-89	10.5			
average				10.5			54.0
(24)							
7.4	Bareco X715 1% stearic acid	heat	32-87	9.3	77		
		cool	81-14	4.0		77	
		reheat	28-85	9.0	81		
		recool	79-10	9.3		77.3	
		reheat	15-84	9.5	79		
average				9.3	79	77	53.0
(26)							
7.3	Bareco X715 w/5% biphenyl	heat	22-87	9.20	80		
		cool	78-5	8.87		77	
		reheat	17-84	8.85	77-82		
		reheat	26-85	8.69	79-82		
	2½ °C min ⁻¹	cool	78-12	8.50		78	
average				8.85	79, 82	82	51.0
(27)							
7.8	Emery 1733-85R	heat	32-98	7.30			
		cool	80-12	6.75		79, 69	
		reheat	30-96	6.00	77		
average				7.00	77	79, 69	38.0

TABLE I (continued)

THERMAL WAX ANALYSIS

Heating or cooling rate, 10 °C min⁻¹ unless notated; flow-rate, 20 ml min⁻¹ of argon.

Weight (mg)	Wax		Temp. range (°C)	Area (in. ²)	Lique- fication temp. (°C)	Solidi- fication temp. (°C)	Heat content cal. g ⁻¹
(28)							
7.7	Standard wax of Knoxville 123	heat	18-86	8.62	70		
		cool	71-60	8.50		69, 63	
		reheat	7-92	8.86	70, 75		
		recool	78-11	8.65		68, 64	
		reheat	12-86	8.87	70, 78		
	2½ °C min ⁻¹	cool	79-30	8.32		69, 65	
	2½ °C min ⁻¹	heat	22-91	8.48	67, 70, 77		
	average for 10 °C min ⁻¹			8.70	70, 77	69, 64	48.0
(29)							
7.6	Bareco X404	heat	25-88	10.00	88		
		cool	81-19	9.10		80	
		reheat	28-86	9.35	86		
		recool	81-23	8.60		80	
		reheat	23-95	9.40	86		
	2½ °C min ⁻¹	cool	83-22			82	
	average			9.89	87	81	55.5
(30)							
7.4	Bareco X404 Batch 110	heat	30-96	10.55	89		
		cool	81-19	9.24		80	
		reheat	25-93	9.80	86		
	2½ °C min ⁻¹	82-24	10.08		88	82	
	average			9.72	87	81	54.0
(31)							
7.2	Biphenyl	reheat	65-77	5.00	69		29.5
		cool	50-45			50	
(32)							
7.6	Bareco X715 10% biphenyl	heat	26-84	9.78	78-81		
		cool	75-10	8.83		74	
		reheat	25-84	9.73	76-80		
		cool at 2½ °C min ⁻¹				75.5	
	average			9.75			54.0
(33)							
8.3	50% Knoxville 123	heat	26-88	9.01	76-81		
		cool	78-10	8.91		74	
	50% Bareco X715	reheat	19-89	9.04		75.6	
	2½ °C min ⁻¹	cool				75.6	
	average			9.00			46.0

(Continued on p. 132)

TABLE I (continued)

THERMAL WAX ANALYSIS

Heating or cooling rate, 10 °C min⁻¹ unless notated; flow-rate, 20 ml min⁻¹ of argon.

Weight (mg)	Wax		Temp. range (°C)	Area (in. ²)	Lique- fication temp. (°C)	Solidi- fication temp. (°C)	Heat content cal. g ⁻¹
(34) 7.25	40% Knoxville 123	heat	21-90	8.87	80		
	60% Bareco X715	cool	83-17	8.40		77	
		reheat	35-89	8.45	79		
	2½ °C min ⁻¹	recool	80-20	8.40		78.3	
	average			8.60			50.2
(35) 8.5	Bareco X715 10% biphenyl	heat	12-86	10.55	77		
		cool	75-5	9.35		75	
		reheat	17-86	10.35	79		
	2½ °C min ⁻¹	recool	77-9	9.76		77	
	average			10.0		76	50.0
(36) 5.9	Amoco Eskar SW-70	cool	75-44	6.77		74	
		reheat	47-82	6.71	79		
		recool	75-36	7.10		74	
	average			6.86	79	74	49.0
(37) 7.5	Ross Candellila	heat	35-76	6.60	68		
		cool	62-22	6.41		62	
		reheat	33-77	6.43	66		
	average			6.50	67	62	37.0
(38) 7.0	5% Bareco X404 in Bareco X715	heat	17-86	9.72	80		
		cool	77-14	8.90		77	
		reheat	9-85	9.82	81		
		cool	78-15	9.00		77	
		reheat	22-86	9.02	80		
	2½ °C min ⁻¹		79-17	8.70		79	
	average			9.50	80	78	57.5
(39) 7.6	Ross wax 561114	heat	28-93	9.00	66, 81		
		cool	79-15	8.90		76, 65	
		reheat	24-90	8.90	67, 80		
	2½ °C min ⁻¹	cool				78, 67	
	average			8.95	67, 81	77, 66	50.0

TABLE I (continued)

THERMAL WAX ANALYSIS

Heating or cooling rate, 10 °C min⁻¹ unless notated; flow-rate, 20 ml min⁻¹ of argon.

