Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Ru	1/4	1/4	1/2	0.01092 (6)
N1	0.1147 (2)	0.12689 (11)	0.43237 (11)	0.0163 (3)
O1	−0.03967 (15)	0.11649 (11)	0.44357 (11)	0.0256 (3)
O2	0.1817 (2)	0.06029 (10)	0.37562 (11)	0.0275 (3)
N2	0.4770 (2)	0.17510 (11)	0.48434 (12)	0.0168 (3)
O3	0.5600 (2)	0.19351 (12)	0.40373 (13)	0.0340 (3)
O4	0.5423 (2)	0.11408 (12)	0.55309 (12)	0.0338 (3)
N3	0.2668 (2)	0.30940 (11)	0.34399 (11)	0.0154 (3)
O5	0.2069 (2)	0.26448 (10)	0.25960 (11)	0.0290 (3)
O6	0.3431 (2)	0.39278 (10)	0.32902 (11)	0.0254 (3)
O1*W*	1/2	−0.0097 (2)	1/4	0.0288 (4)
Na1	0.65317 (9)	0.37543 (6)	0.38096 (6)	0.0232 (2)
Na2	0.74110 (9)	0.08617 (6)	0.30610 (6)	0.0271 (2)

Table 2. *Selected geometric parameters (Å, °)*

Ru—N1	2.0518 (15)	O2—Na2^{iii}	2.369 (2)
Ru—N2	2.0649 (14)	O2—Na1^{iv}	2.402 (2)
Ru—N3	2.0639 (14)	O3—Na1	2.488 (2)
N1—O1	1.249 (2)	O3—Na2	2.385 (2)
N1—O2	1.250 (2)	O4—Na1^{v}	2.481 (2)
N2—O3	1.250 (2)	O5—Na2^{iii}	2.486 (2)
N2—O4	1.232 (2)	O5—Na1^{iii}	2.557 (2)
N3—O5	1.239 (2)	O6—Na1	2.493 (2)
N3—O6	1.257 (2)	O6—Na1^{iii}	2.569 (2)
O1—Na2^{i}	2.341 (2)	O6—Na2^{vi}	2.635 (2)
O1—Na1^{ii}	2.390 (2)	O1*W*—Na2^{iii}	2.3300 (15)
N1—Ru—N2	91.62 (6)	O2—N1—O1	116.83 (15)
N1—Ru—N3	89.51 (6)	O4—N2—O3	115.94 (14)
N2—Ru—N3	87.99 (6)	O5—N3—O6	115.91 (14)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1193). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Diphosphorus Tetraiodide at 120 K

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Abstract

The structure of the title compound, P_2I_4, was determined at 120 K. The structure is composed of isolated P_2I_4 molecules each lying on crystallographic symmetry centres. There are no apparent intermolecular contacts between the molecules. The bond distances are P—I 2.472 (2) and 2.475 (2) Å, P—P 2.230 (3) Å.

Comment

The structure was originally determined at ambient temperature from photographic data estimated visually; the structure was solved from two Patterson projections on the (100) and (010) planes (Yuen Chu Leung & Waser, 1956). Having suitable crystals of P_2I_4 on hand, we considered it useful to redetermine the structure from accurate diffractometer data collected at low temperature.

Despite an absorption correction, there are rather high residual electron densities in the vicinity of the I

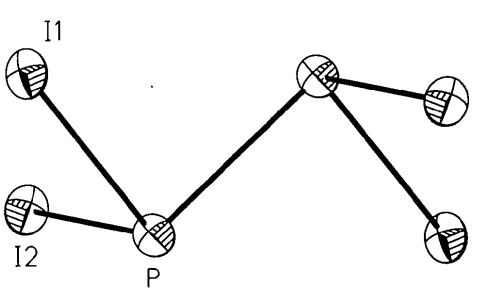

Fig. 1. Molecule of P_2I_4 showing 50% probability displacement ellipsoids.

atoms (0.75–0.93 Å) which can be attributed to residual absorption effects.

There are no basic differences between the two structures: the P—I bond distances are equal within the 2σ limit; the P—P bonds differ by 3σ. No intermolecular contacts among the molecules are apparent in the three-dimensional structure.

Experimental

The compound was obtained as an unexpected product of the reaction of $Me_3SiOP(O)Cl_2$ with Me_3SiI. However, the compound can be easily prepared by the reaction of white phosphorus with iodine in carbon disulfide solution (Germann & Traxler, 1927).

Crystal data

P_2I_4
$M_r = 569.57$
Triclinic
$P\bar{1}$
$a = 4.461\ (1)$ Å
$b = 7.044\ (1)$ Å
$c = 7.346\ (1)$ Å
$\alpha = 80.75\ (1)°$
$\beta = 73.85\ (1)°$
$\gamma = 81.78\ (1)°$
$V = 217.66\ (7)$ Å^3
$Z = 1$
$D_x = 4.345$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 30 reflections
$\theta = 17–23°$
$\mu = 14.438$ mm^{-1}
$T = 120\ (2)$ K
Needle
0.50 × 0.30 × 0.30 mm
Red–orange

Data collection

Kuma KM-4 κ-axis diffractometer with modified Enraf–Nonius LT1 low-temperature device
ω–2θ scans
Absorption correction: ψ scan (*ABSELJ*; Kuma Diffraction, 1994)
$T_{min} = 0.066$, $T_{max} = 0.151$
1722 measured reflections
1294 independent reflections
1221 observed reflections [$I > 2\sigma(I)$]
$R_{int} = 0.0218$
$\theta_{max} = 30.26°$
$h = -5 \rightarrow 6$
$k = -9 \rightarrow 9$
$l = 0 \rightarrow 10$
3 standard reflections monitored every 50 reflections
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0316$
$wR(F^2) = 0.1045$
$S = 1.212$
1286 reflections
29 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0460P)^2 + 1.5293P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 2.284$ e Å^{-3}
$\Delta\rho_{min} = -1.684$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.073 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
P	0.6070 (4)	0.1395 (2)	0.9628 (2)	0.0158 (3)
I1	0.44239 (9)	0.22834 (5)	0.66245 (5)	0.0186 (2)
I2	0.18239 (9)	0.30850 (6)	1.19424 (5)	0.0194 (2)

Table 2. *Selected geometric parameters (Å, °)*

P—P^i	2.230 (3)	P—I1	2.475 (2)
P—I2	2.472 (2)		
P^i—P—I2	94.21 (9)	I2—P—I1	102.71 (6)
P^i—P—I1	94.01 (9)		
I1—P—P^i—I2^i	76.92 (7)		

Symmetry code: (i) $1-x, -y, 2-z$.

Data collection: KM-4 diffractometer software (Kuma Diffraction, 1994). Cell refinement: KM-4 diffractometer software. Data reduction: *DATAREDC* (Kuma Diffraction, 1994). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: DU1134). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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