Cesium – Oxygen – Uranium

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Introduction

The phase equilibria in the Cs-U-O system and the thermodynamic properties of cesium uranates have been extensively investigated in the past decades, because cesium is one of the main volatile elements synthesized in the fission products of uranium. Experimental investigations are listed in Table 1.

Binary Systems

The phase diagram of the Cs-U binary system is unknown. However, no mutual solubility has been reported. The O-U system is accepted from the Calphad assessment of [2004Che]. A precise model of the solid and liquid oxide solutions taking into account the oxygen vacancies in the O-U system may be found in [2002Gue]. The Cs-O binary system is taken from [Mas2].

Solid Phases

Crystallographic data of binary and ternary oxides are listed in Table 2. Uranium has the unusual property of forming with oxygen strong covalent bonds in one dimension and weak electrostatic bonds in the two other dimensions [2002Kin]. This directional anisotropy leads to low dimensional materials consisting of chains or layers constructed from oxygen-uranium networks and explains the easy volatilization of compounds such as $Cs_2U_4O_{12}$ [1999Hua]. Although UO_2 presents a large stability domain in the oxygen potential-temperature diagram, Cs_2O which is known as a basic oxide stabilizes the higher oxidation states of uranium, so that the cesium uranates known to be stable, namely Cs_2UO_4, $Cs_2U_2O_7$, $Cs_4U_5O_{17}$ and $Cs_2U_4O_{13}$ lie on the Cs_2O-UO_3 line in which uranium is in a state of oxidation VI. These compounds may be easily obtained by heating a mixture $U_3O_8 + Cs_2CO_3$ during 48 to 72 h under an air atmosphere at 800°C [2000Ber]. Cs_2UO_4 is non stoichiometric and may loose oxygen with formation of $Cs_2UO_{3.56}$ [1981Lin, 2000Ber, 2005Wal]. Cs_2UO_4 is stable under dry air up to 950°C. It decomposes at 630°C ($2\ Cs_2UO_4 \rightleftharpoons Cs_2U_2O_7 + Cs_2O$) only if some moisture is present [1983Dha]. The role of moisture is to displace the decomposition equilibrium with formation of volatile CsOH. This compound may also be obtained by heating a mixture $4U_3O_8 + 3Cs_2CO_3$ during 12 h at 1100°C under a CO_2 atmosphere [2000Ber]. The formation of $Cs_4U_5O_{17}$ has also been reported and its thermodynamic properties have been measured [1997Jay]. In open air atmosphere, $Cs_4U_5O_{17}$ decomposes around 1000°C with formation of the mixture $Cs_2U_4O_{13} + Cs_2U_2O_7$ [2000Ber]. The structures of the compounds $Cs_2U_4O_{13}$ and $Cs_2U_4O_{12}$ are closely related to that of UO_2 and it is not clear whether $Cs_2U_4O_{13}$ and $Cs_2U_4O_{12}$ may be considered as the two end members of the same solid solution. The formation of $Cs_4U_5O_{17}$ is not observed in the fuel pellets and this compound is often considered as metastable, which is not confirmed by the emf measurements of [1997Jay]. The existence of compounds with high uranium content, namely $Cs_2U_5O_{16}$, $Cs_2U_6O_{18}$, $Cs_2U_7O_{22}$, $Cs_2U_9O_{27}$, $Cs_2U_{15}O_{46}$, and $Cs_2U_{16}O_{49}$ suggested by [1974Cor, 1981Lin] has not been confirmed [2005Wal], notwithstanding that [1978Fee, 1981Lin] proposes an evaluation of the thermodynamic parameters of these phases. [1976Egm1] states that the structure of the so-called $Cs_2U_5O_{16}$ compound is very similar to that of $Cs_2U_4O_{13}$ and shows the existence of a series of solid solution at 600-1000°C with a Cs/U ratio ranging from 0.375 and 0.500.

Quasibinary Systems

The solubility of Cs_2O in UO_2 was measured at 1900°C [1993Kle] by annealing an UO_2-3 mass% Cs_2O pellet in a ThO_2 crucible inserted in a Ta capsule. The chemical potential of oxygen in the capsule was estimated lower than –450 $kJ \cdot mol^{-1}$, which corresponds to the Ta/Ta_2O_5 equilibrium. The solubility of cesium was evaluated by quantitative X-ray microanalysis at 0.07 mass% Cs, which corresponds to

0.08 mol% Cs_2O. No thermodynamic equilibrium was attained between Cs_2O and UO_2 in the annealing experiments at 1000°C.

