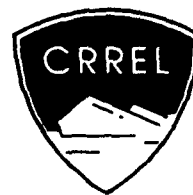


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**Purity Determination of Standard
Analytical Reference Materials
by Differential Scanning Calorimetry**

Patrick B. Black and Dean Pidgeon

May 1990

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**U.S. Army Corps
of Engineers**
Cold Regions Research &
Engineering Laboratory

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PREFACE

This report was prepared by Dr. Patrick B. Black, Soil Physicist, Applied Research Branch, Experimental Engineering Division, and Dean Pidgeon, Research Technician, Geochemical Sciences Branch, Research Division, U.S. Army Cold Regions Research and Engineering Laboratory.

Funding for this work was provided by the U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland (R-90 Multi-Analytical Services), M. Stutz, Project Monitor.

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Purity Determination of Standard Analytical Reference Materials by Differential Scanning Calorimetry

PATRICK B. BLACK AND DEAN PIDGEON

INTRODUCTION

Standard Analytical Reference Materials (SARMs) are a high-purity (>98 mol %) representative set of compounds used in the production of explosives (Table 1). Because the National Bureau of Standards has only a limited amount of these reference materials available for trace organic analysis, the United States Army Toxic and Hazardous Materials Agency (USATHAMA) has initiated a program for developing SARMs for use in its laboratories (USATHAMA 1987). This two-part program consists of procuring and producing the high-purity reference materials and then monitoring their purity level while in storage. This paper, which is concerned with the second part of the program, reports on the method of determining the purity of the seven SARMs noted in Table 1 that are suited to differential scanning calorimetry (DSC). The purity of the remaining SARMs are determined by other methods and will not be discussed further.

The USATHAMA Quality Assurance program manual (USATHAMA 1987) recommends a surveillance program that tests the purity of the SARMs at six-month intervals. For this project the criterion that is used for accepting the standard is that it does not have a purity level below 98 mol %. If the SARM fails to meet this criterion, the central repository is to be contacted and appropriate decisions made on its status.

The surveillance program for each SARM begins when the compound is first produced to the desired purity level, which is determined by successfully undergoing aggravated storage. An aggravated storage test consists of exposing the SARM to 70°C for two weeks in a loosely capped container followed by purity analysis by DSC.* The SARMs are then stored in an explosive-proof freezer, and their purity is monitored by DSC at

Table 1. SARMs analyzed.

SARM	Melting point (°C)	Heat of fusion (cal/gm)	Used DSC
1,3,5 TNB	122.5*		yes
2,4,6 TNBA			yes
2,4 DNT	70†		yes
2,6 DNT			yes
Picric acid	122.5**	18.2**	yes
Tetryl	131*, 129.5,†** 129.45††	22.2††, 19.1**	yes
TNT	80.1†, 80.9,†† 80.8**	23.53††, 23.1**	yes
Nitroglycerine			no
RDX			no
HMX			no
Nitrocellulose			no

*Wentsel et al. (1979)

†Jenkins et al. (1988)

**Meyer (1987)

††Gibbs and Popolato (1980)

six-month intervals. After two years the cycle is repeated by conducting another aggravated storage test (USATHAMA 1987).

This paper first describes the thermodynamic background and the underlying constraints that must be obeyed in order for molar purity to be determined by DSC techniques. Data on the thermophysical properties of the SARMs were collected according to the procedure presented by Pidgeon and Black (in prep.) and each SARM's purity was calculated. The accuracy of these calculated values were then evaluated and the final decision on the purity of each SARM made.

THEORY OF MELTING OF HIGHLY PURE COMPOUNDS

The melting of a pure compound is a first-order transition that occurs abruptly at a specific temperature

* Personal communication between T. Jenkins, CRREL, and M. Stutz, USATHAMA, 1989.

