Thermochimica Acta, 126 (1988) 375–383
Elsevier Science Publishers B.V., Amsterdam – Printed in The Netherlands

THE THERMOCHEMISTRY OF URANYL PHOSPHATES

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(Received 19 November 1987)

ABSTRACT

Standard entropies of uranyl/uranium phosphates are derived by combining their standard enthalpies of formation and high-temperature data. As a result the high-temperature molar thermodynamic values of these compounds have been calculated.

INTRODUCTION

The thermochemistry of the uranyl/uranium ** phosphates has been the subject of study at the Netherlands Energy Research Foundation ECN for quite some time. As a result, we determined for instance their standard enthalpies of formation [1], the enthalpy increment functions at high temperatures [2], and their oxygen pressures [3]. Measurements of low-temperature enthalpy functions, which would have revealed the standard entropies of the uranyl and uranium phosphates, have not been carried out. Since these are necessary to express the molar thermodynamic functions, it was decided to derive these from the available experimental data. The derivations are based on the interrelations obtained from the oxygen pressures measured.

In contrast to the uranyl arsenates [4], the calculations with the decomposition reactions of the uranyl phosphates always involve two unknown standard entropies. As a consequence, the entropies cannot be expressed for single compounds from these relations only; the entropy differences of the two interrelated compounds always being found. Thus additional reference values must be estimated in order to derive the molar entropy values.

This paper gives the results of the derivations of the standard entropies. As a consequence, the high-temperature thermodynamic data of these compounds can be calculated.

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** This indicates uranium with lower valencies than six, which have been met occasionally.

0040-6031/88/$03.50

OVERVIEW OF AVAILABLE DATA

Recently, Cordfunke et al. [1] determined the standard enthalpies of formation of uranyl/uranium phosphates by means of solution calorimetry. The results are summarized in Table 1. It should be noted that the value for the metaphosphate has not been experimentally determined, because the compound did not dissolve in any solvent. In this case an estimated value is given [1], which has been obtained by extrapolation after comparison of the enthalpies of solution of the uranyl phosphates with the arsenates [5].

Additionally, enthalpy increments have been measured of these compounds by drop-calorimetry [2]. The measurements did not reveal enthalpy effects resulting from polymorphy in this system [7]. Table 2 gives the resulting parameters of the enthalpy content functions. From these, the parameters of the heat capacity functions have been obtained by differentiation. Auxiliary data have been taken from the literature [8–10].

DERIVATIONS OF THE ENTROPIES OF URANYL/URANIUM PHOSPHATES

Estimation of the standard entropies of $(UO_2)_3(PO_4)_2$ *and* $UO_2(PO_3)_2$

The entropy of $(UO_2)_3(PO_4)_2$ can be estimated using the method of Latimer [11], in a similar way to that used for the $(UO_2)_3(AsO_4)_2$ [4]. The

TABLE 1

Enthalpies of formation of uranyl/uranium phosphates [1]

Compound	$\Delta H_f^\ominus$(s,298.15 K) (kJ mol^{-1})
$(UO_2)_3(PO_4)_2$	$-(5491.3 \pm 3.5)$
$(UO_2)_2P_2O_7$	$-(4232.6 \pm 2.8)$
$UO_2(PO_3)_2$	$-(2973.0 \pm 3.0)$ (estimated)
UPO_5	$-(2064.3 \pm 2.0)$
UP_2O_7	$-(2852.0 \pm 4.3)$ (recalculated [6])

TABLE 2

Uranyl/uranium phosphates [2]. Parameters of enthalpy content functions $H_T^\ominus - H^\ominus(298.15\text{ K}) = AT + BT^2 + CT^{-1} + D$ (J mol^{-1})

Compound	A	$B \times 10^3$	$C \times 10^{-5}$	D	Temperature range (K)
$(UO_2)_3(PO_4)_2$	326.375	98.219	40.840	−119737.5	298–771
$(UO_2)_2P_2O_7$	250.669	77.149	34.003	−92999.7	298–883
$UO_2(PO_3)_2$	184.304	60.644	38.540	−73267.5	298–900
UPO_5	110.427	42.614	10.379	−40193.0	298–726
UP_2O_7	198.531	30.362	29.011	−71621.2	298–882

