

# C-Cs (Carbon-Cesium) System

James Sangster

## Equilibrium Diagram

There is no phase diagram available for this system. It may be conjectured that it would be similar to that for the C-Li system, and so would show (a) the compound  $C_2Cs_2$ , (b) a liquid and (c) a eutectic of composition very close to 100 at.% Cs and temperature very close to the M.P. of Cs.

The alkali metals exhibit three types of intermediate compounds with carbon. For cesium, these may be designated

- (1) simple C-Cs compounds ( $C_2Cs_2$  in this case)
- (2) cesium-graphite intercalation compounds
- (3) cesium-fullerene exohedral and endohedral compounds

In the chemical literature, molecular formulas for C-Cs compounds have been written with either C or Cs placed first, without much consistency. In the present instance, C will always come before Cs.

$C_2Cs_2$  (cesium acetylide, cesium carbide) was prepared in two steps in liquid ammonia [1903Moi]. Acetylene was added to a solution of cesium in the solvent. The intermediate product was  $C_2Cs_2 \cdot C_2H_2$ , which upon heating in vacuo at 300 °C was converted to  $C_2Cs_2$ . According to a standard inorganic chemistry text [1924Mel], it may also be prepared by the reaction of molten cesium with acetylene. The melting behavior of  $C_2Cs_2$  has not been reported.

## Cesium-Graphite Intercalation Compounds

These are also called “lamellar,” “layered,” “interstitial” or “insertion” compounds. All alkali metals form graphite intercalation compounds, but those with Li or Na are fewer and are formed less easily than those of the heavier alkali metals. The structure of graphite and metal intercalation compounds is shown schematically in Fig. 1. Graphite has a layered structure, in which the carbon atoms are arranged in parallel planes, in each of which they form a network of hexagons [1971Nov]. There is no chemical bonding between the layers and so alkali metals can be inserted rather easily between the layers. The separation of the adjacent layers increases somewhat when alkali metal atoms are inserted.

The conventional method of indicating the stoichiometry of metal-graphite intercalation compounds is  $C_xM$ , where  $x$  may be an integer or fractional number. The formula  $C_xM$  does not represent a molecule of  $(x + 1)$  atoms; rather, it indicates merely the atom ratio C/M in a macroscopic sample.

The intercalation compounds (Fig. 1) are named “first stage,” “second stage,” etc. according to the number of graphite layers separating the metal layers. The stoichiometry  $C_xM$  assigned to a particular stage compound should be

regarded as nominal or average. The overall stoichiometry is established by chemical reaction and analysis, the particular stage by XRD.

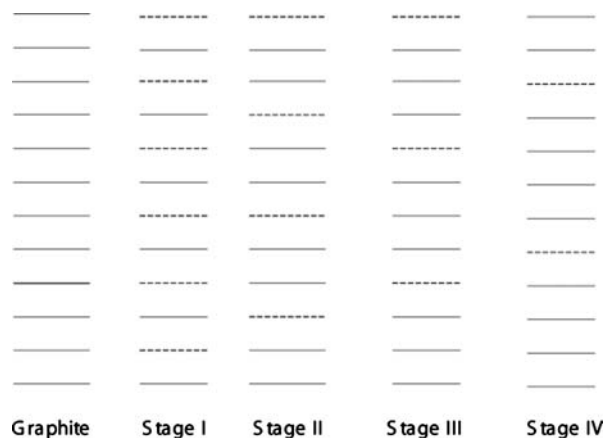
The preparation and properties of alkali metal intercalation compounds have been reviewed extensively [1959Hen, 1959Rud, 1960Cro, 1971Nov, 1975Ber, 1975Boe, 1976Ebe, 1977Her, 1980Sel, 1981Dre, 1981Her, 1982Bar, 1990Lag].