θ_o and involves a characteristic molar latent heat of fusion ΔL (cal/mole). Minor amounts of impurities should lower the transition temperature to θ_F as well as broaden the transition zone (Perkin-Elmer Corporation, n.d.). These changes are expressed by van't Hoff's equation (Plato and Glasgow 1969):

$$\frac{\theta_o - \theta_F}{X_2} = R \frac{\theta_o^2}{\Delta L} \quad (1)$$

where θ_F = fusion temperature of the impure compound ($^{\circ}\text{C}$)

θ_o = melting point of the pure major component ($^{\circ}\text{C}$)

R = gas constant ($\text{cal mole}^{-1} \text{ } ^{\circ}\text{C}^{-1}$)

X_2 = mole fraction of impurity (mol %).

To correctly apply this form of the equation, several constraints must be obeyed (Hunter and Blaine 1984, Widmann and Sommerauer 1988):

- The mole fraction of impurity must be small ($<3\%$);
- The melted impurity must not form additional compounds with the major component as it completes total phase change; and
- The phase transition must be reversible.

The first constraint allows the implementation of the two mathematical simplifications that resulted in eq 1: $\ln(1-X_2) \approx -X_2$; and $\theta_F \theta_o \approx \theta_o^2$. Implicit in eq 1 as well is that ΔL is constant over the temperature range ($\theta_o - \theta_F$). The second constraint assumes that the minor components are totally soluble in the liquid phase of the major component and totally insoluble in the solid phase of the major component. The final constraint is necessary to apply the equilibrium thermodynamics that leads to the van't Hoff's equation.

When a compound satisfies these three requirements, its purity can be determined by measuring, at most, its heat of fusion, its freezing point depression and its melting temperature. Differential scanning calorimetry offers an established method of concurrently measuring these three properties.

EXPERIMENT

A Perkin-Elmer DSC-4 differential scanning calorimeter was used to analyze the seven SARMS suited for DSC analysis (Table 1). The test method consisted of melting the test specimen at a carefully controlled temperature rate while recording the heat flow into the specimen as a function of time. The resulting endotherm was analyzed to determine the specimen's heat of fusion, melting temperature and freezing point depression, which

Table 2. Optimum sample parameters determined by Hunter and Blaine (1984).

Specimen size	1.7 mg
Heating rate	0.5 $^{\circ}\text{C}/\text{min}$
Data acquisition rate	120 data points/ $^{\circ}\text{C}$

were then used in eq 1 to determine mole percent purity. The control of the DSC-4 and necessary data reduction were done using the procedure and program documented by Pidgeon and Black (in prep).

Throughout the several days during which the measurements were taken, an indium reference was regularly employed to calibrate the DSC-4 for temperature and thermal power. The measured calibration constants for the machine from each run were then used in determining the thermal properties of the test specimens measured following that calibration run.

ASTM standards proposed by Hunter and Blaine (1984) recommend optimum values of sample mass, heating rate and sampling rate for computer acquisition of impurity data. These conditions for an impurity <2 mol % are presented in Table 2. In addition to determining optimum parameters, Hunter and Blaine made two important observations. First, deviations from the optimum values result in underestimating the impurity level. Second, slight variations from these values may be tolerated, but the inaccuracies thus generated are cumulative. If deviations from optimum must occur, the "true" impurity will be larger than that calculated, and care must be taken to maintain the remaining parameters at their optimum if any one parameter must deviate.

Ten replicates of each compound were tested. Table 3 summarizes the results. A complete set of all the data and plots of each melting curve are presented in Pidgeon and Black (1989).

DISCUSSION

Based on these measurements alone, all compounds were determined to have a purity greater than 99 mol %. Therefore, these seven compounds meet the USA THAMA QA criterion for Standard Analytical Reference Materials.

The results in Table 3 show that the tests were not conducted at the optimum recommended settings of Table 2. This was due to the limitations of the data collection system used for the measurements. The system's limited memory required that a less-than-optimum number of data points could be collected over the entire temperature range of the melting curve. To compensate, it was necessary to increase the recommended heating rate of 0.5 $^{\circ}\text{C}/\text{min}$ to 2.5 $^{\circ}\text{C}/\text{min}$. Table 3 also

Table 3. Summary statistics of mole percent purity determination of seven SARMS suited to DSC techniques. The heating rate for each analysis was 2.5°C/min.

a. 1,3,5 TNB.

