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$Ni_2Si(P_2O_7)_2$

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Key indicators: single-crystal X-ray study; T = 295 K; mean σ(P–O) = 0.002 Å; R factor = 0.016; wR factor = 0.045; data-to-parameter ratio = 10.4.

Dinickel(II) silicon bis[diphosphate(4−)], $Ni_2Si(P_2O_7)_2$, is isotypic with other phosphates of the formula $M_2Si(P_2O_7)_2$ (M = Co, Cd). All atoms except Si (site symmetry 2) are found in general positions. Ni_2O_{10} dimers formed from edge-sharing NiO_6 octahedra are linked by corners and O—P—O bridges, forming slabs parallel to (100), which are in turn interconnected by O—Si—O contacts.

Related literature

For the structures of isotypic compounds, see: Glaum & Schmidt (1996) for $Co_2Si(P_2O_7)_2$ and Trojan *et al.* (1987) for $Cd_2Si(P_2O_7)_2$. For bond-length and angle data, see: Bostroem (1987); Durif (1995). For the extinction correction, see: Becker & Coppens (1974).

Experimental

Crystal data

$Ni_2Si(P_2O_7)_2$
M_r = 493.3
Monoclinic, $C2/c$
a = 16.8615 (9) Å
b = 4.8948 (2) Å
c = 12.1925 (5) Å
β = 103.693 (4)°
V = 977.69 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.72 mm^{-1}
T = 295 K
0.27 × 0.16 × 0.07 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with Atlas (Gemini ultra Cu) detector
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.438, T_{max} = 0.829
4978 measured reflections
1013 independent reflections
877 reflections with $I > 3\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.016
$wR(F^2)$ = 0.045
S = 1.34
1013 reflections
97 parameters
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—O1	1.9631 (18)	Si1—O7^{vii}	1.5881 (16)
Ni1—O4^{i}	2.2351 (17)	P1—O1	1.4759 (17)
Ni1—O4^{ii}	2.1436 (14)	P1—O2	1.564 (2)
Ni1—O5^{iii}	2.1271 (17)	P1—O3^{viii}	1.5888 (15)
Ni1—O5^{iv}	2.1509 (14)	P1—O4	1.5132 (16)
Ni1—O6	1.9660 (18)	P2—O3	1.5873 (19)
Si1—O2^{v}	1.6018 (18)	P2—O5	1.5017 (17)
Si1—O2^{vi}	1.6018 (18)	P2—O6	1.4768 (18)
Si1—O7	1.5881 (16)	P2—O7	1.5458 (16)

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, y-1, z$; (iv) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z$; (v) $x+\frac{1}{2}, y+\frac{3}{2}, z$; (vi) $-x+\frac{1}{2}, y+\frac{3}{2}, -z+\frac{1}{2}$; (vii) $-x+1, y, -z+\frac{1}{2}$; (viii) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *Superflip* (Palatinus & Chapuis, 2007); program(s) used to refine structure: *JANA2006* (Petříček *et al.*, 2006); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *JANA2006*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: MG2073).

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supporting information

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$Ni_2Si(P_2O_7)_2$

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S1. Comment

Although the isostructural silicodiphosphates $M_2Si(P_2O_7)_2$ have been reported for M = Fe, Ni, Co, Cu, and Cd, full single-crystal structure determinations have been carried out only for the Co and Cd members (Glaum & Schmidt, 1996; Trojan *et al.*, 1987). The structure of $Ni_2Si(P_2O_7)_2$ is reported herein. Its cell parameters are consistent with those obtained earlier from Guinier photographs (Glaum & Schmidt, 1996). The three-dimensional framework is built up of NiO_6 octahedra, SiO_4 tetrahedra, and P_2O_7 diphosphate groups. Ni_2O_{10} dimers, formed from pairs of edge-sharing NiO_6 octahedra, are interconnected through corners (O4) and O–P–O bridges of the diphosphate groups to generate slabs that lie parallel to (100) (Fig. 1). Neighboring slabs are connected through O–Si–O linkages from the SiO_4 groups (Fig. 2). The NiO_6 octahedron, whose apices are formed from one bidentate and four monodentate P_2O_7 groups (Fig. 3), is strongly distorted, with an average Ni–O distance [2.098 (2) Å] that is between those in $Ni_2P_2O_7$ (2.114 Å) and Ni_2SiO_4 (2.080 Å) (Bostroem, 1987). The P_2O_7 group adopts a nearly eclipsed conformation, with an average P–O distance [1.532 (2) Å] that is similar to other diphosphates (Durif, 1995) and a rather small P–O–P angle [132.51 (12) °] owing to its bidentate coordination to the Ni center.

