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CHEMICAL THERMODYNAMICS AND THERMOCHEMISTRY

The Thermodynamic Properties of Four Triphenylsilane Acetylene Peroxides

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Abstract—The enthalpies of formation of four acetylene triphenylsilane peroxides were determined in the condensed and gaseous states. Three silicon-containing fragment increments for the Benson additive scheme and the enthalpies of formation of three radicals formed in the dissociation of the compounds at the O–O bond were calculated.

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INTRODUCTION

Organosilicon compounds have been extensively used in the production of modern materials and compositions. Thanks to their high thermal stability, hydrophobic properties, and stability during storage, siliconcontaining peroxides also find wide application as initiators of various free-radical processes, for instance, in high-temperature (up to 600 K) initiation of polymerization, polymer modification, elastomer vulcanization, and lacquer composition solidification. At the same time, data on the thermodynamic properties of siliconcontaining peroxides are virtually absent. The reason for this is the complexity of preparing organosilicon peroxides sufficiently pure (more than 99% major component) for precision thermodynamic measurements and the great complexity of such measurements themselves.

In [1], we for the first time determined the enthalpies of formation of seven liquid methylsilyl acetylene peroxides by burning the peroxides with an explosion in an isothermic-shell calorimeter. The substances were sealed in Terylene ampules for simultaneously igniting the whole sample surface and increasing temperature at the contact, which ensured maximum combustion completeness. We used double-walled ampules to prevent the diffusion of substance molecules through the film.

We were the first to experimentally determine the enthalpies of formation of four acetylene triphenylsilane peroxides. These were 3-methyl-3-*tert*-butylper-oxy-1-triphenylsilyl-1-butine

$$CH_3)_3COOC(CH_3)_2C\equiv CSi(C_6H_5)_3,$$
 (I)

3-methyl-3-*tert*-amylperoxy-1-triphenylsilyl-1-butine

$$CH_3CH_2(CH_3)_2COOC(CH_3)_2C\equiv CSi(C_6H_5)_3$$
, (II)

3-methyl-3-*tert*-hexylperoxy-1-triphenylsilyl-1-butine $CH_3(CH_2)_2(CH_3)_2COOC(CH_3)_2C\equiv CSi(C_6H_5)_3$, (III) and 4-methyl-4-*tert*-butylperoxy-1-triphenylsiloxy-1-

and 4-methyl-4-*tert*-butylperoxy-1-triphenylsiloxy-1pentine

$$(CH_3)_3COOC(CH_3)_2C \equiv CCH_2OSi(C_6H_5)_3.$$
 (IV)

EXPERIMENTAL

Compounds **I–III** were prepared in the reactions between 3-methyl-3-*tert*-alkylperoxy-1-butines and butyllithium. Next, lithium peroxyacetylenides formed reacted with triphenylchlorosilane [2] to produce the desired substances,

$$\begin{array}{l} \text{ROOCMe}_2\text{C} \equiv \text{CH} \xrightarrow{\text{BuLi}} \text{ROOCMe}_2\text{C} \equiv \text{CLi} \\ \xrightarrow{\text{Ph}_3\text{SiCl}} \text{ROOCMe}_2\text{C} \equiv \text{CSiPh}_3. \end{array}$$

Compound IV [3] was synthesized following the scheme

$$\begin{array}{l} \text{ROOCMe}_2\text{C} \equiv \text{CH} \xrightarrow{\text{BUL}} \text{ROOCMe}_2\text{C} \equiv \text{CLi} \\ \xrightarrow{\text{+CH}_2\text{O}} \quad \text{ROOCMe}_2\text{C} \equiv \text{CCH}_2\text{OLi} \\ \xrightarrow{\text{Ph}_3\text{SiCl}} \text{ROOCMe}_2\text{C} \equiv \text{CCH}_2\text{OSiPh}_3. \end{array}$$

Crystals I were purified by low-temperature recrystallization from hexane. Liquids II and III were purified by removing volatile impurities under evacuation for 3–4 h at 323 K and 0.3 kPa. Liquid IV was evacuated for 2–3 h at 303–313 K and 0.4 kPa.

