

Thermochemical Study of Gaseous Salts of Oxygen-Containing Acids: XVII.¹ Magnesium Salts

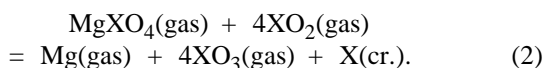
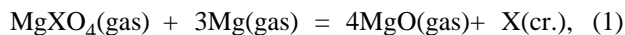
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Abstract—Gas-phase reactions involving magnesium oxide and magnesium salts were studied; the standard enthalpies of formation and atomization of these salts were determined.

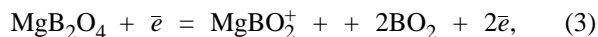
Data on vaporization and thermodynamic properties of oxide systems containing magnesium oxide are important for metallurgy and for technology of magnesia-based refractory and ceramic materials. When heated, MgO mainly vaporizes in the form of atomic Mg and oxygen; the relative content of MgO in the vapor is less than 1% [2–4]. Verhaegen *et al.* [5] showed that, when heated in molybdenum or tungsten cells, magnesium oxide reacts with the cell material to give thermally stable magnesium molybdates and tungstates, which were detected in the vapor phase. Determination of the enthalpies of reactions (1) and (2) involving gaseous magnesium molybdates and tungstates allowed estimation of the standard enthalpies of formation and atomization of $\text{MgMoO}_4(\text{gas})$ and $\text{MgWO}_4(\text{gas})$, equal at 0 K to 2692 ± 96 and 2856 ± 96 kJ mol^{−1}, respectively (here and hereinafter, X = Mo, W).



Kazenas *et al.* [6–8] studied the vaporization of magnesium molybdate and tungstate. They showed that these salts vaporize with partial dissociation to molybdenum or tungsten oxides, atomic magnesium, and oxygen. They determined the heats of vaporization and the standard enthalpies of formation of gaseous MgMoO_4 and MgWO_4 : at 0 K, -911 ± 30 and -867 ± 20 kJ mol^{−1}, respectively.

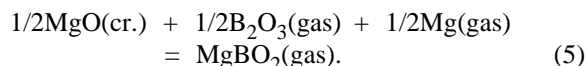
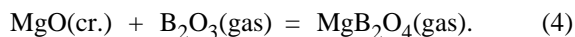
Gusarov [9] studied the vaporization products in the system $\text{MgO}-\text{B}_2\text{O}_3$ (94 mol % MgO). He identified in the mass spectra the Mg^+ , MgBO_2^+ , B_2O_3^+ , and MgB_2O_4^+ ions. Measurement of the appearance poten-

tials of the ions showed that the Mg^+ , B_2O_3^+ , and MgB_2O_4^+ ions originate from direct ionization of the corresponding neutral species. The appearance potentials of MgBO_2^+ and Mg_2O_4^+ were 8.4 and 12.0 eV, respectively. The MgBO_2^+ ion originates both from the direct ionization of the corresponding molecule and from the dissociative ionization of the MgB_2O_4 molecule [Eq. (3)].



which gives rise to an inflection in the curve of the MgBO_2^+ ionization efficiency.

Determination of the equilibrium constants of reactions (4) and (5) allowed calculation of the standard enthalpies of formation of gaseous magnesium meta-borates MgB_2O_4 and MgBO_2 : -1305.4 ± 16.7 and -502 ± 12.6 kJ mol^{−1} at 0 K, respectively.



In this study, we examined gas-phase reactions involving magnesium oxide and typical anion-forming oxides PO , PO_2 , MoO_2 , MoO_3 , WO_2 , and WO_3 , thermally stable in a wide temperature range, and determined the standard enthalpies of formation of six gaseous magnesium salts: MgPO_2 , MgPO_3 , MgMoO_3 , MgMoO_4 , MgWO_3 , and MgWO_4 . To prepare gaseous magnesium molybdates and tungstates, by analogy with gaseous molybdates and tungstates of other alkaline-earth metals and beryllium [10, 11], we studied the vaporization of MgO from molybdenum and tungsten effusion cells. In the mass spectra of the vapor, we detected the Mg^+ , MgO^+ , XO^+ , XO_2^+ , XO_3^+ , MgXO_3^+ , and MgXO_4^+ peaks; their relative intensities depended on the vaporization temperature and time.

¹ For communication XVI, see [1].