Weight (mg)	Wax		Temp. range (°C)	Area (in. ²)	Lique- fication temp. (°C)	Solidi- fication temp. (°C)	Heat content cal. g ⁻¹
(40) 8.0	Ross wax 56-1034	reheat	23-98	8.70	68		
		cool	79-14	8.82		66	
		heat	23-98	9.10	68		
	2½ °C min ⁻¹				68		
average				8.90	68	66	47.0
(41) 8.1	Ross wax 56-1204	cool	76-27	9.00		65	
		reheat	28-73	9.00	69.5		
	2½ °C min ⁻¹					66	
average				9.00	69.5	66	47.0
(42) 8.6	Indramic GLC 69053	heat	29-94	7.76	76		
		cool	81-12	7.50		80	
		reheat	22-73	7.50	82		
	2½ °C min ⁻¹	cool	83-22	7.04		82	
average				7.60			37.50
(43) 9.3	Indramic KPL 6906	reheat	27-90	8.44	73		
		recool	80-18	8.32		73	
	2½ °C min ⁻¹	reheat	23-88	8.60	72		
		recool	79-24	8.40		73	
average				8.40	72	73	38.0
(44) 4.6 3.9	Bareco X715 Ross wax 56114	heat	27-88	10.4	80		
		cool	78-15	9.69		77	
	2½ °C min ⁻¹	reheat	18-88	10.2	81		
						78	
average				10.0			50.0
(45) 6.6	Bareco X715 (MWS00) Batch 652		82-27	8.26		81	
			24-88	8.44	81, 85		
			81-28	8.16		80	
			24-88	8.51	81, 85		
	2½ °C min ^{-1a}		83-22	8.00		82	
	2½ °C min ^{-1a}		82-34	8.00		82	
average				8.34	81, 85	81	53.5

(Continued on p. 134)

TABLE I (continued)

THERMAL WAX ANALYSIS

Heating or cooling rate, $10^{\circ}\text{C min}^{-1}$ unless notated; flow-rate, 20 ml min^{-1} of argon.

Weight (mg)	Wax	Temp. range ($^{\circ}\text{C}$)	Area (in. ²)	Lique- fication temp. ($^{\circ}\text{C}$)	Solidi- fication temp. ($^{\circ}\text{C}$)	Heat content cal. g ⁻¹
(46) 8.0	Bareco X718 (MW400) Batch 649	76-81 73-18 25-80 73-13 25-79 74-27	9.22 9.19 9.60 9.40 9.70 8.40	72, 74 72 72 72	 72 72, 68 73, 71	
	$2\frac{1}{2}^{\circ}\text{C min}^{-1a}$					
	average		9.42	72	72	50.0
(47) 6.8	Bareco X719 Batch 658	55-108 98-58 49-108 98-56 49-108 99-58	9.59 9.36 9.20 9.35 9.40	103 102 102	 95 97 98	
	$2\frac{1}{2}^{\circ}\text{C min}^{-1a}$					
	average		9.38	102	97	58.4
(48) 5.8	Bareco X717 Batch 655	44-101 40-101 94-44 39-101 93-52	8.28 8.07 8.24 8.25 8.30	98 97 97	 93 93	
	$2\frac{1}{2}^{\circ}\text{C min}^{-1a}$					
	average		8.21	97	93	54.5
(49) 6.5	Bareco X900	29-91 25-92 87-26 83-35	8.95 8.50 8.00 7.76	85 81, 85	 80 82	
	$2\frac{1}{2}^{\circ}\text{C min}^{-1a}$					
	average		8.45	85	81	50.0
(50)	Bareco X902	28-94 83-21 23-94 85-35	7.90 7.50 9.43 7.04	87	 82 84	
	$2\frac{1}{2}^{\circ}\text{C min}^{-1a}$					
	average		7.61	87	83	48.5

TABLE 1 (continued)

THERMAL WAX ANALYSIS

Heating or cooling rate, $10^{\circ}\text{C min}^{-1}$ unless notated; flow-rate, 20 ml min^{-1} of argon.

Weight (mg)	Wax	Temp. range ($^{\circ}\text{C}$)	Area (in. ²)	Lique- fication temp. ($^{\circ}\text{C}$)	Solidi- fication temp. ($^{\circ}\text{C}$)	Heat content cal. g ⁻¹
(51) 6.5	Barecco X901	20-93	8.60	85		
		82-26	7.92		81	
		21-91	8.12	82, 86		
		82-26	7.95		81	
	$2\frac{1}{2}^{\circ}\text{C min}^{-1}$ ^a	84-32	8.40		83	
	average		8.15	86	82	48.0
(52) 3.8	Barecco X718	79-21	8.60		79, 73	
3.1	Barecco X715	27-86	8.97	79		
		80-21	8.69		79, 77	
		22-86	8.79	79		
	$2\frac{1}{2}^{\circ}\text{C min}^{-1}$ ^a	80-29	8.56		79, 76	
	average		8.76	79	79, 76	48.5

^a Area not averaged in total.

Solidification temperatures in Table 1 were obtained during a cooling cycle from the temperature at the top of a straight line departure from the baseline when heat was released. The release of heat manifests itself by the solidification of the wax. Temperatures obtained are in close agreement with solidification temperatures observed in the test tubes (Table 1). DSC was normally run at $10^{\circ}\text{C min}^{-1}$; a slower cooling rate of $2\frac{1}{2}^{\circ}\text{C min}^{-1}$, however, results in solidification temperatures which are more accurate and closer to those obtained by observation of the thermal behavior of the wax in an oil bath.

The data appearing in Table 2 was obtained by observing the physical state of the waxes in 16 mm O.D. by 150 mm length test tubes placed in a temperature-controlled silicone oil bath. While the oil is constantly stirred, the temperature is raised 1°C every five minutes for liquefaction measurement and decreased 1°C every ten minutes for solidification measurement. Liquefaction temperatures are reported as the temperature at which the larger part of the solid wax has liquefied. The solidification temperature is reported as the temperature at which a large amount of precipitate appears. The change of state temperatures of the waxes are obtained by means of thermometers placed near each test tube. The greatest temperature gradient between thermometers is $1/2^{\circ}\text{C}$ since the oil is constantly stirred.

