

**THERMODYNAMIC FACTORS AFFECTING $UC_{1-x}N_x$
IRRADIATION AND SYNTHESIS**

T. B. Lindemer

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Nuclear Science and Technology Division

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1. INTRODUCTION

There is interest in attempting to synthesize nearly pure uranium nitride (UN) kernels for high-temperature gas-cooled reactor (HTGR) fuel. Because the proposed process involves carbothermic conversion of a uranium-carbon mixture in nitrogen and because there is a complete ideal solution of uranium carbide (UC) and UN, which is written as $UC_{1-x}N_x$, the practical value of x for fuel irradiation needs to be determined. Insight is to be gained by relevant thermodynamic calculations of carbide-nitride equilibria for the fuel and fission product systems. The equilibria are readily compared on the nitrogen-based Ellingham diagram, which, in turn, provides guidance in interpreting past irradiations and in synthesis of the $UC_{1-x}N_x$ kernels.

2. THE ELLINGHAM DIAGRAM

The Ellingham diagram (Fig. 1), which plots the chemical potential of nitrogen for various equilibria versus temperature, is an aid in understanding the complexity of the HTGR UN-containing fuel. This diagram is exactly analogous to that developed by Homan et al. for the U-C-O-fission product system (ref. 1). In an HTGR coated particle, $UC_{1-x}N_x$ is in a carbon-containing environment. Consequently, the relevant equilibria are carbide plus nitrogen in equilibrium with nitride plus carbon. Thermodynamic data used here are given in Appendix A. It should be noted that thermodynamic data for nitrides are sparse, and some of those are estimated. However, sufficient data exist to permit reasonably accurate calculations for the systems to be considered here. These include the Si-C-N, U-C-N, Sr-C-N, Ce-C-N, Mo-C-N, and Zr-C-N systems. The latter four represent the alkaline earth, yttrium and rare earth (RE), molybdenum, and zirconium fission-product systems; these are the only fission-product groups that may form nitrides.

The Ellingham diagram is constructed from calculated equilibria involving 1 mol of nitrogen gas. The chemical potential of nitrogen is defined as $RT\ln(p^*[N_2])$, in which R is 1.987 cal/(mol · K), T is in kelvins, and $p^*[N_2]$ is the ratio of nitrogen pressure divided by the standard-state pressure of nitrogen, which is 0.101 MPa, or 1 atm. Conveniently for understanding, $p^*[N_2]$, although dimensionless, has values equivalent to atmospheres. The standard Gibbs free energy change for an equilibrium at temperature is given by