Isothermal Sections

The Cs-O-U diagram in the solid state given in Figs. 1a, 1b is taken from [2005Wal]. This diagram may be used in the temperature range between 25 and 727°C, in which no transformation occurs and all the phases remain solid. Each triangle labelled with a small letter (*a, b, ...p*) on the diagram is characterized by an oxygen pressure at equilibrium depending on the temperature. These oxygen pressures between 0 and 500°C are shown in Fig. 2. There is a correspondence between the letters (*a, b, ...m*) in both figures. The equilibrium *i* (oxidation of $Cs_2U_2O_7$ + $Cs_2U_4O_{12}$ in $Cs_4U_5O_{17}$), taken from [1997Jay] is characterized by an oxygen pressure higher than that of equilibrium *e* (oxidation of Cs_2UO_4 + UO_2 in $Cs_2U_4O_{12}$), which is a strong argument for the stability of $Cs_4U_5O_{17}$. The oxygen pressures at equilibrium inside the triangles *n* (oxidation of $Cs_2U_2O_7$ in CsO_2 + $Cs_4U_5O_{17}$), *o* (oxidation of $Cs_4U_5O_{17}$ in CsO_2 + $Cs_2U_4O_{13}$) and *p* (oxidation of $Cs_2U_4O_{13}$ in CsO_2 + UO_3) are much higher than 1 bar. They have not be determined experimentally and thus are not shown in Fig. 2. The triangles in Figs. 1a, 1b which are not labelled with a letter represent a solid solution in equilibrium with a variable oxygen pressure. Under very high oxygen pressures, higher than those given by equilibrium *p*, gaseous oxygen is in equilibrium with CsO_2 and UO_3.

Thermodynamics

The thermodynamic properties of pure uranates are given in Table 3. These data were first measured by [1974OHa, 1975OHa], but a more recent evaluation of [1981OHa, 1986Cor], give the more acceptable results which are presented in Table 3. The thermodynamic properties of the compound $Cs_4U_5O_{17}$ has been measured by [1997Jay] from the oxygen pressure at equilibrium in the triangle $Cs_4U_5O_{17}$-$Cs_2U_2O_7$-$Cs_2U_4O_{12}$. Another evaluation of the thermodynamic properties of cesium uranates may be found in [1978Fee]. They have not been reported in Table 3 because they do not well agree with later evaluations [1997Jay, 2005Wal]. The enthalpies of formation and entropies of the mixed oxides $Cs_2U_5O_{16}$, $Cs_2U_6O_{18}$, $Cs_2U_7O_{22}$, $Cs_2U_9O_{27}$ and $Cs_2U_{15}O_{46}$, estimated by [1981Lin] are reported in Table 3; however, their stability has never been proved experimentally. The enthalpy of the $\alpha Cs_2U_4O_{12}$ /$\beta Cs_2U_4O_{12}$ transition at 625°C was measured at 190 J per mole of atom [1980Cor]. The stability domain of cesium uranates in the (p_{O2}, p_{Cs}) diagram at 727°C (1000 K), calculated by [1980Cor] are reproduced in Fig. 3.

Miscellaneous

Pure cesium uranate phases ($Cs_2U_2O_7$, $Cs_4U_5O_{17}$, $Cs_2U_4O_{13}$ and $Cs_2U_4O_{12}$) were prepared and analyzed by X-ray photoelectron spectroscopy (XPS), a technique which is in principle able of discerning the different oxidation states [2000Ber] of the same element. The last compound ($Cs_2U_4O_{12}$) in which the mean oxidation state of uranium is +5.5 has been shown to hold an equal quantity of U(V) and U(VI). It may thus be written $Cs_2U_2^{(V)}U_2^{(VI)}O_{12}$.

