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Preparation, Crystal Structure, and Physical Properties of the Uranium Nickel Phosphide $U_3Ni_{3.34}P_6$

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The new ternary phosphide $U_3Ni_{3.34}P_6$ was prepared by reaction of the elemental components in a tin flux and its crystal structure was determined from single-crystal X-ray data: $P4/mmm$, a = 381.8(1) pm, c = 1350.1(4) pm, Z = 1, and R = 0.018 for 200 structure factors and 18 variable parameters. The ideal composition is $U_3Ni_4P_6$; however, the nickel site was found to be occupied to only 83.6(5)%. The formation of these defects is rationalized from bonding considerations. One phosphorus site had to be refined with a split position and models suggesting various kinds of short-range order for this position are discussed. $U_3Ni_{3.34}P_6$ contains two different uranium sites, which are assigned the oxidation numbers +3 and +4 on the basis of their differing U–P bond lengths. The structure is related to the structures of several tetragonal transition metal phosphides and arsenides, e.g., $UNi_{1.51}P_2$ with $ThCr_2Si_2$ type structure and $U_2Cu_4As_5$. Magnetic susceptibility measurements suggest ferrimagnetism with the Curie temperature T_C = 139(2) K, the Weiss constant Θ = 107(3) K, and a magnetic moment of $\mu_{exp} = 2.1(1)\mu_B$ per average uranium atom. Four-probe electrical conductivity measurements indicate semimetallic behavior. © 1995 Academic Press, Inc.

INTRODUCTION

Ternary uranium transition metal phosphides and arsenides are the subject of numerous investigations, not only because of their interesting crystal structures, but also because of their intriguing magnetic properties. Especially the compounds with the simple tetragonal $ThCr_2Si_2$ and $CaBe_2Ge_2$ type structures were studied intensively: $UNi_{2-x}P_2$ (1–3), UCo_2P_2 (4, 5), and $UNi_{2-x}As_2$ (6, 7).

SAMPLE PREPARATION AND LATTICE CONSTANTS

Starting materials were platelets of uranium (Merck, "nuklearrein"), powder of nickel (Merck, >99.5%), red phosphorus in the form of small pieces (Hoechst–Knapsack, "ultrapure"), and granules of tin (Merck, 99.9%). Polycrystalline samples of $U_3Ni_{3.34}P_6$ were prepared by annealing the elemental components (the uranium in form of filings) in sealed silica tubes for 1 month at 800°C. These samples contained UP_2 as an impurity phase. A well-crystallized sample of $U_3Ni_{3.34}P_6$ was obtained by the tin flux technique. The elements, in the atomic ratio U : Ni : P : Sn = 3 : 4 : 15 : 32, were sealed in evacuated silica tubes and annealed between 750 and 850°C for about 2 weeks. The tin-rich matrix was then dissolved in cold, moderately diluted (1 : 1) hydrochloric acid, which leaves the crystals of $U_3Ni_{3.34}P_6$ essentially unattacked.

The crystals of the new ternary phosphide have the form of small platelets. They are black with metallic luster and stable in air for long periods of time. Energy-dispersive fluorescence analyses in a scanning electron microscope did not show any impurity elements heavier than sodium.

The products were characterized by the Guinier powder technique using α-quartz (a = 491.30 pm, c = 540.46 pm) as a standard. Indices could be assigned on the basis of the cell obtained from the single-crystal investigations. This was facilitated by comparing the experimental patterns with the calculated ones (8) using the positional parameters of the structure determination. The following tetragonal lattice constants were obtained by least-squares fits: a = 381.8(1) pm, c = 1350.1(4) pm, and V = 0.1968(1) nm^3.

MAGNETIC PROPERTIES AND ELECTRICAL CONDUCTIVITY BEHAVIOR

Susceptibility measurements were performed on polycrystalline samples in the temperature range between 5 and 300 K with a SQUID magnetometer (Quantum Design, Inc.) using magnetic flux densities of up to 1 T as described previously (9, 10). At temperatures above 150 K $U_3Ni_{3.34}P_6$ shows Curie–Weiss behavior. The extrapolation of the linear portion of the $1/\chi$ vs T plot (Fig. 1) resulted in a Weiss constant of Θ = 107(3) K; a magnetic moment of μ_{exp} = 2.1(1) μ_B per uranium atom was calculated from the slope of this plot. At lower temperatures the susceptibility becomes field-dependent, suggesting ferrimagnetism. At low magnetic field strengths the reciprocal susceptibility approaches zero at the Curie tempera-