The common methods for the preparation of cesium-graphite intercalation compounds are

- reaction with cesium vapor at low pressures
- in organic solvent solution
- reaction with liquid cesium at high pressure

The preparation of compounds at high pressure is described later in the “Pressure” section of this paper. Both natural and synthetic graphite have been used in these methods.

In the reaction of graphite with cesium vapor (the “two-bulb method” [1951Her, 1955Her]), graphite in one part of a vacuum apparatus is maintained at a given temperature and is then exposed to cesium vapor from a reservoir of molten metal maintained at a lower temperature. This is a preferred method for preparing these compounds of definite stoichiometry. The stoichiometry of the compound is controlled by the temperature difference between the graphite and metal reservoir. The stoichiometry is ultimately established by chemical reaction. The characterization of cesium-graphite intercalation compounds is routinely done by XRD [1955Her, 1981Her]. Other means include electron diffraction [1977Chu], magnetic susceptibility [1977Gue], NMR [1981Miz] and Raman spectra [1977Ekl, 1977Nem].



**Fig. 1** Schematic representation of the structure of graphite and cesium-graphite intercalation compounds. *Solid lines*: graphite layers. *Dashed lines*: metal layers. The patterns repeat themselves in both directions

## Section II: Phase Diagram Evaluations

The stage I compound is  $C_8Cs$  and the successive stages may be represented by  $C_{12n}Cs$ , where  $n$  is the stage number ( $n \geq 2$ ). Compounds of stages I-X have been prepared by the two-bulb method [1970Car, 1980Bil, 1988Caj]. The higher stage-stage compounds may also be prepared by the careful decomposition of  $C_8Cs$  [1965Sal2]. The synthesis of cesium-graphite intercalation compounds is most successful when well-ordered crystalline graphite is used, from which single crystals may be grown with facility. Annealed pyrolytic graphite yields intercalation compounds whose properties are very close to those prepared with natural crystalline graphite [1965Sal1, 1965Sal2, 1966Sal].

The preparation of a graphite intercalation compound of stoichiometry between  $C_8Cs$  and  $C_{24}Cs$  was reported by [1932Sch, 1964Sal, 1965Sal1, 1965Sal2, 1966Sal]. The stoichiometry  $C_{16}Cs$  was first suggested [1932Sch], based on XRD data. Thermogravimetric studies [1965Sal1, 1965Sal2] established the stoichiometry  $C_{10}Cs$ , analogous to  $C_{10}K$  and  $C_{10}Rb$ . Like the corresponding potassium and rubidium compounds,  $C_{10}Cs$  is a stage I compound in which the atoms in the metal layer are disordered. Vapor pressure data [1966Sal] suggest that  $C_{10}Cs$  is stable only at high temperature; an approximate minimum temperature deduced from these data is 323 °C (see “Thermodynamics” section and Fig. 2).

$C_8Cs$  and  $C_{12}Cs$  may also be prepared at room temperature in solution. The metal is first dissolved in naphthalene or phenanthrene, to which graphite is added in ether solution [1996Miz, 1997Miz1, 1997Miz2]. In another case [1980Kle], the reaction to form  $C_8Cs$  was carried out in pentane solution with the aid of a cobalt-ethylene-trimethylphosphane complex.

### Cesium-Fullerene Compounds

The fullerenes are closed carbon-cage molecules containing only pentagonal and hexagonal rings. They have the general formula  $C_n$ , where  $n$  is an even integer greater than 20 [1995Fow]. The best-known fullerenes are  $C_{60}$  (“soccer ball” shape) and  $C_{70}$ .

$C_{60}$  may be prepared in an arc between graphite electrodes in subatmospheric Ar or He. The  $C_{60}$  is extracted from the smoke of carbon particles by an organic solvent

[1990Hau, 1990Kra, 1990Tay, 1991Bet]. It may also be prepared by a vaporizing laser beam falling on a graphite surface under a high-pressure stream of He [1985Kro] or in a static low pressure Ar atmosphere [1990Mei].