$S^{\ominus}(UO_2^{2+})$ and the $S^{\ominus}(PO_4^{3-})$ parameters have been taken as 87.86 and 71.13 J K^{-1} mol^{-1}, respectively [11]. In this way we obtain for $(UO_2)_3(PO_4)_2$ $S^{\ominus}(298.15\ K) = 405.8 \pm 20.0$ J K^{-1} mol^{-1}.

No $S^{\ominus}(PO_3^-)$ parameter for bivalent cations is given by Latimer [11]. It is, however, necessary to estimate the entropy of $UO_2(PO_3)_2$. Derivation of the parameter value requires entropy values of as many as possible related metaphosphates. Unfortunately, measured entropies of metaphosphates are very scarce; not only at the time that Latimer formulated the estimation values, but also at present. As a result, the parameter mentioned is derived from the entropy of the metaphosphate $Ca(PO_3)_2$, being 146.65 J K^{-1} mol^{-1} [8], which gives $S^{\ominus}(PO_3^-) = 53.87$ J K^{-1} mol^{-1}.

A comparison can be made using the entropy of KPO_3 108.49 J K^{-1} mol^{-1} [8]. From this, we calculate a value for $S^{\ominus}(PO_3^-)$ (monovalent cation!) of 70.00 J K^{-1} mol^{-1}. If we take into consideration the usual correction for the conversion of the monovalent into the bivalent cation parameter, being about 20.9 J K^{-1} mol^{-1}, then we would get a value for $S^{\ominus}(PO_3^-)$ (bivalent cations) of 49.1 J K^{-1} mol^{-1}. This value supports the value initially found, but since this second value is expected to be less accurate, the first value found will be adopted for the estimation. Consequently we find for $UO_2(PO_3)_2$ $S^{\ominus}(298.15\ K) = 195.6 \pm 9.0$ J K^{-1} mol^{-1}.

Entropy difference of $(UO_2)_3(PO_4)_2$ and $2UPO_5$

It can be seen from the reaction

$$(UO_2)_3(PO_4)_2(s) \rightleftharpoons \tfrac{1}{3}U_3O_8(s) + 2UPO_5(s) + \tfrac{2}{3}O_2(g) \qquad (1)$$

that the Gibbs energy of reaction $\Delta G_r^{\ominus}(T)$ can be derived from the oxygen pressures measured [3]. Use will be made of the relation $\Delta G_r^{\ominus}(T) = -nRT \ln pO_2$, which makes it possible to derive the difference in the entropies of $(UO_2)_3(PO_4)_2$ and $2UPO_5$, by combining the measured Gibbs energies of reaction and those cited in the literature. The individual results of these calculations are given in Table 3.

It is noted that a shift takes place in the results in Table 3; this is, however, negligible with respect to the mean value 135.4 ± 2.7 J K^{-1} mol^{-1}

TABLE 3

Derivation of the entropy difference $S^{\ominus}((UO_2)_3(PO_4)_2) - 2S^{\ominus}(UPO_5)$ at 298.15 K

Temperature (K)	Pressure p_{O_2} (atm)	Entropy difference (J K^{-1} mol^{-1})
1280	4.32×10^{-2}	133.76
1255	2.51×10^{-2}	133.75
1211	7.80×10^{-3}	134.63
1089	1.34×10^{-4}	139.40

TABLE 4

Derivation of the entropy difference $S^{\ominus}((UO_2)_2P_2O_7)-2S^{\ominus}(UPO_5)$ at 298.15 K

Temperature (K)	Pressure p_{O_2} (atm)	Entropy difference (J K^{-1} mol^{-1})
1130	$1.00\times10^{+0}$	11.00
1074	4.32×10^{-2}	19.20
1042	7.80×10^{-3}	23.31
979	1.72×10^{-4}	32.69

for the entropy difference $S^{\ominus}((UO_2)_3(PO_4)_2) - 2S^{\ominus}(UPO_5)$ at 298.15 K. Using this result and the estimated entropy of $(UO_2)_3(PO_4)_2$ we obtain for UPO_5 $S^{\ominus}(298.15\ K) = 135.2 \pm 10.0$ J K^{-1} mol^{-1}.