The products were characterized by elemental analyses for carbon, hydrogen, silicon, and active oxygen; their molecular weights were determined by cryoscopy, and their IR, UV, and NMR spectra were recorded. The individual character of the compounds was proved by thin-layer chromatography on Silufol UV-254 plates. The purity of the compounds was checked by highpressure liquid chromatography with the use of a system consisting of a pump, UV and refractometric detectors, and a Separon SGX CN column (length 150 mm and diameter 3.3 mm, particle size 7 μ m). The eluent was a heptane–isopropanol mixture with a 98 : 2 volume ratio. The content of the major component was no less than 99.2 wt % in all the compounds studied. We were unable to determine the nature of impurities.

The temperature dependences of compound saturated vapor pressures and the enthalpies of vaporization were determined by the Knudsen integral effusion method. The results obtained this way for **I**–**III**, the design of the experimental unit, the reliability of its operation, and the procedure for measurements were reported in [4].

Saturated vapor pressure of **IV** was determined in a series of 18 experiments by the extrapolation of the vapor pressure in a cell (membrane orifice diameter was 0.5903 mm) to "zero" orifice area with the use of a correction factor of 1.14. The vacuum line ensured the attainment of a 0.1 Pa pressure in 45 ± 10 s. The mass of the effused substance Δm was determined by weighing the effusion cell with an accuracy of $\pm 5 \times 10^{-6}$ g. Errors in maintaining constant temperature and determining the temperature of measurements *T* and the total effusion time τ were of ± 0.1 K, ± 0.05 K, and ± 1 s, respectively. The effective effusion time (calculated time under stationary conditions during which the effused substance weight equaled that under nonstationary conditions) was 105 s.

The results of effusion experiments with **IV**, including saturated vapor pressure p and pressure in the cell p_c , are listed in Table 1. The temperature dependence of saturated vapor pressure is described by the equation

 $\ln p = (27.3 \pm 1.2) - (105.3 \pm 4.5) \times 10^2 / T,$

with the correlation coefficient $\rho = 99.98$. The enthalpy of vaporization $\Delta_v H$ averaged over the temperature range of measurements was 87.6 ± 3.7 kJ/mol.

The combustion of the compounds was performed in a modernized V-08 MA isothermic-shell (± 0.003 K) calorimeter. The calorimeter was modernized to decrease heat loss and fluctuations. The orifices for resistance thermometers were tightened with foamplastic sockets to a maximum degree possible. A resistance was connected in series to the heaters of the system for maintaining a constant shell temperature to decrease shell temperature fluctuations. The transformer for incandescing the wire that ignited samples was replaced by a set of capacitors. The energy of wire heating was as low as 1.3 ± 0.3 J in calibration and measurement experiments and was therefore excluded from calculations. The energy equivalent of the calorimetric system (W = 14930.5 J/V) was determined with an accuracy of ±0.056% in a series of ten experiments with the combustion of standard benzoic acid, K-1 brand (major component content 99.995 mol %). The heat of

Table 1. Results obtained in experimentally determining thetemperature dependence of saturated vapor pressure over IV

No.	τ, s	$\Delta m \times 10^3$, g	<i>Т</i> , К	$p_{\rm c}$, Pa	<i>p</i> , Pa	
1	4866	0.400	353.5	0.066	0.075	
2	7266	1.320	362.7	0.149	0.170	
3	7266	1.350	362.7	0.152	0.173	
4	5466	1.565	367.8	0.236	0.269	
5	5466	1.550	367.8	0.234	0.267	
6	5466	1.515	367.8	0.228	0.260	
7	5466	1.600	367.8	0.241	0.275	
8	5467	1.875	371.6	0.284	0.324	
9	5467	1.900	371.6	0.288	0.328	
10	3668	1.870	376.0	0.425	0.485	
11	3668	1.950	376.0	0.443	0.505	
12	4566	2.610	379.4	0.479	0.546	
13	4566	2.700	379.4	0.495	0.564	
14	3666	3.025	382.5	0.693	0.790	
15	3666	3.150	382.5	0.722	0.823	
16	3666	2.905	382.9	0.666	0.759	
17	3666	3.050	382.9	0.699	0.797	
18	3666	3.745	386.0	0.862	0.983	

combustion of benzoic acid corrected using the Jessup factor was 26426.9 J/g. The initial pressure of oxygen preliminarily purified from combustible impurities, carbon dioxide, and water was 2.94×10^6 Pa. The initial temperature of the main period was 298.15 K in all experiments.

