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

$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_i^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	$U_{eq}$
P1	0.38412 (5)	0.57986 (5)	0.25518 (4)	0.02344 (14)
P2	0.22845 (5)	0.29839(5)	0.34538 (4)	0.02411 (14)
P3	0.09604 (6)	0.37352 (5)	0.13570 (4)	0.02651 (14)
OE11	0.4015 (2)	0.71604 (15)	0.33127 (11)	0.0356 (3)
OE12	0.5045 (2)	0.58629(15)	0.16791 (11)	0.0333 (3)
OL12	0.3877 (2)	0.43657 (14)	0.32636 (11)	0.0339 (3)
OL13	0.19577 (15)	0.53501 (13)	0.19889 (10)	0.0276 (3)
OE21	0.2976 (2)	0.17137 (15)	0.37985 (11)	0.0368 (3)
OE22	0.1117 (2)	0.3539 (2)	0.41437 (12)	0.0432 (4)
OL23	0.1481 (2)	0.25726 (14)	0.22033 (10)	0.0320 (3)
OE31	-0.0831 (2)	0.3637 (2)	0.14078 (14)	0.0486 (4)
OE32	0.1714 (2)	0.3528 (2)	0.03005 (11)	0.0425 (4)
OW1	0.1433 (2)	0.4755 (2)	0.62967 (15)	0.0477 (4)
OW2	0.2338 (2)	0.9106 (2)	0.24067 (15)	0.0502 (4)
N1	-0.3981 (2)	0.3511 (2)	0.05530(14)	0.0285 (3)
N2	0.6437 (2)	0.2481 (2)	0.44668 (15)	0.0332 (4)
N3	0.0938 (2)	0.2556 (2)	-0.19197 (15)	0.0352 (4)
Cl	0.5011 (2)	0.8048 (2)	-0.0692 (2)	0.0300 (4)
C2	-0.4445 (2)	0.0801 (2)	-0.0031 (2)	0.0327 (4)
C3	-0.2707 (3)	0.1220 (2)	0.4425 (2)	0.0322 (4)
C4	-0.2664 (3)	0.0595 (2)	0.3246 (2)	0.0312 (4)
C5	0.8238 (3)	0.9326 (2)	0.3199 (2)	0.0325 (4)
C6	0 8248 (3)	0.8724(2)	0.2006 (2)	0.0338(4)

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

P1-OE11	1.4727 (13)	P2—P3	2.874 (1)
P1—OE12	1.4796 (13)	OW1—H1W1	0.88 (3)
P1—OL12	1.5993 (13)	OW1—H2W1	0.75 (4)
PI-OL13	1.6031 (13)	OW2—H1W2	0.88 (3)
P2—OE22	1.4700 (14)	OW2—H2W2	0.86 (4)
P2-0E21	1.4755 (13)	N1C1 <sup>i</sup>	1.481 (2)
P2-OL23	1.5964 (13)	N2C3"	1.487 (2)
P2—OL12	1.6098 (13)	N3C6 <sup>iii</sup>	1.482 (3)
P3-0E31	1.462 (2)	C1C2 <sup>1</sup>	1.506 (3)
P3—OE32	1.4712 (14)	C2-C2 <sup>iv</sup>	1.520 (4)
P3-OL13	1.6174 (13)	C3-C4	1.508 (3)
P3-OL23	1.6193 (13)	C4—C5 <sup>v</sup>	1.519 (3)
P1—P2	2.857 (1)	C5C6	1.512 (3)
P1—P3	2.885(1)		
OE11—P1—OE12	118.42 (8)	OE32P3OL23	107.35 (8)
OE11—P1—OL12	109.44 (8)	OL13-P3-OL23	100.17 (6)
OE12-P1-OL12	108.34 (8)	P1—OL12—P2	125.82 (8)
OE11-P1-OL13	107.31 (7)	P1-OL13-P3	127.26 (8)
OE12-P1-OL13	109.81 (7)	P2—OL23—P3	126.68 (8)
OL12-P1-OL13	102.34 (7)	P1-P2-P3	60.46 (2)
OE22-P2-OE21	120.48 (9)	P1—P3—P2	59.46 (2)
OE22-P2-OL23	110.06 (8)	P2—P1—P3	60.06 (2)
OE21-P2-OL23	107.92 (8)	H1W1—OW1—H2W1	103 (4)
OE22-P2-OL12	109.95 (9)	H1W2—OW2—H2W2	100(3)
OE21-P2-OL12	106.08 (8)	N1'C1C2'	110.5 (2)
OL23—P2—OL12	100.44 (7)	$C1^{1}-C2-C2^{1}$	111.8 (2)
OE31-P3-OE32	122.31 (9)	N2 <sup>v1</sup> C3C4	111.7 (2)
OE31-P3-OL13	106.68 (8)	C3-C4-C5*	111.8 (2)
OE32-P3-OL13	109.29 (8)	C6-C5-C4 <sup>vii</sup>	110.3 (2)
OE31-P3-OL23	108.81 (8)	N3 <sup>III</sup> —C6—C5	112.4 (2)

Symmetry codes: (i) -x, 1 - y, -z; (ii) 1 + x, y, z; (iii) 1 - x, 1 - y, -z; (iv) - 1 - x, -y, -z; (v) x - 1, y - 1, z; (vi) x - 1, y, z; (vii) 1 + x, 1 + y, z.