The waxes were tested as submitted by the manufacturers. The manufac-

TABLE 2

TEMPERATURES OF VISUALLY OBSERVED PHASE CHANGES

<i>Ingredient</i>	<i>Heating (°C) liquefaction</i>	<i>Cooling (°C) solidification</i>
1. Sunoco 8810 } TNT/RDX/HNS }	No test	74-72.5 77.5-75
2. TNT 1 HNS	82	79
3. Wax of Knoxville 123/TNT	82	69-7
4. 10% Bareco X715 in TNT 1% Santowax AB-15	80-85	79
5. 15% Bareco in X715 in TNT 2% Holowax 107	80-85	79.5
6. 10% Holowax 105 in X715	79-82	77
7. 10% Santowax AB15 in X75	71-105	79
8. Wax of Knoxville 123	70-76	69
9. X715 Bareco 10% biphenyl	78	77.5
10. X715 Bareco 5% biphenyl	79	79-78.5
11. X715 Bareco 2% biphenyl	79.5	79.5-78.5
12. X715 Bareco 1% biphenyl	80	80-79
13. X715 Bareco 60/Knoxville 40%	79.5	77.5-75
14. 50% Bareco/50% Knoxville	79.5	77-76
15. 80% Bareco/20% Knoxville	81.0	79
16. Bareco X715	81.0	79
17. 85% X715/15% Knoxville	No test	78
18. 90% Bareco X715/10% Knoxville	No test	79
19. 95% Bareco X715/5% Knoxville	No test	80
20. 98% Bareco X715/2% Knoxville	No test	80
21. 50% Bareco X715/Castorwax NFM	81	86, 81
22. Bareco X715/stearic acid 1%	80	80
23. Bareco X715	80	
24. Bareco X715/Knoxville 50/50	78-82	77-75
25. Knoxville/10% Mekon	73-81	80
26. Rosswax 561114	79	78-71
27. TNT +0.1 HNS	83	78-77
28. Rosswax 561114/0.4487 g Bareco X715 0.4560 g	No test	78
29. Rosswax 561204 0.7 g Bareco X715 0.7 g	82	78
30. Rosswax 56-1034 0.6 g Bareco X715 0.6 g	82	79
31. Rosswax 56-1034	Mush till 79	68
31a. Rosswax 56-1204	Mush till 79	68
32. TNT 0.86 g X715 0.16 g Diphenyl 0.02 g HNS 0.01 g	81	76
33. Ross wax 20% Bareco X715 80%	81	79
34. TNT	82	73
35. X715+Polyethylene AC 400	80	82
36. Sunoco 8810+RDX+TNT	82	77
37. Sunoco 8810	80	78-77
38. Biphenyl in X715+TNT+RDX	83	77
39. 2% Bareco X404 in X715 RDX+TNT	82	79.5-78.5

TABLE 2 (continued)

TEMPERATURES OF VISUALLY OBSERVED PHASE CHANGES

Ingredient	Heating (°C) liquefaction	Cooling (°C) solidification
40. 86% TNT biphenyl 2.0 16% X715 HNS 0.1	78	72 77.5
41. TNT+HNS		73 74
42. Polyethylene 400+X715 Bareco	No test	90-82
43. Biphenyl X715 RDX/TNT/HNS	No test	78-74 70-70
44. 95% Bareco X715 5% stearic acid	78-81	80
45. 90% Bareco 10% Ultraflex	80-82	80
46. 90% Bareco 10% Sunoco 1290	80-81	80

turer or the trade name is listed with the wax in Table 1. Bareco waxes were obtained from the Petrolite Corporation.

RESULTS

The results of thermal analysis by means of a Perkin-Elmer DSC-2 are shown in Table 1. Thermal analysis was conducted at a heating rate of $10^{\circ}\text{C min}^{-1}$ and, for more accuracy, at $2\frac{1}{2}^{\circ}\text{C min}^{-1}$. The DSC was calibrated

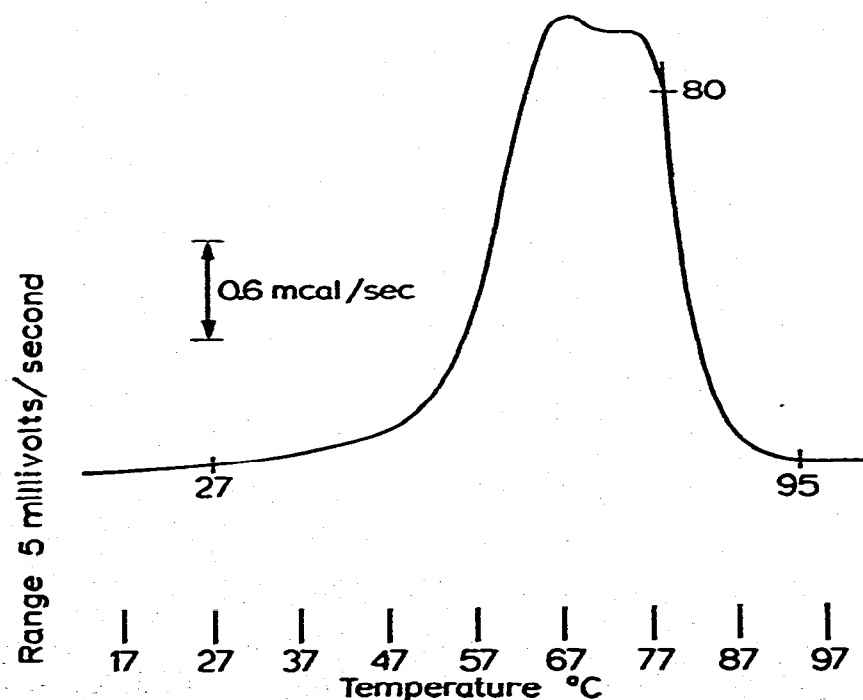


Fig. 1. 7.3 mg Sunoco 8810 heating curve. Heating rate, $10^{\circ}\text{C min}^{-1}$; flow-rate, 20 ml min^{-1} of argon; record speed, 20 mm min^{-1} .