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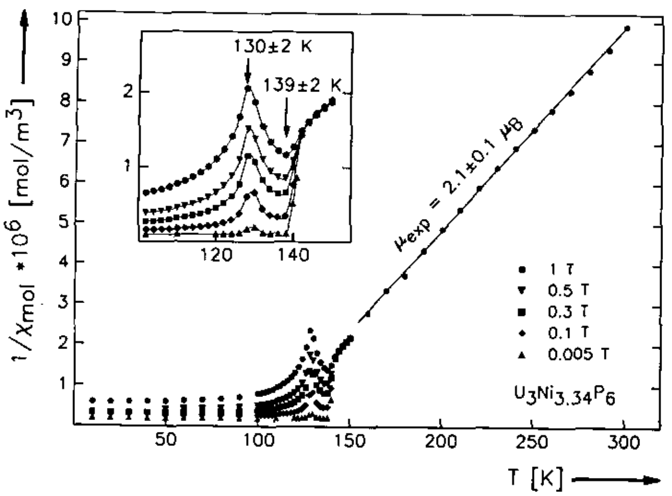

FIG. 1. Temperature dependence of the reciprocal susceptibility of $U_3Ni_{3.34}P_6$ measured with magnetic flux densities of between 0.005 and 1 T.

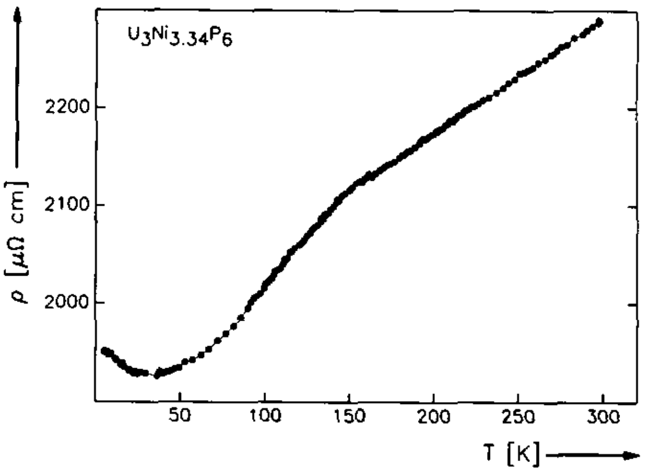

FIG. 2. Temperature dependence of the electrical resistivity of $U_3Ni_{3.34}P_6$.

ture of $T_C = 139(2)$ K. At higher magnetic flux densities the reciprocal susceptibility has a minimum at this temperature. The maximum in the reciprocal susceptibilities at 130(2) K may be rationalized by a change in the magnetic structure, or more likely, as the compensation point, where the absolute values of the magnetic moments of the two sublattices become equal. Preliminary results of a neutron scattering study confirm the ferrimagnetism of this compound in zero magnetic field at 2 K (11). The two uranium sites have different magnetic moments. Each uranium sublattice orders ferromagnetically but the moments of the two sublattices are antiparallel relative to each other. No magnetic moments were found at the nickel sites (11).

The electrical conductivity behavior of $U_3Ni_{3.34}P_6$ was investigated with an ac four-probe device between 4 K and room temperature as described previously (12). The sample consisted of a single crystal with the dimensions 100 × 100 × 500 μm. The specific resistivity of the compound shows a minimum at 30 K (Fig. 2). This behavior may be interpreted as semimetallic (zero-band-gap semiconductor). At low temperatures the material behaves like a semiconductor, where the resistivity decreases with increasing temperature. At higher temperatures the conduction band is sufficiently populated and the resistivity increases with increasing temperature for the same reason as is rationalized for metallic conductors. The absolute value of the conductivity at room temperature is difficult to determine, because of the uncertainty in estimating the contacting areas. The room temperature value of about 2500 μΩ cm may be correct only within a factor of three, however, this resistivity is considerably higher than that of silver (1.6 μΩ cm) or iron (10 μΩ cm) (13). At about 150(10) K, the electrical resistivity of $U_3Ni_{3.34}P_6$ shows a slight deviation from the smooth behavior. This change in the slope of the resistivity curve may be ascribed to the magnetic transition of the structure at the Curie temperature of $T_C = 139(2)$ K. The curve was recorded by heating from low to high temperatures and there is a hysteresis in the measuring device, which explains the poor agreement of the two temperatures.

