

Standard molar enthalpy of formation at 298.15 K of the β -modification of molybdenum ditelluride ^a

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Fluorine-combustion calorimetry of a high-purity sample of molybdenum ditelluride has yielded the standard molar enthalpy of formation: $\Delta_f H_m^\circ(\text{MoTe}_2, \text{cr}, \beta, 298.15 \text{ K}) = -(84.2 \pm 4.6) \text{ kJ} \cdot \text{mol}^{-1}$. At 298.15 K, the enthalpy of the hypothetical β -to- α transition in MoTe_2 is approximately $-6 \text{ kJ} \cdot \text{mol}^{-1}$. The present result for $\Delta_f H_m^\circ(\text{MoTe}_2)$ has been combined with literature values for the decomposition pressures of MoTe_2 to yield $\Delta_f H_m^\circ(\text{Mo}_3\text{Te}_4, \text{cr}, 298.15 \text{ K}) = -(185 \pm 10) \text{ kJ} \cdot \text{mol}^{-1}$. Our previously published $\Delta_f H_m^\circ(\text{TeF}_6)$ (*Trans. Faraday Soc.* **1966**, 62, 558) has been revised slightly to $-(1371.8 \pm 1.8) \text{ kJ} \cdot \text{mol}^{-1}$.

1. Introduction

There are but two well documented^(1,2) distinct solid compositions in (molybdenum + tellurium): $\text{MoTe}_{(2-x)}$ and $\text{Mo}_3\text{Te}_{(4+y)}$. The composition range of the ditelluride has been somewhat uncertain, but Brewer and Lamoureaux⁽³⁾ recommended the work of Vellinga *et al.*,⁽²⁾ who gave $0.01 \leq x \leq 0.09$ at $1023 \leq T/\text{K} \leq 1223$, as being more reliable than earlier phase studies. A very recent investigation by Krabbes and Oppermann⁽⁴⁾ and Krabbes⁽⁵⁾ revealed an even narrower range of homogeneity: $0.01 \geq x \geq 0$, at $1100 \leq T/\text{K} \leq 1373$. Therefore, in the present study, the composition of molybdenum ditelluride will be taken as, exactly, MoTe_2 .

Two forms of MoTe_2 have been identified: the low-temperature α -modification (hP6) is converted to the high-temperature β -modification (mP12) at $(1150 \pm 10) \text{ K}$, according to the results of d.t.a. experiments reported by Krabbes.⁽⁵⁾

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Modern technological applications of molybdenum ditelluride include its use as a catalyst,⁽⁶⁾ as a cathode for intercalation of lithium in batteries,⁽⁷⁾ as sputtered antifriction coatings,⁽⁸⁾ in photoelectric solar cells,⁽⁹⁾ and as a semiconductor.⁽¹⁰⁾ There is also considerable interest in (molybdenum + tellurium) compounds formed from the high-yield fission-product elements Mo and Te in irradiated fast-reactor fuel pins.⁽¹¹⁾

Some reliable thermodynamic investigations of MoTe₂ have been reported recently. They include measurements of the low-temperature heat capacity of the α -phase by Kiwia and Westrum,⁽¹²⁾ tensimetric studies at $\langle T \rangle = 1235$ K of the gas-phase in equilibrium with (molybdenum + tellurium) by Krabbes and Oppermann⁽⁴⁾ and Krabbes;⁽⁵⁾ and a determination of the Gibbs energy of formation at $\langle T \rangle = 860$ K of α -MoTe₂ by Mallika and Sreedharan⁽¹³⁾ who used an e.m.f. technique. The $\Delta_f H_m^\circ(298.15 \text{ K})$ values deduced from the high-temperature studies are rather uncertain ($\pm 10 \text{ kJ} \cdot \text{mol}^{-1}$) because the auxiliary thermodynamic quantities used in the derivations have been estimated.⁽³⁾

Thus, the purpose of the present investigation was to determine $\Delta_f H_m^\circ(\beta\text{-MoTe}_2)$ with a high precision ($\pm 4 \text{ kJ} \cdot \text{mol}^{-1}$) as we have done in our other studies of chalcogenides.