To prepare gaseous magnesium phosphates, we provided conditions for the coexistence of magnesium and phosphorus oxides in the vapor, using a double two-temperature cell [12]. As shown in [13], when vaporized from a one-temperature cell, magnesium phosphates fully dissociate with vaporization of phosphorus oxides and accumulation of magnesium oxide in the condensed phase. In the mass spectrum of the vapor over MgO evaporated from a two-temperature Mo cell in the range 1740–1940 K, we detected the ion currents of Mg^+ , MgO^+ , PO^+ , PO_2^+ , MgPO_2^+ , and MgPO_3^+ ; their relative intensities depended on the vaporization temperature and time. Also, in the mass spectrum of the vapor we detected the MoO^+ , MoO_2^+ , MoO_3^+ , MgMoO_3^+ , and MgMoO_4^+ ions formed by ionization of the products of the reaction of MgO with the cell material.

To determine the origin of the ions in the vapor mass spectrum, we measured their appearance potentials and obtained the following results (eV, ± 0.5): 7.6 (Mg^+), (MgO^+), 9.5 (MoO_2^+), 12.1 (MoO_3^+), 10.0 (WO_2^+), 11.6 (WO_3^+), 11.7 (PO_2^+), 9.2 (PO^+ , MgMoO_4^+), 10.0 (MgWO_4^+ , MgPO_2^+ , MgPO_3^+). For the Mg^+ , MgO^+ , XO_2^+ , XO_3^+ , PO^+ , and PO_2^+ ions, the appearance potentials coincide within the determination error with the ionization potentials of the corresponding molecules [14], and for MXO_4^+ they are comparable with the appearance potentials of the corresponding molecular ions of alkaline-earth metal molybdates, tungstates, and phosphates [10, 15, 16].

The mass spectra of the vapor and the appearance potentials of the ions indicate that the vapor over MgO vaporized from molybdenum or tungsten cells consists of MgO, molybdenum (or tungsten) oxides XO_2 and XO_3 , oxygen, atomic Mg, and magnesium molybdates (or tungstates) MgXO_3 and MgXO_4 . The vapor over the system MgO-MgHPO_4 vaporized from a two-temperature cell consists of MgPO_3 , MgPO_2 , MgO, PO_2 , and PO molecules and of atomic Mg.

To determine the standard enthalpies of formation and atomization of gaseous magnesium phosphates, molybdates, and tungstates, we measured the equilibrium constants of gas-phase reactions (6)–(9):



The partial pressures of molecular species in the vapor were determined by comparison of the ion currents, using gold as a pressure reference [17]. To eval-

uate the contribution of the dissociative ionization to the total intensities of the XO_2^+ and PO^+ ion currents, we measured the $\text{XO}_3^+/\text{XO}_2^+$ and $\text{PO}_2^+/\text{PO}^+$ intensity ratios at an ionizing voltage of 25 V, exceeding the ionization threshold by 3 V. The measurement results were taken into account when determining the partial pressures of PO, PO_2 , XO_2 , and XO_3 . The correction for the fragmentation of $\text{Mg}^+(\text{MgO})$ and the salts was not made, because at low ionizing voltages the MgO^+ , MgPO_2^+ , MgPO_3^+ , and MgXO_4^+ ion currents were too low to be measured. The errors thus introduced into the measured equilibrium constants of the gas-phase reactions were discussed in detail in [1]. The enthalpies of gas-phase reactions (6)–(9) were determined by Eq. (10):

$$\Delta_r H^0(0) = T[\Delta_r \Phi^0(T) - R \ln K_{\text{eq}}(T)]. \quad (10)$$

For reaction (8), we measured the temperature dependence of the equilibrium constant in a fairly wide temperature range and determined the enthalpy of the reaction $\Delta_r H(T)$ for the midpoint of the examined temperature range by Eq. (11):

$$\Delta_r H^0(T) = -R \frac{\partial \ln K_{\text{eq}}(T)}{\partial (1/T)}, \quad (11)$$

where $\Delta_r H^0(0)$, $\Delta_r H^0(T)$, and $\Delta_r \Phi^0(T)$ are the enthalpy and reduced Gibbs potential of the reaction at 0 K and T , respectively; R is the gas constant; and K_{eq} is the reaction equilibrium constant.