TABLE 3

PROJECTILE IMPACT TEST

fps = feet per second; melt T = temperature when all wax is liquid; solid T = solidification temperature of a wax; cal = calories; g = grams.

% Wax in Composition B	Velocity (fps)		ΔH of wax (cal g ⁻¹)	Melt T (°C)	Solid T (°C)
	Go	No Go			
0	3080	2900	—	—	—
1 Bareco X715	3310	3130	50	82	77
1 Indramic 170C	3280	3070	40	60, 82	75, 54
1 Sunoco 8810	3110	2900	45	77	75
1 Castorwax NFM	2950	2900	29	87	65
1 Petrolite ES670	2800	2570	36	80	78

for both temperature and energy equivalence, using pure indium (m.p. 156.60 °C; heat of fusion 6.80 cal g⁻¹) and pure lead (m.p. 327.47 °C, heat of fusion 5.50 cal g⁻¹). The temperature was also checked with naphthalene (m.p. 80.5 °C). Temperature accuracy was always within ± 0.2 °C.

Confirming temperature analysis was conducted on some of the more promising waxes and wax mixtures in a thermostatically-controlled, silicone oil bath which was constantly stirred. These results, obtained mostly at a heating rate of 1 °C per five minutes and a cooling rate of 1 °C per ten minutes, are reported in Table 2. The waxes were put in test tubes, capped and placed in the oil bath. Solidification of blends of waxes stretches over ranges of temperature. Beginning and final temperatures of solidification of wax blends are reported in Table 2.

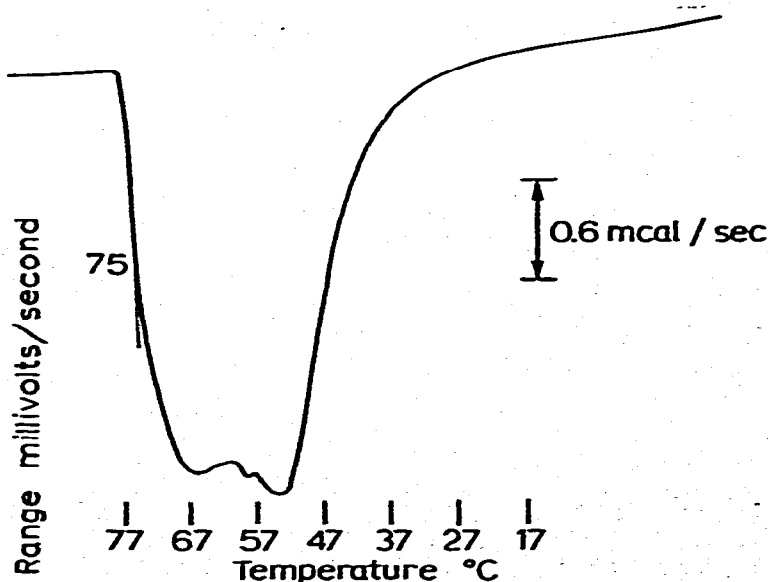


Fig. 2. 7.3 mg Sunoco 8810 cooling curve. Cooling rate, 10 °C min⁻¹; flow-rate, 20 ml min⁻¹ of argon; record speed, 20 mm min⁻¹.

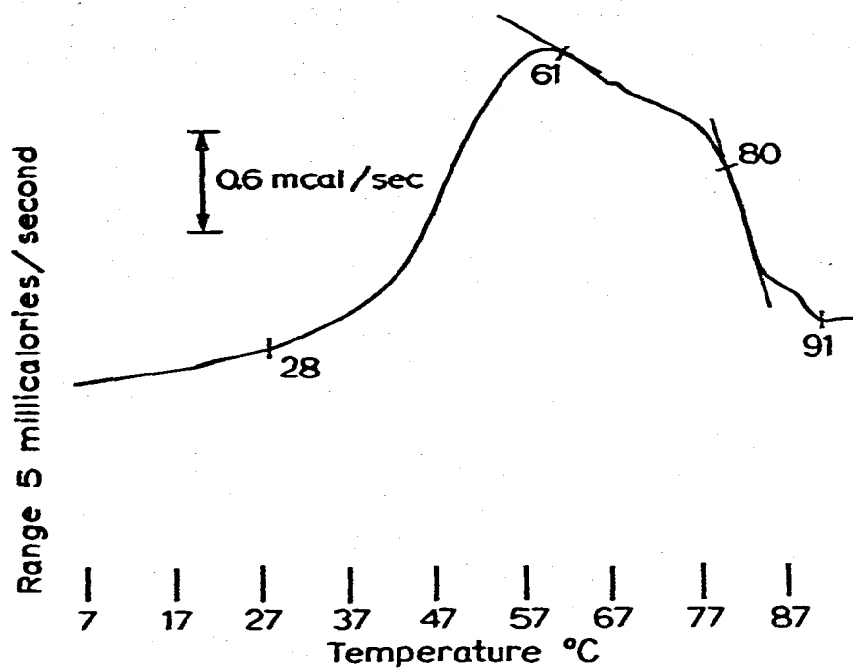


Fig. 3. 9.3 mg Petrolite ES670 heating curve. Heating rate, $10^{\circ}\text{C min}^{-1}$; flow-rate, 20 ml min^{-1} of argon; record speed, 20 mm min^{-1} .