S2. Experimental

$Ni_2Si(P_2O_7)_2$ in the form of powder was prepared by stoichiometric reaction of SiO_2 (99.9%, Aldrich) and $Ni_2P_4O_{12}$ (the latter being obtained from a reaction of NiO and $NH_4H_2PO_4$ in a 2:4 ratio at 750 °C for 24 h under O_2 atmosphere). The mixture was heated at 500 °C under Ar atmosphere for 2 days and 700 °C for 36 h with intermediate grindings to ensure complete reaction. Subsequent melting at 1300 °C followed by slow cooling to room temperature at a rate of 5 ° h^{-1} resulted in green crystals of the title compound.

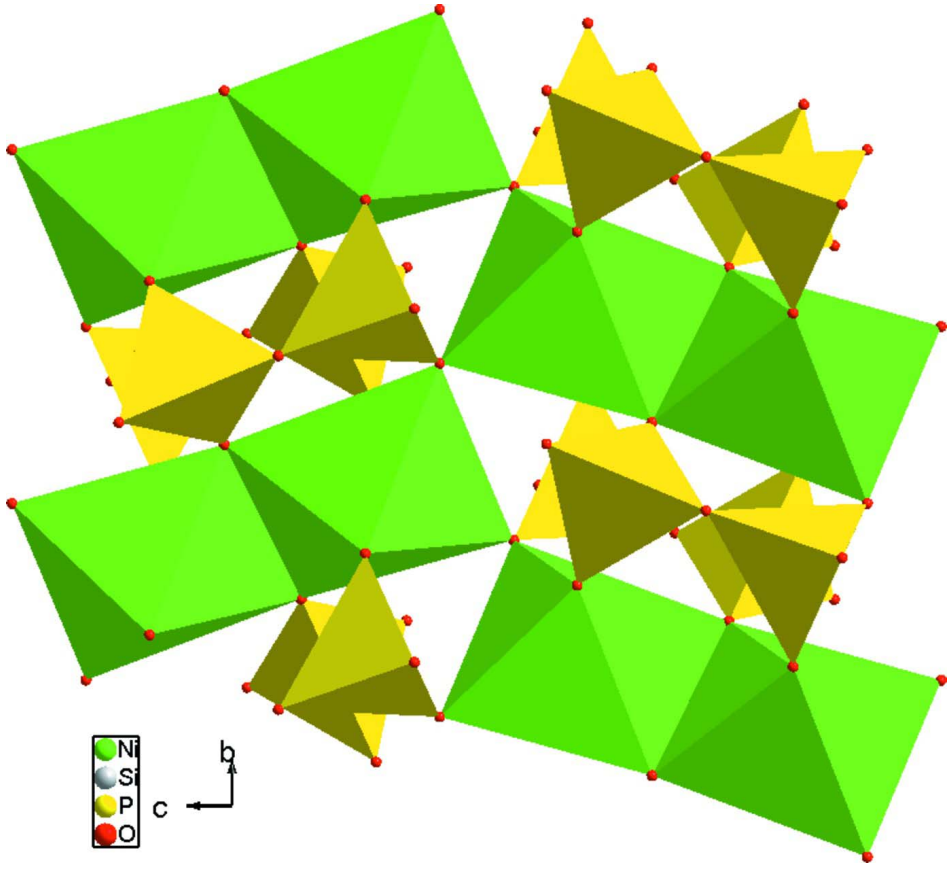

Figure 1

Ni_2O_{10} dimers and their connection in $Ni_2Si(P_2O_7)_2$.

