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C-Rb (Carbon-Rubidium) System

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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_2Rb_2, (b) a liquid, and (c) a eutectic of composition very close to 100 at.% Rb and temperature very close to the M.P. of Rb.

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

(1) simple C-Rb compounds (C_2Rb_2 in this case)
(2) rubidium-graphite intercalation compounds
(3) rubidium-fullerene exohedral and endohedral compounds.

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

C_2Rb_2 (rubidium acetylide, rubidium carbide) was prepared in two steps in liquid ammonia [1903Moi]. Acetylene was added to a solution of rubidium in the solvent. The intermediate product was $C_2Rb_2{\cdot}C_2H_2$, which upon heating in vacuo at 300 °C is converted to C_2Rb_2. According to a standard inorganic chemistry text [1924Mel], it may also be prepared by the reaction of molten rubidium with acetylene. The meting behavior of C_2Rb_2 has not been reported.

Rubidium-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 rubidium-graphite intercalation compounds are:

- reaction with rubidium vapor at low pressures
- in organic solvent solution
- reaction with liquid rubidium 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 rubidium 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 rubidium 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 stage I compound is C_8Rb and the successive stages may be represented by $C_{12n}Rb$, where n is the stage number ($n \geq 2$). Compounds of stages I-XII have been prepared by the two-bulb method [1926Fre, 1932Sch, 1951Her, 1954Rud2, 1955Her, 1977Gue, 1980Bil]. The higher stage compounds may also be prepared by the careful decomposition of C_8Rb [1954Rud1, 1954Rud2]. The compounds are commonly characterized by XRD.

A rubidium-graphite intercalation compound of stoichiometry between C_8Rb and $C_{24}Rb$ was reportedly prepared by [1932Sch] ("$C_{16}Rb$") and by [1966Sal] ("$C_{10}Rb$"). In the earlier work, the identification was based on equilibrium rubidium vapor pressure measurements and XRD. However, 1954Rud2 later described his earlier assignment [1954Rud1] as erroneous, and the stoichiometry $C_{10}Rb$ was confirmed by a combination of Knudsen effusion and radioactive tracer techniques [1966Sal]. Like the corresponding potassium and cesium compounds, $C_{10}Rb$ is a stage I compound in which the intercalated metal layers are disordered [1966Sal]. According to the more complete data of [1988Met], C_8Rb exists in two disordered forms, which are stable only above 340 °C and 364 °C, respectively.

More recently, low stage intercalation compounds (C_nRb, $n \leq 4$) were prepared with oxygen as an impurity in the graphite [1994Her, 1995Skl]. These are not considered here.

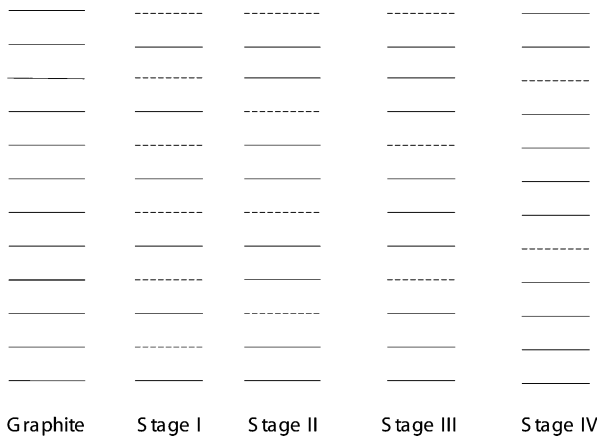

Fig. 1 Schematic representation of the structure of graphite and rubidium-graphite intercalation compounds. *Solid lines*: graphite layers. *dashed lines*: metal layers. The patterns repeat themselves in both directions

C_8Rb 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 was carried out in pentane solution with the aid of a cobalt-ethylene-trimethylphosphane complex.

Rubidium-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 ac 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 rubidium compounds, the Rb atoms and fullerene molecules occupy sites on a crystalline lattice; in endohedral compounds, the Rb atoms are inside the fullerene cages [1998Lah]. The endohedral compound of rubidium is conventionally indicated by $Rb@C_{60}$.

$C_{60}Rb$ is prepared commonly by the direct reaction of solid C_{60} with Rb vapor at elevated temperatures [1993Zhu, 1994Teg, 1995Win]. Crystals were also prepared from co-evaporation of both starting materials in a zone furnace [2000Hal]. An electrochemical method was used [1998End] with a toluene solution of fullerene and a dimethyl formamide solution of $RbClO_4$. The direct reaction was also carried in toluene solution at 111 °C [1993Sch]. The R.T. solid transforms in the interval 340-405 K to a high temperature structure, as determined by DSC, XRD, and Raman spectroscopy [1994Teg, 1995Win, 1995Gra, 1998Win].

