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Thermodynamic Properties of Tetraphenylantimony Benzophenoxymate in the Region of 0–450 K

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Abstract—Heat capacity of tetraphenylantimony benzophenoxymate $Ph_4SbONCPh_2$ is measured for first time using adiabatic calorimeter in the range from 6K to 350K and differential scanning calorimeter in the range from 330 K to 450 K. In the range of 400–450 K is revealed a melting accompanied with partial decomposition of the substance. Standard thermodynamic functions of crystalline $Ph_4SbONCPh_2$ in the range from $T \to 0$ K to 440 K are calculated. Enthalpy of combustion of this compound is measured in a combustion calorimeter with isothermal cover and static bomb. Standard thermodynamic formation functions of crystalline $Ph_4SbONCPh_2$ at 298.15 K are calculated. Fractal dimension D is revealed.

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Nowadays has increased significantly an interest to organic compounds of antimony(V) due to the possibility of their application to preparative organoelement chemistry. Moreover, synthesis of earlier unknown organic antimony derivatives attracts attention because it has been shown that in some such compounds the central antimony atom can have coordination number equal to six [1], seven [2] and even nine [3]. Study of derivatives with highly coordinated antimony undoubtedly will promote better understanding of the nature of chemical bonding. Although synthesis of organic antimony(V) compounds and investigation of their structure are actively developed [4], physicochemical characteristics of this class compounds have been studied poorly [5]. Earlier we have studied [6] standard thermodynamic properties of pentaphenylantimony.

The purpose of this work is calorimetric investigation of thermal dependence of tetraphenylantimony benzophenoxymate $Ph_4SbONCPh_2$ (**I**) heat capacity in the region of 6–450 K, revealing the possible transformations and estimation of thermodynamic characteristics of the latter, calculation from the experimental data of standard thermodynamic functions C_p^0, $H^0(T)–H^0(0)$, $S^0(T)$, $–[G^0(T)–H^0(0)]$ of the crystalline compound **I** in the region from $T \to 0$ K to 440 K, determining combustion energy of this compound at T = 298.15 K, and calculation from the obtained data of standard enthalpy, entropy and Gibbs function of formation of crystalline compound **I** at the same temperature, and elucidation of fractal dimension D and characteristic temperature θ_{max} from the experimental data on low temperature heat capacity.

Heat capacity. Experimental values of heat capacity of **I** in the region from 6.21 K to 446.9 K (Table 1) and smoothed $C_p^0 = f(T)$ plot are shown in figure. The heat capacity of **I** grows smoothly with temperature and has no features up to T = 400 K, then C_p^0 increases sharply with temperature, that is defined by the beginning of the crystals **I** melting. The melting is accompanied with damaging the substance that restricts obtaining the heat capacity value for its liquid state and estimating thermodynamic characteristics of the melting. After the calorimetric experiment the sample mass diminished by 3% and its color and physical state changed visually. As the melting temperature T_{mp}^0 =

Table 1. Experimental values of tetraphenylantimony benzophenoxymate heat capacity. M 626.401 g mol^{-1}