2. Experimental

Crystals of MoTe₂ were grown, at Griffith University, by vapor transport from the elements in a sealed quartz tube. Molybdenum sheet (Alfa Products, 99.9 mass per cent) and tellurium (Alfa Products, 99.5 mass per cent) were cleaned in NH₃(aq) and 5 mol·dm⁻³ HCl(aq), respectively, washed with distilled water and ethanol, dried, and placed in a 20 cm long quartz tube of diameter 16 mm. Stoichiometric amounts of Mo and Te were used (total mass: 25 g). The pressure in the tube was then reduced to less than 1.3×10^{-3} Pa with a turbomolecular pumped vacuum system, and the (molybdenum + tellurium) was pumped on for 10 h at 423 K to remove water adsorbed on the surface and other volatile contaminants. Afterwards, the tube was filled with dry N₂(g), removed from the vacuum line, and transferred to a glovebox where the transport agent, 0.1 g of TeCl₄, was introduced. Next, the tube was once again evacuated to a base pressure of less than 1.3×10^{-6} Pa for 24 h, after which it was sealed under vacuum and placed in a horizontal tube furnace.

The Mo and Te were made to react by heating the tube to 873 K for 12 h. The molybdenum foil expanded and disintegrated to yield a grey powder, while the tellurium chips evaporated. Crystalline MoTe₂ was grown by increasing the maximum temperature of the furnace to (1148 ± 1) K. This produced a temperature profile along the quartz tube, the central region being at the furnace setting and the ends 50 K cooler than the center. Flat crystals, up to 1 cm² in size and usually in clusters, were formed in the cooler regions of the transport tube. Crystal growth was continued for approximately 7 d, then the tube was removed from the furnace and its central section was quenched in a stream of cold water. Vapor condensed on the cold wall of the tube and did not deposit to any significant extent on the MoTe₂ crystals. After washing with H₂O, the MoTe₂ was dried in a desiccator on filter paper. Other

washing agents such as ethanol or acetone could not be used because they are readily intercalated into the telluride.

Surface examination of the crystals using X-ray photoelectron spectroscopy identified only molybdenum and tellurium in the spectra. X-ray diffraction analysis showed the preparation to be the high-temperature (β) monoclinic form of MoTe_2 ; there was excellent agreement between the diffraction pattern and that reported by Brown.⁽¹⁴⁾ Emission spectrochemical analysis revealed no significant metallic impurities, and inert-gas fusion showed mass fraction $(250 \pm 50) \times 10^{-6}$ of oxygen. Thus, our specimen was deemed to be suitable for calorimetric study.

As in our previous thermochemical studies of chalcogenides at Argonne National Laboratory, we have employed the technique of fluorine-combustion calorimetry in the present work. Trial combustion experiments showed that MoTe_2 reacted spontaneously with high-pressure fluorine at room temperature. However, this reaction was by no means vigorous, and did not bring about complete conversion of the telluride to the corresponding hexafluorides. Thus, the following experimental tactics were adopted: a two-chamber combustion vessel⁽¹⁵⁾ was used to isolate the F_2 from MoTe_2 until reaction was desired, and tungsten foil was used as an auxiliary combustant as in our recent study of vanadium monosulfide.⁽¹⁶⁾

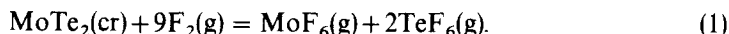
Tungsten sheet, approximately 0.13 mm thick, was purchased from Schwarzkopf Development Corporation; the rhombohedral sulfur was part of U.S. Bureau of Mines reference material USBM-P-1b; and high-purity (≈ 99.99 moles per cent) fluorine was prepared by distillation of a commercial sample in a low-temperature still.⁽¹⁷⁾ The MoTe_2 was removed from the sealed tube in which it had been shipped from Australia, and was subsequently stored in our high-purity helium-atmosphere glovebox.

