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K-P (Potassium-Phosphorus) System

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Equilibrium Diagram

There are no liquidus or solubility data available for this system [2001Bor]. A large number of K-P compounds has been prepared and identified. Phosphorus forms many polyatomic groups (rings and cages), and Baudler [1982Bau, 1987Bau] has pointed out the analogies between phosphorus and carbon chemistry.

The preparation and structure of phosphorus compounds with alkali metals were reviewed [1958Waz, 1973Sch, 1977Sch, 1981Sch, 1983Sch1, 1983Sch2]; a later review [1988Sch] is both more detailed and more extensive.

In early work, the reported stoichiometry of some compounds was confused. This was because elemental compositions were not easily distinguished by analytical techniques of the time.

It is appropriate here to distinguish between compounds isolated and characterized as solids and those prepared only in solution (or those studied only by theoretical calculations). Unless otherwise noted, red phosphorus was used in preparing K-P compounds. Most K-P compounds decompose before melting.

Compounds Isolated as Solids

K_3P (25 at.% P) was prepared by reaction of the elements in liquid ammonia [1895Hug] or in liquid hydrocarbon [1862Cah]. [1913Vou] prepared this compound by the reduction of PCl_3 or PCl_5 with K. It is better prepared by reaction of K with white or red P in stoichiometric amounts without solvent [1961Gnu]. It was characterized by powder XRD [1961Gnu].

K_4P_3 (42.9 at.% P) was prepared from the elements, K:P = 1.5-2.0 [1989Sch]. It was characterized by single-crystal XRD [1989Sch].

KP (50 at.% P) is prepared by the reaction of the elements [1979Sch]. It was characterized by single-crystal XRD [1978Hon, 1979Sch].

K_4P_6 (60.0 at.% P) is prepared by reaction of the elements in stoichiometric ratio [1984Abi, 2005Kra]. This compound exists in two forms; the room temperature compound αK_4P_6 transforms by a first-order process at ~577 °C [1984Abi] to the high-temperature form βK_4P_6. Both forms were characterized by single-crystal XRD [1984Abi]. βK_4P_6may be quenched from high temperature and exists metastably at ambient temperature; it can then revert to αK_4P_6 by heating to 475 °C [1984Abi].

K_3P_7 (70.0 at.% P) in early work was known as K_2P_5 (71.4 at.% P). The misidentification was pointed out later [1983Sch1]. K_3P_7 was prepared [1940Leg] from decomposition of KH_2P (a product of the reaction of PH_3 + K in liquid ammonia). Ether-solvated K_3P_7 resulted from reaction of K_4P_6 with ether in liquid ammonia [2005Kra]. The compound described as "$K_{0.43}P$" [1979Sch] might have been K_3P_7. The pure phosphide was prepared by direct reaction of the elements without solvent [1912Hac]. It is best prepared from stoichiometric amounts of the elements [1985Ten, 1986San, 1995Hon]. It undergoes a first-order crystalline to plastic-crystalline transition $\alpha K_3P_7 \rightarrow \beta K_3P_7$ at ~ 245 °C [1983Hon, 1983Sch1, 1983Sch2], ~260 °C [1985Ten] or ~265 °C [1986San]. The melting point was given as 982 °C [1985Ten]. Only βK_3P_7 has been characterized by XRD [1983Hon, 1983Sch1, 1983Sch2].

K_3P_{11} (78.6 at.% P) is prepared by reaction of the elements [1991Sch]. The room temperature crystalline form (αK_3P_{11}) transforms to a plastic-crystalline form (βK_3P_{11}) in a first-order process at 450 °C [1991Sch]. Both forms were characterized by XRD [1983Sch1, 1983Sch2, 1991Sch]. Raman and infrared spectra of αK_3P_{11}in ethylenediamine solution were reported [1991Sch].

$KP_{10.3}$ (91.1 at.% P). This compound, prepared by direct reaction of the elements, was described by [1988Sch] in preliminary work. The structure, from XRD data, could not be completely elucidated.

KP_{15} (93.8 at.% P). The crystalline compound is prepared from the elements in a solid or liquid state reaction or by vapor transport [1967Sch, 1985Mar, 1985Ole1]. Crystalline or amorphous thin films were prepared from this product by vapor transport or vapor deposition [1984Ole, 1985Ole2, 1984Sch, 1985Sch]. The compound was characterized by single-crystal [1967Sch, 1985Ole1] or powder [1985Mar] XRD. Results of Raman, photoluminescence, photoconductivity, and transmission spectroscopy were reported [1984Ole, 1984Sch, 1985Mar, 1985Ole1, 1985Ole2, 1985Ole1].

Other Compounds

K_6P_4 (40.0 at.% P) was prepared by reaction of the elements in liquid ammonia [1951Eve]. This author gave it the empirical formula K_3P_2 and stated that it was "probably dimeric." [1979Sch] identified a by-product of the preparation of KP as "$K_{1.5}P$" .