Spec. no.	Mass (mg)	θ_0 (°C)	θ_0 (K)	Fraction(K)		Corr. (X%)	Hf (cal/mole)	Hf (cal/gm)	Purity (%)
				start	end				
1	1.55	122.941	396.101	395.948	396.197	22.3	4340.263	20.366	99.954
2	2.34	123.131	396.291	396.114	396.364	20.1	4284.198	20.103	99.953
3	2.29	123.042	396.202	396.031	396.322	35.0	4816.054	22.599	99.925
4	1.65	122.958	396.118	395.962	396.253	24.6	4457.232	20.915	99.948
5	2.89	123.011	396.171	396.003	396.336	24.2	4515.091	21.187	99.945
6	1.65	123.067	396.227	396.045	396.295	24.3	4530.101	21.257	99.938
7	2.45	123.131	396.291	396.128	396.503	16.1	4159.037	19.516	99.964
8	2.10	123.225	396.385	396.211	396.545	24.0	4335.729	20.345	99.944
9	1.71	123.173	396.333	396.170	396.461	30.4	4729.276	22.192	99.935
10	2.38	123.136	396.296	396.128	396.461	23.1	4463.889	20.946	99.948
Average	2.10	123.08	396.242	396.074	396.374	24.4	4463.087	20.943	99.945
Std. Dev.	0.44	0.09	0.093	0.089	0.114	5.2	199.562	0.937	0.011
Variance	0.20	0.01	0.009	0.008	0.013	26.9	39825.108	0.877	0.000

b. 2,4,6 TNBA.

Spec. no.	Mass (mg)	θ_0 (°C)	θ_0 (K)	Fraction(K)		Corr. (X%)	Hf (cal/mole)	Hf (cal/gm)	Purity (%)
				start	end				
1	2.16	118.884	392.044	391.779	392.196	12.7	4455.758	18.479	99.891
2	2.42	118.713	391.873	391.654	392.487	36.3	5728.564	23.758	99.833
3	1.24	118.701	391.861	391.638	392.221	23.2	5755.129	23.868	99.837
4	1.81	119.447	392.607	392.404	393.070	19.3	5015.647	20.801	99.882
5	1.75	119.158	392.318	392.071	392.404	2.3	4047.713	16.787	99.916
6	2.36	119.239	392.399	392.162	392.829	32.3	5102.442	21.161	99.841
7	2.02	118.888	392.048	391.854	392.854	44.2	5659.786	23.473	99.840
8	1.46	118.589	391.749	391.461	391.794	13.7	3986.815	16.535	99.898
9	1.99	118.579	391.739	391.494	392.077	34.0	4549.465	18.868	99.864
10	2.19	119.098	392.258	391.961	392.294	19.0	4138.200	17.162	99.865
Average	1.94	118.93	392.090	391.848	392.423	23.7	4843.952	20.089	99.867
Std. Dev.	0.38	0.30	0.295	0.304	0.394	12.8	705.443	2.926	0.029
Variance	0.14	0.09	0.087	0.092	0.155	164.4	497649.636	8.559	0.001

c. 2,4 DNT.

Spec. no.	Mass (mg)	θ_0 (°C)	θ_0 (K)	Fraction(K)		Corr. (X%)	Hf (cal/mole)	Hf (cal/gm)	Purity (%)
				start	end				
1	2.03	70.21	343.370	342.767	343.392	28.6	5285.832	29.021	99.531
2	2.00	69.659	342.819	342.351	343.392	30.9	5041.063	27.677	99.686
3	1.46	70.331	343.491	343.184	343.600	15.8	4986.223	27.376	99.823
4	1.69	70.459	343.619	343.184	343.600	26.1	5182.906	28.456	99.645
5	1.92	70.27	343.430	342.976	343.600	20.1	4948.470	27.168	99.765
6	2.20	69.422	342.582	342.143	343.808	38.1	4816.913	26.446	99.656
7	2.06	70.654	343.814	343.392	343.808	18.0	4865.404	26.712	99.762
8	2.54	70.64	343.800	343.184	343.808	21.4	4755.163	26.107	99.647
9	1.95	70.184	343.344	342.767	343.808	45.1	4896.278	26.882	99.506
10									
Average	1.98	70.20	343.363	342.883	343.646	27.1	4975.361	27.316	99.669
Std. Dev.	0.30	0.42	0.416	0.418	0.173	9.7	171.960	0.944	0.105
Variance	0.09	0.17	0.173	0.175	0.030	94.3	29570.328	0.892	0.011

Table 3 (cont'd). Summary statistics of mole percent purity determination of seven SARMS suited to DSC techniques. The heating rate for each analysis was 2.5°C/min.

d. 2,6 DNT.

