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Thermochemical Study of Gaseous Salts of Oxygen-containing Acids: XXIII.[1] Molecules MnB_2O_4, $MnNbO_2$, $MnNbO_3$ and $MnTiO_3$

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Abstract—Gas-phase equilibria involving manganese borate, niobates, and titanate were studied. Standard enthalpies of formation and atomization of the gaseous molecules MnB_2O_4, $MnNbO_2$, $MnNbO_3$, and $MnTiO_3$ were determined.

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It has been shown earlier [2–4] that gaseous manganese(II) oxide has strongly pronounced basic properties and acts as cation-forming oxide in the synthesis of gaseous salts of oxygen-containing acids from gaseous oxides. When studying $MnCO_3$ vaporization from molybdenum and tungsten effusion cells it has been found [2, 3] that the vapor at temperatures higher than 1800 K in addition to monatomic manganese, oxygen, and MnO contains gaseous molybdenum and tungsten oxides MO_2 and MO_3 (M = Mo, W) that are products of the manganese oxide reaction with a cell material, and also manganese molybdates and tungstates $MnMO_4$ and $MnMO_3$. The application of the double two-temperature cell technique [5] providing conditions for the coexistence of manganese(II) oxide with phosphorus oxides PO and PO_2 [4] allowed us to obtain gaseous manganese phosphates $MnPO_2$ and $MnPO_3$.

In this work we have selected as objects of the study gaseous manganese borates, niobates, and titanate that, according to the calculations of reactivity [6] and the estimate of thermal stability of MnO, BO, B_2O_3, NbO, NbO_2, and TiO_2 oxides [7], should be stable in a sufficiently wide temperature range.

Borates occupy a special position in the series of gaseous salts as they form the most numerous and well studied group of compounds. It originates from the unique features of boron oxide B_2O_3 with respect to the high-temperature chemistry, as it is thermally stable in a wide temperature range and dissociates into BO and oxygen only at temperatures higher than 2000 K [7, 8]. Possessing amphoteric properties with acid properties predominating, B_2O_3 can participate in the formation of borates as an anion-forming oxide. The examination of acid–base properties and relative volatility of MnO and B_2O_3 [9] has shown that crystalline manganese borate MnB_2O_4 should be sufficiently thermally stable and pass into vapor on heating either without decomposition or with a partial dissociation. Gaseous salts $MnNbO_2$, $MnNbO_3$, and $MnTiO_3$, by analogy with chromium gaseous salts [10], can be obtained by joint evaporating of the manganese oxide MnO with Nb_2O_5 or Ti_3O_5 from molybdenum or tungsten effusion cells.

We synthesized manganese borate in aqueous solution [reaction (1)].

$$(CH_3COO)_2Mn + 2NaBO_2$$

$$= MnB_2O_4\downarrow + 2CH_3COONa. \quad (1)$$

Manganese borate MnB_2O_4 was the main phase in the resulting sample, whereas the content of admixtures did not exceed 10%. To obtain manganese borate with a lower impurity level we have made an attempt to synthesize it by a ceramic method using

[1] For communication XXII, see [1].

reaction (2) of boron oxide B_2O_3 with manganese carbonate $MnCO_3$.

$$MnCO_3 + B_2O_3 = MnB_2O_4 + CO_2\uparrow. \quad (2)$$

To study processes of manganese metaborate vaporization, we have chosen a sample synthesized by Eq. (1).

Ionic currents of Mn^+, $B_2O_3^+$, MnO^+, $MnBO_2^+$, and $MnB_2O_4^+$ were recorded in the mass spectrum of vapor over manganese borate evaporating from a tungsten cell. Their relative intensities depended on temperature and the evaporation duration. To determine molecular precursors of the ions in the mass spectrum, we have measured their appearance energies. In none of the ionization effectiveness curves noticeable breaks, which would point to a double nature of the ion formation, were observed. The appearance energies of the ions measured with an accuracy of ±0.3 eV at 1657 K were: $MnB_2O_4^+$ 11.5, $MnBO_2^+$ 8.2, Mn^+ 7.3, MnO^+ 7.8, and $B_2O_3^+$ 14.0 eV. The appearance energies of Mn^+, MnO^+, and $B_2O_3^+$ ions coincided with the ionization energies of the corresponding molecules [11] within the limits of experimental error. The appearance energies of the $MnB_2O_4^+$ and $MnBO_2^+$ ions have been measured for the first time. The comparison of the obtained values with the appearance energies of borates of alkali-earth metals $MB_2O_4^+$ and MBO_2^+ (M = Ca, Sr, Ba) [8, 12] suggests that they have a molecular origin and appear as a result of direct ionization of the corresponding molecules.