STRUCTURE DETERMINATION

The intensity data for the structure determination of $U_3Ni_{3.34}P_6$ were collected on a four-circle diffractometer with $\theta/2\theta$ scans, graphite monochromated Mo$K\alpha$ radia-

TABLE 1
Crystal Data for $U_3Ni_{3.34}P_6$

Space group	$P4/mmm$ (No. 123)
Formula units/cell	$Z = 1$
Formula weight	1096.02
a (pm)	381.8(1)
c (pm)	1350.1(4)
V (nm³)	0.1968(1)
Calculated density (g/cm³)	9.24
Crystal size (μm³)	20 × 20 × 10
$\theta/2\theta$ scans up to	$2\theta = 70°$
Range in h k l	±6 ±6 ±21
Highest/lowest transmission	1.72
Total number of reflections	3341
Inner residual	$R_i = 0.050$
Unique reflections	322
Reflections with $I_0 > 3\sigma(I_0)$	200
Number of variables	18
Conventional residual	$R = 0.018$
Weighted residual	$R_W = 0.027$

TABLE 2
Atomic Parameters of $U_3Ni_{3.34(2)}P_6$

Atom	*P*4/*mmm*	Occupancy	*x*	*y*	*z*	B_{11}	B_{22}	B_{33}	*B*
U1	2*h*	1	1/2	1/2	0.34794(6)	0.31(1)	B_{11}	0.32(2)	0.317(7)
U2	1*a*	1	0	0	0	0.21(2)	B_{11}	0.53(3)	0.31(1)
Ni	4*i*	0.836(5)	0	1/2	0.1733(1)	0.37(7)	0.51(8)	0.69(6)	0.53(3)
P1	2*g*	1	0	0	0.2702(4)	—	—	—	0.44(8)
P2	2*h*	1	1/2	1/2	0.0857(4)	—	—	—	0.50(8)
P3	4*m*	0.5	0	0.451(2)	1/2	—	—	—	0.42(9)

Note. The last column contains the isotropic thermal parameters (×100 in units of nm^2) of the phosphorus atoms and the equivalent isotropic thermal parameters of the metal atoms. The anisotropic thermal parameters are defined by $\exp[-0.25 \cdot (B_{11}h^2a^{*2} + \cdots)]$. The values of B_{12}, B_{13}, and B_{23} are zero for the metal atoms of this structure.

tion, and a scintillation counter with pulse-height discriminator. The crystallographic data and some results are summarized in Table 1.

The reciprocal lattice had the Laue symmetry 4/*mmm*. There were no systematic extinctions and the structure was eventually refined in the space group with the highest symmetry, *P*4/*mmm*. The positions of the uranium atoms were obtained from a Patterson synthesis; those of the other atoms were located by difference Fourier maps. The structure was refined with a full-matrix least-squares program using atomic scattering factors (14) corrected for anomalous dispersion (15). The weights consisted of a constant and a term accounting for the counting statistics. A parameter correcting for isotropic secondary extinction was also refined.

The P3 atom was originally refined in the position 2*e* 0, 1/2, 1/2; however, its displacement parameter U_{22} was very large and we therefore refined this atom with a split position off the mirror plane. In the final least-squares cycles the metal atoms were refined with ellipsoidal displacement parameters and the phosphorus atoms with isotropic ones. To check for the correct composition, we also refined occupancy parameters together with the thermal parameters using a fixed scale factor. For the nickel atom, a serious deviation from the full occupancy was found. The occupancy factors of the other atoms were all within four standard deviations at the ideal values. Therefore, in the final least-squares cycles only the occupancy parameter of the nickel position was allowed to vary. The atomic parameters and interatomic distances are given in Tables 2 and 3.