K_5P_4 (44.4 at.% P) was identified as "$K_{1.25}P$," by-product of the preparation of KP [1979Sch].

KP_x ($x < 1$, >50 at.% P) was mentioned by [1977Sch], without further identification.

K_2P_4 (66.7 at.% P) was prepared as an ether-ammoniate in liquid ammonia solution [2006Kra]. Density functional theory was used to deduce the geometry of K_2P_4 itself [2004Jin].

KP_5 (83.3 at.% P) was prepared by reaction of the elements in liquid ammonia solution [1895Hug]. It has not been isolated as a solid product.

K_3P_{19} (86.4 at.% P) was prepared in glyme or tetrahydrofuran solution by the reaction of K with white P [1986Bau]. The solvated species was identified through its ^{31}P-NMR spectrum [1986Bau].

KP_7 (87.5 at.% P) was identified initially as "KP_x," the product of the reaction between K and white P [1955Kre]. The corresponding Na compound could be identified by its structure and lattice parameters as most probably NaP_7. The designation "KP_7" is therefore tentative.

Crystal Structures and Lattice Parameters

Crystal structures and lattice parameters of potassium phosphides are summarized in Table 1 and 2, respectively. These tables include only those compounds isolated and characterized in the pure solid state. The text below includes a description also of compounds incompletely described ("Other Compounds"). Some are descriptions of the

Table 1 K-P crystal structure data

Phase	Composition at. % P	Pearson symbol	Space group	Strukturbericht designation	Prototype	Temperature, °C	Reference
K	0	*cI*2	$Im\bar{3}m$	*A*2	W	25	[King1]
K_3P	25.0	*hP*8	$P6_3/mmc$	DO_{18}	Na_3As	25	[1961Gnu]
K_4P_3	42.9	*oC*28	*Cmcm*		W_3CoB_3	25	[1989Sch]
KP	50.0	*oP*16	$P2_12_12_1$		NaP	20	[1978Hon]
αK_4P_6	60.0	*oF*40	*Fmmm*		Rb_4P_6	20	[1974Sch]
βK_4P_6	60.0	*oF*80	*Fddd*		βK_4P_6	>575	[1984Abi]
βK_3P_7	70.0	*cF*40	$Fm\bar{3}m$		βRb_3P_7	>265	[1983Hon]
αK_3P_{11}	78.6	*oP*56	*Pnab*		αNa_3P_{11}	20	[1983Sch1, 1983Sch2]
βK_3P_{11}	78.6	*cP*28	$Fm\bar{3}m$		βRb_3P_{11}	>450	[1988Sch]
KP_{15}	93.8	*aP*64(a)	$P\bar{1}$		KP_{15}	20	[1967Sch]
P (black)	100	*oC*8(b)	*Cmca*			25	[Pearson2]
P (white)	100	*c***				25	[Pearson2]
P (red)	100	*c**66				25	[Pearson2]

(a) In [1988Sch], this is given as *aP*32

(b) P exists in rhombohedral and cubic forms at high pressures and room temperature

Table 2 K-P lattice parameter data

Phase	Composition, at.% P	Lattice parameters, nm *a*	*b*	*c*	α	β	γ	Temperature, °C	Reference
K	0	0.5321						25	[King2]
K_3P	25.0	0.5691		1.005				20	[1961Gnu]
K_4P_3	42.9	0.5049	1.1197	1.4788				20	[1989Sch]
KP	50.0	0.6500	0.6016	1.1288				20	[1978Hon]
αK_4P_6	60.0	0.9347	1.4253	0.8624				20	[1984Abi]
		0.9361	1.4268	0.8644				20	[1974Sch]
βK_4P_6	60.0	1.8650	1.4772	0.8305				20	[1984Abi]
βK_3P_7	70.0	1.0603						267	[1983Hon]
αK_3P_{11}	78.6	1.0315	1.3940	1.0450				20	[1983Sch1, 1983Sch2]
βK_3P_{11}	78.6	0.9282						25(a)	[1991Sch]
KP_{15}	93.8	2.374	0.969	0.721	116.7°	97.5°	90.0°	20	[1967Sch]
		1.1945	0.9705	0.7157	116.34°	101.46°	81.93°	20	[1985Ole1, 1985Ole2]
P (black)	100	0.33136	1.0478	0.43763				25	[Pearson2]
P (white)	100	0.718						25	[Pearson2]
P (red)	100	1.131						25	[Pearson2]

(a) Metastably quenched from high temperature phosphorus anions, and are given here to document the ability of P to form cage structures.

Compounds Isolated in the Pure Solid State

K_3P has hexagonal structure with isolated P^{3-} anions [1961Gnu] from powder XRD data . It is isostructural with Na_3P. The fractional ionic character of K_3P, calculated by two different methods, was 0.74 or 0.96 [1990Sha].