The examination of the vapor mass spectra and the values of the appearance energies of ions has allowed us to assert that the vapor phase over the substance under study consists of a mixture of boron and manganese oxides, monatomic manganese, and two manganese borates, MnB_2O_4 and $MnBO_2$. To calculate standard enthalpies of formation and atomization of gaseous manganese borate MnB_2O_4 by Eq. (3), we have determined the temperature dependences of equilibrium constants of reactions (4) and (5).

$$\frac{d\ln K_p}{dT} = \frac{\Delta_r H^0(T)}{RT^2}, \quad (3)$$

$$MnO\ (gas) + B_2O_3\ (gas) = MnB_2O_4\ (gas), \quad (4)$$

$$MnB_2O_4\ (cr) = MnB_2O_4\ (gas). \quad (5)$$

In Eq. (3) $\Delta_r H^0(T)$ and K_p are the enthalpy and equilibrium constant of the reaction at a temperature T, and R is the gas constant. We did not determine absolute values of partial pressures for the vapor of molecular forms, and substituted the values of the intensities of MnO^+, $B_2O_3^+$, and $MnB_2O_4^+$ ionic currents proportional to them in the expression for the equilibrium constant [13]. Thus the results of determination of enthalpies of reactions (4) and (5) are presented in Table 1.

The data for gaseous boron and manganese oxides required for the calculations were taken from the handbooks [14, 15], and thermodynamic functions of gaseous manganese metaborate were calculated by the statistical thermodynamics method in the approximation "rigid rotator–harmonic oscillator" on the basis of molecular parameters calculated by quantum chemistry methods. The temperature dependence of heat capacity for crystalline MnB_2O_4 was determined by Landia's method [16]. The quantum-chemical calculations were carried out using the GAMESS program complex [17] both by the ab initio self-consistent field method (SCF) and by the density functional method (DFT) in the B3LYP approximation [18, 19] with the 6-31G* basis. It was found that, similarly to borates of alkaline-earth metals [20], gaseous manganese borate MnB_2O_4 has a linear structure of the $D_{\infty h}$ symmetry. A sextet state has a minimal energy, and the spin density is concentrated

Table 1. Values of enthalpies of reactions (4), (5)

Experiment no.	Number of measurements	Temperature range (average temperature of experiment), K	$-\Delta_r H^0(T)$, kJ		$-\Delta_r H^0(298)$, kJ	
			(4)	(5)	(4)	(5)
1	27	1510–1690 (1600)	435±13	467±16	456±13	549±16
2	19	1526–1727 (1627)	441±23	502±17	463±23	–
3	26	1535–1687 (1611)	441±29	–	463±29	–
4	22	1579–1735 (1657)	434±28	485±21	456±29	
Average value:					459±23	549±16

exclusively on the manganese atom. The values of interatomic distances determined by the DFT calculations agree well with the data of [20]: r(Mn–O) 1.82, r(B–O) 1.30, and r(B=O) 1.22 Å; normal vibration frequencies: 2102, 2081, 1198(2), 548, 526(2), 525(2), 354, 218(2), 149(2), 30(2) cm^{-1}.

The combination of the weighted average value of the enthalpy of reaction (4) based on four experiments and reduced to 298 K and of the values of standard enthalpies of formation of gaseous manganese(II) and boron oxides [14, 15] allowed us to determine the standard enthalpies of formation and atomizations of MnB_2O_4(gas): –1132±24 and 3532±25 kJ mol^{-1}, respectively.

The absence of the standard enthalpy of formation of the crystalline manganese borate MnB_2O_4 in the reference publications did not allow us to determine the enthalpy of formation of gaseous MnB_2O_4 by reaction (5). The obtained value of the enthalpy of this salt sublimation together with the enthalpy of the gaseous salt formation determined by Eq. (4) have allowed us to estimate the standard enthalpy of formation of crystalline manganese borate at (–1681 kJ mol^{-1}).