Solid peroxide I was tabletted prior to combustion. Liquid substances II-IV were sealed in double-walled ampules following the procedure described in [1]. Combustion was performed in platinum crucibles to decrease carbon black formation. Insignificant carbon black deposits on platinum crucible walls were weighed with an accuracy of $\pm 5 \times 10^{-6}$ g. Each combustion experiment was followed by quantitative analyses of combustion products for carbon mono- and dioxides, carbon black, and nitric acid. The weight of the substance burned was determined from the amount of carbon dioxide [5] with an accuracy of $\pm 1 \times 10^{-4}$ g. The reliability of our gas analyses was substantiated in a series of calibration experiments with the combustion of standard benzoic acid. The amounts of carbon dioxide formed in the combustion of 1 g of the film and 1 g of the cotton thread were experimentally found to be 2.2872 and 1.6284 g, respectively. The content of carbon monoxide in combustion products was beyond the sensitivity of our method for determining CO ($\pm 1 \times 10^{-6}$ g). The amounts of nitric acid formed in combustion experiments were determined by titration with a 0.1 N solution of NaOH. When samples were prepared and experiments conducted as described above, the completeness of combustion (k) was of 99.05–99.97%, which was

<i>m</i> , g	$\Delta T, V$	Q_{Σ},J	$Q_{\rm t}, { m J}$	$Q_{\rm a}, { m J}$	$Q_{\rm n}, { m J}$	<i>Q</i> _c , J	k, %	$-\Delta U$, J/g
	1		(CH ₃) ₃ CO	OC(CH ₃) ₂ C≡	CSi(C ₆ H ₅) ₃		<u>I</u>	
0.127610	0.3306	4936.2	49.36	965.95	2.95	6.56	99.76	38413.3
0.11690	0.3022	4512.1	46.90	971.34	2.36	6.70	99.80	38319.5
0.221580	0.5733	8559.9	49.44	948.63	1.77	6.89	99.86	38486.4
0.208400	0.5365	8010.5	47.52	921.09	4.72	7.87	99.82	38312.3
0.223425	0.5760	8600.3	51.20	930.27	3.54	10.99	99.55	38476.9
0.173020	0.4463	6663.7	37.58	1078.72	2.95	9.84	99.82	38411.7
0.155925	0.4020	6002.3	37.58	987.40	0.89	6.56	99.73	38390.8
	I	C	H ₃ CH ₂ (CH ₃)	2COOC(CH ₃)	$_2C \equiv CSi(C_6H_5)$)3	I	I
0.128565	0.3998	5969.4	47.61	965.95	2.95	11.97	99.48	38815.9
0.117135	0.3692	5512.5	36.75	971.34	2.36	10.50	99.59	38675.8
0.123600	0.3856	5757.4	42.01	948.63	1.77	15.58	99.84	38735.6
0.166170	0.4937	7371.4	41.18	921.09	4.72	20.17	99.95	38677.4
0.127825	0.3945	5890.3	42.60	930.27	3.54	18.20	99.46	38794.9
0.129925	0.4106	6130.7	42.26	1078.72	2.95	12.30	99.63	38774.0
0.128450	0.4110	6136.6	43.60	987.40	0.89	9.68	99.65	38792.3
	1	CH	$H_3(CH_2)_2(CH_3)$) ₂ COOC(CH ₃) ₂ C≡CSi(C ₆ H	5)3	Į.	1
0.102105	0.3360	5016.8	50.11	1000.71	1.18	11.97	99.80	39018.5
0.107570	0.3503	5230.3	49.61	1012.07	0.59	7.87	99.58	38981.5
0.11290	0.3594	5366.2	49.95	1001.06	0.59	13.45	99.63	39032.7
0.120710	0.3820	5703.6	51.28	955.74	0.59	9.68	99.83	39044.2
0.119675	0.3841	5735.0	48.02	1040.75	0.59	6.89	99.95	38897.9
0.122320	0.3870	5778.3	37.42	992.22	1.18	10.99	99.82	38977.3
0.121305	0.3836	5727.5	45.69	966.52	2.36	10.99	99.97	38951.2
$(CH_3)_3COOC(CH_3)_2C\equiv CCH_2OSi(C_6H_5)_3$								
0.107365	0.3301	4928.7	57.96	950.46	1.18	7.87	99.56	36731.9
0.150230	0.4364	6515.9	62.47	973.52	1.18	7.05	99.58	36669.9
0.152825	0.4441	6630.9	73.08	1017.80	1.18	10.82	99.05	36655.1
0.145165	0.4227	6311.3	59.38	963.77	0.59	9.51	99.52	36662.3
0.144890	0.4280	6390.5	57.71	1042.47	1.77	6.23	99.52	36720.3
0.155300	0.4489	6702.5	58.80	971.46	0.59	14.92	99.52	36791.2
0.146095	0.4283	6394.9	45.85	1012.18	1.18	7.71	99.25	36844.5
0.118120	0.3626	5414.0	49.69	1045.68	1.18	10.33	99.74	36725.1
0.141610	0.4156	6205.3	50.20	994.63	1.18	11.97	99.75	36603.0
0.108865	0.3351	5003.4	46.86	983.27	1.18	8.36	99.81	36624.9
0.120600	0.3622	5408.0	47.36	974.33	1.18	10.00	99.52	36622.9