DISCUSSION

The crystal structure of $U_3Ni_{3.34}P_6$ represents a new structure type, which belongs to a large family of tetragonal structures, of which the $ThCr_2Si_2$ and $CaBe_2Ge_2$ type structures are well known representatives. In Fig. 3, we show some of them: $LaNi_{1.51}Sb_2$ with $CaBe_2Ge_2$ type structure (16), $UNi_{1.51}P_2$ with $ThCr_2Si_2$ type structure (1–3, 17), $U_2Cu_2P_3O$ (18, 19), $Eu_2Pt_7AlP_{4-x}$ (20), and $U_2Cu_4As_5$ (21). Many more representatives are known, mostly with silicon, germanium, and tin as the anionic component (22). A recent example is $U_3Co_4Ge_7$ (23), which actually has the same space group symmetry *I*4/*mmm* and atomic sites as $Eu_2Pt_7AlP_{4-x}$ (20), but with a different atom distribution. In some of these structures with more complicated stacking sequences layers of AlB_2 and the Cu_3Au type are intergrown with $ThCr_2Si_2$ and $CaBe_2Ge_2$ slabs (20, 21, 23). It can be seen from Fig. 3 that the $U_3Ni_{3.34}P_6$ structure is built up from $ThCr_2Si_2$ type ($Z = 2$) and Cu_3Au type ($Z = 1$) slabs of the composition $U_2Ni_4P_{4-2/2}$ and UP_3. The P1 atoms are actually slightly outside the Cu_3Au building block; however, since we count them as belonging to both, the Cu_3Au layers and the $ThCr_2Si_2$ layers, we cannot count them fully at the $ThCr_2Si_2$ layer, which therefore must be counted with the

TABLE 3
Interatomic Distances in the Structure of $U_3Ni_{3.34}P_6$

U1:	8/2P3	281.0(2)	P1:	4Ni	231.4(3)
	4P1	289.7(2)		4U1	289.7(2)
	4Ni	303.4(2)	P2:	4Ni	224.6(3)
	4U1	381.8(1)		1P2	231.3(8)
U2:	8P2	293.7(2)		4U2	293.7(2)
	8Ni	302.0(2)	P3:	(1P3	37.8(12))
	4U2	381.8(1)		2/2P3	243.3(8)
Ni:	2P2	224.6(3)		4/2P3	271.3(8)
	2P1	231.4(3)		4U1	281.0(2)
	4Ni	270.0(1)		2/2P3	296.7(8)
	2U2	302.0(2)			
	2U1	303.4(2)			

Note. All distances shorter than 400 pm (U–U), 350 pm (U–Ni, U–P, Ni–Ni, Ni–P), and 340 pm (P–P) are listed. Standard deviations computed from those of the positional parameters and the lattice constants are given in parentheses.

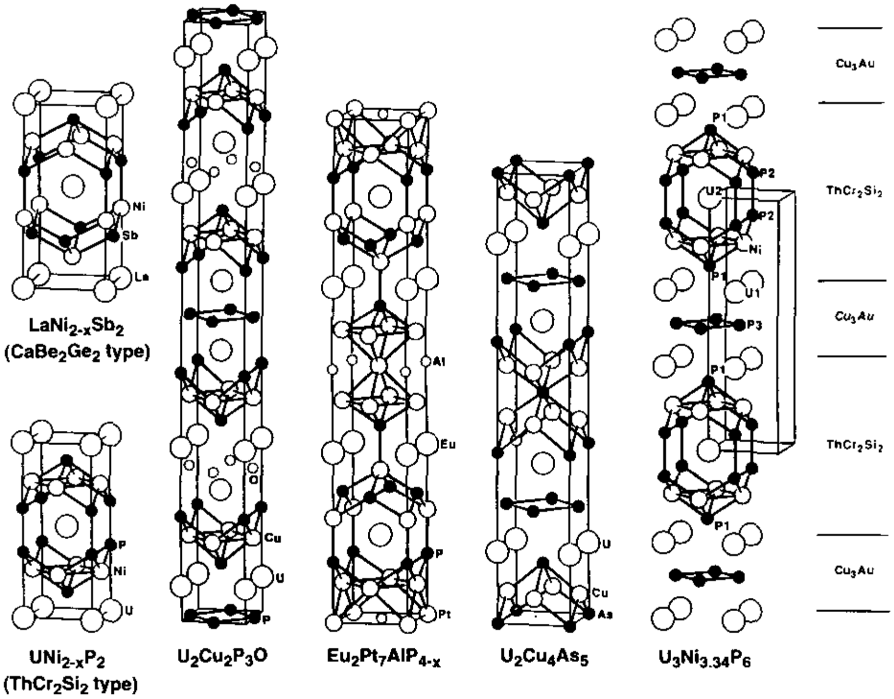

FIG. 3. The crystal structure of $U_3Ni_{3.34}P_6$ as compared to several closely related tetragonal structures with similar compositions. Several short T–T, T–Pn, and Pn–Pn distances between the transition metal (T) and pnictogen atoms (Pn) are outlined to emphasize similar polyanionic building elements of the structure. On the right-hand side, the Cu_3Au and $ThCr_2Si_2$ layers of the $U_3Ni_{3.34}P_6$ structure are emphasized.

differing composition $U_2Ni_4P_{4-2/2}$ = $U_2Ni_4P_3$. Thus, the correct composition $U_3Ni_{\sim 4}P_6$ is arrived at by adding $U_2Ni_4P_3$ and UP_3.