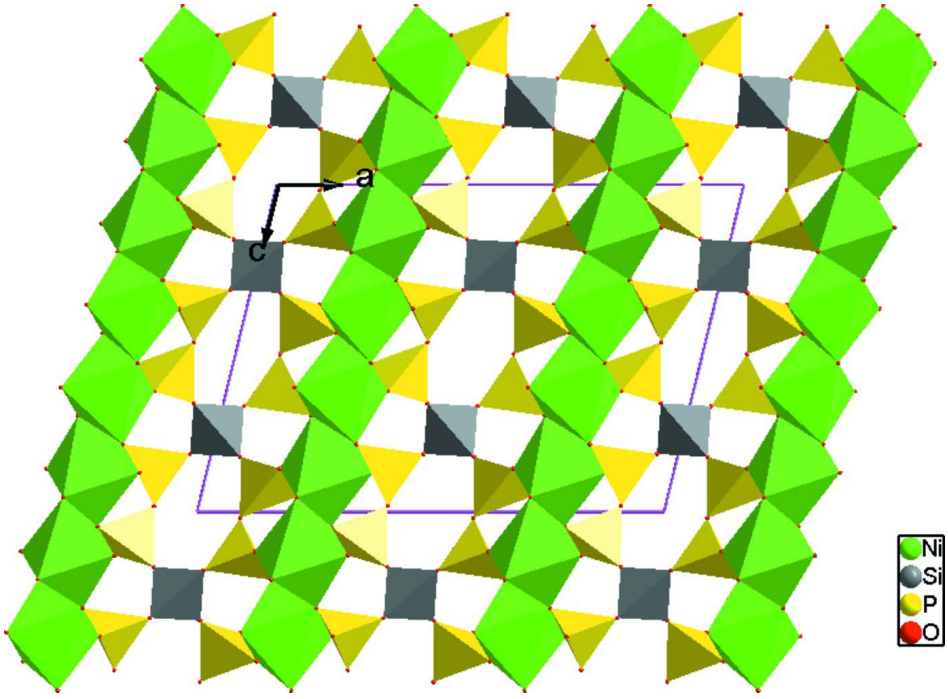

Figure 2

Projection of $Ni_2Si(P_2O_7)_2$ along [010].