All the alkali metals form *exohedral* and *endohedral* compounds with fullerene. In exohedral cesium compounds, the Cs atoms and fullerene molecules occupy sites on a crystalline lattice; in endohedral compounds, the Cs atoms are inside the fullerene cages [1998Lah]. The endohedral compound of cesium is conventionally indicated by  $Cs@C_{60}$ .

A fulleride  $C_{60}Cs_x$  of undetermined stoichiometry were prepared by exposure of  $C_{60}$  to cesium vapor [1991Had, 1991Kel, 1991Tan]. Cesium fullerides were generally characterized by XRD.

$C_{60}Cs$  was prepared from diluting  $C_{60}Cs_6$  with the required amount of  $C_{60}$  [1993Zhu]. Like the corresponding potassium and rubidium compounds, it undergoes a transition in the range 350-425 K. Though this phase is thermodynamically metastable, it persists long enough at lower temperatures to be characterized. The polymer phase was characterized by XRD [1994Pek, 1995Osz].

The dipole moment of  $C_{60}Cs$  was measured [2000Ant] and was found to be similar to that of the corresponding potassium and rubidium compounds. Like the K and Rb compounds, it too is ionic in nature.

There are conflicting reports about the existence of  $C_{60}Cs_3$ . Certain general statements [1991Fle1, 1991Fle2, 1992Mur] would imply that this compound could be prepared by the reaction of fullerene with cesium vapor. This was not confirmed by XRD data [1991Fle1, 1991Fle2, 1992Mur, 1993Tan]. [1992Mur] in fact concluded that the compound was actually a mixture of  $C_{60}$  and  $C_{60}Cs_4$ . In contrast, [1993Mes] reported the successful preparation of this compound by reaction of fullerene and metal at elevated temperatures. This was not, however, a strictly direct reaction. The  $C_{60}$  fullerene was a mixture with about 10%  $C_{70}$  fullerene, and the cesium was de-intercalated from the  $C_8Cs$  graphite compound. The “ $C_{60}Cs_3$ ” was characterized by XRD [1993Mes]. In addition, [1995Pal] reported that this compound could be prepared by direct reaction in liquid ammonia. This question is further discussed in the section “Crystal Structures and Lattice Parameters.”

[1995Her] attempted the preparation of  $C_{60}Cs_3$  by true direct reaction at 200 °C, but concluded, from X-ray diffractogram evidence, that the compound was instead a mixture of  $C_{60}$  and  $C_{60}Cs_6$ . Concerning  $C_{60}Cs_4$ , it was stated categorically [1995Her] that “ $C_{60}Cs_4$  has never been mentioned.” The authors evidently overlooked the fact that the compound was prepared from stoichiometric amounts of  $C_{60}$  and  $C_{60}Cs_6$  at elevated temperature [1992Mur] and characterized by XRD.

$C_{60}Cs_6$  is sometimes called the “saturated” cesium-fullerene compound and is prepared by the reaction of fullerene with excess Cs vapor (250-350 °C) [1991Duc, 1992Mur]. The compound was characterized by X-ray and Raman spectra.

The preparation of the endohedral compound  $Cs@C_{60}$  has not been reported, although the analogous compounds with Li, Na, K, and Rb are known [1996Cam, 1996Tel].

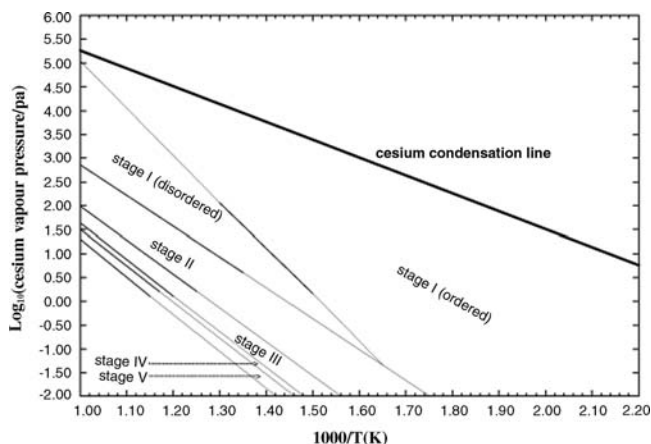


Fig. 2 Pressure of cesium vapor above cesium-graphite intercalation compounds [1966Sal]