The bomb-and-tank apparatus had already been used in sequential studies of other compounds and had not been exposed for several weeks to any atmosphere other than high-purity helium and fluorine: thus, the customary preconditioning experiments were not necessary. Approximately 0.6 g specimens of MoTe_2 were weighed in the glovebox on approximately 0.7 g tungsten saucers, and milligram quantities of rhombohedral sulfur were sprinkled on the MoTe_2 to ignite it. This ensemble rested on a 25 g prefluorinated nickel crucible around whose circumference several 5 mm holes were drilled to increase circulation of F_2 close to the (sample + W). The (crucible + sample), bomb, and tank were assembled in the glovebox and then placed in the calorimeter, the prototype of which has been described by Hubbard *et al.*⁽¹⁸⁾

After the fore-rating period had been recorded by means of a Hewlett-Packard 2804-A quartz-crystal thermometer, the fluorine storage tank, charged with fluorine to a pressure of approximately 1.5 MPa at 297 K, was opened remotely. Expanding fluorine reacted with the sulfur fuse which, in turn, ignited the MoTe_2 and W saucer. When the after-rating period had been recorded, the reaction vessel was removed from the calorimeter and the bomb gases were condensed into a liquid-nitrogen cooled trap. The sealed reaction vessel was then transferred to the glovebox and opened for inspection. No evidence of unreacted MoTe_2 was found. In some experiments, minor fragments (< 1 mg) of shiny unreacted tungsten remained in the

crucible; they had evidently broken off the tungsten saucers whose edges were quite fragile due to the brittleness of high-purity tungsten.

Infrared analysis of the bomb gases showed the presence of just MoF_6 , TeF_6 , SF_6 , and WF_6 . Thus, as expected, the sulfur had reacted to give SF_6 and the tungsten to give WF_6 and, since we had chosen the mass of MoTe_2 in each experiment such that all the MoF_6 produced was gaseous, we concluded that the combustion of MoTe_2 took place as follows:



3. Results

In table 1, we have given detailed results for combustion reaction (1). The mass of MoTe_2 reacted and the corrected temperature rise of the calorimeter⁽¹⁹⁾ are denoted by m and $\Delta\theta_c$, respectively. In separate experiments, we determined the specific energy of combustion of our W sample and the standard deviation to be $-(9371.0 \pm 1.5) \text{ J} \cdot \text{g}^{-1}$. Multiplication of this value by the mass of the tungsten saucer corrected for unreacted fragments gave $-\Delta U(\text{W})$. In an earlier study,⁽²⁰⁾ we determined the specific energy of combustion of USBM-P-1b rhombohedral sulfur to be $-(37930.6 \pm 11.9) \text{ J} \cdot \text{g}^{-1}$; it is on this value that the correction for the energy of combustion of the fuse $\Delta U(\text{S})$ is based. Earlier publications^(20, 21) have detailed the calculation of $\Delta U(\text{cont})$, the correction for the contents of the bomb; $\Delta U(\text{gas})$, the correction for compression and decompression of F_2 and the combustion gases; and $\Delta U(\text{blank})$, the correction for expansion of F_2 from the storage tank into the combustion bomb. Summation of the energy quantities in the table and division by m gave $\Delta_c U_m^\circ/M(\text{sample})$, the specific energy of combustion of our MoTe_2 specimen.

As stated earlier, oxygen was the only significant contaminant found in the

TABLE 1. Results for the energy of combustion in fluorine of $\beta\text{-MoTe}_2$ ($T = 298.15 \text{ K}$, $p^\circ = 101.325 \text{ kPa}$)