This result demonstrates the propagation of the uncertainty from the estimated entropy value of the orthophosphate, as well as the number of significant figures maintained in this derived entropy. The following entropy values will be influenced in a similar way.

Entropy difference of $(UO_2)_2P_2O_7$ and $2UPO_5$

The measured oxygen pressures [3] also give values of $\Delta G_r^{\ominus}(T)$ for the reaction

$$(UO_2)_2P_2O_7(s) \rightleftharpoons 2UPO_5(s) + \tfrac{1}{2}O_2(g) \qquad (2)$$

From this reaction, the entropy difference of $(UO_2)_2P_2O_7$ and $2UPO_5$ can be derived in a similar way to that used above. Individual results of the calculations are given in Table 4, where it is shown that the shift in the derived values of the indicated entropy difference is rather high. This is also demonstrated in the uncertainty of the mean value 21.55 ± 9.02 J K^{-1} mol^{-1}.

The background for the observed shift is not clear and many reasons may be indicated. The deviations could be the result of errors introduced by extrapolating the heat capacity functions, such as neglecting phase transitions and/or deviations from the indicated reaction caused by non-stoichiometry. Since further observations fail in this respect, it was decided to accept the foregoing derived mean value as the best value currently available for the indicated entropy difference. In this way we obtain a value for the entropy of $(UO_2)_2P_2O_7$ of $S^{\ominus}(298.15\ K) = 292.0 \pm 21.0$ J K^{-1} mol^{-1}.

For comparison, the entropy of the pyrophosphate has also been estimated. To do so, use is made of the known standard entropies of $Ca_2P_2O_7$ and $Mg_2P_2O_7$, being 189.24 and 154.89 J K^{-1} mol^{-1}, respectively [8], and of the appropriate estimation parameters of Latimer [11] for the cations. Of these, the value for Mg has not been mentioned yet, this being 31.80 J K^{-1} mol^{-1}.

TABLE 5

Derivation of the entropy difference $S^\ominus(UO_2(PO_3)_2) - S^\ominus(UP_2O_7)$

Temperature (K)	Pressure p_{O_2} (atm)	Entropy difference (J K^{-1} mol^{-1})
1118	$1.00 \times 10^{+0}$	3.47
1093	2.14×10^{-1}	−0.61
1052	7.80×10^{-3}	−7.39
963	1.96×10^{-4}	−15.35

Exchange of the uranyl parameter for the Ca or Mg parameters results in a mean estimated entropy value for $(UO_2)_2P_2O_7$ of $S^\ominus$(298.15 K) = 277.0 ± 15.0 J K^{-1} mol^{-1}. This value is somewhat lower than the mean experimental value, but both values agree reasonably within the given uncertainties. Since both uncertainties are about the same magnitude, the mean value of both results has been chosen as the best value currently available, the entropy of $(UO_2)_2P_2O_7$ thus being $S^\ominus$(298.15 K) = 284.0 ± 13.0 J K^{-1} mol^{-1}.