Calculations (see “Thermodynamics” section) suggest that Cs@C<sub>60</sub> is as thermodynamically stable as those of the other alkali metal compounds.

Although no global C-Cs phase diagram is available, C<sub>60</sub>-Cs phase diagrams have been proposed. The diagram for C<sub>60</sub>-C<sub>60</sub>Cs<sub>x</sub> ( $x = 0-2$ ) shows a eutectoid and peritectoid arising from the structural change in C<sub>60</sub>Cs [1994Poi, 1995Poi]. These authors include the line compounds C<sub>60</sub>Cs<sub>4</sub> and C<sub>60</sub>Cs<sub>6</sub> though they are not shown on the diagram. By implication, the phase diagram of [1995Her] would show only C<sub>60</sub>Cs and C<sub>60</sub>Cs<sub>6</sub>.

## Crystal Structures and Lattice Parameters

These are presented in Tables 1 and 2, respectively.

The structure of C<sub>2</sub>Cs<sub>2</sub> has not been reported.

The structure of cesium-graphite intercalation compounds is derived from the structure of graphite itself (Fig. 1). The data have been reviewed extensively [1959Hen, 1959Rud, 1971Nov, 1975Ber, 1975Boe, 1976Ebe, 1979Fis, 1979Her, 1980Sel, 1981Dre, 1982Bar]. The order of the carbon atoms in the graphite layers is very little affected by the process of intercalation and by temperature, but the metal atoms in the intercalated layers are more or less labile, depending on the temperature. The degree of order in the metal layers has received very extensive study and is partially reviewed in [1979Fis]. Some investigators describe “phase changes” in the metal layers from “solid” to “liquid” and transition temperatures have been reported [1979Fis]; these are usually below ambient temperature. In general, much more is known about the interlayer spacing of the graphite layers than of the cesium layers [1979Fis, 1979Her].

In the stage I compound C<sub>8</sub>Cs the metal atoms are completely ordered at room temperature. The unit cell symmetry of C<sub>8</sub>Cs was first proposed to be C222<sub>1</sub> orthorhombic [1960Wol]. Later more careful XRD studies [1978Gue, 1978Lag] led to the conclusion that the unit cells are hexagonal. The space group is P6<sub>2</sub>22 or P6<sub>4</sub>22.

The progressive ordering of the intercalated cesium layers in stage II and higher compounds as the temperature is lowered from ambient has received extensive investigation, and the results have been reviewed by [1979Fis, 1990Mos]. For example, transitions were reported for C<sub>24</sub>Cs, as found by XRD, at 50, 160 (or 163 or 165) and 228 K [1969Par, 1979Cla, 1986Win].

The molecular structure of fullerene C<sub>60</sub> was suggested by [1985Kro, 1987Kro, 1990Kra] to resemble closely that of a soccerball. Ab initio calculations [1988Sch] indicated that a cluster of C atoms having 60 vertices and 32 faces (20 hexagonal and 12 pentagonal) would be energetically favored. This structure, a truncated icosahedron, would have I<sub>h</sub> overall symmetry, i.e., all the C atoms are equivalent. This assignment was confirmed by NMR measurements [1990Joh, 1991Yan] and Raman/IR spectra [1991Bet]. The arrangement of the C<sub>60</sub> molecules in the crystal lattice was deduced from powder XRD [1990Kra, 1991Hei]. Originally it was thought that the spheres were stacked in a hexagonal close-packed lattice, with  $a = 1.002$  nm and  $c = 1.636$  nm [1990Kra]. Later more careful examination showed that the crystal structure is fcc [1991Hei] at ambient temperature.