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Table 1: Investigations of the Cs-O-U Phase Relations, Structures and Thermodynamics

Reference	Method/Experimental Technique	Temperature/Composition/Phase Range Studied
[1974Cor]	X-ray diffraction, DTA, emf measurements,	600-1000°C, $p(O_2)$ < 10 MPa UO_3-Cs_2UO_4
[1974OHa]	Enthalpies measurements by solution calorimetry in HCl	25°C, Cs_2UO_4
[1975Cor]	X-ray analysis, thermal analysis	< 1000°C, UO_3-Cs_2UO_4
[1975OHa]	Enthalpies measurements by solution calorimetry in HCl	25°C, $Cs_2U_2O_7$
[1976Egm1]	X-ray diffraction	< 1000°C, $Cs_2U_5O_{16}$-$Cs_2U_4O_{13}$
[1976Egm2]	X-ray diffraction	< 1000°C, $Cs_2U_2O_7$
[1976Osb]	Heat capacity, entropy and Gibbs energy measurements	5-350 K, Cs_2UO_4
[1978Fee]	Powder X-ray analysis	600-1000°C, Cs-Cs_2O-UO_2
[1980Cor]	Heat capacity, entropy and enthalpies measurements	5-1070 K, α and β$Cs_2U_4O_{12}$
[1981OHa]	Heat capacity, entropy and enthalpies measurements	5-350 K, β$Cs_2U_2O_7$
[1983Dha]	Thermogravimetric analysis, vapor pressure evaluations	Cs_2UO_4-$Cs_2U_2O_7$ 725-1125°C
[1986Cor]	Enthalpies measurements by solution calorimetry with H_2SO_4 and with HF	25°C, Cs_2UO_4
[1993Kle]	Solubility measurements by X-Ray diffraction	1900°C, 200 bar ($p(O_2)$ < 10 MPa UO_2-3 mass% Cs_2O
[1997Jay]	Emf and calorimetric measurements	< 1000°C $Cs_4U_5O_{17}$-$Cs_2U_2O_7$-$Cs_2U_4O_{12}$
[1999Hua]	Mass spectrometric measurements with Knudsen' effusion cell	1000-1300°C $Cs_2U_4O_{12}$

Reference	Method/Experimental Technique	Temperature/Composition/Phase Range Studied
[2000Ber]	X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS)	25°C, $Cs_2U_2O_7$, $Cs_4U_5O_{17}$, $Cs_2U_4O_{13}$, $Cs_2U_4O_{12}$
[2002Ber1]	Powder neutron and X-ray diffraction	25°C, α$Cs_2U_4O_{12}$
[2002Ber2]	XRD, XPS, Extended X-ray Absorption Fine Structure (EXAFS)	25°C, Cs_2UO_4, $Cs_2U_2O_7$, $Cs_4U_5O_{17}$, $Cs_2U_4O_{12}$
[2005Wal]	Solid phase equilibria	350°C, Cs-O-U