The coordination polyhedra of $U_3Ni_{3.34}P_6$ are shown in Fig. 4. Both uranium atoms are surrounded by nickel and phosphorus atoms. The U1 atoms have eight phosphorus neighbors in a distorted square-antiprismatic arrangement with four additional nickel atoms. The U2 atoms also have eight phosphorus neighbors, however, in the arrangement of a compressed cube. Their bonding distance of 293.7 pm is greater than the average U1–P distance of 285.3 pm. This fact could be rationalized with the greater coordination number of the U2 atoms, since the U2 atoms have eight nickel neighbors, while the U1 atoms have four. However, the uranium–nickel interactions are certainly rather weak, considering the rather large U–Ni distances of 303.4 pm (U1–Ni) and 302.0 pm (U2–Ni) as compared to the sum of the coordination number 12 radii of 281 pm (24), and we believe that the differing U–P bond lengths reflect differing oxidation states of the two uranium atoms. We suggest that these are +3 and +4 for the following reasons. The structurally best-characterized uranium phosphides are UP and U_3P_4 (25), where the phosphorus atoms are isolated from each other, and therefore the uranium atoms in these binary phosphides have the oxidation numbers +3 and +4, respectively. Actually, these oxidation states are the most frequently encountered valencies for uranium atoms in combination with pnictogens (26). Assuming that the free parameter x in the cubic structure of U_3P_4 is the same as in the prototype Th_3P_4 (27), we obtain an U–P distance of 284.2 pm for the eight phosphorus neighbors of an uranium atom in U_3P_4. This is the same coordination number and practically the same U–P distance as found for the U1 atoms in $U_3Ni_{3.34}P_6$ with an average U1–P distance of 285.3 pm. We therefore assign the oxidation number +4 to the U1 atoms of $U_3Ni_{3.34}P_6$. Since the U2–P distances of 293.7 pm are greater than the U1–P distances, an oxidation number of +3 fits well for the U2 atoms in $U_3Ni_{3.34}P_6$.

The nickel atoms in that compound occupy only one atomic site, which is coordinated by four phosphorus atoms forming a compressed tetrahedron (Fig. 4) at an average distance of 228.0 pm. This distance is only slightly larger than the Ni–P distance of 225.3 pm of the nickel atoms in $UNi_{1.51}P_2$ (17), which have practically the same coordination. The other neighbors of the nickel atoms of both compounds are virtually at the same distances: four Ni at 270.0(1) pm and 270.6(1) pm, and four U at an average of 302.7(2) pm and 303.8(1) pm in $U_3Ni_{3.34}P_6$ and $UNi_{1.51}P_2$, respectively. In both compounds the nickel sites are partially occupied with 83.6(5) and 75.4(3)%, respectively. As was argued before (28, 29), the formation of these defects can be ascribed to the relatively high electron count in the environment of the