From measurements of the dipole moment [2000Ant], it was concluded that $C_{60}Rb$ is an ionic compound.

$C_{60}Rb_3$ is prepared by direct reaction of Rb vapor with fullerene at elevated temperatures [1991Duc, 1991Fle2, 1992Tan, 1993Sch]. The direct reaction may also be carried out in solution. Solvents which have been used are liquid ammonia [1993Buf], methylamine [1996Coo], and a toluene/benzonitrile mixture [1993Sch]. Another method is by dilution of $C_{60}Rb_6$ with C_{60} [1991Mcc, 1991Zhu, 1992Mur].

$C_{60}Rb_4$ is prepared by the dilution method [1991Fle1, 1991Zhu, 1992Mur, 1997Kun] and also by the direct reaction with Rb vapor [1991Fle1].

$C_{60}Rb_6$ is sometimes called the “saturated” rubidium-fullerene compound and is prepared by the reaction of fullerene with excess Rb vapor (250-350 °C) [1991Duc, 1992Mur].

The compounds $C_{60}Rb_3$, $C_{60}Rb_4$, and $C_{60}Rb_6$ were all characterized by XRD.

A compound $C_{60}Rb_x$ of undetermined stoichiometry was prepared by direct reaction in toluene solution [1991Wan].

The endohedral compound $Rb@C_{60}$ was prepared by bombardment of fullerene with ionized rubidium atoms [1996Cam, 1996Tel].

Although no global C-Rb phase diagram is available, C_{60}-Rb phase diagrams have been proposed. A preliminary diagram was given by [1991Zhu] covering the composition range x = 0-6 in $C_{60}Rb_x$. This diagram was based on limited XRD data on stoichiomteric and non-stoichiometric mixtures. Poirier and co-workers [1994Poi, 1995Poi1, 1995Poi2] used X-ray photoemission spectroscopy and high temperature decomposition and fractional sublimation [1995Poi2] for the composition range x = 0-3. The diagram of Poirier et al. incorporates the high- and low-temperature modifications of $C_{60}Rb$ as giving rise to eutectoid and peritectoid temperatures at $x \sim 1$.

Crystal Structures and Lattice Parameters

These are presented in Tables 1 and 2 respectively.

The structure of C_2Rb_2 has not been reported.

The structure of rubidium-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.

Table 1 C-Rb crystal structure data

Phase	Composition, at. % Rb	Pearson symbol	Space group	Strukturbericht designation	Prototype	Reference
C	0	*hP*4	$P6_3/mmc$	A9	C (graphite)	[King1]
C	0	*cF*8	$Fd\bar{3}m$	A4	C (diamond)	[King1]
C	0	*cF*240	$Fm\bar{3}m$	…	C_{60}	[1991Hei]
$C_{60}Rb$(a)	1.6	*cF**		…	…	[1993Zhu]
$C_{60}Rb_3$	4.8	*cF*252	$Fm\bar{3}m$	…	…	[1991Fle2]
$C_{60}Rb_4$	6.3	*tI**	*I*4/*mmm*	…	…	[1991Fle1]
$C_{60}Rb_6$	9.1	*cI**	*Im*3	…	…	[1991Zhu]
C_8Rb	11.1	*oF*72	*Fddd*	…	…	[1978Lag1, 1978Lag2]
Rb	100	*cI*2	$Im\bar{3}m$	A2	W	[King1]

(a) Above 420 K

Table 2 C-Rb lattice parameter data

		Lattice parameter, nm			
Phase	Composition, at. % Rb	*a*	*b*	*c*	Reference
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]
$C_{60}Rb$(a)	1.6	1.408 (a)	…	…	[1993Zhu]
	…	1.4072 (b)	…	…	[1994Cha]
	…	1.4077 (b)	…	…	[1994Teg]
$C_{60}Rb_3$	4.8	1.4384	…	…	[1992Tan]
	…	1.439	…	…	[1991Mcc]
	…	1.442	…	…	[1991Zhu]
	…	1.4436	…	…	[1991Fle2]
	…	1.443	…	…	[1996Coo]
	…	1.4384	…	…	[1992Tan]
$C_{60}Rb_4$	6.3	1.196	…	1.098	[1991Zhu]
	…	1.1962	…	1.1022	[1991Fle1]
	…	1.1966	…	1.1015	[1992Mur]
$C_{60}Rb_6$	9.1	1.1536	…	…	[1991Zhu]
	…	1.1548	…	…	[1992Mur]
C_8Rb	11.1	0.4926	0.8532	2.2472	[1978Lag2]
Rb	100	0.5705	…	…	[King1]

(a) At 200 °C
(b) At 192 °C

In the stage I compound C_8Rb, the metal atoms are completely ordered at room temperature. In the metal layers the rubidium atoms are ordered in a centered hexagonal arrangement [1954Rud1]. The resulting three-dimensional structure may be described as based on a hexagonal unit cell, Pearson hP36 with $a = 0.4926$ nm, $c = 2.278$ nm [1932Sch, 1954Rud1, Pearson 3] showing direct similarity to graphite itself. An alternative more accurate description is an orthorhombic unit cell, which takes into account the four orientations of the rubidium sublattice [1960Wol, 1978Lag1, 1978Lag2, 1981Her]. The space group of this structure was identified as $C222_1$ [1960Wol] or *Fddd* [1978Lag1, 1978Lag2]. Since the structure of rubidium intercalation compounds is entirely analogous to that of the potassium compounds, the assignment *Fddd* is probably more accurate [1981Her].