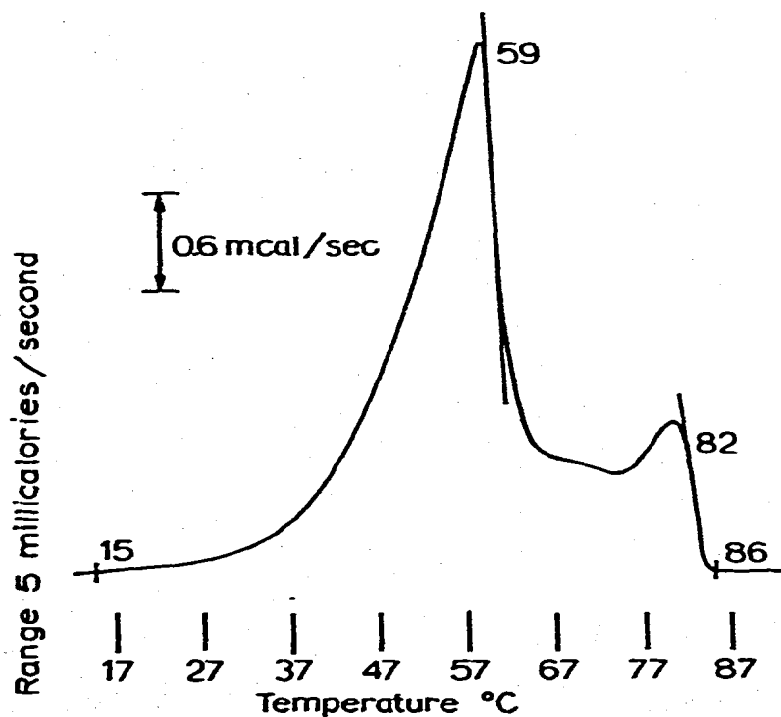


Fig. 4. 7.7 mg Indramic 170C heating curve. Heating rate, $10^{\circ}\text{C min}^{-1}$; flow-rate, 20 ml min^{-1} ; record speed, 20 mm min^{-1} .

Figures 1-10 are thermal heating curves of the waxes appearing in Table 1.

Results from a projectile impact test from some published and unpublished results are listed in Table 3 (ref. 3).

DISCUSSION OF RESULTS

Sunoco Wax 8810 was one of the desensitizer waxes used in many explosive compositions for over 15 years. A discontinuance of its production resulted in research to find a substitute wax. The thermal characteristics of the ideal replacement would be similar in changes of state temperatures and have a similar or greater heat content (Table 2, No. 1; Figs. 1 and 2).

One synthetic wax, Bareco Wax X715, comes closest to possessing all the desirable thermal characteristics and develops a greater heat content than does Sunoco 8810. (Table 1, No. 19-22; Figs. 8 and 9). Solidification temperature, however, is slightly higher. Some wax solidification occurred before the molten explosive solidified. Most additives to the Bareco Wax failed to lower the change of state temperature and while biphenyl additive lowered the change of state temperatures of the wax, it also lowered the change of state temperature of TNT.

Since the Sunoco 8810 is a blend of Sunoco 985 (Table 1, No. 14; Fig. 6) and Sunoco 1290 (Table 1, No. 17; Fig. 7) blending of other waxes was attempted.

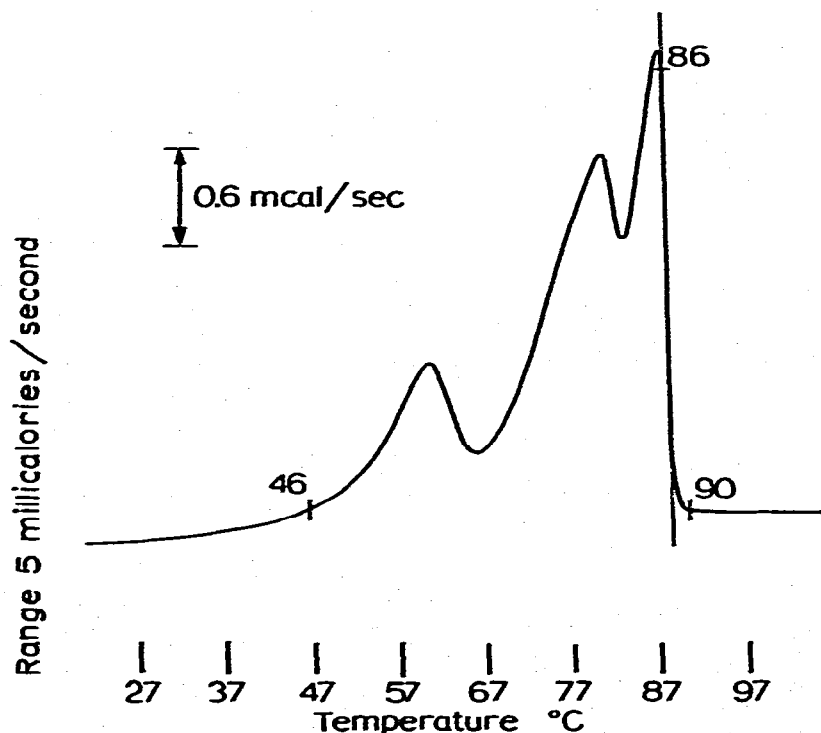


Fig. 5. 8.0 mg Castorwax NFM heating curve. Heating rate, $10^{\circ}\text{C min}^{-1}$; flow-rate, 20 ml min^{-1} of argon; record speed, 20 mm min^{-1} .