C<sub>60</sub> fullerene—like many unsaturated organic compounds—polymerizes under certain conditions [1993Rao, 1994Iwa]. C<sub>60</sub>Cs behaves similarly. When this compound is cooled from high temperature, a metastable polymer phase may appear before room temperature is reached [1995Pus, 1995Ste]. C<sub>60</sub>Cs, in common with the corresponding potassium and rubidium compounds, also polymerizes; the polymer is orthorhombic [1994Pek] with lattice parameters [1995Osz, 1995Pek]  $a = 0.910$  nm,  $b = 1.021$  nm,  $c = 1.417$  nm.

As mentioned earlier, the existence of C<sub>60</sub>Cs<sub>3</sub> is in dispute. In the two reported successful preparations [1993Mes, 1995Pal], the compound is described as bcc with a lattice parameter of 1.182 nm [1993Mes]. [1995Pal] cites Raman spectra at room temperature of the compound “as made” as evidence for its existence. Somewhat confusingly, these authors also present XRD evidence that the structure at 1 bar and room temperature corresponds to a mixture of the A15 phase ( $a = 1.1770$  nm) and a bct phase

**Table 1 C-Cs Crystal Structure Data(a)**

Phase	Composition at.% Cs	Pearson symbol	Space group	Strukturbericht designation	Prototype	Reference
C	0	<i>hP4</i>	<i>P6<sub>3</sub>/mmc</i>	A9	C(graphite)	[King1]
C	0	<i>cF8</i>	<i>Fd<math>\bar{3}m</math></i>	A4	C(diamond)	[King1]
C	0	<i>cF240</i>	<i>Fm<math>\bar{3}m</math></i>	...	C <sub>60</sub>	[1991Hei]
C <sub>60</sub> Cs(b)	1.6	<i>cF*</i>	...	...	...	[1993Zhu]
C <sub>60</sub> Cs <sub>4</sub>	6.3	<i>tI*</i>	...	...	...	[1991Fle1]
C <sub>60</sub> Cs <sub>6</sub>	9.1	<i>cI*</i>	<i>Im<math>\bar{3}</math></i>	...	...	[1991Zho]
C <sub>8</sub> Cs	11.1	<i>hP27</i>	<i>P6<sub>2</sub>22</i>	...	...	[1978Gue]
Cs	100	<i>cI2</i>	<i>Im<math>\bar{3}m</math></i>	A2	W	[King1]

(a) The existence and crystal structure of C<sub>60</sub>Cs<sub>3</sub> are in dispute. See sections “Equilibrium Diagram” and “Crystal Structures and Lattice Parameters”

(b) Above 420 K [1993Zhu]

## Section II: Phase Diagram Evaluations

( $a = 1.2057$  nm,  $c = 1.1432$  nm), but that at 26 kbar the structure can be indexed as a bcc phase. The corresponding unit cell volumes are close to those of the bcc  $C_{60}Cs_6$ , and the bct  $C_{60}Cs_4$  compounds.

Ultimately, the evidence concerning  $C_{60}Cs_3$  is ambiguous. One can, as does [1995Her], conclude that the compound does not exist *tout court*. Alternatively, one could claim that it is a metastable compound ([1995Pal] states this explicitly) which is thermodynamically stable under certain conditions (perhaps at low temperature, or under pressure at higher temperature).

Like the analogous potassium and rubidium compounds,  $C_{60}Cs_4$  is bct [1991Fle1, 1992Mur]. The same is true for  $C_{60}Cs_6$ , which is bcc [1991Zho, 1992Mur, 1993Mes, 1995Her, 1995Pal].