TABLE 6

Thermodynamic functions of solid $(UO_2)_3(PO_4)_2$

T (K)	$C_p^\ominus(T)$ (J K^{-1} mol^{-1})	$S^\ominus$(T) (J K^{-1} mol^{-1})	−GEF [a] (J K^{-1} mol^{-1})	$H^\ominus$(T) − $H^\ominus$(298 K) (kJ mol^{-1})	$\Delta H_f^\ominus(T)$ (kJ mol^{-1})	$\Delta G_f^\ominus(T)$ (kJ mol^{-1})	log $K_p^\ominus$
298.15	339.00	405.80	405.80	0.00	−5491.30	−5114.95	896.11
300	339.93	407.90	405.80	0.63	−5491.29	−5112.62	890.17
317.3	348.14	427.19	406.45	6.58	−5491.19	−5090.79	838.05
317.3	348.14	427.19	406.45	6.58	−5492.51	−5090.79	838.05
400	379.43	511.51	419.66	36.74	−5491.31	−4986.19	651.12
500	408.26	599.39	447.05	76.17	−5488.09	−4860.25	507.74
550	420.92	638.90	462.70	96.91	−5485.93	−4797.57	455.63
550	420.92	638.90	462.70	96.91	−5510.21	−4797.57	455.63
600	432.89	676.04	478.96	118.25	−5507.07	−4732.92	412.03
700	455.55	744.49	512.08	162.69	−5500.09	−4604.44	343.58
800	477.14	806.74	545.08	209.33	−5492.37	−4477.01	292.31
900	498.13	864.15	577.37	258.10	−5484.09	−4350.58	252.50
942	506.82	887.07	590.68	279.20	−5480.50	−4297.76	238.31
942	506.82	887.07	590.68	279.20	−5488.87	−4297.76	238.31
1000	518.73	917.71	608.77	308.94	−5481.82	−4224.64	220.67
1049	528.73	942.76	623.79	334.60	−5475.37	−4163.19	207.30
1049	528.73	942.76	623.79	334.60	−5489.65	−4163.19	207.30
1100	539.08	968.10	639.17	361.83	−5477.77	−4098.98	194.64

[a] −GEF is the Gibbs energy function $[G_T^\ominus - H_{298\,K}^\ominus]/T$.

Entropy difference of $UO_2(PO_3)_2$ and UP_2O_7

In a similar way, the reaction

$$UO_2(PO_3)_2(s) \rightleftharpoons UP_2O_7(s) + \tfrac{1}{2}O_2(g) \qquad (3)$$

is considered. Table 5 gives the results of calculations in which we use relevant thermodynamic data given in Tables 1 and 2, and the measured oxygen pressures [3].

The values of the entropy difference appear to shift to more positive values at higher temperatures. Since no specific cause has been found for this phenomenon, it is expected that the reason for the observed shift is essentially the same as in the case of the reduction of the pyrophosphate (resulting from the extrapolation of the heat capacity functions and/or from nonstoichiometry). If the last value in the table is rejected as an excessive value, then the mean value obtained is -1.5 ± 7.8 J K^{-1} mol^{-1} for the entropy difference. Consequently, the entropy of the pyrophosphate can be derived using the estimated entropy of the uranyl metaphosphate, to give for UP_2O_7 $S^\ominus(298.15\ K) = 197.1 \pm 11.9$ J K^{-1} mol^{-1}.

TABLE 7

Thermodynamic functions of solid $(UO_2)_2P_2O_7$

T (K)	$C_p^\ominus(T)$ (J K^{-1} mol^{-1})	$S^\ominus(T)$ (J K^{-1} mol^{-1})	$-$GEF (J K^{-1} mol^{-1})	$H^\ominus(T) - H^\ominus(298\ K)$ (kJ mol^{-1})	$\Delta H_f^\ominus(T)$ (kJ mol^{-1})	$\Delta G_f^\ominus(T)$ (kJ mol^{-1})	log $K_p^\ominus$
298.15	258.42	284.00	284.00	0.00	−4232.60	−3926.61	687.92
300	259.18	258.60	284.00	0.48	−4232.61	−3924.71	683.34
317.3	265.85	300.32	284.50	5.02	−4232.67	−3906.95	643.16
317.3	265.85	300.32	284.50	5.02	−4233.99	−3906.95	643.16
400	291.14	364.88	294.61	28.11	−4233.75	−3821.71	499.06
500	314.22	432.42	315.58	58.42	−4231.94	−3718.88	388.50
550	324.29	462.84	327.59	74.39	−4230.55	−3667.64	348.32
550	324.29	462.84	327.59	74.39	−4254.84	−3667.64	348.32
600	333.80	491.47	340.07	90.84	−4252.51	−3614.36	314.65
700	351.74	544.29	365.53	125.13	−4247.22	−3508.41	261.80
800	368.80	592.38	390.93	161.16	−4241.21	−3403.27	222.21
900	385.34	636.78	415.81	198.87	−4234.64	−3298.92	191.46
942	392.19	654.51	426.06	215.20	−4231.73	−3255.31	180.51
942	392.19	654.51	426.06	215.20	−4237.31	−3255.31	180.51
1000	401.57	678.22	440.00	238.22	−4232.43	−3194.99	166.89
1049	409.44	697.62	451.59	258.09	−4227.95	−3144.27	156.57
1049	409.44	697.62	451.59	258.09	−4237.47	−3144.27	156.57
1100	417.59	717.24	463.44	279.18	−4232.00	−3091.25	146.79