Table 2: Crystallographic Data of Solid Phases

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
(Cs) < 28.39	*cI*2 *Im*$\bar{3}$*m* W	*a* = 614.1	at 25°C [Mas2]
(αU) < 668	*oC*4 *Cmcm* αU	*a* = 285.37 *b* = 586.95 *c* = 495.48	at 25°C [Mas2]
(βU) 776 - 668	*tP*30 $P4_2/mnm$ βU	*a* = 1075.9 *c* = 565.6	at 25°C [Mas2]
(γU) 1135 - 776	*cI*2 *Im*$\bar{3}$*m* W	*a* = 352.4	[Mas2]
Cs_7O < 4.33	*hP*24 *P*$\bar{6}$*m*2 Cs_7O	*a* = 1639.3 *c* = 919.3	at 0°C [Mas2, V-C2]
Cs_4O < 11.5	-	-	[Mas2]
$Cs_{11}O_3$ < 52.5	*mP*56 $P2_1/c$ $Cs_{11}O_3$	*a* = 1761.0 *b* = 921.8 *c* = 2404.7 β = 100.14°	[Mas2, V-C2]
Cs_3O < 166	-	-	23 to 25 at.% O [Mas2]
Cs_2O < 490	*hR*9 *R*$\bar{3}$*m* Sm	*a* = 425.6 *c* = 1899.2	[Mas2, V-C2]
Cs_2O_2 < 590	*oI*8 *Immm* Cs_2O_2	*a* = 432.2 *b* = 751.7 *c* = 643.0	[Mas2, V-C2]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
Cs_2O_3 < 502	*cI*28 $I\bar{4}3d$ Th_3P_4	*a* = 988	[Mas2, V-C2]
αCsO_2 (r) < 200	*tI*6 *I*4/*mmm* CaC_2	*a* = 446.2 *c* = 732.6	[Mas2, V-C2]
βCsO_2 (h) 432 - 200	*cF*8 $Fm\bar{3}m$ NaCl	*a* = 662	[Mas2, V-C2]
UO_2	*cF*12 $Fm\bar{3}m$ CaF_2	*a* = 547.0	from 62.5 to 66.7 at.% O [2002Gue]
U_3O_8 < 1870	*oC*44 *Cmcm*	*a* = 706.9 *b* = 1144.5 *c* = 830.3	[2004Che]
UO_3 < 669	*cP*4 $Pm\bar{3}m$ ReO_3	*a* = 414.6	[2004Che]
* Cs_2UO_4 < 950	*tI** *I*4/*mmm*	*a* = 439.1 *c* = 1482.3	[2002Ber2] [1983Dha]
* $Cs_2UO_{3.56}$	-	-	Structure unknown [2000Ber]
* α$Cs_2U_2O_7$ (r) < 300	*mC*22 *C*2/*m*	*a* = 1452.8 ± 0.3 *b* = 426.38 ± 0.07 *c* = 760.5 ± 0.1 β = 112.93°	[1975Cor, 1976Egm2]
* β$Cs_2U_2O_7$ (h) 900 - 300	*mC*22 *C*2/*m*	*a* = 1452.93 *b* = 432.33 *c* = 748.99 β = 113.852°	[2002Ber2]. decomposes at 900°C in open atmosphere [2000Ber]
* γ$Cs_2U_2O_7$	*hP*11 $P6_3/mmc$	*a* = 410.8 ± 0.1 *c* = 1464.6 ± 0.5	metastable. Gives rapidly β$Cs_2U_2O_7$ at 800°C [1976Egm2]
* $Cs_4U_5O_{17}$	*oP*104 *Pbcn*	*a* = 1875.99 *b* = 706.38 *c* = 1495.48	[2002Ber2]
* $Cs_2U_4O_{13}$ < 1000	*oC*95 *Cmcm*	*a* = 1349.4 ± 0.2 *b* = 1547.6 ± 0.2 *c* = 791.1 ± 0.2	[1975Cor] above 1000°C, gives an orthorhombic solid solution with $Cs_2U_5O_{16}$ [1976Egm1]
* α$Cs_2U_4O_{12}$ (r) < 625	*hR*108 $R\bar{3}$ $RbNiCrF_6$	*a* = 1542.32 *c* = 1918.16	modified pyrochlore structure [2002Ber1]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
* $\beta Cs_2U_4O_{12}$ (h1) 695 - 625	*m***	-	[1974Cor]
* $\gamma Cs_2U_4O_{12}$ (h2) > 695	*cF*72 $Fd\bar{3}m$	a = 1122.95	[1974Cor]
* $Cs_2U_5O_{16}$	*mC*92 *C*2/*m*	a = 1346.5 ± 0.2 b = 1556.1 ± 0.2 c = 796.4 ± 0.2 β = 92.78°	[1975Cor] above 1000°C, gives a solid solution with $Cs_2U_4O_{13}$ [1976Egm1]
* $Cs_2U_6O_{18}$ 1050 - ~900	*mP*26 $P2_1/c$	a = 413.7 ± 0.1 b = 1347.1 ± 0.1 c = 808.9 ± 0.2 β = 90.37°	[1975Cor, 1976Egm1]
* $Cs_2U_7O_{22}$ < 720	*oP*62 *Pbam*	a = 694.9 ± 0.1 b = 1971.1 ± 0.2 c = 739.55 ± 0.08	[1975Cor]
* $Cs_2U_9O_{27}$ 900 - 720	*oP*38 *P***	a = 1495.6 ± 0.4 b = 1057.1 ± 0.3 c = 398.56 ± 0.07	[1975Cor]
* $Cs_2U_{15}O_{46}$ < 720	*oC*252 *Cmca*	a = 1469.0 ± 0.3 b = 1343.5 ± 0.2 c = 1974.1 ± 0.4	[1981Lin] labelled $Cs_2U_{16}0_{49}$ in [1975Cor]