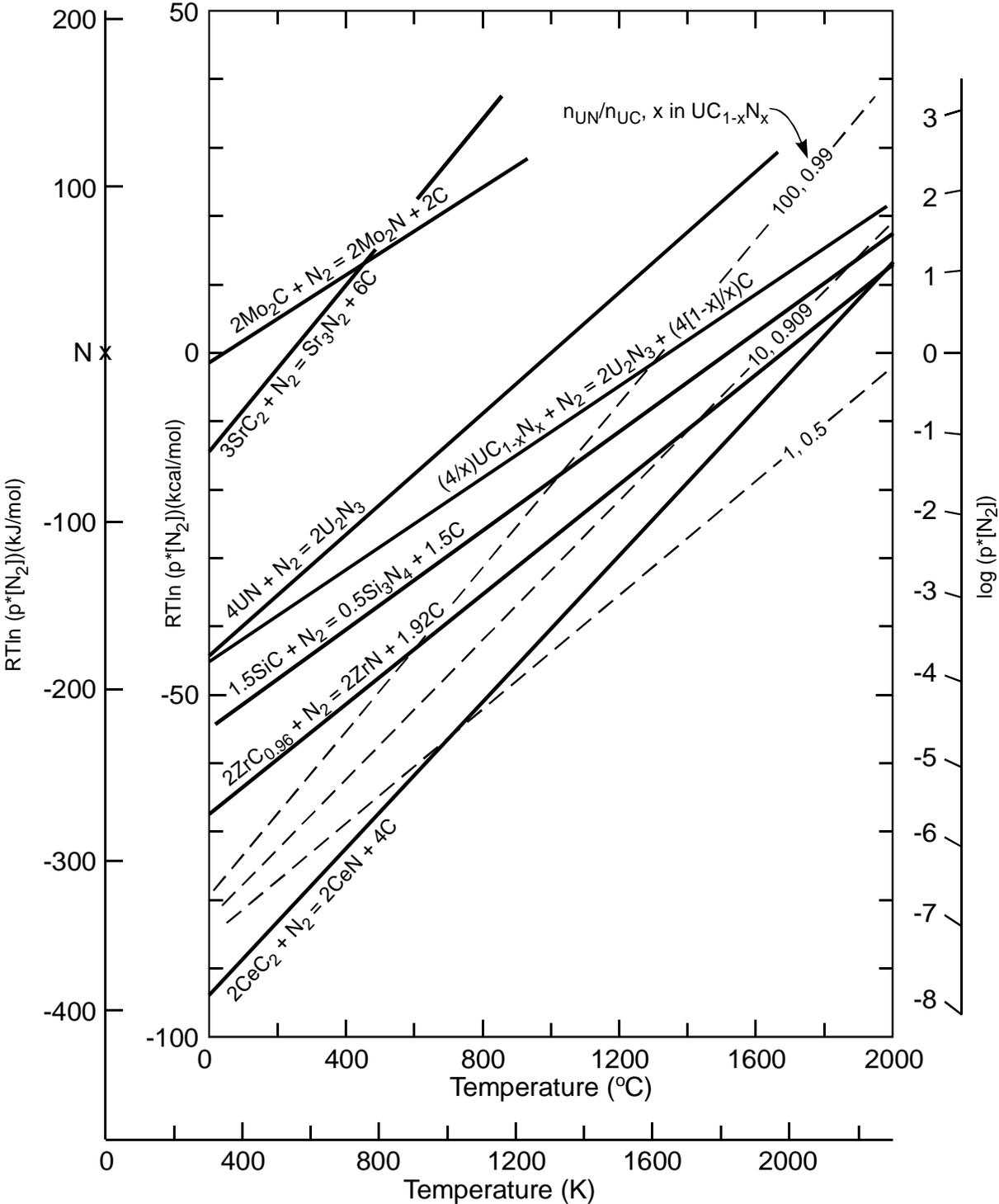
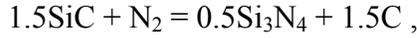


Fig. 1. The Ellingham diagram for nitrogen-containing systems relevant to the fission of UN-containing fuel in a gas-cooled reactor. Phases and equilibria at a given nitrogen pressure (in atm) and temperature can be obtained by placing a ruler on “X” near “N” on the vertical scale at 0 K and on the desired log pressure given on the vertical scale at the extreme right of the figure.

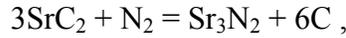
$$\Delta G^{\circ}_T = \Delta H^{\circ}_{f,298} - T\Delta S^{\circ}_{298},$$

in which $\Delta H^{\circ}_{f,298}$ and ΔS°_{298} are the usual differences in heat of formation and absolute entropy, respectively, between products and reactants at 298 K and all phases have unit activity. The accuracy of the data is on the order of 1 kcal/mol.

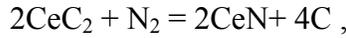
The equilibria and corresponding nitrogen potentials for the silicon and fission-product systems are listed below.



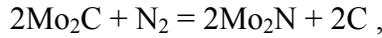
$$RT\ln(p^*[\text{N}_2]) = -65,000 + 36.14T,$$



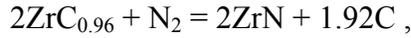
$$RT\ln(p^*[\text{N}_2]) = -32,800 + 59.05T,$$



$$RT\ln(p^*[\text{N}_2]) = -109,600 + 53.09T,$$



$$RT\ln(p^*[\text{N}_2]) = -11,200 + 32.43T,$$

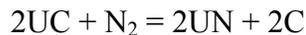


and

$$RT\ln(p^*[\text{N}_2]) = -79,460 + 40.42T.$$

The nitrogen potentials are plotted in the Ellingham diagram given in Fig. 1. It can be seen from the thermodynamic data in Appendix A that the nitride-carbide equilibria for the Sr and Ba systems would plot similarly in Fig. 1, as would those for the Y, Ce, and La systems (and, presumably, the rest of the RE systems).