Table 2. Results obtained in experimentally determining the energies of combustion of the compounds

quite acceptable for compounds with such complex structures.

To take into account the energy (J/g) released in experiments as a result of side processes, we introduced corrections for the combustion of Terylene ampule $(Q_a = 22944.2 [6])$ and cotton thread $(Q_t = 16704.2 [7])$ and the formation of carbon black $(Q_c = 32800)$ and a

solution of nitric acid ($Q_n = 59 \text{ kJ/mol} [5]$). The results obtained in determining the energy of combustion of **I–IV** are listed in Table 2. In addition to the notation specified above, Table 2 contains the following notation: *m* is the burned substance weight, Q_{Σ} is the total amount of energy released, ΔT is the corrected temperature rise during measurements, and ΔU is the energy

Compound	-ΔU° (298.15 K)	π	$-\Delta nRT$	–Δ _c H° (298.15 K)	$\Delta_{\rm f} H^{\circ}$ (cond., 298.15 K)	$\Delta_{\rm f} H^{\circ} ({ m g},298.15~{ m K})$	
						experimental	calculated
I	15914 ± 26	8	19	15933 ± 26	81 ± 26	238 ± 27	233
II	16603 ± 23	8	20	16623 ± 23	92 ± 23	212 ± 23	212
III	17250 ± 21	9	21	17271 ± 21	61 ± 21	187 ± 21	192
IV	16305 ± 21	9	19	16324 ± 21	-207 ± 21	-119 ± 21	-119

Table 3. Standard enthalpies of combustion and formation of acetylene peroxides, kJ/mol

of substance combustion under bomb conditions at the initial temperature of measurements. The enthalpies of combustion of the compounds were taken to correspond to the reaction

$$C_a H_b O_c Si_d(1, s) + (a + b/4 - c/2 + d)O_2(g)$$

→ $a CO_2(g) + (b/2)H_2O(1) + dSiO_2(am. hydr.).$ ⁽¹⁾

The standard energies of combustion $\Delta U^{\circ}(298.15 \text{ K})$ and the enthalpies of combustion $\Delta_{c}H^{\circ}(298.15 \text{ K})$ and formation $\Delta_{\rm f} H^{\circ}(298.15 \text{ K})$ of the peroxides in the condensed state (Table 3) were calculated with the inclusion of the Washburn correction π and correction for the work of expansion ΔnRT (Δn is the change in the number of gas moles in reaction (1)). We used the following reference $\Delta_{\rm f} H^{\circ}(298.15 \,{\rm K})$ values (kJ/mol): CO₂(g), -393.514 ± 0.046 ; H₂O(1), -285.830 ± 0.042 [9]; and $SiO_2(am. hydr.), -939.39 \pm 0.52$ [10]. The standard deviation of the mean combustion energy value was calculated using the Student test value for a 5% significance level. Table 3 also contains the standard enthalpies of formation of the peroxides in the gas phase $\Delta_{\rm f} H^{\rm o}({\rm g}, 298.15 {\rm K})$. These were determined using the enthalpies of vaporization of IV and I-III; the latter were 115.9 ± 3.2 , 120.3 ± 5.8 , and 126.3 ± 3.0 kJ/mol, respectively [1]. We also used the enthalpy of fusion of I: 41 ± 1 kJ/mol [7].