Table 3: Thermodynamic Properties of Single Phases

Phase	Temperature Range [°C]	Property, per mole of atoms [J, mol, K]	Comments
1/7 (Cs_2UO_4)	25	C_p = 21.82 ± 0.05	[1976Osb]
		$S°$ = 31.38 ± 0.06	[1976Osb]
		$\Delta_f S°$ = – 58.70 ± 0.10	[1976Osb]
		$\Delta_f H°$ = – 275 500 ± 500	[1986Cor]
	750-900	$\Delta_f G°$ = – 275 150 + 31.57 T	[1997Jay]
1/6.65 ($Cs_2UO_{3.56}$)	25	$S°$ = 36.43 ± 0.06	[1981Lin]
		$\Delta_f H°$ = – 265 200 ± 500	[1981Lin]
	750-900	$\Delta_f G°$ = – 267 540 + 52.31 T	[1997Jay]
1/11 ($\alpha Cs_2U_2O_7$)	25	C_p = 21.02 ± 0.04	[1981OHa]
		$S°$ = 29.80 ± 0.06	[1981OHa]
		$\Delta_f H°$ = – 292 750 ± 500	[1981OHa]
1/11 ($\beta Cs_2U_2O_7$)	750-900	$\Delta_f G°$ = – 290 800 + 59.64 T	[1997Jay]
1/26 ($Cs_4U_5O_{17}$)	25	C_p = 21.15 ± 0.05	[1997Jay]
		$S°$ = 29.90 ± 0.06	[1997Jay]
		$\Delta_f H°$ = – 294 000 ± 500	[1997Jay]
	750-900	$\Delta_f G°$ = – 297 270 + 64.65 T	[1997Jay]

Phase	Temperature Range [°C]	Property, per mole of atoms [J, mol, K]	Comments
1/19 ($Cs_2U_4O_{13}$)	25	$S°$ = 28.21 ± 0.06	[1981Lin]
		$\Delta_f H°$ = – 300 700 ± 500	[1981Lin]
	750-900	$\Delta_f G°$ = – 310 580 + 73.21 T	[1997Jay]
1/18 ($Cs_2U_4O_{12}$)	25	C_p = 21.33 ± 0.05	[1980Cor]
		$S°$ = 29.25 ± 0.06	[1980Cor]
		$\Delta_f H°$ = – 309 660 ± 500	[1980Cor]
	25-625	$\Delta_f G°$ = – 318 100 + 69.72 T	[1997Jay]
1/23 ($Cs_2U_5O_{16}$)	25	$S°$ = 27.65 (evaluation)	[1981Lin]
		$\Delta_f H°$ = – 302 500 (evaluation)	[1981Lin]
1/26 ($Cs_2U_6O_{18}$)	25	$S°$ = 27.04 (evaluation)	[1981Lin]
		$\Delta_f H°$ = – 311 000 (evaluation)	[1981Lin]
1/31 ($Cs_2U_7O_{22}$)	25	$S°$ = 26.90 (evaluation)	[1981Lin]
		$\Delta_f H°$ = – 304 300 (evaluation)	[1981Lin]
1/38 ($Cs_2U_9O_{27}$)	25	$S°$ = 26.26 (evaluation)	[1981Lin]
		$\Delta_f H°$ = – 310 800 (evaluation)	[1981Lin]
1/63 ($Cs_2U_{15}O_{46}$)	25	$S°$ = 25.83 (evaluation)	[1981Lin]
		$\Delta_f H°$ = – 306 000 (evaluation)	[1981Lin]