For $C_{24}Rb$ and higher stage compounds, the metal layers are (partially) disordered at room temperature, becoming progressively more ordered as the temperature is lowered.

The molecular structure of fullerene C_{60} 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_h 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_{60} 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.

Like $C_{60}K$, $C_{60}Rb$ is fcc at high temperature, and orthorhombic at R.T. C_{60} fullerene—like many unsaturated organic compounds—polymerizes under certain conditions [1993Rao, 1994Iwa]. $C_{60}Rb$ behaves similarly. When this compound is cooled from high temperature, a number of metastable phases may appear before room temperature is reached [1995Ste]. Among them are a dimer phase and a polymer phase. Though these are thermodynamically metastable, they persist long enough at lower temperatures to be characterized. Both are orthorhombic, space group *Pmnn* or *Pnnm* or *Immm* (*I*2/*m* is preferred [1998Lau]). The lattice parameters of the polymer were determined by XRD [1994Teg, 1994Ste, 1994Cha, 1995Osz, 1998End]: the parameter values of these authors agree within 0.02 nm. For the dimer at this temperature, the parameters are similar: a = 0.963 nm, b = 0.992 nm, c = 1.415 nm [1995Osz].

$C_{60}Rb_3$, $C_{60}Rb_4$, and $C_{60}Rb_6$ are all isostructural with the potassium compounds (Table 1). In these compounds the rubidium atoms fill the space between the C_{60} cages and occupy tetrahedral and octahedral sites.

Endohedral compounds have been prepared only as thin films, and no crystalline structure data are available. Since the Rb atoms are inside the C_{60} cages, it is expected that the crystalline structure of $Rb@C_{60}$ would be very similar to C_{60} itself.

Thermodynamics

The thermodynamic properties of rubidium-graphite intercalation compounds were reviewed by [1959Hen, 1968Tak, 1971Nov, 1979Her]. The heat of reaction between graphite and the liquid metal was measured by at 50 ^C by direct calorimetry [1964Sae]; for the reaction with natural graphite the result was –46.6 ± 0.7 kJ per mole of C_8Rb. For artificial graphite, the value was –49.8 ± 1.9 kJ per mole of C_8Rb.

The vapor pressure above rubidium-graphite intercalation compounds was measured as a function of temperature by [1966Sal] by Knudsen effusion, and by [1988Met] under equilibrium conditions. The results are summarized in Fig. 2, which is analogous to the corresponding potassium diagram. The derived thermodynamic quantities for the equilibrium reactions among the various stages are presented in Table 3, and were calculated from the equation

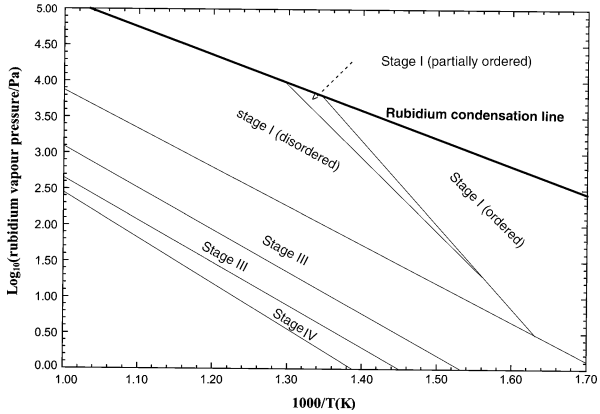

Fig. 2 Pressure of rubidium vapor above rubidium-graphite intercalation compounds [1988Met]

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

where P is the measured vapor pressure and P° is a reference pressure (1 atm). The ΔS values for [1988Met] in Table 3 are not those tabulated by [1988Met] in their original article; it was found that those tabulated values did not reproduce the equilibrium values in their diagram. Instead, ΔS data were read off from the intercepts of their vapor pressure equilibrium lines (Fig. 2). The entries of [1966Sal] in Table 3 indicate that their data lie below those of [1988Met] in all cases, probably as a consequence of the fact that the Knudsen effusion method is a non-equilibrium process.