The partial pressures and enthalpies of reactions (6)–(9) are listed in Tables 1 and 2. The enthalpy of reaction (8) determined by Eq. (11) appeared to be -411 ± 34 kJ at 1845 K (middle of the examined temperature range). Recalculation of this quantity to the standard temperature of 0 K gives -400 ± 34 kJ. The weighted average enthalpy of reaction (8), determined by Eqs. (10) and (11), is -426 ± 12 kJ. The enthalpy of reaction (9) was determined only by Eq. (10), although the partial pressures of species participating in reaction (9) were determined in the same temperature range as for reaction (8). The poor measurement accuracy (due to low intensity of the MgPO_3^+ ion current) did not allow $\Delta_r H^0(T)$ to be determined with an error smaller than ± 70 kJ.

To recalculate the enthalpies of gas-phase reactions (6)–(9) to the standard temperature of 298 K and to determine the standard enthalpies of formation and atomization of the gaseous salts, we took the reference thermodynamic data [18] for gaseous atoms and oxides of Mg, Mo, W, and P. The thermodynamic functions of formation of gaseous magnesium salts were calculated by the method of statistical thermodynamics in the rigid rotator–harmonic oscillator

Table 1. Partial pressures of molecular species in the vapor over MgO vaporized from molybdenum and tungsten cells and enthalpies of the reactions involving MgO

| T, K | p_i , atm | | | | | $-\Delta_f H^0(0)$, kJ | |
|---------|-----------------------|------------------------------------|------------------------------------|--------------------------------------|--------------------------------------|-------------------------|-----------------|
| | MgO ($\times 10^8$) | WO ₂ ($\times 10^6$) | WO ₃ ($\times 10^6$) | MgWO ₃ ($\times 10^8$) | MgWO ₄ ($\times 10^8$) | Eq. (7) | Eq. (6) |
| 2042 | 8.26 | 1.05 | 0.96 | 1.70 | 1.70 | 488.6 | 484.1 |
| 2073 | 12.92 | 1.58 | 1.45 | 2.30 | 2.15 | 486.5 | 480.8 |
| 2136 | 22.67 | 3.55 | 2.96 | 4.73 | 4.44 | 485.7 | 485.6 |
| 2148 | 28.23 | 4.63 | 3.65 | 7.14 | 4.46 | 487.1 | 480.8 |
| 2197 | 18.88 | 20.42 | 19.92 | 12.17 | 8.67 | 488.3 | 480.3 |
| 2111 | 8.68 | 3.26 | 3.98 | 3.89 | 1.46 | 494.8 | 472.0 |
| 2130 | 6.37 | 4.56 | 5.06 | 5.89 | 3.44 | 506.2 | 492.7 |
| 2120 | 6.74 | 4.75 | 5.18 | 5.86 | 3.42 | 502.0 | 488.9 |
| 2093 | 9.33 | 5.07 | 5.23 | 7.08 | 4.42 | 496.2 | 481.3 |
| 2111 | 7.6 | 4.05 | 4.73 | 6.54 | 4.01 | 502.5 | 489.1 |
| 2120 | 9.08 | 5.33 | 5.84 | 8.36 | 4.48 | 501.0 | 486.3 |
| 2117 | 6.89 | 4.55 | 5.42 | 7.16 | 4.92 | 505.2 | 493.4 |
| 2135 | 7.69 | 5.56 | 6.15 | 6.62 | 4.06 | 502.6 | 490.0 |
| 2153 | 7.01 | 15.64 | 23.17 | 9.10 | 5.91 | 495.7 | 478.8 |
| Average | | | | | | 495.9 \pm 7.5 | 484.6 \pm 6.0 |
| | MgO ($\times 10^8$) | MgO ₂ ($\times 10^5$) | MgO ₃ ($\times 10^5$) | MgMoO ₃ ($\times 10^8$) | MgMoO ₄ ($\times 10^8$) | Eq. (9) | Eq. (8) |
| 2196 | 7.26 | 6.65 | 10.00 | 4.61 | 6.35 | 486.4 | 460.9 |
| 2189 | 6.82 | 6.40 | 10.20 | 4.46 | 4.22 | 486.1 | 452.8 |
| 2079 | 2.02 | 1.76 | 3.44 | 0.32 | 4.01 | 459.6 | 468.8 |
| 2089 | 1.96 | 1.36 | 2.53 | 0.37 | 3.65 | 469.2 | 475.3 |
| 2144 | 3.53 | 2.37 | 4.01 | 1.05 | 3.74 | 479.8 | 469.6 |
| 2147 | 3.78 | 2.51 | 4.39 | 1.33 | 3.75 | 482.5 | 467.4 |
| 2141 | 3.52 | 2.43 | 4.01 | 1.08 | 2.49 | 479.2 | 461.8 |
| 2080 | 1.34 | 1.33 | 2.56 | 0.37 | 2.76 | 473.9 | 474.8 |
| 2128 | 1.82 | 2.51 | 4.43 | 1.14 | 2.37 | 488.4 | 468.0 |
| 2117 | 1.77 | 2.37 | 4.29 | 1.09 | 2.36 | 486.5 | 466.6 |
| 2121 | 1.70 | 2.38 | 4.30 | 1.09 | 2.36 | 488.0 | 468.1 |
| 2120 | 1.64 | 2.07 | 3.73 | 0.89 | 2.36 | 487.5 | 471.1 |
| 2147 | 2.07 | 2.16 | 3.55 | 1.03 | 1.37 | 491.3 | 464.0 |
| 2094 | 0.85 | 0.70 | 2.74 | 0.08 | 1.75 | 476.1 | 476.9 |
| 2103 | 0.92 | 1.42 | 2.64 | 0.39 | 1.05 | 485.5 | 469.2 |
| 2133 | 1.36 | 2.18 | 3.57 | 0.99 | 2.13 | 494.6 | 476.2 |
| 2161 | 1.38 | 2.28 | 3.86 | 0.90 | 2.52 | 498.4 | 483.8 |
| Average | | | | | | 483.1 \pm 7.2 | 469.1 \pm 9.4 |