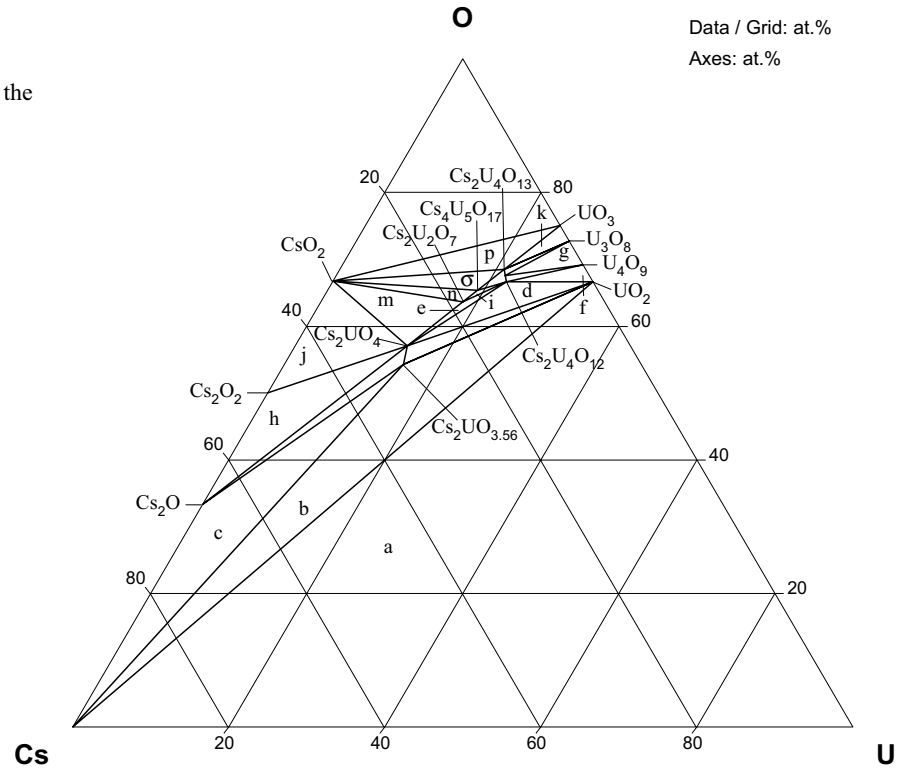

Fig. 1a: Cs-O-U. Phase equilibria in the solid state

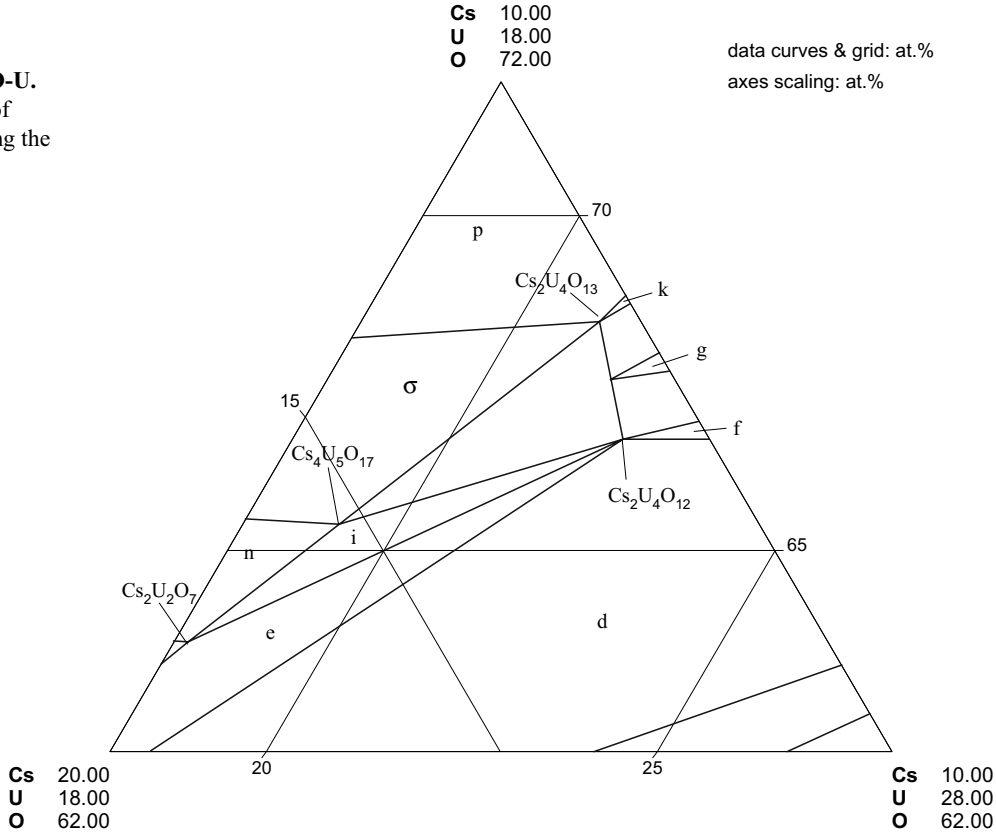

Fig. 1b: Cs-O-U. Enlargement of Fig. 1a showing the "i" domain