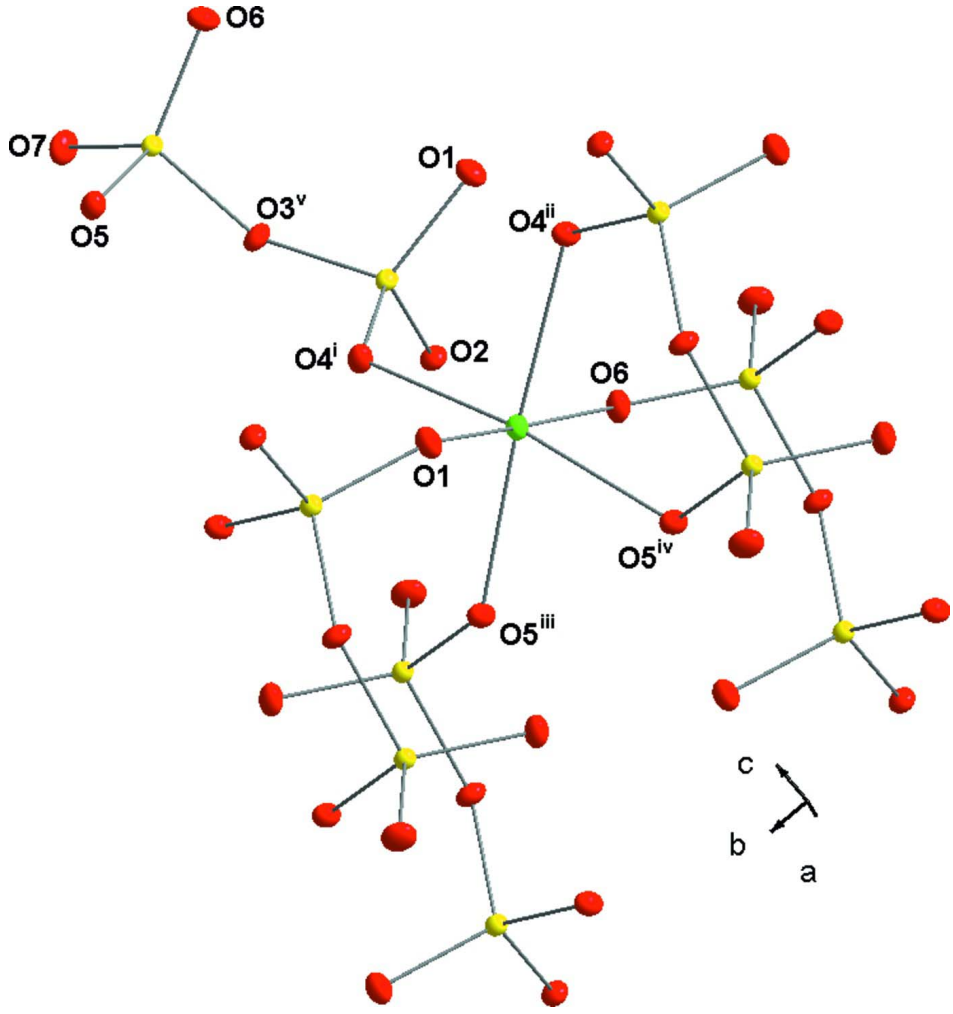

Figure 3

Local coordination geometry around the Ni atom in $Ni_2Si(P_2O_7)_2$. Symmetry codes: (i) x, 1 + y, z, (iii) x, 1 - y, z (ii) -x + 1/2,y + 1/2,-z + 1/2, (iv) -x + 1/2,-y + 3/2,-z, (v) -x + 1/2,-y + 1/2,-z. Displacement ellipsoids are drawn at the 50% probability level.

dinickel(II) silicon bis[diphosphate(4-)]

Crystal data

$Ni_2Si(P_2O_7)_2$
M_r = 493.3
Monoclinic, $C2/c$
Hall symbol: -C 2yc
a = 16.8615 (9) Å
b = 4.8948 (2) Å
c = 12.1925 (5) Å
β = 103.693 (4)°
V = 977.69 (8) Å^3
Z = 4
$F(000)$ = 968
D_x = 3.351 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3598 reflections
θ = 2.7–26.5°
μ = 4.72 mm^{-1}
T = 295 K
Plate, yellow
0.27 × 0.16 × 0.07 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with Atlas (Gemini ultra Cu) detector
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 20.7567 pixels mm^{-1}
Rotation method data acquisition using ω scans
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.438, T_{max} = 0.829
4978 measured reflections
1013 independent reflections
877 reflections with $I > 3\sigma(I)$
R_{int} = 0.023
θ_{max} = 26.5°, θ_{min} = 3.4°
h = −20→20
k = −6→6
l = −15→15

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.016
$wR(F^2)$ = 0.045
S = 1.34
1013 reflections
97 parameters
0 restraints
0 constraints
Weighting scheme based on measured s.u.'s $w = 1/[\sigma^2(I) + 0.0004I^2]$
$(\Delta/\sigma)_{max}$ = 0.010
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: B–C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
Extinction coefficient: 370 (80)

Special details

Experimental. Absorption correction CrysAlisPro; Oxford Diffraction, 2009; Version 1.171.33.34d (release 27-02-2009 CrysAlis171.NET) Absorption correction: analytical, implemented in CrysAlis RED (Oxford Diffraction, 2006).

Refinement. The refinement was carried out against all reflections. The conventional *R*-factor is always based on *F*. The goodness of fit as well as the weighted *R*-factor are based on *F* and F^2 for refinement carried out on *F* and F^2, respectively. The threshold expression is used only for calculating *R*-factors *etc*. and it is not relevant to the choice of reflections for refinement.
The program used for refinement, Jana2006, uses the weighting scheme based on the experimental expectations, see _refine_ls_weighting_details, that does not force *S* to be one. Therefore the values of *S* are usually larger than the ones from the *SHELX* program.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.247526 (19)	0.35079 (6)	0.12916 (2)	0.00715 (11)
Si1	0.5	1.16111 (18)	0.25	0.0054 (3)
P1	0.12621 (4)	−0.13090 (12)	0.13612 (5)	0.00608 (18)
P2	0.36378 (4)	0.83352 (12)	0.08433 (5)	0.00583 (18)
O1	0.14379 (10)	0.1598 (3)	0.12002 (13)	0.0100 (5)
O2	0.04526 (11)	−0.1475 (3)	0.17733 (12)	0.0088 (5)
O3	0.40484 (11)	0.7811 (3)	−0.01825 (12)	0.0091 (5)
O4	0.19424 (10)	−0.3018 (3)	0.20675 (12)	0.0088 (5)
O5	0.29494 (10)	1.0316 (3)	0.04486 (12)	0.0091 (5)
O6	0.34569 (10)	0.5703 (3)	0.13237 (12)	0.0101 (5)
O7	0.43615 (10)	0.9697 (3)	0.16829 (13)	0.0116 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.00662 (18)	0.00720 (17)	0.00757 (17)	−0.00117 (13)	0.00154 (12)	0.00048 (11)
Si1	0.0046 (5)	0.0065 (4)	0.0053 (4)	0	0.0012 (3)	0