### Table 3. Hydrogen-bonding geometry (Å, °)

D—H···A	DH	$\mathbf{H} \cdot \cdot \cdot \mathbf{A}$	$D \cdots A$	D—H···A
OW1—H1W1···OE22	0.88 (3)	1.87 (4)	2.737 (2)	171 (3)
$OW1 - H2W1 \cdot \cdot \cdot OE22^{i}$	0.75 (4)	2.24 (4)	2.946 (2)	158 (4)
OW2H1W2···OE21 <sup>ii</sup>	0.88 (3)	1.87 (3)	2.750 (2)	178 (2)
OW2—H2W2···OE11	0.86(4)	1.88 (4)	2.732 (2)	169 (3)
$N1 - H1N1 \cdot \cdot \cdot OE12^{iii}$	0.88 (2)	1.91 (3)	2.780 (2)	170 (2)
$N1 - H2N1 \cdot \cdot \cdot OE12^{iv}$	0.88 (3)	2.05 (3)	2.906 (2)	163 (2)
N1—H3N1···OE31	0.90(2)	1.82 (3)	2.710(2)	168 (2)
$N2 - H1N2 \cdot \cdot \cdot OE11^{v}$	0.93 (3)	1.82 (3)	2.734 (2)	166 (2)
N2_H2N2OW1	0.89 (3)	2 02 (3)	2,907(3)	172 (2)

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Symmetry codes: (i) -	-x, 1-y, 1	– z; (ii) x, l	+ y, z; (iii) x –	- 1, y, z; (iv)
N3—H3N3···O <i>W</i> 2 <sup>™</sup>	0.89 (3)	1.89(3)	2.757 (2)	163 (2)
N3—H2N3···OE32	0.93 (3)	1.84 (3)	2.759 (2)	169 (2)
N3—H1N3···OW1 <sup>vi</sup>	0.94 (3)	2.09 (3)	2.982 (3)	159 (2)
N2—H3N2···OE21	0.88(3)	1.97 (3)	2.820(2)	165 (2)

-x, 1-y, -z; (v) 1-x, 1-y, 1-z; (vi) x, y, z-1.

Data collection: CAD-4 Software (Enraf-Nonius, 1989). Cell refinement: CAD-4 Software. Data reduction: MolEN (Fair, 1990). Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Software used to prepare material for publication: SHELXL93.

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Lists of structure factors, anisotropic displacement parameters, Hatom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1152). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# 1,6-Hexanediammonium cyclo-Hexaphosphate Hexahydrate

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#### Abstract

The atomic arrangement of the title compound,  $3C_6H_{18}N_2^{2+}P_6O_{18}^{6-}.6H_2O$ , is described as a stacking of  $P_6O_{18}$  groups and organic entities. The stability of such an arrangement results from a network of weak hydrogen bonds  $[N(O)-H \cdots O]$  connecting organic groups and water molecules to the external O atoms of the ring anions.

# Comment

The *cyclo*-hexaphosphate class has been investigated little when compared, for example, with the body of knowledge collected on condensed phosphates containing the  $P_2O_7$ ,  $P_3O_9$  or  $P_4O_{12}$  groups (Durif, 1995). Among the few structurally well characterized compounds, a small number of *cyclo*-hexaphosphates including organic cations are known. All were synthesized by Boullé's process (Boullé, 1938). The previously reported ( $N_3C_4H_{16}$ )<sub>2</sub> $P_6O_{18}$ .2H<sub>2</sub>O (Gharbi, Jouini & Durif, 1995) and the title compound were synthesized with the aid of the ion-exchange resins. This work describes the structural investigation of 1,6-hexanediammonium *cyclo*-hexaphosphate hexahydrate, (I).