The expression for the nitrogen potential for the U-C-N system is different because $\text{UC}_{1-x}\text{N}_x$ is nearly an ideal solid solution of UC and UN (ref. 2). Thus, for the equilibrium

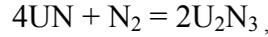


the nitrogen potential is

$$RT\ln(p^*[\text{N}_2]) = -97,400 + 41.25T + 2RT\ln(n_{\text{UN}}/n_{\text{UC}}),$$

in which n_{UN} and n_{UC} are the mole fractions of UN and UC, respectively, and $n_{UC} = 1 - n_{UN}$. The nitrogen potentials at n_{UN}/n_{UC} values of 1, 10, and 100 (i.e., at x values of 0.5, 0.909, and 0.990, respectively) are plotted in Fig. 1.

As is explained later, U_2N_3 may be present either during synthesis or upon irradiation and the equilibrium and nitrogen potential respectively, are as follows:



$$RT\ln(p^*[N_2]) = -57,200 + 44.05T .$$

The latter value is plotted in Fig. 1. A more exact calculation of the conditions for the appearance of U_2N_3 was derived from ref. 3, which provided an assessment of the U-C-N system that appears in Fig. 9064 of ref. 4 and is based on the critical analysis of ref. 2. The relevant equilibrium is



which is shown as locus “b” in Fig. 9064 of ref. 4. That figure gives $\log(p[N_2]\{\text{MPa}\})$ versus $10,000/T(\text{K})$, and locus “b” is at 2250 and 1053 K at 100 and 0.0001 atm N_2 , respectively. Converting the latter two values to nitrogen potential and fitting the T - $RT\ln(p^*[N_2])$ data give

$$RT\ln(p^*[N_2]) = -54,300 + 33.3T .$$

Figure 1 illustrates that the latter nitrogen potential is somewhat lower than that for the more approximate values for the equilibrium $4UN + N_2 = 2U_2N_3$. However, the nitrogen potentials for both equilibria illustrate that the value of x must reach at least 0.99 before U_2N_3 appears at normal fuel temperatures. Figure 1 also illustrates that if the inner pyrocarbon were breached in the UN-containing HTGR particle, the SiC may react with N_2 to produce Si_3N_4 at sufficiently high nitrogen potentials. This is analogous to CO corrosion of SiC in oxide-containing particles at sufficiently high CO pressures but differs in that there is no Si-N gaseous species analogous to the silicon-transporting species, SiO.

3. IRRADIATION OF $UC_{1-x}N_x$

In general, the methodology for deducing the change of kernel composition during the irradiation of $UC_{1-x}N_x$ is similar to that developed for the UO_2 - UC_2 kernel (ref. 1). In particular, as fission proceeds, not all of the nitrogen originally combined in the UN component can be captured by the fission products. Consequently, the value of x in the remaining $UC_{1-x}N_x$ increases and thus the nitrogen potential increases. Furthermore, depending on the initial value of x , the carbide-nitride equilibria for the REs and zirconium also come into play. If the nitrogen potential becomes sufficiently high, as certainly would occur with fission of nearly pure UN, then the nitrogen potential would be high enough to permit formation of U_2N_3 at fuel temperatures below 1000°C. Above that temperature, as seen in Fig. 1, the nitrogen pressure could exceed 1 atm. Figure 1 also shows that the Mo and Sr carbides are always stable for any irradiation condition because the relevant carbide-nitride equilibria are above that for the UN- U_2N_3 equilibrium.

The nitrogen release per fission for pure UN is calculated in the same way as is the oxygen release during fission of UO_2 (ref. 5). The nitrogen from fission of 100 mol of UN would combine to give 54.57 mol of (RE)N and 30.37 mol of ZrN, for a total of 84.94 mol of combined nitrogen. The remaining 15.06 mol of nitrogen atoms are not combined and are available, for instance, to increase the nitrogen potential (i.e., the pressure of N_2 , and thus the value of x) and, ultimately, to form U_2N_3 .