Expt. no.	4	6	7	8	9
m/g	0.64099	0.65576	0.65944	0.65815	0.62576
$\Delta\theta_c/\text{K}$	1.02396	1.04549	1.03221	1.03749	1.01901
$s(\text{calor})(-\Delta\theta_c)/\text{J}^a$	-14238.6	-14538.0	-14353.3	-14426.7	-14169.7
$\Delta U(\text{W})/\text{J}$	6523.8	6657.2	6467.1	6525.5	6657.7
$\Delta U(\text{S})/\text{J}$	64.1	48.2	14.0	53.9	45.1
$\Delta U(\text{cont})/\text{J}$	-15.4	-15.7	-15.5	-15.6	-15.3
$\Delta U(\text{gas})/\text{J}$	-0.2	-0.2	-0.2	-0.2	-0.2
$\Delta U(\text{blank})/\text{J}$	-3.5	-3.5	-3.5	-3.5	-3.5
$\{\Delta_c U_m^\circ/M(\text{sample})\}/(\text{J} \cdot \text{g}^{-1})$	-11965.6	-11973.9	-11966.8	-11952.6	-11962.9
$\langle \Delta_c U_m^\circ/M(\text{sample}) \rangle = -(11964.4 \pm 3.5) \text{ J} \cdot \text{g}^{-1},^b$ Impurity correction $= -(3.0 \pm 0.3) \text{ J} \cdot \text{g}^{-1},^{b,c}$					
$\langle \Delta_c U_m^\circ/M(\text{MoTe}_2) \rangle = -(11967.4 \pm 4.0) \text{ J} \cdot \text{g}^{-1},^b$					
$\Delta_c U_m^\circ = -(4202.2 \pm 2.8) \text{ kJ} \cdot \text{mol}^{-1},^d$ $\Delta nRT = -14.9 \text{ kJ} \cdot \text{mol}^{-1}$; $\Delta_c H_m^\circ = -(4217.1 \pm 2.8) \text{ kJ} \cdot \text{mol}^{-1},^d$					

^a $s(\text{calor}) = (13905.4 \pm 0.6) \text{ J} \cdot \text{K}^{-1}$, determined by combustion in pure oxygen of N.B.S. Standard Benzoic Acid (sample 39 i).

^b Uncertainty is the standard deviation of the mean.

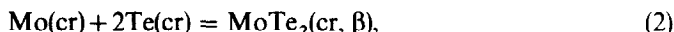
^c Impurity corrections due solely to oxygen which was assumed to be intercalated in the MoTe_2 .

^d Uncertainty corresponds to the uncertainty interval equal to twice the overall standard deviation of the mean.

MoTe₂, but we do not know how it was present in the sample. Thus, the impurity correction was based on the simplest assumption, namely, that the oxygen was uncombined (intercalated or in solid solution) in the MoTe₂. It is possible, of course, that the oxygen was chemically bound, for example, as TeMo₅O₁₆ or Te₂MoO₇,⁽²²⁾ but there are no experimental $\Delta_f H_m^\circ$ s of (molybdenum + tellurium + oxygen), and it would not be possible to calculate a meaningful value for the corresponding impurity correction.

The scatter of the experimental results contributes $\pm 3.5 \text{ J} \cdot \text{g}^{-1}$ to $\Delta_c U_m^\circ/M$. Additional uncertainties and their origins are as follows: the impurity, $\pm 0.3 \text{ J} \cdot \text{g}^{-1}$; $\Delta U(\text{blank})$, $\pm 0.8 \text{ J} \cdot \text{g}^{-1}$; $\epsilon(\text{calor})$, $\pm 0.6 \text{ J} \cdot \text{g}^{-1}$; $\Delta U(\text{gas})$, $\pm 0.1 \text{ J} \cdot \text{g}^{-1}$; and $\Delta U(W)$, $\pm 1.6 \text{ J} \cdot \text{g}^{-1}$. Thus, the combined uncertainty $(\Sigma \sigma^2)^{1/2}$ is $\pm 4.0 \text{ J} \cdot \text{g}^{-1}$ and $\Delta_c U_m^\circ/M(\text{MoTe}_2) = -(11967.4 \pm 8.0) \text{ J} \cdot \text{g}^{-1}$, where the uncertainty is now expressed as *twice* the standard deviation of the mean. From this result, we have deduced the molar thermochemical quantities for reaction (1) given in the lower portion of table 1; they are based on a molar mass $M = 351.14 \text{ g} \cdot \text{mol}^{-1}$ for MoTe₂.