approximation. For the MgXO₃ and MgPO₃ molecules, we assumed the cyclic structure of C_{2v} symmetry, similar to the structures suggested in [19–21] for EuMoO₃ and alkali metal phosphate molecules. The interatomic distances are as follows: $r(\text{Mo}=\text{O})$ 1.90, $r(\text{Mo}-\text{O})$ 1.80 Å; $\angle\text{OMoO}$ 116°; the normal mode frequencies for the MoO₃ group (883, 589, 557, 545, 508, 458 cm⁻¹) were taken the same as for the EuMoO₃ molecule. To calculate the enthalpy of reaction (8) involving MgWO₃ molecules, we used the thermodynamic functions of MgMoO₃ molecules.

For MgXO₄ molecules, we assumed the cyclic structure of C_{2v} symmetry, similar to the structure of GeWO₄ [22], with a regular XO₄ tetrahedron coordinating the alkaline-earth metal atom in the bidentate fashion via two oxygen atoms of the tetrahedron edge. The interatomic distances are as follows: $r(\text{Mo}=\text{O})$ 1.80, $r(\text{W}=\text{O})$ 1.82 Å; $\angle\text{OMoO}$ 104°, $\angle\text{OWO}$ 101°. The characteristic frequencies for the MoO₄ group are 930, 845, 832(2), 350, 330, 320, and 316(2) cm⁻¹, and for the WO₄ group, 930, 845, 830(2), 355, 330, 325, and 320(2) cm⁻¹. These frequencies were calcu-

Table 2. Partial pressures of molecular species in the vapor over MgO vaporized from a double two-temperature cell and enthalpies of the reactions involving MgO

