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Redetermination of Ni_5P_4

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(P–P) = 0.001 Å
R factor = 0.027
wR factor = 0.034
Data-to-parameter ratio = 25.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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The binary phosphide pentanickel tetraphosphide, Ni_5P_4, has been reinvestigated, leading to improved reliability factors than found previously [Elfström (1965), *Acta Chem. Scand.* **19**, 1694–1704] and inclusion of anisotropic displacement parameters.

Comment

The structure of Ni_5P_4 was first determined by Elfström (1965) from Weissenberg photography (R_F = 0.10). The present determination from diffractometer data is significantly improved (R_F = 0.027) and includes anisotropic displacement parameters.

Fig. 1 shows the structure of Ni_5P_4, which has been described in detail previously in terms of the coordination polyhedra around each P and Ni atom (Elfström, 1965). It is the most metal-rich nickel phosphide that exhibits close P—P contacts [P2—P3 = 2.1907 (10) Å] within tetrahedral phosphorus clusters.

An interesting feature of the title compound is the presence of peculiar fragments formed by the condensation of three distorted square pyramids with Ni atoms at the vertices and P1 atoms at the centres (Fig. 2). The shortest distance, Ni4—P1 = 2.1645 (7) Å, is indicative of strong bonding.

Experimental

Nickel (99.99%) and red phosphorus (99.98%) powders were mixed and cold-pressed, then introduced into an alumina crucible within a sealed and evacuated silica tube. The sample was annealed at 1173 K for 120 h and then slowly cooled to room temperature. Powder X-ray diffraction patterns collected on an INEL CPS 120 diffractometer

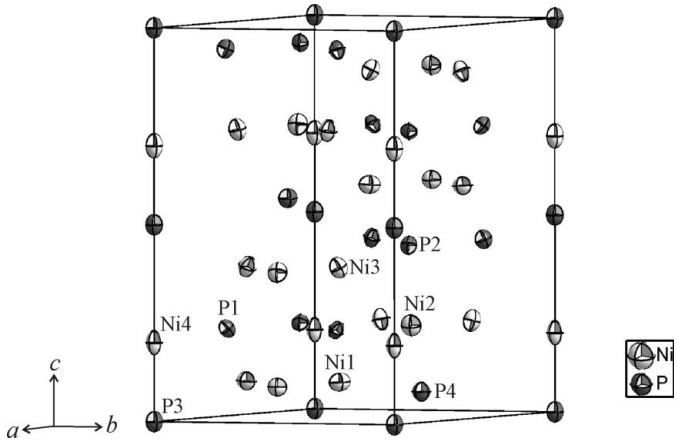

Figure 1
A clinographic projection of the title compound, with displacement ellipsoids drawn at the 99% probability level.

confirmed the presence of single-phase Ni_5P_4. Single crystals were extracted mechanically by crushing the sample.

Crystal data

Ni_5P_4
M_r = 417.33
Hexagonal, $P6_3mc$
a = 6.7921 (1) Å
c = 10.9922 (2) Å
V = 439.16 (1) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 22.31 mm^{-1}
T = 293 (2) K
0.05 × 0.03 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.472, T_{max} = 0.745
19002 measured reflections
1079 independent reflections
896 reflections with $I > 2\sigma(I)$
R_{int} = 0.082

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.034
S = 1.06
1079 reflections
42 parameters
1 restraint
$\Delta\rho_{max}$ = 1.15 e Å^{-3}
$\Delta\rho_{min}$ = −1.06 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter: 0.557 (16)

The value of the Flack parameter [0.557 (16); Flack, 1983] indicates that the crystal was twinned by inversion (Flack & Bernardinelli, 1999). The maximum peak and deepest hole are located 0.93 and 0.65 Å, respectively, from P4 and P3.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DIRAX*/LS (Duisenberg *et al.*, 2003); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

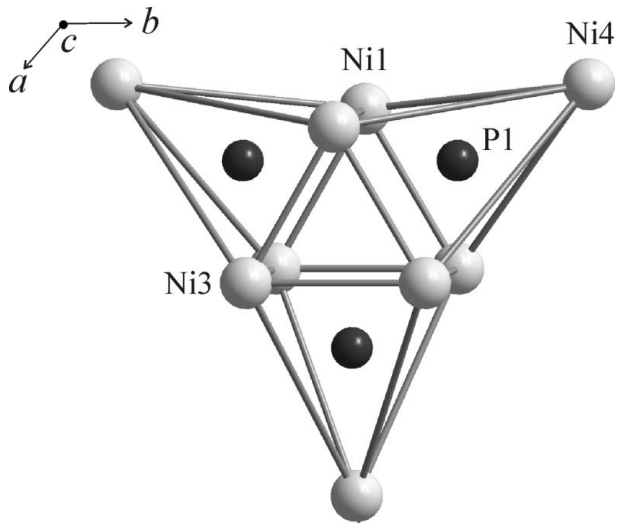

Figure 2
Fragments of condensed Ni square pyramids centred by P atoms.

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