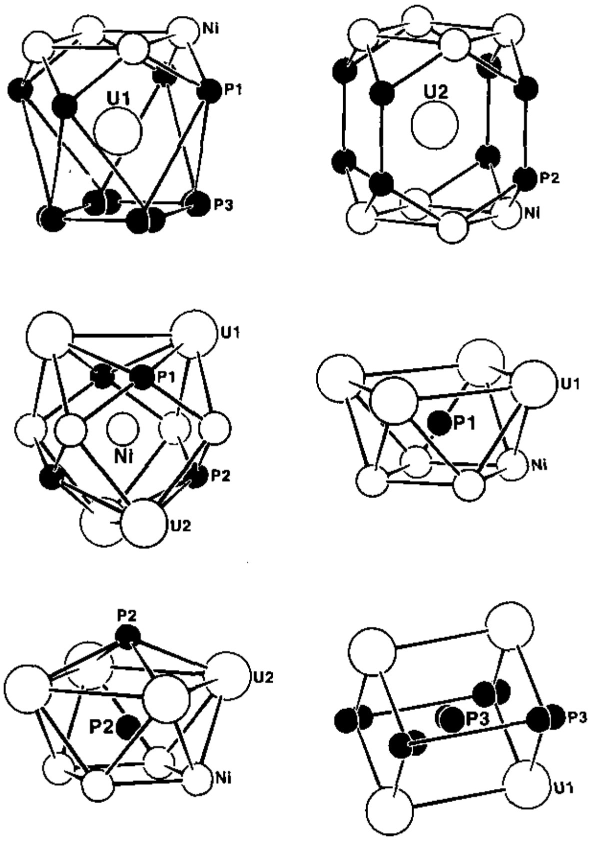

FIG. 4. Coordination polyhedra in the structure of $U_3Ni_{3.34}P_6$.

nickel atoms. For each missing nickel atom four electrons can be accommodated in nonbonding electron pairs of the four phosphorus atoms surrounding the vacant site.

The phosphorus atoms in $U_3Ni_{3.34}P_6$ occupy three different atomic sites. The P1 atoms are coordinated by four uranium and four nickel atoms, forming a (distorted) square antiprism. They have no phosphorus neighbors and since phosphorus is the most electronegative component of the compound the valence orbitals of the P1 atoms will be fully participating in chemical bonding to the metal atoms; i.e., the P1 atoms obtain the oxidation number −3. This environment of the phosphorus atoms is actually the same as that found in the $ThCr_2Si_2$ type phosphides with a high *c*/*a* ratio (4). The P2 atoms also have four uranium and four nickel neighbors forming a square antiprism. However, these phosphorus atoms form pairs (Fig. 3) with a P–P distance of 231.3(8) pm, which is only slightly longer than the typical two-electron bond distance of 222.8 pm (30). In assigning oxidation numbers and in aiming for integer numbers, the P2 atoms therefore obtain the oxidation number −2, i.e., we count −4 per P_2 pair (analogous to the O_2^{2-} ion of hydrogen peroxide). While the P1 and P2 atoms are coordinated by eight metal atoms, the P3 atoms have only four uranium neighbors and accordingly the P3–U bonds of 281.0(2) pm are shorter than the P1–U and P2–U bonds of 289.7(2) pm and 293.7(2). In addition to the uranium neighbors, the P3 atoms have phosphorus neighbors. However, since the structure refinement resulted in a split position for the P3 atoms, it is not immediately clear how many P3 neighbors each P3 has and at what distances they are. In Fig. 5 various models for the distribution of occupied and unoccupied P3 sites are indicated. In (A), all possible P3 sites are shown and the result of the structure refinement indicated, that each of these sites has an average occupancy of 50%. In (B, C, D, F), all of the short P3–P3 bonds of 243 pm are shown. In (B) and (C), each of the P3 atoms has one P3 neighbor at 243 pm and two additional ones at 271 pm. (The neighbors at 297 pm are too far away to be considered as bonded.) In (D) one-third of the P3 atoms have two neighbors at 243 pm, and two-thirds have one P3 neighbor at 243 pm and two additional ones at 271 pm. (E) presents a special case in that there are no short P3–P3 distances of 243 pm. Instead each P3 atom has four P3 neighbors at 271 pm. This situation, we suggest, might be stable at high temperatures in a dynamic form, and a snapshot might show this distribution. At lower temperatures this situation might become unstable and the structure may distort, as it is known for Jahn–Teller and Peierls phase transitions. In (F), finally, the P3 atoms form four-membered rings, where each P3 atom has two P3 neighbors at 243 pm. For symmetry reasons this is possibly the actual distribution at low temperatures.