| <i>T</i> , K | <i>p_i</i> , atm | | | | | $-\Delta_f H^0(0)$, kJ | |
|--------------|----------------------------|----------------------|-----------------------------------|-------------------------------------|--|-------------------------|------------------|
| | MgO ($\times 10^8$) | PO ($\times 10^5$) | PO ₂ ($\times 10^7$) | MgPO ₂ ($\times 10^8$) | MgPO ₃ ($\times 10^{10}$) | Eq. (8) | Eq. (9) |
| 1680 | 0.04 | 0.04 | 1.15 | 0.11 | 1.11 | 437.9 | 449.0 |
| 1741 | 0.15 | 0.28 | 1.42 | 0.32 | 2.68 | 426.1 | 453.7 |
| 1830 | 0.36 | 0.93 | 4.99 | 0.71 | 4.83 | 428.8 | 457.6 |
| 1826 | 0.28 | 0.17 | 1.45 | 0.20 | 2.01 | 438.1 | 466.1 |
| 1853 | 1.14 | 0.52 | 3.92 | 0.76 | 7.34 | 426.6 | 455.9 |
| 1862 | 1.19 | 0.33 | 2.22 | 0.42 | 4.43 | 425.7 | 458.4 |
| 1866 | 1.32 | 0.30 | 2.00 | 0.42 | 3.75 | 426.9 | 456.8 |
| 1908 | 2.30 | 0.42 | 2.67 | 0.61 | 7.06 | 428.2 | 463.8 |
| 1914 | 2.35 | 0.41 | 2.39 | 0.47 | 6.07 | 425.5 | 464.3 |
| 1987 | 4.23 | 0.73 | 3.13 | 0.78 | 11.55 | 431.4 | 478.6 |
| 2002 | 4.54 | 0.46 | 2.03 | 0.72 | 8.46 | 439.8 | 483.2 |
| 1876 | 3.94 | 11.67 | 71.57 | 16.64 | 78.18 | 412.3 | 433.8 |
| 1877 | 1.17 | 3.18 | 31.95 | 8.79 | 44.00 | 441.9 | 456.7 |
| 1952 | 3.49 | 13.23 | 76.38 | 20.44 | 101.68 | 432.8 | 456.7 |
| 1951 | 4.24 | 7.81 | 51.53 | 15.14 | 86.38 | 433.0 | 457.1 |
| 1969 | 4.13 | 5.29 | 38.14 | 10.55 | 61.54 | 438.0 | 461.1 |
| 2000 | 6.53 | 5.71 | 41.09 | 12.20 | 88.55 | 440.3 | 467.3 |
| 2008 | 5.62 | 1.60 | 10.81 | 2.10 | 16.92 | 435.1 | 464.9 |
| 2009 | 5.62 | 1.60 | 10.81 | 2.10 | 16.92 | 435.1 | 464.9 |
| Average | | | | | | 431.9 \pm 7.4 | 460.8 \pm 10.7 |

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

| Molecule | $-\Delta_f H^0_{298}$, kJ mol ⁻¹ | $-\Delta_{at} H^0_{298}$, kJ mol ⁻¹ | S^0_{298} , J mol ⁻¹ K ⁻¹ |
|--------------------|---|--|--|
| MgWO ₄ | 773 \pm 22 | 2768 \pm 23 | 356.1 |
| MgWO ₃ | 435 \pm 22 | 2181 \pm 23 | 342.0 |
| MgMoO ₄ | 803 \pm 22 | 2604 \pm 23 | 350.8 |
| MgMoO ₃ | 467 \pm 23 | 2020 \pm 24 | 336.5 |
| MgPO ₃ | 712 \pm 11 | 1923 \pm 11 | 317.7 |
| MgPO ₂ | 429 \pm 12 | 1386 \pm 13 | 304.3 |

lated from the vibration frequencies of the free MO₄²⁻ and WO₄²⁻ anions [23], taking into account splitting of the *E* and *F*₂ terms [24, 25], and from the experimental frequencies given in the above papers. To calculate the enthalpy of reaction (7) involving MgWO₄ molecules, we used the thermodynamic functions of MgMoO₄ molecules.

The Mg–O interatomic distance and Mg–O normal mode frequencies in the MgMoO₄ molecule were calculated ab initio (SCF, GAMESS program complex). The calculation procedure is described in detail in [1].

For the Mg–O interatomic distance, we obtained the value of 1.93 Å, and for the Mg–O normal mode frequencies, 87, 100, and 178 cm⁻¹.

By combining the enthalpies of reactions (6)–(9), recalculated to 298 K, with the heats of formation of gaseous oxides of magnesium, molybdenum, tungsten, and phosphorus, we calculated the standard enthalpies of formation and atomization of gaseous magnesium phosphates, molybdates, and tungstates. The results are listed in Table 3.

The enthalpies of formation of MgPO₂, MgPO₃, MgMoO₃, and MgWO₃ were not determined previously. The enthalpies of formation of gaseous MgMoO₄ and MgWO₄ that we determined differ from those given in [5–8] by ~100 kJ mol⁻¹. The major disagreement may be due to the following facts.

(1) The enthalpies of reactions (1) and (2) obtained in [2] are tentative. The number of the measured equilibrium constants is from 1 to 5, and measurements were made at a single temperature, which is insufficient for statistical treatment, especially in the case when the condensed phase rapidly changes, as indicated in [2]. In this study, we made 15–17 measurements of the equilibrium constants of reactions (5) in a fairly wide temperature range.