Spec. no.	Mass (mg)	θ_0 (°C)	θ_0 (K)	Fraction (K)		Corr. (X%)	Hf (cal/mole)	Hf (cal/gm)	Purity (%)
				start	end				
1	2.39	65.052	338.212	338.120	338.370	22.6	3689.047	20.254	99.962
2	2.09	65.426	338.586	338.478	338.644	14.1	1294.827	7.109	99.992
3	1.31	65.265	338.425	338.299	338.466	20.6	4150.906	22.790	99.936
4									
5	2.45	57.923	331.083	330.823	331.239	10.7	5345.079	29.346	99.919
6	2.10	57.945	331.105	330.887	331.137	35.9	6528.501	35.843	99.761
7									
8	2.99	57.787	330.947	330.802	330.969	13.2	5418.076	29.747	99.945
9	2.76	57.807	330.967	330.803	331.136	21.6	5596.084	30.724	99.914
10	2.16	68.969	342.129	341.926	342.759	23.4	5384.466	29.562	99.872
Average	2.28	62.02	335.182	335.017	335.340	20.3	4675.873	25.672	99.913
Std. Dev.	0.51	4.61	4.609	4.633	4.723	7.9	1623.960	8.916	0.071
Variance	0.26	21.24	21.239	21.469	22.310	63.0	2637244.679	79.493	0.005

e. Picric acid.

Spec. no.	Mass (mg)	θ_0 (°C)	θ_0 (K)	Fraction (K)		Corr. (X%)	Hf (cal/mole)	Hf (cal/gm)	Purity (%)
				start	end				
1	1.74	122.009	395.169	394.962	395.295	23.0	4649.824	20.305	99.909
2	3.02	122.030	395.190	394.962	395.295	28.3	4907.968	21.432	99.881
3	2.04	121.647	394.807	394.626	395.042	23.7	4933.367	21.543	99.913
4	2.00	121.978	395.138	394.876	395.126	32.2	5324.762	23.252	99.828
5	1.69	122.362	395.522	395.281	395.614	30.6	5031.882	21.973	99.863
6	2.37	122.103	395.263	395.034	395.367	24.1	5003.511	21.849	99.881
7	1.99	121.933	395.093	394.876	395.209	29.4	5039.895	22.008	99.877
8	1.33	121.857	395.017	394.826	395.117	28.5	5335.077	23.297	99.887
9	2.37	121.503	394.663	394.459	395.042	37.4	5602.834	24.467	99.868
10	1.90	121.952	395.112	394.909	395.201	25.8	5118.580	22.352	99.883
Average	2.05	121.94	395.097	394.881	395.231	28.3	5094.770	22.248	99.879
Std. Dev.	0.46	0.24	0.236	0.222	0.173	4.4	267.407	1.168	0.024
Variance	0.21	0.06	0.056	0.049	0.030	19.7	71506.328	1.364	0.001

f. Tetryl.

Spec. no.	Mass (mg)	θ_0 (°C)	θ_0 (K)	Fraction (K)		Corr. (X%)	Hf (cal/mole)	Hf (cal/gm)	Purity (%)
				start	end				
1	2.86	129.085	402.245	402.130	402.771	9.4	6045.691	21.054	99.975
2	2.34	129.102	402.262	402.130	402.563	7.6	5954.854	20.738	99.975
3	2.66	129.348	402.508	402.380	402.963	11.6	6226.205	21.683	99.966
4	3.03	129.101	402.261	402.130	402.671	9.1	6083.879	21.187	99.970
5	1.91	129.189	402.349	402.213	402.630	9.9	6133.364	21.359	99.965
6	1.73	129.187	402.347	402.213	402.646	9.2	6079.107	21.170	99.967
7	2.23	129.172	402.332	402.213	402.763	9.4	6207.048	21.616	99.973
8	2.42	129.525	402.685	402.546	403.021	12.5	6276.334	21.857	99.959
9	2.54	129.298	402.458	402.338	402.896	8.5	6068.322	21.133	99.976
10	1.67	129.272	402.432	402.296	402.713	9.0	6145.558	21.402	99.967
Average	2.34	129.23	402.388	402.259	402.764	9.6	6122.036	21.320	99.969
Std. Dev.	0.46	0.14	0.137	0.134	0.152	1.4	95.760	0.333	0.005
Variance	0.21	0.02	0.019	0.018	0.023	2.1	9170.015	0.111	0.000