General spreadsheet calculations were performed for $UC_{1-x}N_x$ to reveal the phases present at burnups ranging from 0.1 to 0.9 at various initial values of x . These calculations ignore the thermodynamics of the fuel and fission-product system but give some insight into the system behavior. For example, if x equaled 0.5457, the yield of REs, the system remained at the RE carbide-nitride equilibrium. Similarly, if x equaled 0.8494, the yield of REs and zirconium, all the REs were nitrides and the system remained at the ZrC-ZrN equilibrium. These results are expected since, like the UN component of $UC_{1-x}N_x$, REs and zirconium form mononitrides. When $0.85 < x < 0.95$ initially, all the REs and zirconium were present as nitrides. The value of x in the remaining fuel increased from 0.99 to 0.999 and, at burnups above 60% fissions per initial metal atom (FIMA), resulted in some U_2N_3 formation and/or increase in nitrogen pressure above 1 atm, possibly to 50 atm, depending on temperature.

Including the thermodynamics of the system, as shown in Fig. 1, results in a more complicated interpretation of burnup. For example, at an irradiation temperature of 900°C and an initial value of x of 0.4, the nitrogen released by fission would first increase the x value of the remaining fuel until it reached 0.5457, at which point the nitrogen potential of the RE system, as represented by the equilibrium $2\text{CeC}_2 + \text{N}_2 = 2\text{CeN} + 4\text{C}$, would be attained, and the RE carbides would begin to convert to nitrides. If further burnup released enough nitrogen to completely convert the RE carbides to nitrides, then x of the remaining fuel would again increase to approximately 0.96, at which point the nitrogen potential of the ZrC-ZrN equilibrium would be attained and the system would remain at that nitrogen potential until all the ZrC was converted to ZrN. Then the value of x of the remaining fuel would again increase until uranium sesquinitride began to form. As another example, at 1300°C, the value of x of the fuel would have to reach about 0.75 before the RE carbide-nitride equilibrium was attained and about 0.91 before the zirconium carbide-nitride was attained. These calculations are more complicated than those for the $\text{UO}_2\text{-UC}_2$ kernel (ref. 1) because of the increase of x during burnup; calculations more precise than those done here would best be performed in a generalized thermodynamics program such as SOLGASMIX-PV (ref. 6) or its more modern successors.

The RE equilibria are the most relevant. The possible errors in the 298 K heat of formation for CeN and CeC_2 shown in Appendix A may lead to an error of $\pm 2(1.7 + 6)$ kcal/mol in the nitrogen potential for that equilibrium. If the true value of the equilibrium lies 15.4 kcal/mol more positive, it can be seen from Fig. 1 that no practical value of x would preclude the formation of RE carbides and thus $\text{UC}_{1-x}\text{N}_x$ would not be a useful HTGR fuel.

At this point, one can analyze the irradiation data of weak-acid-resin (WAR) $\text{UC}_{1-x}\text{N}_x$ in HRB-9 and -10 in tristructural isotropic (TRISO) particles (ref. 7). The 360- μm kernels contained 3.21 mol of free carbon and $\text{UC}_{0.47}\text{N}_{0.53}$ by chemical analysis and $\text{UC}_{0.37}\text{N}_{0.63}$ by lattice parameter measurements based on data in ref. 8. The fuel experienced 85% FIMA for ^{235}U . The design operating temperatures for HRB-9 were 1250°C centerline and 1000°C at the compact surface. Those for HRB-10 were 1500°C centerline and 1250°C at the compact surface. Metallographic exam showed no thermal migration but showed fission product attack of the SiC layer on the cold side of the particle and many failures due to attack of the

SiC by RE carbides, much like the attack observed in irradiations of WAR UC₂ and dense UC₂ particles.