T, K	C_p^0, J mol^{-1} K^{-1}	T, K	C_p^0, J mol^{-1} K^{-1}	T, K	C_p^0, J mol^{-1} K^{-1}	T, K	C_p^0, J mol^{-1} K^{-1}	T, K	C_p^0, J mol^{-1} K^{-1}	T, K	C_p^0, J mol^{-1} K^{-1}
Series 1		27.98	77.22	136.53	319.5	288.47	624.6	352.4	778	401.0	959
6.21	3.57	31.03	88.67	141.48	328.4	292.87	633.1	353.8	782	402.4	972
6.50	4.04	34.08	99.01	146.19	337.2	297.22	644.3	355.3	792	403.9	975
6.81	4.62	37.14	108.8	150.90	346.2	301.57	654.5	356.7	794	405.4	978
7.11	5.20	40.20	117.7	155.61	354.6	305.72	664.9	358.2	793	406.9	986
7.41	5.84	44.64	130.8	160.32	363.7	310.02	676.4	359.7	809	408.3	989
7.68	6.46	47.16	138.2	165.03	372.1	314.26	685.8	361.2	807	409.8	990
7.97	7.16	50.06	145.8	169.75	380.4	318.50	695.9	362.7	818	411.3	998
8.24	7.84	52.62	152.3	174.46	390.4	322.76	708.1	364.2	816	412.8	1025
8.54	8.67	55.46	160.0	179.17	398.5	327.02	718.7	365.7	822	414.3	1044
8.87	9.58	58.08	166.0	183.88	408.0	331.28	729.3	367.2	829	415.8	1041
9.21	10.6	61.14	173.1	188.58	417.5	335.54	739.9	368.8	842	417.3	1076
9.57	11.7	64.21	180.2	193.30	426.1	339.80	750.4	370.3	837	418.8	1071
10.13	13.7	67.28	187.7	198.00	435.5	344.06	761.0	371.9	849	420.3	1116
10.54	14.8	70.21	194.3	202.72	444.8	348.32	771.6	373.4	847	421.8	1135
10.95	16.1	72.88	199.8	207.43	454.5	352.58	782.2	375.0	852	423.3	1167
11.35	17.5	75.96	206.0	212.31	464.1	Series 3		376.6	863	424.8	1205
11.78	18.9	79.02	213.0	217.15	474.3	330.0	728	378.1	874	426.3	1252
12.36	20.3	83.34	221.7	221.85	484.3	331.6	736	379.7	874	426.8	1267
13.09	23.0	88.22	232.1	227.10	494.4	333.1	732	381.2	886	428.6	1342
13.82	25.40	Series 2		233.10	506.0	334.7	748	382.8	887	430.4	1423
14.55	28.10	82.24	220.1	238.79	516.0	336.2	745	384.3	884	432.2	1517
15.28	30.78	87.11	229.9	243.39	526.0	337.7	759	385.9	892	433.9	1651
16.01	33.33	91.98	238.9	247.98	534.5	339.2	745	387.4	911	435.6	1834
16.74	36.16	96.67	248.0	252.54	545.0	340.7	749	388.9	906	437.3	2084
17.48	39.22	101.36	256.7	257.10	555.0	342.2	744	390.5	917	438.9	2408
18.18	41.95	106.05	265.7	261.65	564.5	343.6	758	392.0	924	440.5	2792
18.85	44.20	110.75	274.3	266.17	574.7	345.1	758	393.5	926	442.0	3579
19.51	46.89	116.40	283.6	270.68	584.4	346.6	760	395.0	935	443.4	4233
20.16	48.83	121.11	291.7	275.17	594.6	348.0	764	396.5	942	444.9	2845
22.00	55.10	126.05	300.6	279.62	605.5	349.5	774	398.0	949	446.9	1180
24.96	66.27	131.00	309.2	284.06	614.0	350.9	782	399.5	954		

443.4±0.3 K is taken the value that corresponds to the minimum of apparent heat capacity in the range of melting. Thus, under conditions of calorimetric experiment the substance initially remained in crystalline state (figure, ABC region) and then damaged.

It seems interesting to obtain for the studied compound the value of fractal dimension D [7] from the experimental data on low temperature heat capacity. The fractal dimension is an exponent index at the temperature in the basic equation of fractal version of the Debye theory of the solid state heat capacity. The D value gives some ideas on the character of topology of a solid body structure and can be obtained from the ln C_v–ln T plot for the respective substance [7]. In part, this follows from the formula (1).

$$C_v = 3D(D + 1)kN\gamma(D + 1)\xi(D + 1)(T/\theta_{max})^D. \quad (1)$$

Here k is Boltzmann constant, N is number of atoms in the molecule, $\gamma(D + 1)$ and $\xi(D + 1)$ are respectively Riman's γ- and ξ-functions, θ_{max} is maximal characteristic temperature. For a given solid body $3D(D + 1)kN\gamma(D + 1)\xi(D + 1)(T/\theta_{max})^D = A$, where A is a constant, and Eq. (1) can be rewritten as Eq. (2).