The standard enthalpy of the formation of gaseous manganese borate $MnBO_2$ that was found in vapor over the studied samples can be determined if the constant of gas-phase reaction (6) is calculated.

$$MnO + BO = MnBO_2. \quad (6)$$

Unfortunately, we failed to create conditions for the coexistence of MnO and BO in vapor.

Gaseous niobates $MnNbO_2$ and $MnNbO_3$ and manganese titanate $MnTiO_3$ were obtained by joint evaporation of manganese carbonate $MnCO_3$ with niobium and titanium oxides, Nb_2O_5 Ti_3O_5, respectively, from ordinary molybdenum and tungsten cells. It is known that Nb_2O_5 and Ti_3O_5 oxides dissociate on heating to form gaseous oxides XO and XO_2 (hereinafter X = Nb, Ti) and oxygen [7]. At a temperature about 1200–1300 K manganese carbonate dissociated to form crystalline MnO and gaseous CO_2. To obtain gaseous manganese niobates, we have applied both an ordinary one-temperature cell and a double two-temperature cell [21] for creating optimal conditions for the coexistence of gaseous manganese and niobium oxides.

We recorded Mn^+, MnO^+, XO^+, XO_2^+, $MnNbO_2^+$, $MnNbO_3^+$, and $MnTiO_3^+$ ions in the mass spectra of vapor over the systems MnO–Nb_2O_5 and MnO–Ti_3O_5. The relative intensity of Mn^+ and MnO^+ ions decreased during the experiment, which is connected with a higher volatility of MnO as compared to niobium and tantalum oxides.

To determine the nature of the ions in the mass spectra we have measured their appearance energies. The resulting values for Mn^+, MnO^+, XO^+, and XO_2^+ coincided with the ionization energies of Mn, MnO, XO, and XO_2 within the limits of experimental errors [11]. The appearance energies of $MnTiO_3^+$ and $MnNbO_3^+$ ions (9.6 ± 1 and 10.0 ± 1 eV, respectively) have been measured for the first time. The values obtained agree well by the order of magnitude with those for niobates and titanates of alkaline-earth metals [22, 23] suggesting the molecular origin of the $MnTiO_3^+$ and $MnNbO_3^+$ ions. We did not measure the appearance energy of the $MnNbO_2^+$ ion because of the low intensity of the corresponding ionic current but by analogy with the mass spectra of vapor over niobates of alkaline-earth metals [22], we also assigned these ions to the direct ionization of $MnNbO_2$ molecules.

The examination of the vapor mass spectra, their temperature dependence, and the appearance energies of ions allows us to assert that the vapors over the systems MnO–Nb_2O_5 and MnO–Ti_3O_5 contain manganese, niobium, and titanium oxides, monatomic manganese, and also gaseous manganese niobates and titanate. We calculated partial pressures of the vapor components by the method of comparing ionic currents using gold as a pressure interior reference [24].

The values of enthalpies of reactions (7) and (8) were determined by Eq. (9).

$$MnO + XO_2 = MXO_3, \quad (7)$$

$$MnO + NbO = MnNbO_2, \quad (8)$$

$$\Delta H^0(0) = T[\Delta_r\Phi^0(T) - R\ln K_p(T)]. \quad (9)$$

Here $\Delta_rH^0(0)$ and $\Delta_r\Phi^0(T)$ are changes in the enthalpy and reduced Gibbs energy of the reaction at temperatures 0 and T K, respectively, R is the gas constant, K_p is the equilibrium constant of the reaction. Partial pressures of the main molecular forms of vapor over the studied systems and the enthalpies of reactions (7), (8) are presented in Table 2.

The values of enthalpies of reactions were reduced to the standard temperature 298 K using the reference data [14, 15] for gaseous oxides, and the data calculated by the statistical thermodynamics method in

the approximation "rigid rotator–harmonic oscillator" for gaseous salts. Structures of the molecules $MnNbO_3$, $MnNbO_2$, and $MnTiO_3$, similarly to the structures offered in [22, 23, 25, 26] for titanates and niobates of europium and alkaline-earth metals, were assigned to the C_{2v} symmetry.

Interatomic distances in anionic groups were taken from europium titanate and niobates [25, 26]: r(Ti–O) 1.83, r(Ti=O) 1.69, r(Nb–O) 1.91, and r(Nb=O) 1.77 Å, angle OXO 120°.