DISCUSSION

It was found that the individually derived entropy values reproduced quite closely in the case of uranyl ortho-phosphate. This applies to a lesser extent for the pyro- and metaphosphates. It must be kept in mind that the selected entropy values have been taken to fit calculated and experimental oxygen pressures at high temperatures. Deviations from the applied model, e.g. the reaction, stoichiometry, and extrapolation of the C_p functions, result in a contribution to the derived standard entropy values. A consequence of this is that, in these cases, the derived entropies deviate from the standard entropies. These deviations are expected to be within the indicated uncertainties.

Using the present data, the high-temperature thermodynamic functions for uranyl/uranium phosphates have been calculated and are given in Tables 6–10.

TABLE 8

Thermodynamic functions of solid $UO_2(PO_3)_2$

T (K)	$C_p^\ominus(T)$ ($J\ K^{-1}\ mol^{-1}$)	$S^\ominus(T)$ ($J\ K^{-1}\ mol^{-1}$)	$-$GEF ($J\ K^{-1}\ mol^{-1}$)	$H^\ominus(T) - H^\ominus(298\ K)$ ($kJ\ mol^{-1}$)	$\Delta H_f^\ominus(T)$ ($kJ\ mol^{-1}$)	$\Delta G_f^\ominus(T)$ ($kJ\ mol^{-1}$)	$\log K_p^\ominus$
298.15	177.11	195.60	195.60	0.00	−2973.00	−2747.32	481.31
300	177.87	196.70	195.60	0.33	−2973.03	−2745.92	478.10
317.3	184.51	206.86	195.96	3.46	−2973.25	−2732.82	449.88
317.3	184.51	206.86	195.96	3.46	−2974.57	−2732.82	449.88
400	208.73	252.48	203.01	19.79	−2974.95	−2669.73	348.53
500	229.53	301.40	217.90	41.75	−2973.74	−2593.54	270.64
550	238.27	323.69	226.51	53.45	−2972.61	−2555.57	242.70
550	238.27	323.69	226.51	53.45	−2996.89	−2555.57	242.70
600	246.37	344.76	235.48	65.57	−2994.80	−2515.54	218.99
700	261.34	383.89	253.93	90.97	−2989.90	−2436.04	181.78
800	275.31	419.71	272.45	117.81	−2984.17	−2357.29	153.91
900	288.70	452.92	290.69	146.01	−2977.69	−2279.32	132.29
942	294.21	466.21	298.22	158.25	−2974.77	−2246.79	124.58
942	294.21	466.21	298.22	158.25	−2977.56	−2246.79	124.58
1000	301.74	484.01	308.48	175.53	−2972.97	−2201.93	115.02
1049	308.03	498.60	317.03	190.47	−2968.80	−2164.25	107.77
1049	308.03	498.60	317.03	190.47	−2973.56	−2164.25	107.77
1100	314.53	513.37	325.78	206.35	−2968.71	−2125.01	100.91