The standard molar enthalpy of formation, $\Delta_f H_m^\circ(\text{MoTe}_2, \text{cr}, \beta, 298.15 \text{ K}) = -(84.2 \pm 4.6) \text{ kJ} \cdot \text{mol}^{-1}$, refers to the reaction:



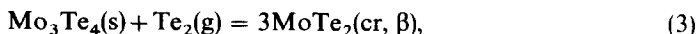
and has been derived by combining $\Delta_c H_m^\circ(\text{MoTe}_2) = -(4217.1 \pm 2.8) \text{ kJ} \cdot \text{mol}^{-1}$ (table 1), $\Delta_f H_m^\circ(\text{MoF}_6, \text{g}) = -(1557.7 \pm 0.9) \text{ kJ} \cdot \text{mol}^{-1}$,⁽²³⁾ and $\Delta_f H_m^\circ(\text{TeF}_6, \text{g}) = -(1371.8 \pm 1.8) \text{ kJ} \cdot \text{mol}^{-1}$. The latter quantity is a revised value based on our earlier determination⁽²⁴⁾ and the new method⁽²⁰⁾ now being used by us to calculate $\Delta U(\text{blank})$. The recalculated $\Delta U(\text{blank})$, 47.7 J, is 27 J smaller than that given in reference 24; it is still quite large, and is due, we believe, to the presence in the bomb of a porous disk of B₄C used as a thermal insulator to promote combustion of Te in F₂. Our recalculated $\Delta_f H_m^\circ(\text{TeF}_6)$ agrees well with a subsequent determination, $-(1381 \pm 25) \text{ kJ} \cdot \text{mol}^{-1}$ reported by Bousquet *et al.*,⁽²⁵⁾ but is clearly more precise.

4. Discussion

Apart from the low-temperature adiabatic calorimetric study by Kiwia and Westrum,⁽¹²⁾ all other thermodynamic investigations of (molybdenum + tellurium) have been performed at elevated temperatures.

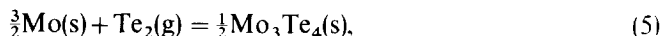
In 1966, Opalovskii and Fedorov⁽²⁶⁾ reported that MoTe₂ decomposed to Mo₃Te₄ and Te₂(g) when heated in a vacuum. Workers from the same laboratory⁽²⁷⁾ subsequently used a manometric technique to deduce $\Delta_f H_m^\circ$ for the dissociation of MoTe₂.

By far the most comprehensive tensimetric study of (molybdenum + tellurium) was described by Krabbes and Oppermann⁽⁴⁾ in a communication that contained⁽²⁸⁾ a number of errors. The same work was subsequently corrected and reported in greater detail by Krabbes.⁽⁵⁾ He has given the following results ($p^\circ = 1 \times 10^5 \text{ Pa}$): for



$$\lg\{p(\text{Te}_2)/p^\circ\} = 8.398 - 11790(\text{K}/T), \quad (4)$$

where $1100 \leq T/K \leq 1333$; and for



$$\lg\{p(\text{Te}_2)/p^\circ\} = 5.56 - 9879(\text{K}/T), \quad (6)$$

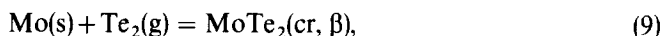
where $1173 \leq T/K \leq 1373$. For reaction (3), we derive the relation:

$$\Delta_r G_m^\circ/(\text{kJ} \cdot \text{mol}^{-1}) = -225.8 + 0.1608(T/\text{K}), \quad (7)$$

and for reaction (5):

$$\Delta_r G_m^\circ/(\text{kJ} \cdot \text{mol}^{-1}) = -189.2 + 0.1065(T/\text{K}). \quad (8)$$

From the combination of reactions (3) and (5) we obtain from equations (7) and (8):



$$\Delta_r G_m^\circ/(\text{kJ} \cdot \text{mol}^{-1}) = -201.4 + 0.1246(T/\text{K}). \quad (10)$$