One seemingly successful mix results from a blend of Bareco X715 and Standard Wax of Knoxville 123, which possesses an acceptable heat of fusion but a change of state temperature which is unacceptable (Table 1, No. 28). Blends of 40–50% Knoxville Wax 123 and 50–60% Bareco X715 provide satisfactory thermal characteristics (Table 1). Another wax which possesses the desired properties is Amoco Eskar SW-70 (Table 1, No. 36). This wax, described in ref. 1, is not available, however, in large amounts. Rosswax 561114, an ozokerite wax, was promising in that it has a large heat of fusion, but it was found to possess a high percentage of low-melting components which could lead to exudation (Table 1, No. 40) and did not mix with Barexo X715.

Sunoco 1290 wax appeared to have a melting point at 70°C when initially heated which would be too low to prevent exudation in storage. After the wax was cooled at 10°C min⁻¹ and reheated, it showed a melting point at 73°C which would be acceptable. It solidified at 74°C which is desirable and compares favorably with the Amoco SW-70 used by the Navy. The curve for 1290 wax shows a preliminary melting at 67°C, but it has been found, particularly with Indramic

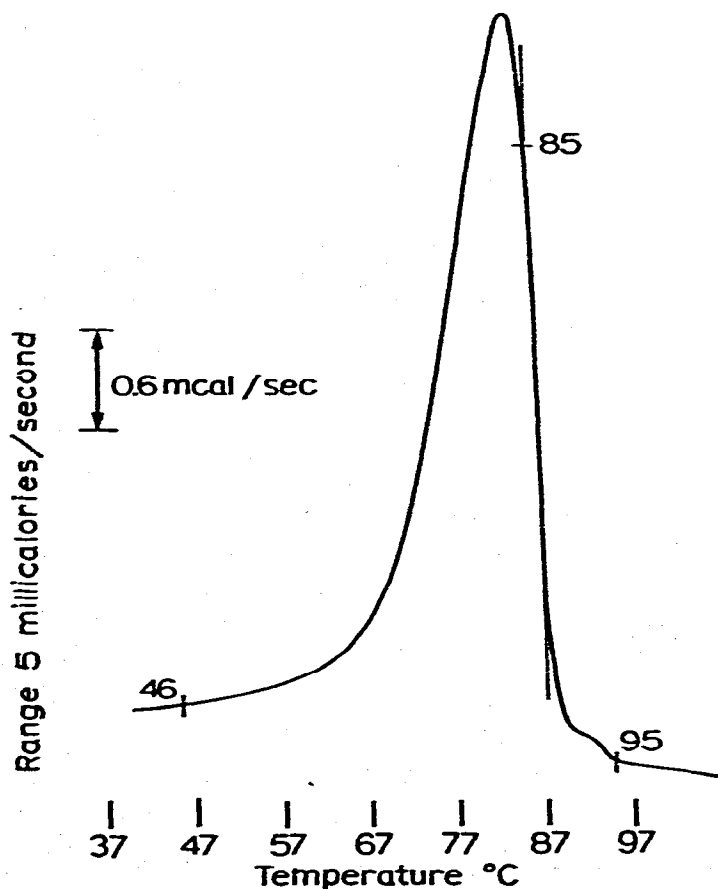


Fig. 6. 7.6 mg Sunoco 985 heating curve. Heating rate, 10°C min⁻¹; flow-rate 20 ml min⁻¹ of argon; recorder speed 20 ml min⁻¹.

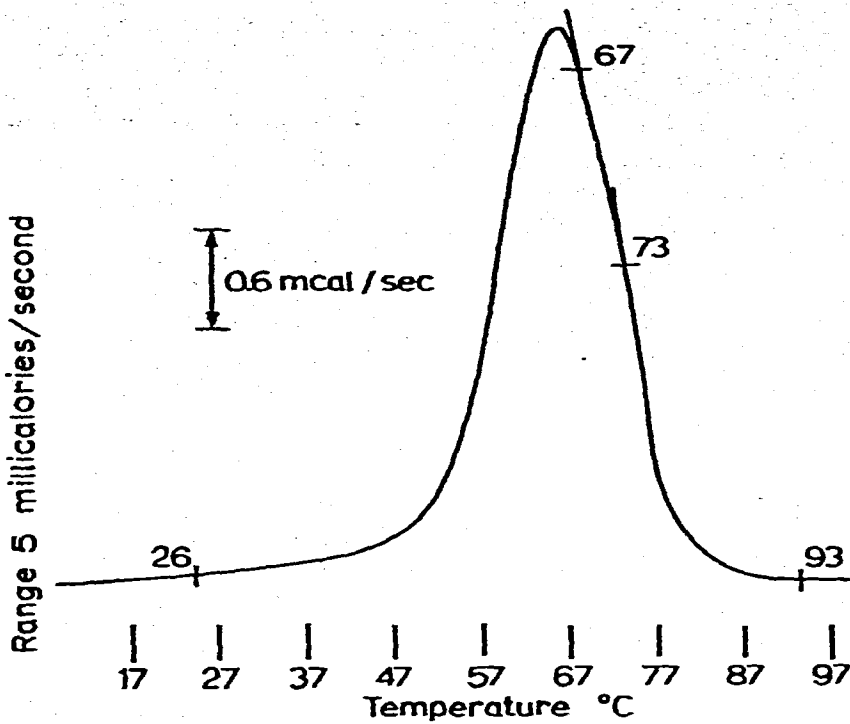


Fig. 7. 7.9 mg Sunoco 1290 heating curve. Heating rate, $10^{\circ}\text{C min}^{-1}$; flow-rate, 20 ml min^{-1} of argon; record speed, 20 mm min^{-1} .