The SiC corrosion by the RE carbides can be supported by the data in Fig. 1 and the burnup calculations. The initial kernel composition at a value of x of 0.53 is that where RE carbides are clearly present at the irradiation temperatures. The value of 0.63 would also apparently place the nitrogen potential slightly under that for the RE carbide-nitride equilibrium at the beginning of irradiation, and thus only RE carbides would be present. As fission takes place, x would increase from 0.63 and beyond the value of x for the RE carbide-nitride equilibrium. Whether the irradiations suggest that the RE nitrides also attack SiC is uncertain. If RE carbides are to be precluded, Fig. 1 indicates that the initial value of x must be above about 0.8 (i.e., above the value at which RE carbides are stable at any normal irradiation temperature).

This analysis of UC_{1-x}N_x irradiation leads to several conclusions. The observed corrosion of SiC in the HRB-9 and -10 irradiations was the probable result of the presence of RE carbides that were formed because the value of x was too low in the initial fuel. It is unknown whether irradiation of UC_{1-x}N_x with $0.85 < x < 0.95$, as suggested by the present analysis, would be successful, but one can state with reasonable confidence that the RE carbides would not be present and that U₂N₃ formation and nitrogen overpressure would be minimal. It is not known whether RE nitrides would migrate to the SiC and cause corrosion. Irradiation experience with ZrC, either as a fission-product phase or as an intentional additive to the fuel particle, shows that ZrC does not lead to SiC attack. The effect of ZrN is not known, but it should not attack the SiC either. Therefore, the choice of x is probably not dependent on whether ZrC and ZrN are present.

Given the low molar volume of UN, 17.61 cm³/mol and the general absence of nitrogen pressures above 1 atm during irradiation, a more compact HTGR fuel particle is possible. The buffer layer needs only to accommodate the Kr + Xe overpressure.

4. SYNTHESIS OF NEARLY PURE UN

From the irradiation experience analyzed above, it would appear that the initial x value in UC_{1-x}N_x fuel must place the nitrogen potential above that at which the RE carbides are present. The combination of nitrogen inventory calculations and RE thermodynamic data

suggests $0.85 < x < 0.95$ for the initial fuel. Figure 1 illustrates that n_{UN}/n_{UC} should be at least 10 (i.e., x should be at least 0.91).

The following paragraphs outline the thermodynamic parameters needed to guide the synthesis of the fuel with a high value of x . The direct method is the conversion of a UO_2 -C mixture in flowing nitrogen at temperatures high enough to generate a significant pressure of CO. The relevant equilibrium is



and the equilibrium constant leads to

$$(p^*[CO])^2 = (p^*[N_2])^{0.5} (n_{UN})^{-1} \exp([-135,760 + 65.19T]/RT) ,$$

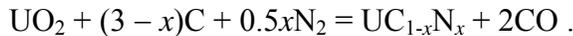
in which n_{UN} is x in $UC_{1-x}N_x$. Fortunately, in the desired process the flowing nitrogen is somewhere between 0.5 and 1 atm. Under these conditions x is close to unity and the product of the first two terms is close to unity. Thus, the above equation reduces to

$$(p^*[CO])^2 = \exp([-135,760 + 65.19T]/RT) ,$$

which is rearranged to give

$$\log(p^*[CO]) = (-135,760 + 65.19T)/(2RT \ln[10]) = -14,880/T + 7.12 .$$

From the latter equation, $p^*[CO]$ values of 1, 0.1, and 0.01 atm are attained at 2090, 1833, and 1632 K, respectively. As seen in Fig. 1, at $p^*[N_2]$ of 0.5 to 1 atm or $RT \ln(p^*[N_2])$ of about zero, the desired x values can be attained by processing between 1632 and 1833 K. These temperatures are probably too low to achieve sufficient reaction rates. Higher temperatures would lead to lower values of x but would have higher reaction rates and $1-x$ additional moles of carbon might need to be added to the initial mix: that is, the initial carbon content would be $2 + (1 - x)$ and the overall reaction would then be



After the UO_2 is completely reacted, the temperature could be lowered in flowing nitrogen so that the x value would increase while the excess carbon was rejected.