At $T = 1300$ K, a temperature common to reactions (3) and (5), $\Delta_r G_m^\circ = -39.4 \text{ kJ} \cdot \text{mol}^{-1}$ and $(\Delta_0^T S_m^\circ - \Delta_{298.15 \text{ K}}^T H_m^\circ/T) = 46.82 \text{ J} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$ for Mo(s) ,⁽³⁾ $286.61 \text{ J} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$ for $\text{Te}_2(\text{g})$,⁽²⁹⁾ and $172.62 \text{ J} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$ for MoTe_2 , as estimated by Brewer and Lamoreaux.⁽³⁾ By means of a third-law calculation, $\Delta_r H_m^\circ(298.15 \text{ K}) = -248.4 \text{ kJ} \cdot \text{mol}^{-1}$ for reaction (9). We add to this result $\Delta_f H_m^\circ(\text{Te}_2, \text{g}, 298.15 \text{ K}) = 163.2 \text{ kJ} \cdot \text{mol}^{-1}$,⁽²⁹⁾ and derive $\Delta_f H_m^\circ(\text{MoTe}_2, \text{cr}, \beta) = -85.2 \text{ kJ} \cdot \text{mol}^{-1}$. Thus, the high-temperature and calorimetric results are in excellent agreement. It may well be that the $\pm 10 \text{ kJ} \cdot \text{mol}^{-1}$ uncertainty in $\Delta_f H_m^\circ$ given by Krabbes is too large and that the probable error in the estimated thermodynamic properties suggested by Brewer and Lamoreaux may be too conservative. A high-temperature calorimetric determination of the heat capacity of MoTe_2 would clearly be of value.

For $\Delta_f H_m^\circ(\text{MoTe}_2, \text{cr}, \alpha)$, Mallika and Sreedharan⁽¹³⁾ obtained a value of $-90 \text{ kJ} \cdot \text{mol}^{-1}$ at 298.15 K . Thus, the enthalpy of the (hypothetical) β -to- α transition at 298.15 K is approximately $-6 \text{ kJ} \cdot \text{mol}^{-1}$, close to the estimated $-4.8 \text{ kJ} \cdot \text{mol}^{-1}$ given by Krabbes⁽⁵⁾ for the transition temperature of $(1150 \pm 10) \text{ K}$.

Krabbes has also reported $\Delta_f H_m^\circ(\text{Mo}_3\text{Te}_4)$, but we do not agree with his result. For reaction (5) at $\langle T \rangle = 1300 \text{ K}$, $\Delta_r G_m^\circ = -50.7 \text{ kJ} \cdot \text{mol}^{-1}$ which we combine with $(\Delta_0^T S_m^\circ - \Delta_{298.15 \text{ K}}^T H_m^\circ/T)$ values given earlier for Mo and $\text{Te}_2(\text{g})$ along with $398.2 \text{ J} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$,⁽³⁾ for $\text{Mo}_3\text{Te}_4(\text{cr})$ to calculate by the third-law method $\Delta_r H_m^\circ(5) = -255.7 \text{ kJ} \cdot \text{mol}^{-1}$ at 298.15 K . Addition of $\Delta_f H_m^\circ(\text{Te}_2, \text{g}) = 163.2 \text{ kJ} \cdot \text{mol}^{-1}$,⁽²⁹⁾ yields $\Delta_f H_m^\circ(\text{Mo}_3\text{Te}_4, \text{cr}, 298.15 \text{ K}) = -(185 \pm 10) \text{ kJ} \cdot \text{mol}^{-1}$, where the uncertainty has been estimated. This result is approximately $10 \text{ kJ} \cdot \text{mol}^{-1}$ less negative than that given by Krabbes. The discrepancy is due, in all probability, to a difference between the values used for $\Delta_f H_m^\circ(\text{Te}_2, \text{g})$. Unfortunately, Krabbes does not mention his source for this quantity.

Opalovskii *et al.*⁽²⁷⁾ evidently used the second-law method to deduce $\Delta_r H_m^\circ(T) = -(186.6 \pm 2.9) \text{ kJ} \cdot \text{mol}^{-1}$ for reaction (3) at $T \approx 1200 \text{ K}$. This result is approximately $40 \text{ kJ} \cdot \text{mol}^{-1}$ less negative than that given by Krabbes and leads to an unlikely $\Delta_f H_m^\circ(\text{MoTe}_2) = -71 \text{ kJ} \cdot \text{mol}^{-1}$.

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