RESULTS AND DISCUSSION

Compounds **I–III** make up a homologous series each member of which is one CH_2 group longer than the preceding one. The contribution of the $C-(C)_2(H)_2$ group to the enthalpy of formation of compounds in the liquid and gaseous states¹ and to the enthalpy of vaporization is -24, -20, and 4.8 kJ/mol according to the Benson [11], Kizin *et al.* [12], and Lebedev–Miroshnichenko [13] schemes, respectively. Along the homologous series **I–III**, changes in the enthalpy of formation of the liquid and gaseous compounds and in their enthalpies of vaporization are -30 and -31, -26 and -25, and 4.4 and 6.0 kJ/mol; they coincide with the corresponding contribution of the CH_2 group to within

¹ Parenthesized atoms in the denotations of group contributions describe the environment of the central atom, and subscripts indicate the number of atoms of a given kind in the environment; C_t is the C atom at the triple bond, and C_b is the C atom in the benzene ring.

measurement errors. This is evidence that the enthalpy properties of the compounds under study are additive. Comparing the structures of compounds **I–III** with those from [1] pairwise shows that the former can be obtained from the latter and vice versa by the formal replacement of the trimethylsilyl and triphenylsilyl groups. The differences of the enthalpies of formation for the pairs of substances that we compare are 492, 481, and 471 kJ/mol, respectively. These values coincide to within the error of enthalpy determinations and substantiate the additive character of enthalpy properties.

This circumstance allows us to calculate the contributions of silicon-containing groups, which have been unknown in additive schemes. We were able to determine such contributions only to the enthalpy of formation of compounds in the gas phase for the Benson scheme, which contains the most complete list of the contributions of groups present in the compounds studied. In addition, we used the increments (kJ/mol) $O_{-}(C)(O), -19.32; C_{-}(O)(C)_{2}(C_{t}),$ -9.55; and C-(Si)(H)₃, -42.17 kJ/mol. The increments were calculated from the enthalpies of formation reliably determined for fairly large numbers of compounds [14–16]. Substituting the group contributions into the enthalpies of formation of compounds I-III yielded an overdetermined system of three equations. Solving this system allowed us to obtain the contribution of the $(C_t) = CSi(C_b)_3$ fragment equal to 128 kJ/mol. The contribution of the Si-(O)(C_b)₃ group (-211 kJ/mol) was found by substituting the corresponding group contributions to the enthalpy of formation of **IV**.

The peroxide bond energy was calculated by constructing a system of peroxide homolytic decomposition reactions and the corresponding system of thermochemical equations,

$$R_1OOR_2 \longrightarrow R_1O' + R_2O',$$

$$D(O-O) = \Delta_f H^{\circ}(R_1O') + \Delta_f H^{\circ}(R_2O') - \Delta_f H^{\circ}(I);$$

$$R_3OOR_2 \longrightarrow R_3O' + R_2O',$$

$$D(O-O) = \Delta_f H^{\circ}(R_3O') + \Delta_f H^{\circ}(R_2O') - \Delta_f H^{\circ}(II);$$

$$R_4OOR_2 \longrightarrow R_4O' + R_2O',$$

$$D(O-O) = \Delta_f H^{\circ}(R_4O') + \Delta_f H^{\circ}(R_2O') - \Delta_f H^{\circ}(III);$$

$$R_1OOR_5 \longrightarrow R_1O + R_5O$$
,

$$D(O-O) = \Delta_{f}H^{\circ}(R_{1}O^{\bullet}) + \Delta_{f}H^{\circ}(R_{5}O^{\bullet}) - \Delta_{f}H^{\circ}(IV),$$

where $R_1O' = Me_3CO'$, $R_2O' = Ph_3SiC \equiv CCMe_2O'$, $R_3O' = t-C_5H_{11}O'$, $R_4O' = t-C_6H_{13}O'$, and $R_5O' = (Ph)_3SiOCH_2C \equiv CCMe_2O'$.

The known enthalpies of formation $\Delta_f H^\circ$ of the radicals produced in the dissociation of the compounds at the peroxide bond are -96.2 kJ/mol for Me₃CO[•] [17] and -121.3 kJ/mol for $t-C_5H_{11}O'$ [18]. These data were insufficient for solving the above system of equations. However, considering the structural similarity of compounds I-III and compounds studied in [1] and taking into account that differences in structure between IV and the other compounds begin with the atoms separated by four bonds from the peroxide bond, we may quite rigorously use the peroxide bond energy [1] equal to 148 kJ/mol. Solving the system of equations then yields the enthalpies of formation $\Delta_{f}H^{\circ}$ of the radicals $Ph_3SiC \equiv CCMe_2O'$, $(Ph)_3SiOCH_2C \equiv CC(CH_3)_2O'$, and CH₃(CH₂)₂(CH₃)₂CO[•] (482, 125, and -147 kJ/mol, respectively). The enthalpy of formation of the last radical obtained in [1] was -131 kJ/mol. We recommend using the average value, -139 kJ/mol.

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