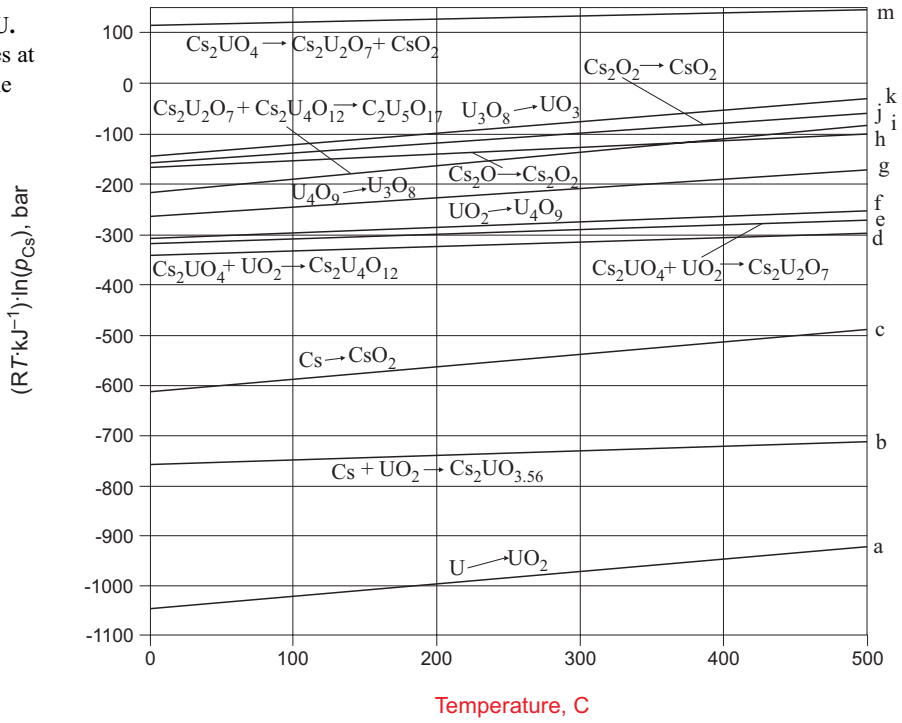

Fig. 2: Cs-O-U. Oxygen pressures at equilibrium in the solid state

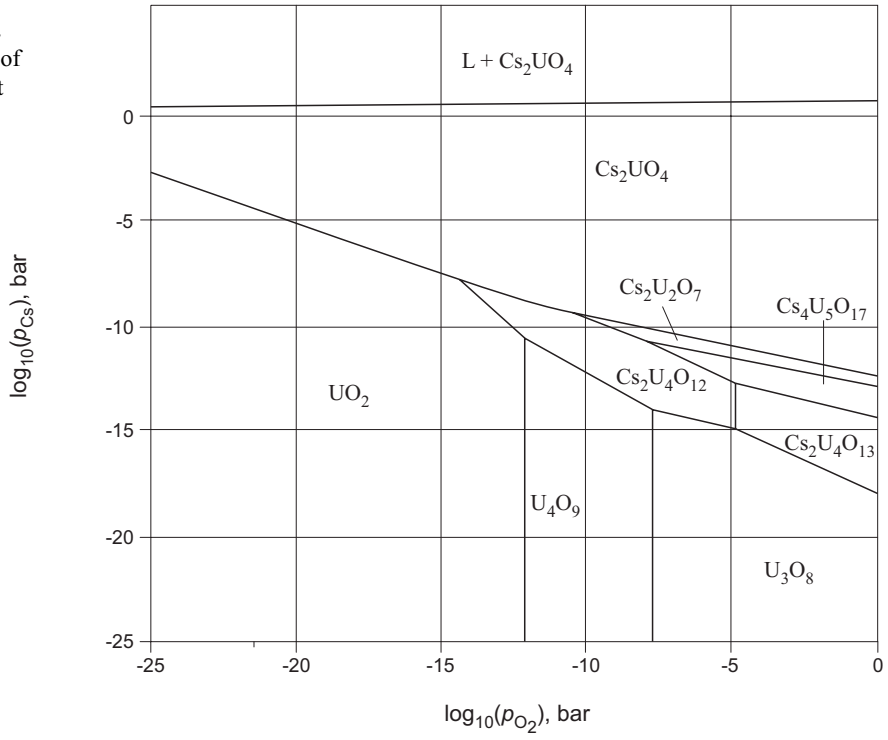

Fig. 3: Cs-O-U. Stability domains of cesium uranates at 727°C (1000 K)