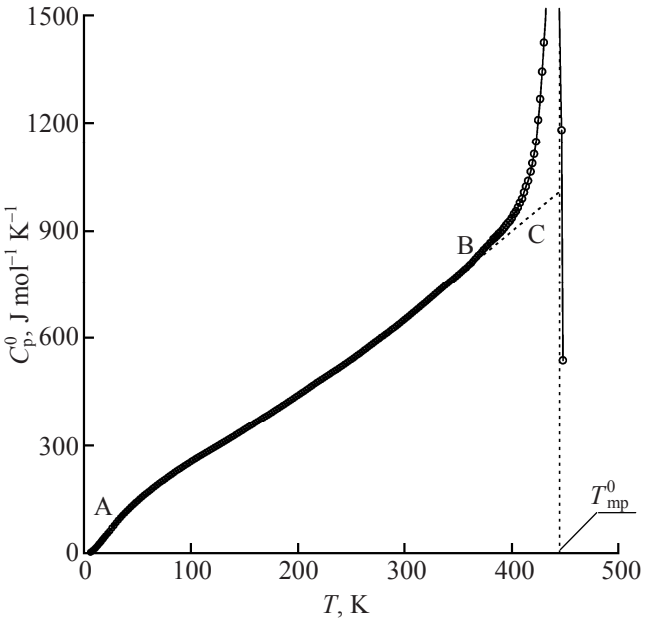
$$\ln C_v = \ln A + D\ln T. \quad (2)$$

The experimental C_p^0 value at the temperature $T <$ 50 K can with negligible error be suggested equal to C_v. Then, using experimental data on the heat capacity for the range 20–50 K and Eq. (2) one can obtain D value. We found that for compound I the fractal dimension D = 1.5 in the region of 20–50 K, and characteristic temperature θ_{max} of the heat capacity fractal theory is 202.8 K. With the found values of fractal dimension and characteristic temperature the Eq. (1) reproduces C_p^0 values in this range with error 0.5% or less. According to Tarasov's theory of solid state heat capacity [8] the value D = 1 corresponds to the substances with chain structure, D = 2 to laminated structure and D = 3 to spatial structure. The obtained value of D corresponds to laminate-chain topology of structure **I**.

Standard thermodynamic functions. For the calculation of standard thermodynamic functions (Table 2) we extrapolated the plot of heat capacity against temperature from 6 K to $T \to 0$ K using the functions of Debye's theory of solid state heat capacity:

$$C_p^0 = nD(\theta_D/T), \quad (3)$$

where D is Debye's heat capacity function, θ_D is Debye's characteristic temperature, n = 10 and θ_D =



Temperature plot of heat capacity of tetraphenylantimony benzophenoxymate.

76.14 K, are specially chosen parameters. With these parameters the Eq. (3) describes experimental values of heat capacity C_p^0 in the region from 6 K to 11 K with error ±0.4%. We suppose that in the region from $T \to$ 0 K to 6 K the Eq. (3) reproduces the value of heat capacity with the same error.

The calculations of enthalpy and entropy were carried out by the numerical integration of the curves $C_p^0 = f(T)$ and $C_p^0 = \ln f(T)$, respectively. The Gibbs function was calculated with the Gibbs–Helmholtz equation from the enthalpy and entropy values at the respective temperatures. The calculation procedure has been described [9] in details. We suggest that error of calculated values of this function is ±2% at $T <$ 15 K, ±0.5% in the T range 15–40 K, ±0.2% in the range of 40–350 K and ±1.5% in the region of from 350 K to 450 K.

Combustion enthalpy and standard thermodynamic formation characteristics of $Ph_4SbONCPh_2$ at T = 298.15 K. Combustion energy of crystalline compound **I** was measured in six experiments. The sample weight was ~0.2 g. The experimental results are listed in Table 3. After the experiment the products of combustion were analyzed. Visually, on the crucible walls occurred dark spot traces attesting formation of carbon. From the data of X-ray phase analysis the solid products after combustion of **I** contained antimony tetraoxide Sb_2O_4, trioxide Sb_2O_3 and free antimony.

Table 2. Thermodynamic functions of tetraphenylantimony benzophenoxymate. M 626.401 g mol^{-1}

T, K	C_p^0, J mol^{-1} K^{-1}	$H^0(T)$–$H^0(0)$, kJ mol^{-1}	$S^0(T)$, J mol^{-1} K^{-1}	–[$G^0(T)$–$H^0(T)$], kJ mol^{-1}
5	1.83	0.00230	0.612	0.000764
10	13.1	0.0349	4.69	0.0120
15	29.6	0.141	13.1	0.0549
20	48.41	0.3366	24.19	0.1472
30	84.82	0.9994	50.55	0.5171
40	117.2	2.017	79.60	1.167
50	145.6	3.333	108.9	2.110
60	170.3	4.918	137.7	3.343
70	193.7	6.740	165.7	4.861
80	215.0	8.783	193.0	6.655
90	235.3	11.04	219.5	8.718
100	254.3	13.48	245.3	11.04
120	290.1	18.93	294.8	16.45
140	325.6	25.09	342.2	22.82
160	363.0	31.97	388.1	30.13
180	400.3	39.60	433.0	38.34
200	439.7	48.00	477.2	47.44
220	480.3	57.20	521.0	57.42
240	519.0	67.19	564.5	68.28
260	561.1	77.98	607.6	80.00
280	605.3	89.65	650.8	92.58
298.15	646.4	101.0	690.1	104.8
300	650.9	102.2	694.1	106.0
320	700.7	115.7	737.7	120.4
340	751.0	130.2	781.7	135.5
360	798	146	826	152
380	847	162	870	169
400	897	180	915	186
440	995	217	1005	225