P1	0.0054 (3)	0.0070 (3)	0.0061 (3)	−0.0008 (2)	0.0019 (2)	−0.0002 (2)
P2	0.0055 (3)	0.0064 (3)	0.0054 (3)	−0.0008 (2)	0.0010 (2)	−0.0003 (2)
O1	0.0076 (9)	0.0082 (8)	0.0146 (8)	−0.0006 (7)	0.0035 (7)	0.0012 (7)
O2	0.0085 (9)	0.0103 (8)	0.0088 (7)	−0.0003 (7)	0.0045 (6)	0.0001 (6)
O3	0.0086 (9)	0.0128 (9)	0.0066 (7)	−0.0004 (7)	0.0030 (6)	−0.0013 (6)
O4	0.0079 (9)	0.0094 (8)	0.0085 (8)	0.0019 (7)	0.0009 (7)	−0.0009 (6)
O5	0.0091 (9)	0.0098 (8)	0.0081 (7)	0.0015 (7)	0.0015 (6)	−0.0008 (7)
O6	0.0090 (9)	0.0099 (8)	0.0112 (8)	−0.0022 (7)	0.0019 (7)	0.0025 (6)
O7	0.0092 (10)	0.0145 (9)	0.0099 (8)	−0.0035 (7)	−0.0002 (7)	−0.0037 (7)

Geometric parameters (Å, º)

Ni1—O1	1.9631 (18)	Si1—O7^{vii}	1.5881 (16)
Ni1—O4^{i}	2.2351 (17)	P1—O1	1.4759 (17)
Ni1—O4^{ii}	2.1436 (14)	P1—O2	1.564 (2)
Ni1—O5^{iii}	2.1271 (17)	P1—O3^{viii}	1.5888 (15)
Ni1—O5^{iv}	2.1509 (14)	P1—O4	1.5132 (16)
Ni1—O6	1.9660 (18)	P2—O3	1.5873 (19)
Si1—O2^{v}	1.6018 (18)	P2—O5	1.5017 (17)
Si1—O2^{vi}	1.6018 (18)	P2—O6	1.4768 (18)
Si1—O7	1.5881 (16)	P2—O7	1.5458 (16)
O1—Ni1—O4^{i}	86.81 (7)	O2^{vi}—Si1—O7	110.56 (9)
O1—Ni1—O4^{ii}	95.28 (6)	O2^{vi}—Si1—O7^{vii}	109.83 (8)
O1—Ni1—O5^{iii}	93.12 (7)	O7—Si1—O7^{vii}	107.67 (10)
O1—Ni1—O5^{iv}	89.28 (6)	O1—P1—O2	108.10 (10)
O1—Ni1—O6	174.93 (7)	O1—P1—O3^{viii}	111.07 (9)
O4^{i}—Ni1—O4^{ii}	90.65 (6)	O1—P1—O4	117.37 (9)
O4^{i}—Ni1—O5^{iii}	176.27 (5)	O2—P1—O3^{viii}	98.09 (9)
O4^{i}—Ni1—O5^{iv}	98.09 (6)	O2—P1—O4	112.97 (9)
O4^{i}—Ni1—O6	89.81 (7)	O3^{viii}—P1—O4	107.57 (9)
O4^{ii}—Ni1—O5^{iii}	93.07 (6)	O3—P2—O5	107.46 (9)
O4^{ii}—Ni1—O5^{iv}	170.37 (7)	O3—P2—O6	109.95 (10)
O4^{ii}—Ni1—O6	88.53 (6)	O3—P2—O7	99.69 (9)
O5^{iii}—Ni1—O5^{iv}	78.19 (6)	O5—P2—O6	118.30 (10)
O5^{iii}—Ni1—O6	90	O5—P2—O7	111.28 (9)
O5^{iv}—Ni1—O6	87.45 (6)	O6—P2—O7	108.55 (9)
O2^{v}—Si1—O2^{vi}	108.39 (10)	Si1^{ix}—O2—P1	140.73 (11)
O2^{v}—Si1—O7	109.83 (8)	P1^{viii}—O3—P2	132.51 (12)
O2^{v}—Si1—O7^{vii}	110.56 (9)	Si1—O7—P2	168.95 (12)

Symmetry codes: (i) x, y+1, z; (ii) −x+1/2, y+1/2, −z+1/2; (iii) x, y−1, z; (iv) −x+1/2, −y+3/2, −z; (v) x+1/2, y+3/2, z; (vi) −x+1/2, y+3/2, −z+1/2; (vii) −x+1, y, −z+1/2; (viii) −x+1/2, −y+1/2, −z; (ix) x−1/2, y−3/2, z.