Further insight into the complexities of the above reaction can be found in refs. 9, 10, 11, and 12. Reference 12 in particular describes beneficial gas-phase transport of carbon by

HCN in N₂-H₂ atmospheres. As much as 4% hydrogen is safe in mixed gas. The resulting HCN would be useful in transporting carbon in the initial oxide-carbon mixture and may also be useful in stripping excess carbon from the UC_{1-x}N_x product, although the ~0.001-atm pressure of HCN at 1700°C would require a flow of ~100 mol of N₂-4% H₂ gas to remove 0.1 mol of carbon from 1 mol of UC_{1-x}N_x.

5. CONCLUSIONS

The HRB-9 and -10 irradiations of UC_{1-x}N_x with $0.53 \leq x \leq 0.63$ resulted in SiC corrosion. Thermochemical analysis of the fission-product carbide-nitride system suggests that the corrosion was caused by RE carbides at $x = 0.53$ and probably during the early stages of irradiation at $x = 0.63$. The present analysis suggests that UC_{1-x}N_x with $0.85 \leq x \leq 0.95$ would be an appropriate HTGR fuel composition. This conclusion is dependent on the present thermodynamic values of (RE)C₂ and (RE)N being correct. A more compact TRISO particle design may be possible because of the low molar volume of UC_{1-x}N_x. Irradiation of this fuel is calculated to preclude U₂N₃ formation and nitrogen overpressures above 1 atm until 60% FIMA is attained. Irradiation of materials with x values above 0.85 would resolve the question of whether RE nitrides would corrode SiC. High-temperature experiments with TRISO-coated RE nitride in a temperature gradient would also be useful and considerably less expensive than irradiations.

General guidelines have been given for the synthesis of UC_{1-x}N_x with $0.85 \leq x \leq 0.9$ from UO₂-C mixtures in flowing N₂ or N₂-4% H₂.

6. ACKNOWLEDGMENTS

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APPENDIX A: THERMODYNAMIC DATA

Table A.1. Thermodynamic data

	$\Delta H_{f,298}^{\circ}$ (kcal/mol)	S_{298}° [cal/(mol·K)]	$\Delta S_{f,298}^{\circ}$ (per N ₂) [cal/(mol·K)]
Elements			
Ba	0	14.92	
C	0	1.37	
Ce	0	17.2	
La	0	13.6	
Mo	0	6.85	
N ₂	0	45.77	
Sc	0	8.28	
Si	0	4.5	
Sr	0	12.5	
U	0	12.02	
Y	0	10.63	
Zr	0	9.32	
Carbides			
BaC ₂	-17.7 ± 3.0	21 ± 2.0	
CeC ₂	-23.2 ± 1.7	21.5 ± 2.0	
Mo ₂ C	-11.0 ± 0.7	15.7 ± 0.2	
SiC	-16.0 ± 1.5	3.95 ± 0.05	
SrC ₂	-20.2 ± 4.0	17.0 ± 2.0	
UC	-21.7 ± 1.0	14.08	
ZrC _{0.96}	-48.27 ± 0.6	7.93 ± 0.5	
Nitrides			
Ba ₃ N ₂	-81.5 ± 7.5	36.4 ± 2.0	-54.13
CeN	-78 ± 6.0	15.1	-50.0 ^a
LaN	-71.5 ± 4.0	10.6 ± 1.1	-51.77
Mo ₂ N	-16.6 ± 0.5	21.0	-31.17
ScN	-75.0 ± 4.0	7.1 ± 1.0	-48.13
Si ₃ N ₄	-178.0 ± 3.0	27.0 ± 2.0	-39.02
Sr ₃ N ₂	-93.4 ± 5.0	29.5 ± 2.5	-53.7
UN	-70.4 ± 1.0	14.97 ± 0.1	-39.87
U ₂ N ₃	-169.4 ± 2.5	30.8 ± 0.3	-41.27
YN	-71.5 ± 5.0	9.0 ± 1.2	-49.03
ZrN	-88.0 ± 0.6	9.29 ± 0.3	-45.83
Oxides			
CO	-26.42	47.22	
UO ₂	-259.0	18.6	

^aEstimated by comparison with ScN, YN, and LaN, then S_{298}° derived.

Source: O. Kubaschewski and C. B. Alcock, *Metallurgical Thermochemistry*, Pergamon Press, New York, 1979, Table A.

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