Qualitative and quantitative analyses of solid products after combustion of the studied compound were the same as after combustion of Me_3Sb under the similar conditions determined in [10]. Because of detection of the some amount of free antimony, corrections for incomplete oxidation of the metal were introduced (Table 3). At the calculation of ΔU_c were introduced ordinary thermochemistry corrections: for the combustion of cotton fiber used for ignition of the weighted sample, for the combustion of used paraffin

Table 3. Experimental results on measuring combustion enthalpy of $Ph_4SbONCPh_2$[a]

m, g	Q, J	q(paraffin), J	q(fiber), J	q(HNO_3), J	q(Sb_2O_3), J	q(C+Sb), J	$-\Delta_c U$, J g^{-1}	$-\Delta_c U$, kJ mol^{-1}
0.21167	39952.5	33240.3	36.8	11.7	10.5	143.2	32206.8	20174.4
0.20282	39667.3	33263.7	33.1	10.0	10.0	152.3	32160.7	20145.5
0.19916	39521.3	33244.1	31.3	6.4	9.8	170.1	32232.7	20190.6
0.20548	39658.3	33084.7	42.7	5.9	10.2	90.9	32246.9	20199.5
0.19902	39524.2	33216.0	34.8	11.1	9.8	133.4	32185.0	20160.7
0.20156	39596.3	33223.3	34.6	10.0	10.0	141.5	32149.0	20138.2

[a] (m) is combusted sample mass, g; (Q) is totally liberated energy, J; [q(paraffin), q(fiber), q(HNO_3), q(Sb_2O_3), q(C+Sb)] are corrections on respective combustion; ($\Delta_c U$) is energy of sample combustion in the conditions of calorimetric bomb, average value: $\Delta_c U(Ph_4SbONCPh_2) = -20168.2 \pm 20.0$ kJ mol^{-1}

and for the formation of HNO_3 solution. The process in the bomb was suggested corresponding to the Eq. (4):

$$Ph_4SbONCPh_2(cryst.) + 91/2\ O_2(gas) \rightarrow 37\ CO_2(gas) + 15\ H_2O(l) + 1/2\ NO_2(gas) + 1/2\ Sb_2O_4(cryst.). \quad (4)$$

The solid combustion products besides Sb_2O_4 contained Sb_2O_3, therefore at the calculation of the pentaphenylantimony combustion enthalpy we accounted for corrections on incomplete oxidation of the studied compound [11]. Also, was accounted for the Washbern correction ($\pi = -0.041$ %) and correction for the change in the number of moles of gaseous products in the combustion reaction ($\Delta n = -8$ mol). Then, standard combustion enthalpy of **I** at $T = 298.15$ K:

$$\Delta_c H^0(298.15, Ph_4SbONCPh_2, cryst.) = -20179.8 \pm 20.0 \text{ kJ mol}^{-1}.$$

From the value of standard combustion enthalpy of crystalline compound **I** and the data on standard formation enthalpy of gaseous compounds CO_2 [12, 13] and NO_2 [14], liquid water [12, 13] and crystalline Sb_2O_4 [15] was calculated standard enthalpy of formation of compound **I** at $T = 298.15$ K:

$$\Delta_f H^0(298.15, Ph_4SbONCPh_2, cryst.) = 895.4 \pm 25.6 \text{ kJ mol}^{-1}.$$

With the value of absolute entropy of crystalline compound **I** (Table 2) and of simple substances: carbon, [12], hydrogen [12, 13], oxygen [14], nitrogen [14] and antimony [16], we calculated its standard formation entropy:

$$\Delta_f S^0 = -1767.7 \pm 2.1 \text{ J mol}^{-1} \text{ K}^{-1} \text{ at } T\ 298.15 \text{ K}.$$

From the values of standard formation enthalpy and entropy of crystalline compound **I** at $T = 298.15$ K with the Gibbs–Helmholtz equation was calculated standard Gibbs function of its formation: $\Delta_f G^0(298.15, Ph_4SbONCPh_2, cryst.) = 1422.3 \pm 25.6$ kJ mol^{-1} at the same temperature.