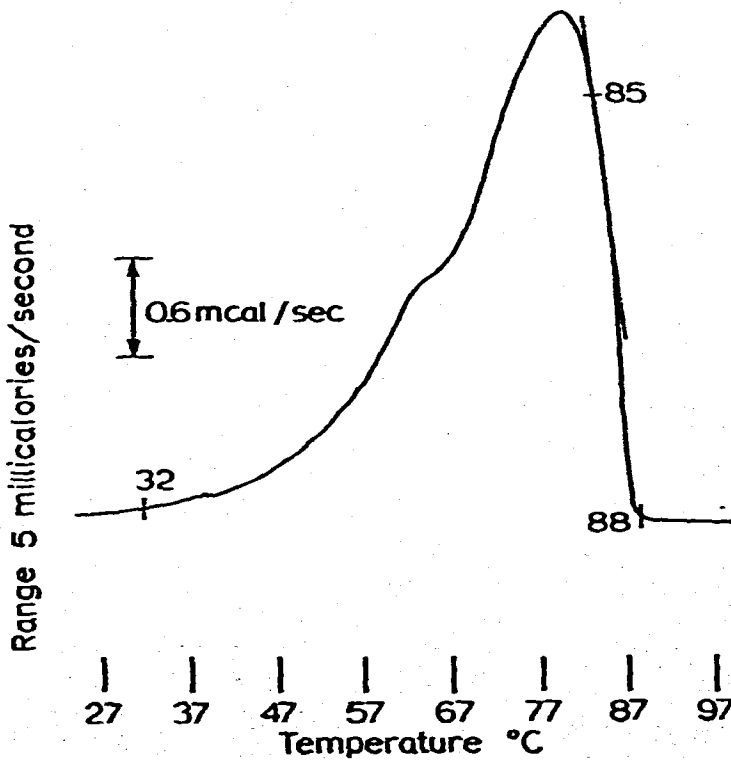


Fig. 8. 7.2 mg Barco X715, batch 110 heating curve. Heating rate, $10^{\circ}\text{C min}^{-1}$; flow-rate, 20 ml min^{-1} ; record speed, 20 mm min^{-1} .

Wax 170 °C, that the higher melting component in this case 73 °C (Fig. 7) can encapsulate the already soft or liquid component. The encapsulation by the higher melting component would tend to preclude exudation of wax at 70 °C storage condition from an explosive composition.

Waxes 45–52 (Table 1) are synthetics. Waxes X715, X717, X718, X719 are straight-chain paraffinic waxes synthesized by the Petrolite Corporation. No. 52 represents an attempt to mix X715, which may solidify at a temperature slightly too high, and X718 (Table 1, No. 46) which is decidedly too low in temperature of solidification. The results of mixing (Table 1, No. 52) seem encouraging from the point of view of wax compatibility and temperature of solidification.

To ascertain whether DSC reported changes of state temperatures are accurate, samples of wax were weighed and placed in test tubes in a temperature-controlled, stirred oil bath. Pilot plant conditions were simulated in the oil bath, and changes of state visually observed. Data obtained from these experiments appears in Table 2. The oil bath was subsequently utilized to observe the behavior of RDX, TNT, and wax during cycling.

Visual observation of the solidification of wax and explosive in test tubes indicates that separation of wax and explosive occurs on solidification. If a wax solidified at nearly the same temperature as the TNT, a mechanical mixture of

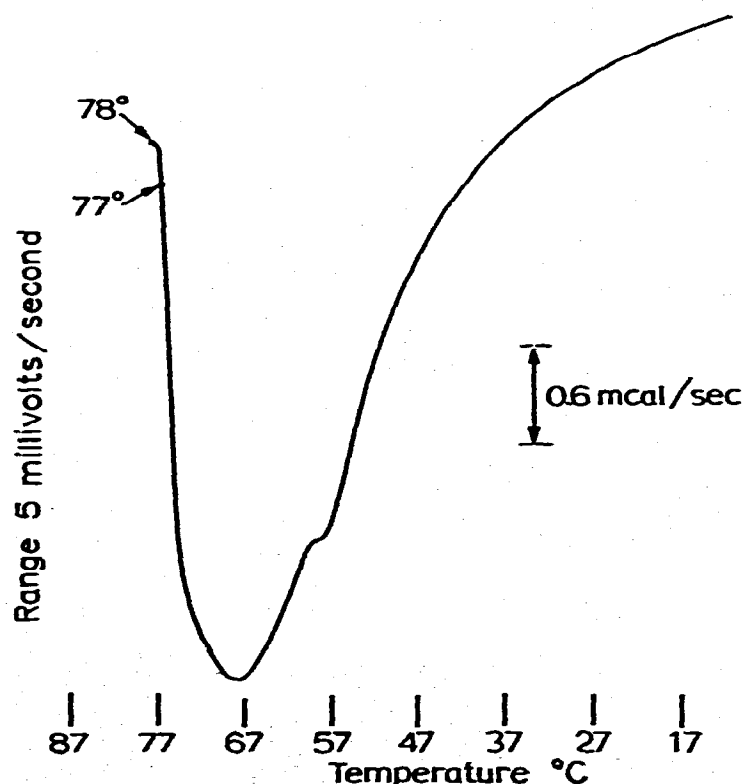


Fig. 9. 8.5 mg Bareco X715 cooling curve. Cooling rate $10\text{ }^{\circ}\text{C min}^{-1}$; flow-rate, 20 ml min^{-1} of argon; range 5 mV sec^{-1} ; record speed, 20 mm min^{-1} .