TABLE 9

Thermodynamic functions of solid UPO_5

T (K)	$C_p^\ominus(T)$ (J K^{-1} mol^{-1})	$S^\ominus(T)$ (J K^{-1} mol^{-1})	$-$GEF (J K^{-1} mol^{-1})	$H^\ominus(T) - H^\ominus(298\ \mathrm{K})$ (kJ mol^{-1})	$\Delta H_f^\ominus(T)$ (kJ mol^{-1})	$\Delta G_f^\ominus(T)$ (kJ mol^{-1})	log $K_p^\ominus$
298.15	124.16	135.20	135.20	0.00	−2064.30	−1924.56	337.17
300	124.46	135.97	135.20	0.23	−2064.30	−1923.69	334.94
317.3	127.16	143.02	135.42	2.41	−2064.30	−1915.58	315.34
317.3	127.16	143.02	135.42	2.41	−2064.96	−1915.58	315.34
400	138.02	173.74	140.27	13.39	−2064.77	−1876.66	245.06
500	148.89	205.73	150.23	27.75	−2063.87	−1829.73	191.15
550	153.87	220.16	155.94	35.32	−2063.19	−1806.35	171.55
550	153.87	220.16	155.94	35.32	−2077.31	−1806.35	171.55
600	158.68	233.75	161.87	43.13	−2076.49	−1781.74	155.11
700	167.97	258.92	173.96	59.47	−2074.49	−1732.77	129.30
800	176.99	281.94	186.04	76.72	−2072.06	−1684.11	109.96
900	185.85	303.30	197.90	94.86	−2069.26	−1635.79	94.94
942	189.54	311.86	202.78	102.75	−2067.99	−1615.59	89.58
942	189.54	311.86	202.78	102.75	−2070.78	−1615.59	89.58
1000	194.62	323.33	209.44	113.89	−2068.21	−1587.63	82.93
1049	198.89	332.74	214.98	123.53	−2065.84	−1564.14	77.88
1049	198.89	332.74	214.98	123.53	−2070.60	−1564.14	77.88
1100	203.32	342.29	220.67	133.78	−2067.71	−1539.58	73.11

TABLE 10

Thermodynamic functions of solid UP_2O_7

T (K)	$C_p^\ominus(T)$ (J K^{-1} mol^{-1})	$S^\ominus(T)$ (J K^{-1} mol^{-1})	$-$GEF (J K^{-1} mol^{-1})	$H^\ominus(T) - H^\ominus(298\ \mathrm{K})$ (kJ mol^{-1})	$\Delta H_f^\ominus(T)$ (kJ mol^{-1})	$\Delta G_f^\ominus(T)$ (kJ mol^{-1})	log $K_p^\ominus$
298.15	184.00	197.10	197.10	0.00	−2852.00	−2657.33	465.55
300	184.51	198.24	197.11	0.34	−2851.99	−2656.12	462.47
317.3	188.98	208.71	197.46	3.57	−2851.86	−2644.83	435.39
317.3	188.98	208.71	197.46	3.57	−2853.18	−2644.83	435.39
400	204.69	254.37	204.62	19.90	−2852.31	−2590.62	338.30
500	217.29	301.48	219.40	41.04	−2850.37	−2525.41	263.82
550	222.34	322.44	227.84	52.03	−2849.17	−2492.97	236.76
550	222.34	322.44	227.84	52.03	−2873.44	−2492.97	236.76
600	226.91	341.98	236.55	63.26	−2871.44	−2458.47	214.03
700	235.12	377.59	254.20	86.37	−2867.22	−2389.98	178.34
800	242.58	409.48	271.66	110.26	−2862.79	−2322.10	151.62
900	249.60	438.46	288.60	134.87	−2858.25	−2254.79	130.86
942	252.46	449.91	295.54	145.42	−2856.32	−2226.67	123.47
942	252.46	449.91	295.54	145.42	−2859.11	−2226.67	123.47
1000	256.35	465.11	304.94	160.17	−2856.06	−2187.82	114.28
1049	259.59	477.45	312.71	172.81	−2853.36	−2155.14	107.31
1049	259.59	477.45	312.71	172.81	−2858.12	−2155.14	107.31
1100	262.93	489.85	320.63	186.14	−2854.95	−2121.03	100.72

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