To check the chosen molecular parameters we have carried out quantum-chemical calculations of structure and vibration spectrum of the molecule $MnTiO_3$. The C_s-symmetry structure is minimal in energy for manganese titanate. The angle $O_{term}TiMn$ in this structure is 154°, the interatomic distances are as follows: r(Ti–O) 1.84, r(Ti=O) 1.63, and r(Mn–O) 1.95 Å, the angle $O_{cycl}TiO_{cycl}$ 93°; frequencies of normal vibrations of the TiO_3 group are: 963, 689, 673, 573, 306, and 262 cm^{-1}. Thermodynamic functions of gaseous manganese titanate calculated on the basis of the molecular parameters given in [25] and on the basis of our quantum-chemical calculations differ from each other by no more than 2 J mol^{-1} K^{-1} at 2000 K. It allowed us to use the thermodynamic functions of manganese niobates and titanate calculated on the basis of the molecular parameters given in [22, 23, 25, 26]. Frequencies of the normal vibrations of the groups NbO_2 and NbO_3 were taken from europium niobates [26]; NbO_3: 928, 795, 730, 520(2), and 350; NbO_2: 668, 660, and 292; TiO_3: 961, 907, 705, 349, 331, and 304 cm^{-1}.

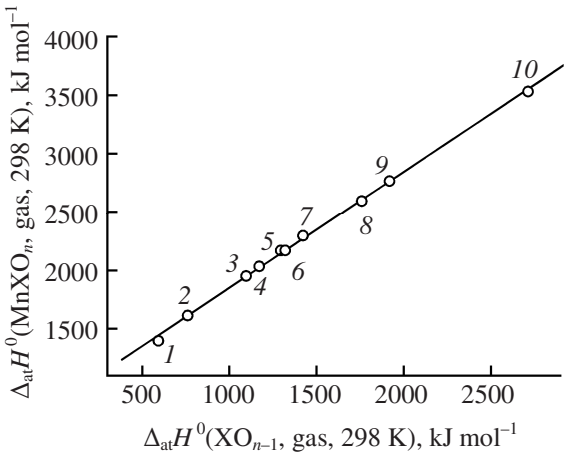
The interatomic distance r(Mn–O) was accepted to be 1.97 Å [3]. The interatomic distance manganese-oxygen estimated as the sum of the ionic radius of Mn^{2+} [27] and effective ionic radius of oxygen [28] is 1.96 Å that confirms the validity of our choice. The frequencies of normal vibrations of the manganese-oxygen bond in the cation (456, 196, and 118 cm^{-1}) were taken from the manganese molybdate molecule [3].

The calculated standard enthalpies of formation and atomization of gaseous manganese niobates and titanate are presented in Table 3.

As shown earlier, the enthalpies of atomization of isocation gaseous salts linearly depend on the enthalpies of atomization of the corresponding gaseous anion-forming oxides. The dependence can be presented in the form of Eq. (10).

Table 2. Partial pressures of vapor of molecular forms over the systems $MnO–Ti_3O_5$ and $MnO–Nb_2O_5$ and enthalpies of reactions (7), (8)

T, K	p_i, atm			$-\Delta_r H^0(0)$, kJ (7)
	MnO (×10^7)	TiO_2 (×10^6)	$MnTiO_3$ (×10^8)	
2177	73.12	3.33	11.59	493.6
2339	5.98	5.24	0.35	500.7
2356	3.89	4.92	0.18	500.4
2304	9.15	3.19	0.52	502.4
2286	10.94	21.45	3.69	497.4
Average value:				498.9±3.5
	MnO (×10^7)	NbO (×10^7)	$MnNbO_2$ (×10^9)	$-\Delta_r H^0$ (0), kJ (8)
2039	4.02	1.77	1.18	477.5
2033	4.13	2.17	1.76	479.1
2034	3.40	2.13	1.18	476
2035	3.28	2.05	1.18	477.5
2031	2.67	2.11	0.59	467.9
Average value:				476±4
	MnO (×10^7)	NbO_2 (×10^6)	$MnNbO_3$ (×10^8)	$-\Delta_r H^0$ (0), kJ (8)
1948	3.25	1.12	1.06	504.1
1986	3.11	2.01	3.78	525.6
2009	2.56	3.67	1.09	504.1
2012	1.31	3.88	0.68	507.2
2034	4.01	1.40	2.05	529.5
2027	3.76	1.20	1.54	526.5
2034	3.77	1.51	1.67	525.8
2039	4.02	1.32	2.41	529.4
2033	4.13	1.74	2.02	518.8
2034	3.40	1.70	2.15	524.2
2035	3.28	1.49	1.90	524.3
2031	2.67	1.58	1.89	525.6
2034	2.55	1.51	0.53	513
2033	2.43	1.55	0.28	502.2
2027	2.42	1.50	0.40	507.6
2032	2.31	1.55	0.28	502.9
2030	2.18	1.51	0.15	493.5
2080	2.98	3.01	1.32	525.3
2060	2.59	3.10	0.67	510.7
2079	2.24	2.97	0.41	510.2
2047	1.24	3.33	0.62	517.5
2047	1.06	3.37	0.33	509.4
2260	0.91	30.96	0.13	503.7
2158	11.00	19.25	1.63	491.5
2157	7.85	18.97	1.09	490.3
2159	7.34	18.72	0.82	487.1
2155	54.87	21.48	27.15	510.5
2148	36.38	43.57	24.83	502.0
2163	31.61	60.97	15.59	493.5
2162	20.10	74.33	14.09	496.0
2169	18.01	82.02	11.24	493.7
2170	9.61	84.55	9.75	502.1
2165	8.39	89.31	7.11	496.8
2164	5.75	101.70	6.35	499.0
Average value:				509±13