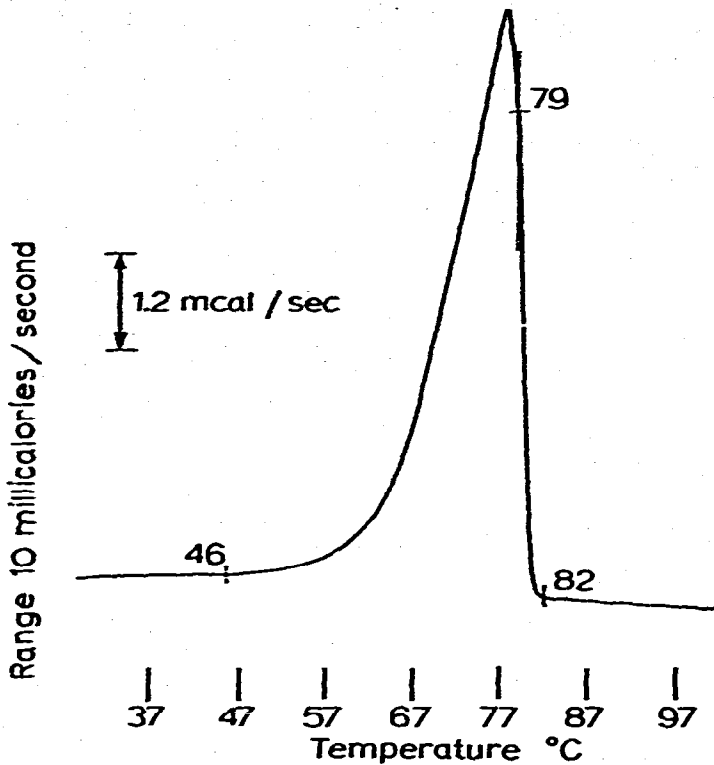


Fig. 10. 7.4 mg Amoco-EskaR SW-70 heating curve. Heating rate $10^{\circ}\text{C min}^{-1}$; flow-rate, 20 ml min^{-1} of argon; recorder speed, 20 mm min^{-1} .

the majority of the wax and explosive can be produced with stirring. With consideration for both the eutectic formed by RDX and TNT and the super cooling observed in the test tube experiments (Table 2, No. 32, 34, 36, 38), a wax solidification temperature of $72\text{--}78^{\circ}\text{C}$ would be recommended to produce the mechanical mixture.

Since waxes melt over a range of temperature rather than at one particular point, the melting point is a nebulous requirement. Some waxes reputed to melt at 80°C were found to have large amounts of their components melting at 60°C (Table 1, No. 2 and 3). These low-melting components would likely exude when a composition is exposed to the 71°C environmental test. Other waxes reputed to have melting points of 80°C were found to have components which remained solid until 86 to 87°C .

Projectile impact test consists of $1/2 \times 1/2$ inch cylindrical projectiles propelled by increments of 4 or more grams of propellant fired from a 0.50 caliber gun⁵. The data are reported as the velocity of the projectile which caused no ignitions in ten successive shots into 2×1 inch cylinders of explosive composition (No GO) (Table 3), and the next higher level of velocity of the projectile which brought about an ignition of the explosive composition. The criteria of ignition was a visual indication of smoke or fire.

The Go velocity shows some correlation with enthalpy of the waxes (Table 3). The wax with the largest enthalpy also desensitized the explosive to the extent that the greatest projectile velocity was required for ignition. The temperature at which an increase in enthalpy also desensitized the explosive to the extent that the greatest projectile velocity was required for ignition. The temperature at which an increase in enthalpy occurs also affects the desensitization. Indramic 170c is a low temperature heat absorber and seems to desensitize better than would be indicated by its enthalpy value. A possible incompatibility is indicated by the decreased velocity which ignited the Composition B with the Petrolite ES 670 wax. Actual bomb tests have indicated that 1° wax changes the ignition characteristics of bombs⁶.

In conclusion the DSC provides a means of determining the enthalpy, liquefaction and solidification temperatures and a means to screen waxes.

It is recommended that a wax used as a desensitizer in a composition containing TNT has a solidification temperature of 72 to 78°C and an enthalpy value of at least 40 cal g⁻¹ from 25°C to the melt temperature.

REFERENCES

- 1 R. C. Bowers, J. G. Romans and W. A. Zisman, *Ind. Eng. Chem., Prod. Res. Dev.*, 12 (1973) 2.
- 2 *Military Specification, Wax Desensitizing MIL-W-20553B*, 18 Mar. 1962.
- 3 D. E. Seeger, *Composition B Wax Study*, Picatinny Arsenal, Dover, N.J., presented at *Annual Meeting of Loading Section*, American Defense Preparedness Association, Apr. 74, AD918877.
- 4 *Perkin-Elmer, Instruction Manual DSC-2 Differential Scanning Calorimeter*, Norwalk, Conn.
- 5 M. L. Weiss and E. L. Litchfield, *Projectile Impact Initiation of Condensed Explosives*, Bureau of Mines Report 6986, July 1967.
- 6 J. Rubin, *Properties of RDX Composition B Without Desensitizing Wax*, Picatinny Arsenal, Dover, N.J., PATR 1435, July 1944.