(2) An additional source of error in the equilibrium constants of reactions (1) and (2) is the fact that Verhaegen *et al.* [5] did not take into account the possible deviation of the activity of metallic Mo or W from unity.

(3) Variation of the composition of the condensed phase in the vaporization experiments made in [6–8] can also affect the results obtained. The partial pressures of molybdenum (or tungsten) oxides are much higher than the vapor pressure of atomic magnesium, which is indicative of the thermal dissociation of magnesium molybdate and tungstate with a change in the condensed phase composition. The major components of the gas phase are molybdenum (or tungsten) oxides, and the condensed phase gets enriched with magnesium oxide. The partial pressures of gaseous magnesium molybdate and tungstate should gradually decrease in the course of the experiment; this will result in underestimation of the enthalpy of vaporization, proportional to the slope of the linear dependence of the logarithm of the MgXO_4 partial pressure on the reciprocal temperature, and overestimation of the enthalpy of formation of the salt.

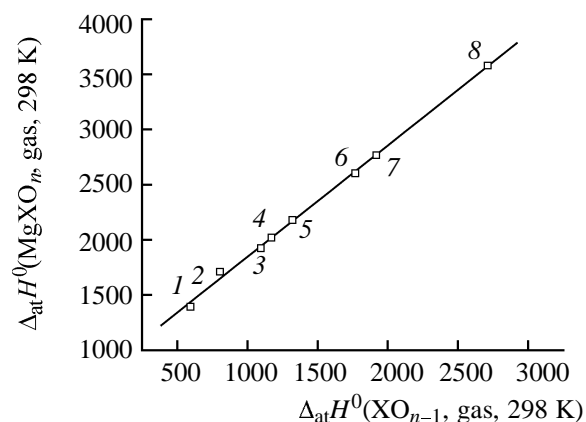
(4) The molecular parameters of gaseous magnesium molybdate and tungstate MgSO_4 , used to calculate the thermodynamic functions of these salts in this study and in [5], are essentially different. Kazenas *et al.* [6–8] did not indicate how they obtained the thermodynamic functions of gaseous salts.

From the enthalpies of atomization of gaseous MgPO_2 , MgPO_3 , MgMoO_3 , MgWO_3 , MgMoO_4 , and MgWO_4 , determined in this study, in combination with the data from [9], we constructed a correlation between the enthalpies of atomization of gaseous magnesium salts and those of the gaseous anion-forming oxides (see figure). This correlation can be described by Eq. (12). The high linear correlation coefficient ($r = 0.99897$) confirms the reliability of our results. The coefficients k and b are 1.009 ± 0.0187 and 837.2 ± 29.2 , respectively. This correlation allows estimation of the enthalpies of other, not studied yet, gaseous magnesium salts.

$$\Delta_f H^0(\text{MgXO}_n, \text{gas}, 298) = k \Delta_f H^0(\text{XO}_{n-1}, \text{gas}, 298) + b. \quad (12)$$

EXPERIMENTAL

The study was performed with an MS-1301 mass spectrometer at an ionizing energy of 25 eV. Samples of MgO were vaporized from single molybdenum or tungsten effusion cells and from a double two-temperature molybdenum cell [12] providing conditions for the coexistence in the vapor phase of



Correlation between the enthalpies of atomization of gaseous magnesium salts and those of gaseous anion-forming oxides: (1) MgPO_2 , (2) MgBO_2 , (3) MgPO_3 , (4) MgMoO_3 , (5) MgWO_3 , (6) MgMoO_4 , (7) MgWO_4 , and (8) MgB_2O_4 .

oxides essentially differing in the volatility. The cells were heated by electron bombardment; the temperature was monitored with an EOP-66 optical pyrometer. The partial pressures of vapor components were determined by comparing the ion currents, using gold as internal pressure reference [17]. The ionization cross sections were calculated additively from the ionization cross sections of the atoms [26]; the ionization cross sections of gaseous salts were multiplied by 0.7 [27]. To determine the molecular composition of the vapor, we determined the appearance potentials of the ions by the method of vanishing ion current, using the ionization potentials of gold as reference [14]. The device was preliminarily calibrated by the CaF_2 vapor pressure [18].

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