Dependence of atomization enthalpies of gaseous Mn salts on atomization enthalpies of anion-forming oxides: (*1*) $MnPO_2$; (*2*) $MnNbO_2$; (*3*) $MnPO_3$; (*4*) $MnMoO_3$; (*5*) $MnWO_3$; (*6*) $MnTiO_3$; (*7*) $MnNbO_3$; (*8*) $MnMoO_4$; (*9*) $MnWO_4$; and (*10*) MnB_2O_4.

$$\Delta_{at}H^0(M_mXO_n, \text{gas}, 298) = k\Delta_{at}H^0(XO_{n-1}, \text{gas}, 298) + b. \quad (10)$$

The results obtained in combination with the data of [2–4] have allowed us to construct such dependence for gaseous manganese salts (see the figure). The coefficients k and b are 0.996 ± 0.014 and 853 ± 21, respectively. The high correlation coefficient (0.9993) and the standard deviation value (24.82) confirm the validity of the results obtained and allow us to reliably estimate the enthalpies of atomization and formation of yet not investigated manganese gaseous salts. In particular, the standard enthalpy of formation of the $MnBO_2$(gas) was estimated by Eq. (10) at –312 kJ mol^{-1}.

EXPERIMENTAL

The study was carried out on an MS-1301 mass-spectrometer at ionizing voltage of 25 V. The salt MnB_2O_4 and also mixtures of manganese carbonate with niobium and titanium oxides were evaporated from molybdenum and tungsten effusion cells heated by electron impact. The temperature was measured by an EOP-66 optical pyrometer. The equipment was beforehand calibrated by the pressure of CaF_2 vapor [14]. Appearance energies of ions were measured by the vanishing ionic current method using gold with ionization energy of 9.22 eV as a reference[11].

Table 3. Standard enthalpies of formation and atomization of gaseous manganese salts

Molecule	$-\Delta_fH^0(298)$, kJ mol^{-1}	$\Delta_{at}H^0$ (298), kJ mol^{-1}
MnB_2O_4	1132±24	3532±25
$MnBO_2$	312 (estimate)	1659 (estimate)
$MnNbO_3$	551±17	2306±20
$MnNbO_2$	105±17	1610±20
$MnTiO_3$	662±21	2167±21

Manganese borate. *a*. Equivalent amounts of manganese acetate and $NaBO_2$ were mixed. The initial components and sodium acetate are readily soluble in water. Manganese borate precipitated during the reaction. The precipitate formed was filtered, washed with distilled water several times, and dried in air at room temperature. Residual water was removed by heating the sample up to 400 K in a drying box, and the sample was analyzed by the X-ray method.

b. Finely ground reagents $MnCO_3$ and BrO_3, taken in a molar ratio 1:1, were ground together in an agate mortar, placed in a platinum crucible, and slowly heated in air in a muffle furnace up to 1300 K. Visual observation has shown that the mixture was not fused at this temperature, and its color changed from pale-brown up to brownish black, which is caused by the manganese(II) oxidation up to manganese(IV) by air oxygen. The X-ray phase analysis has confirmed the presence of manganese oxide MnO_2 in the condensed phase.

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