Table 3 (cont'd).

g. TNT.

Spec. no.	Mass (mg)	θ_0 (°C)	θ_0 (K)	Fraction (K)		Corr. (X%)	Hf (cal/mole)	Hf (cal/gm)	Purity (%)
				start	end				
1	2.36	81.62	354.780	354.442	354.858	33.7	6795.214	29.918	99.731
2	2.06	81.771	354.931	354.546	355.067	36.6	6761.890	29.771	99.686
3	2.04	81.593	354.753	354.442	354.858	31.8	6749.360	29.716	99.765
4	2.15	80.858	354.018	353.816	354.149	20.3	6279.014	27.645	99.889
5	2.57	80.91	354.070	353.899	354.399	27.5	6457.268	28.430	99.893
6	1.43	80.8	353.960	353.816	354.149	11.2	5794.607	25.512	99.957
7	1.59	80.621	353.781	353.592	353.925	20.0	6469.962	28.486	99.902
8	2.37	80.693	353.853	353.675	354.175	24.0	6769.176	29.803	99.892
9	1.71	80.563	353.723	353.509	353.842	32.2	7028.742	30.946	99.825
10	2.60	80.564	353.724	353.509	353.842	22.3	6595.779	29.040	99.871
Average	2.09	81.00	354.159	353.925	354.326	26.0	6570.101	28.927	99.841
Std.Dev.	0.41	0.47	0.473	0.403	0.452	7.8	346.268	1.525	0.087
Variance	0.16	0.22	0.224	0.163	0.204	61.2	11901.794	2.325	0.008

shows that the average sample mass was approximately 2 mg instead of the recommended 1.7 mg. This was due in part to poor laboratory technique, as well as the granular nature of the compounds, which made it difficult to control the specimen's mass in that small range. According to Hunter and Blaine (1984) the combined effect of this heating rate and mass deviation should cause a 0.20 mol % overestimation of the purity. The values listed in Table 3 accordingly must be adjusted downward and the number of significant figures reduced to two. Table 4 presents the results of these adjustments to the values in Table 3 and shows that each SARM's calculated purity is still well within the 98 mol % criterion of the USATHAMA QA manual.

Table 4. Final purity values of SARMS.

SARM	Purity (mol %)
1,3,5 TNB	99.74
2,4,6 TNBA	99.47
2,4 DNT	99.67
2,6 DNT	99.71
Picric acid	99.68
Tetryl	99.77
TNT	99.64

CONCLUSION

It is interesting to note the effect that compounds such as the SARMS, with large values of melting temperature and heat of fusion, have on eq 1. The most apparent is that temperature inaccuracies must be significant (>2 K) to affect the predicted purity. Likewise, but to a lesser degree, there must be a large uncertainty in the calcu-

lated heat of fusion to modify the predicted purity value. These artifacts might indicate that the DSC approach is inherently well suited for determining the purity of the SARMS.

The procedure used to analyze the SARMS (Pidgeon and Black, in prep.) in this experiment was found to be accurate as seen by a comparison of calculated values with the values listed in Table 1 from other sources. The melting temperatures determined in these tests were all well within the range of literature values. The same is true of the calculated heats of fusion, but they were not as close to literature values.

Differential scanning calorimetry is a suitable technique for determining mole percent purity of the seven Standard Analytical Reference Materials that meet the three constraints imposed by eq 1. In addition, the SARMS tested were found to exceed the minimum purity value set by USATHAMA and therefore are suitable standards for their program.

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