No crystalline structure data are available for the endohedral compound  $Cs@C_{60}$ . Since the Cs atoms are inside the  $C_{60}$  cages, it is expected that the crystalline structure of  $Cs@C_{60}$  would be very similar to  $C_{60}$  itself.

## Thermodynamics

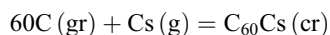
The thermodynamic properties of cesium-graphite intercalation compounds were reviewed by [1959Hen, 1968Tak, 1971Nov, 1979Her]. The heat of reaction between graphite and the liquid cesium was measured by at 40 °C by direct calorimetry [1964Sae]. On the assumption that the product was  $C_8Cs$ , the measured enthalpy change was  $-63.9 \pm 1.2$  kJ mol<sup>-1</sup>.

The vapor pressure above stages I-V cesium-graphite intercalation compounds was measured at 400-800 °C by the Knudsen effusion method [1965Sal1, 1966Sal]. The compounds were prepared from annealed pyrolytic graphite

[1965Sal1] and natural graphite [1966Sal]; the measured vapor pressures generally agreed within 30%. For the following discussion, only the results for natural graphite are used and are reproduced in Fig. 2. Thermodynamic properties for the equilibria among the stages were deduced from the equation

$$\ln(P/P^\circ) = \Delta H/RT - \Delta S/R \quad (\text{Eq 1})$$

where  $P$  is the measured vapor pressure and  $P^\circ$  is a reference pressure (1 atm). The enthalpies and entropies thus determined [1966Sal] are listed in Table 3. The enthalpies and entropies of formation of the compounds from graphite and Cs (g) were calculated from these data [1966Sal] by summing the stages and assuming that the values for  $C_{60}Cs$  according to the equation



are  $-154$  kJ and  $-78.7$  J K<sup>-1</sup> per g-atom of Cs (g). The corresponding quantities (Table 4) referring to Cs (l) at 277 °C were calculated from the Cs (g) values by use of the vaporization data  $\Delta_{\text{vap}}H^\circ(\text{Cs}, l) = 74.2$  kJ g-atom<sup>-1</sup> and  $\Delta_{\text{vap}}S^\circ(\text{Cs}, l) = 79.9$  J K<sup>-1</sup> g-atom<sup>-1</sup> [1985Ohs].

The enthalpy of formation of the stage I compound determined by direct calorimetry [1964Sae] is  $-63.9 \pm 1.2$  kJ mol<sup>-1</sup>, in good agreement with the results in Table 4.

Similar data were obtained for stage I and stage II compounds by an emf method [1972And]. The results were quoted for a temperature of 250 °C, but the temperature interval over which the emf was measured was not stated. There is only qualitative agreement between these results and those of [1966Sal]. In principle, the emf method would

**Table 2 C-Cs lattice parameter data**

Phase	Composition at.% Cs	Lattice parameter, nm			Reference
		<i>a</i>	<i>b</i>	<i>c</i>	
C(diamond)	0	0.54308	...	...	[Pearson3]
C(graphite)	0	0.2464	...	0.6711	[Pearson3]
C( $C_{60}$ )	0	1.411	...	...	[1991Ste]
	...	1.4161	...	...	[1992Tan]
	...	1.417	...	...	[1991Hei]
	...	1.4155	...	...	[1992Aga]
$C_{60}Cs(a)$	1.6	1.412(a)	...	...	[1993Zhu]
$C_{60}Cs_4$	6.3	1.196	...	1.098	[1991Zhu]
	...	1.2057	...	1.1443	[1991Fle1]
	...	1.2057	...	1.1432	[1995Pal]
	...	1.1966	...	1.1015	[1992Mur]
$C_{60}Cs_6$	9.1	1.1790	...	...	[1991Zho]
	...	1.180	...	...	[1995Her]
$C_8Cs$	11.1	0.4945	...	1.776	[1978Gue]
Cs	100	0.6141	...	...	[King1]