It is now possible to assign oxidation numbers also to the P3 atoms. For simplicity we assign a bond order of 1/2 to each P3–P3 distance of 243 pm, and a bond order of 1/4 for each P3–P3 interaction of 271 pm. Then it can be seen (Fig. 5) that in all distributions (B–F), each P3 atom obtains a total P–P bond order of 1. Since two electrons are counted for each bond with this bond order, and since the phosphorus atoms are expected to obey the octet rule, each phosphorus atom also has six additional electrons, which are involved in bonding toward the uranium atoms. In counting these at the P3 atoms these atoms might be ascribed the oxidation number −2. We now have assigned oxidation numbers to all uranium and phosphorus atoms and consequently the formula of the compound may be written as $U1^{+4}U1^{+4}U2^{+3}(Ni_{3.34})^{+3}P1^{-3}P1^{-3}(P2\text{–}P2)^{-4}P3^{-2}P3^{-2}$. In a compound with a band structure, oxidation numbers do not need to be integers. Actually, the P2–P2 bonds are somewhat weaker than what is expected for a two-electron bond (see above). Therefore one might even assign a higher negative oxidation number to the P2 atoms and therefore each nickel atom may obtain an oxidation number of +1. This is the oxidation number of the nickel atoms in $CaNi_2P_2$ (31), where the P atoms form pairs with a P–P distance of

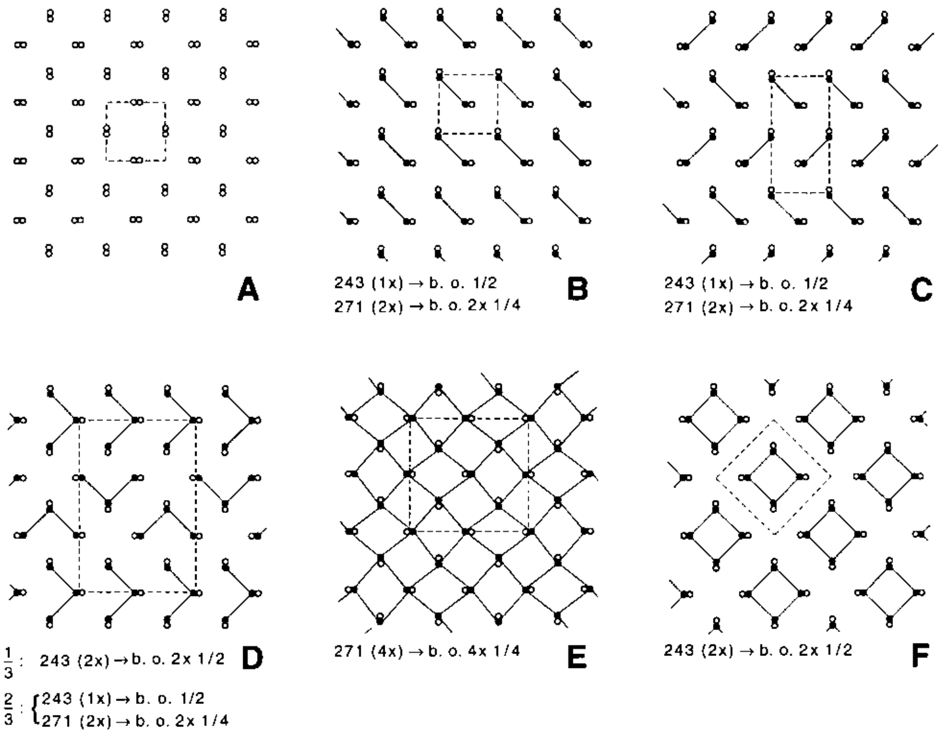

FIG. 5. Possible distributions of the phosphorus atoms on the P3 sites. All P3 sites (with a statistical occupancy of 50%) are shown in (A), in a view along the tetragonal axis. The unit cells are indicated with dashed lines and the cell shown in (A) corresponds to the cell (or subcell, respectively) actually found for $U_3Ni_{3.34}P_6$ with $a = 381.8$ pm. Various possibilities for an ordered arrangement of occupied (filled circles) and unoccupied sites (open circles) are shown in the other drawings. Short P–P bonds of 243 pm are indicated (B, C, D, F). In (E, F), the tetragonal symmetry is preserved albeit with larger cells. In (E), all P–P distances are 271 pm. The number of P3 neighbors for an average P3 atom and the corresponding distances (pm), as well as the suggested bond orders (b.o.), are also indicated.

229.7 pm. The formula of that compound may therefore be written with $Ca^{+2}Ni^{+1}Ni^{+1}(P\text{–}P)^{-4}$. The nickel atoms in that compound have also four phosphorus neighbors in tetrahedral coordination with Ni–P distances of 229.3 pm, which compares favorably with the average Ni–P distance of 228.0 pm in $U_3Ni_{3.34}P_6$. Admittedly, such a rationalization of chemical bonding is crude; nevertheless, it correlates the observed interatomic distances with plausible oxidation numbers.

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