The enthalpy and entropy of formation of rubidium-graphite intercalation compounds were calculated from the data in Table 3, in the manner described in [1966Sal]. The values were obtained by summing the stages, together with the assumptions that the enthalpy and entropy of the reaction

$$60C\,(gr) + Rb(g) = C_{60}Rb\,(cr) \qquad \text{(Eq 2)}$$

are −141 kJ and −75.7 J K^{-1}, respectively [1966Sal]. The data were converted to refer to Rb(l) at 400 °C with the use of the data $\Delta_{vap}H$ = 77.2 kJ and $\Delta_{vap}S$ = 81.0 J K^{-1} per g-atom of rubidium [1985Ohs].

From these results (Table 4), the direct calorimetric determination of the enthalpy of formation of the stage I compound [1964Sae] would correspond to the partially or completely disordered form, rather than the completely ordered C_8Rb, as might be expected. The validity of this conclusion is contingent upon the assumptions made in this calculation (Table 3), that the $C_{10}Rb$ of [1966Sal] corresponds to the stage I (disordered) of [1988Met], and the C_8Rb of [1966Sal] to the stage I (partially ordered) of [1988Met]. Standard enthalpies of formation at 25 °C are

Table 3 Thermodynamic properties of rubidium-graphite intercalation reactions, per g-atom of Rb (g) (Eq 1)

	[1988Met]		[1966Sal]	
Stage equilibrium reaction	**$-\Delta H$, kJ**	**$-\Delta S$, J K^{-1}**	**$-\Delta H$, kJ**	**$-\Delta S$, J K^{-1}**
I (partially ordered) = I (ordered)	219	271	…	…
I (disordered) = I (partially ordered)	189	225	142	177
II = I (disordered)	103	81.67	106	80.3
III = II	111	74.5	114	71.1
IV = III	112	66.1	123	73.6
V = IV	122	72.4	130	76.6
VI = V	…	…	133	75.7

Table 4 Thermodynamic formation properties of rubidium-graphite intercalation compounds, per g-atom of Rb(l) at 400 °C

	$-\Delta_f H°$, kJ		**$\Delta_f S°$, J K^{-1}**	
Stage	**[1988Met]**	**[1966Sal]**	**[1988Met]**	**[1966Sal]**
I (ordered)	269	…	−300	…
I (partially ordered)	50.0	43.3	−29.3	−16.5
I (disordered)	34.6	37.9	−0.6	3.6
II	46.9	50.8	7.6	7.3
III	53.4	57.5	8.2	5.7
IV	59.7	61.3	5.9	5.2
V	…	63.4	…	5.2

Table 5 Calculated and experimental enthalpies of formation of exohedral rubidium fullerides according to the equation C_{60}(cr) + xRb(cr) = $C_{60}Rb_x$(cr)

		$-\Delta_f H°$, kJ mol^{-1}		
Method	**Reference**	**C_{60}Rb**	**$C_{60}Rb_3$**	**$C_{60}Rb_6$**
Calculation Born-Haber cycle	[1993Tom]	133	…	…
Calculation Born-Haber cycle	[1993Tom]	…	609	…
Calculation Born-Haber cycle	[1993Tom]	…	…	1086
Experimental DSC	[1993Che]	…	490	1034

listed in [1982Wag], and are −44.4 kJ mol^{-1} for C_8Rb, increasing to −28.5 kJ mol^{-1} for C_{72}Rb.

Thermodynamic properties of the phases of C_{60}Rb were determined by DSC [1995Pek, 1996Gra]. Enthalpy changes for the reactions polymer → monomer and dimer → monomer were reported to be 26 kJ mol^{-1} and 11 kJ mol^{-1}, respectively. These authors also presented a plot of the Gibbs energies of C_{60}Rb phases relative to that of the fcc phase as a function of temperature.

Calculated and experimental enthalpies of formation of exohedral rubidium fullerides are shown in Table 5. This quantity for the endohedral compound Rb@C_{60} was calculated by the Born-Haber cycle as −184.4 kJ mol^{-1} [1993Wan].

Pressure

The preparation of rubidium-graphite intercalation compound under pressure has been investigated in some detail. A recent review of preparative methods is [1995Gue], and properties were reviewed by [1981Sol, 1984Cla]. Reported reaction conditions are: pressure ≥0.2 GPa, temperature ≥293 K [1982Mor,1990Avd, 1991Nal, 1992Nal]. Reported stoichiometries were C_nRb ($n \leq 8$). The compounds were examined by DTA, IR spectroscopy, and density measurement. Compounds prepared under pressure are not stable under atmospheric pressure.

The lattice parameter of $C_{60}Rb_3$ was measured by XRD as a function of pressure up to 29 kbar and the relation may be represented by the equation

$$d(\ln a)/dP = -1.52E - 3\ \text{kbar}^{-1}$$

The corresponding value for C_{60} itself is approximately −2.3E−3 kbar^{-1} [1992Zho].

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