In K_4P_3, the P-P-P bond angle in the P_3^{4-} anion is 118.1°, and each P atom lies in the center of a trigonal prism built up of six K^+ ions with tricapped rectangular faces [1989Sch].

KP may be regarded as containing one-dimensional infinite helices of P^- anions [1978Hon, 1979Sch].

In K_4P_6, both forms are face-centered orthorhombic; in both there are isolated planar parallel P_6^{4-} rings. Each P atom is surrounded trigonal-prismatically by six K^+ ions [1974Sch, 1984Abi, 1981Sch, 1987Sch]. Alternatively, the structure may be envisioned as a defect-structure derivative of the Al_4B_8 (AlB_2) type: $K_4P_6\square_2$. In this case, two P sites remain vacant in an ordered way and leave isolated planar P_6 rings [1988Sch]. This scheme is explained more fully in [1983Nes].

K_3P_7. The structure of the room temperature (α-form) solid has not been fully elucidated; the high-temperature (β-form) is fcc. In both forms, there are P_7^{3-} cage groups (formally analogous to nortricyclene in carbon chemistry) [1983Hon, 1983Sch1, 1983Sch2]. The β-form is of the Li_3Bi type, where the P_7^{3-} anions occupy the Bi^{3-} sites.

K_3P_{11} at room temperature is orthorhombic (α-form), whereas the high-temperature form is cubic [1983Sch1, 1983Sch2]. Both forms contain P_{11}^{3-} cages, highly internally connected [1991Sch]. The corresponding carbon cage compound was given the trivial name "ufosan" [1973Wic, 1988Sch].

KP_{15} contains infinite P_{15}^- tubes, resulting from a polymerization of alternating P_7^- and P_8^0 units [1967Sch, 1981Sch]. The K atoms are situated between the tubes, and each K atom has six nearest neighbors.

Other Compounds

$KP_{10.3}$ was given [1988Sch] the Pearson symbol *tP*1488 and the anionic structure was described as "complex tubular superstructure."

K_3P_{19} contains P_{19}^{3-} anions made up of a central P_5^- pentagon, to which are attached two P_7^- groups formally analogous to deltacyclane [1986Bau].

K_2P_4 has been investigated only as an ether ammoniate [2006Kra]. The compound K_2P_4 itself has a dipyramidyl structure, with a square P_4^{2-} anion. This was corroborated by theoretical calculations [2005Kra]. MINDO/3 calculations suggest that the P_4^{2-} anion by itself has D_{4h} symmetry, i.e., tetrahedral [1981Cut].

Thermodynamics

Experimental data are summarized in Table 3. Standard thermodynamic quantities of K_3P were estimated [1980Suu] as: $\Delta_f H°_{298} = 1.77$ kJ mol^{-1} and $S°_{298} = 49.8$ J mol^{-1} K^{-1}. Vapor pressure data for K_3P_7 were obtained by Knudsen effusion/mass spectrometry [1986San] in the range 744-889 K. From these data were deduced the standard thermodynamic properties (second law) $\Delta_f H°_{298}$, $\Delta_f G°_{298}$, and $S°_{298}$. Heat capacity data of this compound were measured in the range 120-770 K by differential scanning calorimetry [1985Ten]. From these data were deduced the solid transition temperature and the enthalpy of transition [1985Ten]. The melting temperature and enthalpy of fusion were determined by differential thermal analysis [1985Ten].

Heat capacity data for KP_{15} were obtained in the range 2-470 K by adiabatic and differential scanning calorimetry [1983San]. A value for $S°_{298}$ for KP_{15} was deduced from these data. Vapor pressure of this compound was measured by Knudsen effusion/mass spectrometry [1987San] in the range 502-600 K. From these data were deduced the standard formation properties $\Delta_f H°_{298}$ and $\Delta_f G°_{298}$.

Table 3 Experimental thermodynamic properties of K phosphides

Compound	$\Delta_f H^0_{298}$, kJ mol^{-1}	$\Delta_f G^0_{298}$, kJ mol^{-1}	S^0_{298}, J mol^{-1} K^{-1}	$\Delta_{trs}H$, kJ mol^{-1}	$\Delta_{fus}H$, kJ mol^{-1}
…	…	…			
αK_3P_7	−124(a)	−302(a)	596(a)	10.1(b)	…
βK_3P_7	−66(a)	−291(a)	756(a)	…	10(b)
KP_{15}	−126(c)	−245(c)	411.4(e)	…	…
	−118(d)	−241(d)	398(g)	…	…
	− 651(f)	…	…	…	…

(a) [1986San]
(b) [1985Ten] (heating mode)
(c) [1987San], second law
(d) [1987San], third law
(e) [1983San]
(f) [1985Mar]. Reference state of elements: ideal gas at 298 K and 1 atm
(g) [1988Sch]

[1985Mar] measured the pressure of P_4 (gas) over KP_{15} with a Bourdon gauge. From these data were deduced the enthalpy of formation of KP_{15} (cr) from the gaseous elements at 298.15 K.

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