The values obtained fit Eq. (5):

$$37\ C(graphite) + 15\ H_2(gas) + Sb(cryst.) + 1/2\ O_2(gas) + 1/2\ N_2(gas) \rightarrow Ph_4SbONCPh_2(cryst.). \quad (5)$$

EXPERIMENTAL

Compound **I** was synthesized in the Chemistry division of Blagoveshchensk State Pedagogical University along the procedure described in [4]. For the synthesis of **I** a mixture of pentaphenylantimony, benzophenone oxime [or bis(benzophenonoximate)] and toluene was kept for 1 h at $T = 90$°C, then the solvent was removed and residue was recrystallized from a mixture heptane–benzene 3:1. Yield of crystalline compound **I** was 90–97%. Compound **I** appears as light-yellow crystals stable in air under ambient conditions. Structure of compound **I** is revealed by the method of X-ray structural analysis. The crystals **I**, $C_{37}H_{30}NOSb$, are monoclinic, at 20°C: $a = 11.565(1)$, $b = 16.545(8)$, $c = 15.713(4)$ Å; $\beta = 94.09(2)$°, $V = 2999(2)$ Å^3, $Z = 4$, $d_{calc} = 1.39$ g cm^{-3} , spatial group $P2_1/n$. The structure is decoded by direct method with SIR program [17] and refined in isotropic and then in anisotropic approximation. By the data of elemental and IR spectroscopic analyses, the content of main component in the studied sample was 99.5 mol %.

For the study of thermal dependence of heat capacity of the sample **I** in the region from 6 K to 350 K was used completely automated adiabatic vacuum calorimeter BKT-3. The device construction and

operation procedure have been described in [18]. Temperature was measured by means of iron–rhodium resistance thermometer calibrated in correspondence with MTSh-90. As cold carriers were used liquid helium (for the temperature 5–6 K and higher) and nitrogen (for 80 K and higher). For testing the measuring procedure we measured heat capacity of benzoic acid of K-1 sort [19]. The result obtained coincided with the heat capacity value of reference benzoic acid within ±2% in the region of 6–15 K, ±0.5% between 15 and 40 K and ±0.2% in the range of 40–350 K. Error at the measuring of phase transitions temperature was ±01 0. K, of phase transition enthalpy ±0.2%.

Measuring the heat capacity, temperature and phase transition enthalpy in the region 330 K to 450 K was carried out with automatic differential scanning calorimeter operating on the pricnciple of triple heat bridge (ADKTTM) [20]. The calorimeter construction and measuring procedure are described in [20]. The calorimeter operating reliability was tested by measuring the heat capacity of a sample of synthetic corundum and thermodynamic characteristics of indium, tin and lead melting. The maximal error at the heat capacity measuring is evaluated as ±2%, of transition temperature ±0.5 K. Since the experiment of the heat capacity measuring in the temperature range from 330 K to 350 K was carried out with adiabatic vacuum calorimeter with error ±0.2% and conditions of measuring with dynamic scanning calorimeter were optimized for coincidence of results on C_p^0 on both calorimeters in the same temperature range, we suggest that at $T > 350$ K the error at measuring C_p^0 value is ±(0.5–2.0)%.

The C_p^0 heat capacity measuring was carried out in the temperature range from 6 K to 450 K. The sample weight of the samples loaded to ampoules for BKT-3 and ADKTTM were, respectively, 0.7555 g and 0.7499 g. With BKT-3 were obtained 110 experimental values of heat capacity in two series, with ADKTTM 78 values in one series (Table 1). The substance heat capacity was 20–70% of the total heat capacity of the ampoule with the substance in the range from 6 K to 115 K and 25–35 % in the range from 115 K to 450 K. The experimental C_p^0 values were averaged using exponential and semi-logarithmic polynomials. The mean square deviation of experimental heat capacity values from averaging curve $C_p^0 = f(T)$ was ±0.1% in the region of 19–80 K, ±0.18% for 75–210 K and ±0.15% for 210–360 K. Molecular weight of the studied object was calculated using IUPAC Table of Atomic Weights [21].

Combustion energy of **I** was measured in the modernized calorimeter B-08-MA with isothermal cover and static bomb [22]. Oxygen pressure in the bomb was 25×10^5 Pa. The calorimeter energetic factor $W = 14805.1\pm2.5$ J Ω^{-1} was measured at combusting reference benzoic acid of K-2 sort. Testing of reliability by combustion of reference succinic acid showed that its combustion energy actually corresponds to property data sheet within error ±0.017%.

The studied sample was combusted in a thin-walled quartz crucible being mixed with melted paraffin, the combustion energy of the latter measured in preliminary experiments was $\Delta U_c = -46744\pm8$ J g^{-1}. Application of paraffin as auxiliary substance provided both necessary increase of temperature in the experiment and conditions for complete oxidation of the studied substances.

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