(a) At 200 °C

**Table 3 Thermodynamic properties of cesium-graphite intercalation reactions, per g-atom of Cs (g) [1966Sal] according to Eq 1**

Stage equilibrium reaction	$-\Delta H$ (kJ)	$-\Delta S$ , J (K <sup>-1</sup> )
I (disordered) = I (ordered)	183	182
II = I (disordered)	124	82.0
III = II	137	78.2
IV = III	143	77.8
V = IV	146	77.8
VI = V	150	78.7

**Table 4 Thermodynamic formation properties of cesium-graphite intercalation compounds, per g-atom of Cs(l) at 277 °C [1966Sal]**

Stage	$-\Delta_f H^\circ$ (kJ)	$\Delta_f S^\circ$ (J K <sup>-1</sup> )
I (ordered)	68.9	-20.6
I (disordered)	60.5	-0.5
II	71.4	1.6
III	75.6	1.6
IV	78.1	1.2
V	79.3	1.2

**Table 5** Calculated and experimental enthalpies of formation of exohedral cesium fullerides according to the equation  $C_{60}(cr) + xCs(cr) = C_{60}Cs_x(cr)$ 

Method	Reference	$-\Delta_f H^\circ$ , kJ mol <sup>-1</sup>		
		C <sub>60</sub> Cs	C <sub>60</sub> Cs <sub>3</sub>	C <sub>60</sub> Cs <sub>6</sub>
Calculation Born-Haber cycle	[1993Tom]	133	...	...
Calculation Born-Haber cycle	[1993Tom]	...	597	...
Calculation Born-Haber cycle	[1993Tom]	...	...	1062
Experimental DSC	[1993Che]	...	587	1182

be superior to the Knudsen effusion method, since the pressure measurement [1966Sal] is not performed under equilibrium conditions. It is possible therefore that the equilibrium lines in Fig. 2 lie somewhat too low. The general disposition of the P-T phase diagram, Fig. 2, is however quite similar to those for potassium and rubidium compounds.

Standard enthalpies of formation at 25°C were listed for stages I-VI [1982Wag]; these values increase monotonically from -61.9 kJ mol<sup>-1</sup> for C<sub>8</sub>Cs to + 3.3 kJ mol<sup>-1</sup> for C<sub>72</sub>Cs. Thermodynamic properties of the phases of C<sub>60</sub>Cs were determined by DSC [1995Pek, 1996Gra, 1997Gra]. Enthalpy change for the reaction monomer → polymer was reported to be 22.7 kJ mol<sup>-1</sup> [1995Pek]. [1996Gra, 1997Gra] presented a plot of the Gibbs energies of C<sub>60</sub>Cs phases relative to that of the fcc phase as a function of temperature. Calculated and experimental enthalpies of formation of exohedral cesium fullerides are shown in Table 5. This quantity for the endohedral compound Cs@C<sub>60</sub> was calculated by the Born-Haber cycle as -158 kJ mol<sup>-1</sup> [1993Wan].

## Pressure

The preparation and properties of cesium-graphite intercalation compounds were reviewed by [1981Sol, 1982Sol, 1995Gue]. The compounds reported on were C<sub>n</sub>Cs ( $n = 2, 4, 6, 8, 24, 36$ ). Methods used in study included XRD, DTA, and Raman spectroscopy [1981Sol, 1982Mor, 1985Wad, 1989Sya, 1990Avd, 1992Nal]. The conditions for preparation of these compounds from C<sub>60</sub> and cesium solid or liquid varied and were in the ranges: pressure, to 100 kbar; temperature, to 300 °C. Structural information was one item of interest in this work, but there was no attempt at systematic comprehensive study.

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C-Cs evaluation contributed by **J. Sangster**, Sangster Research Laboratories, P.O. Box 49562, 5122 Cote des Neiges, Montreal, Quebec, Canada, H3T 2A5. Literature searched through 2004.