This atomic arrangement, depicted in Fig. 1 by a projection along the c axis, can be described as a simple stacking of P<sub>6</sub>O<sub>18</sub> groups and organic entities. Both of the  $P_6O_{18}^{6-}$  anions and one of the organic cations are located around the (0,1/2,1/2) and the (1/2,0,0) inversion centers of the triclinic cell, respectively, and are interconnected along the *a* axis by hydrogen bonds from one water molecule. Connection by hydrogen bonds, along the b and c axes, from the remaining two organic groups and the water molecules gives rise to a three-dimensional network. All hydrogen bonds establishing the cohesion between the different components of the structure are rather weak, with  $O(N) \cdots O$  distances ranging from 2.715 (4) to 3.019 (3) Å (Blessing, 1986; Brown, 1976). The main geometric features of a phosphoric ring anion  $[P_n O_{3n}]^{n-}$  are the P...P distances and the P—O—P and  $P \cdots P \cdots P$  angles. The observed  $P \cdots P$  distances are quite similar whatever the *n* value. The P—O—P angles also exhibit values typical for  $[P_nO_{3n}]^{3-}$  anions. The situation is very different when the observed  $P \cdots P \cdots P$  angles in  $[P_6O_{18}]^{6-}$  (n = 6) are compared with those of  $[P_3O_9]^{3-}$  (n = 3) and  $[P_4O_{12}]^{4-}(n = 4)$ . For this purpose the P···P···P angles do not depart significantly from their ideal values of  $60 \pm 2^{\circ}$  for n = 3 and  $90 \pm 4^{\circ}$  for n = 4. However, for n = 6 we observe, in most of the available structural data, very large deviations from the ideal value of  $120^{\circ}$ . The average  $P \cdots P \cdots P$  angle in (I) is  $116.96^{\circ}$  with values ranging from 107.68 (4) to 125.55 (4)°. It should be noted that these large deviations are commonly observed in *cyclo*-hexaphosphates with a low local symmetry ring ( $\overline{1}$ ) like that in the title compound. Details of the main geometrical features of the anionic group and the organic cation are reported in Table 2. The (N—C, C—C) distances and (N—C—C, C—C—C) angles are similar to those previously reported for condensed phosphates with organic cations. Thus, they are within the ranges 1.481 (3)–1.522 (3) Å and 110.2 (2)–115.4 (2)°, respectively.



Fig. 1. Projection along the [001] direction of the  $P_6O_{18}$  groups, in polyhedral representation and of the organic entities. By order of decreasing sizes, circles represent water molecules, N and C atoms. Hydrogen bonds are denoted by full and dotted lines.

## Experimental

The title compound was synthesized by the action of *cyclo*hexaphosphoric acid on an aqueous solution of 1,6-diaminohexane. The acid was produced from  $\text{Li}_6\text{P}_6\text{O}_{18}$  solution through the use of ion-exchange resins. Colourless single crystals appeared as thick prisms after some days of evaporation of the solution at room temperature.

### Crystal data

$3C_6H_{18}N_2^{2+}.P_6O_{18}^{6-}.6H_2O$	Mo $K\alpha$ radiation
$M_r = 936.59$	$\lambda = 0.71069 \text{ Å}$
Triclinic	Cell parameters from 25
PĪ	reflections
a = 11.085(3) Å	$\theta = 10 - 14^{\circ}$
$b = 11.678 (4) \text{\AA}$	$\mu = 0.349 \text{ mm}^{-1}$
c = 8.995 (4)  Å	T = 293 (2)  K

377 parameters

refined

All H-atom parameters

 $w = 1/[\sigma^2(F_o^2) + (0.0317P)^2$ 

$ \begin{array}{llllllllllllllllllllllllllllllllllll$	$\alpha = 112.98 (3)^{\circ}$	Prism	Table 2. Se	lected geom	etric parameters (Å	, °)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	$\beta = 87.85 (3)^{\circ}$	$0.30 \times 0.25 \times 0.20$ mm	P1—OE11	1.473 (2)	N2—C6	1.483 (3)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$\gamma = 104.90(2)^{\circ}$	Colourless	P1-OE12	1.487 (2)	N3—C7	1.482 (3)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$V = 10334(6) Å^3$		P1-OL13	1.604 (2)	C1-C2 <sup>ii</sup>	1.505 (4)
$ \begin{array}{c} Z = 1 \\ D_x = 1.505 \text{ Mg m}^{-3} \\ D_m = 1.50 \text{ Mg m}^{-3} \\ D_m measured by pycnometry \\ (toluene as pycnometric liquid) \\ Data collection \\ Enraf-Nonius CAD-4 \\ diffractometer \\ \theta_{max} = 24.96^{\circ} \\ 0 \\ diffractometer \\ none \\ I = 0 \rightarrow 10 \\ 0 \\ 2885 measured reflections \\ 3885 measured reflections \\ 1 \ standard reflection (031) \\ 0 \\ cost = 1 \\$	7 1		P1OL12	1.609 (2)	C2—C3	1.519 (3)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Z = 1		P2—OE22	1.472 (2)	C3—C4 <sup>ini</sup>	1.518 (4)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	$D_x = 1.505 \text{ Mg m}^{-3}$		P2—OE21	1.497 (2)	C4—C5 <sup>i</sup>	1.522 (3)
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	$D_m = 1.50 \text{ Mg m}^{-3}$		P2—OL23	1.591 (2)	C5—C6	1.517 (3)
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	$D_m$ measured by pycnometry		P2—OL12	1.599 (2)	C7—C8	1.496 (4)
$\begin{array}{c} p_{3} = -0E32 & 1.488 (2) & C9 = -C9^{\circ} & 1.506 (5) \\ p_{3} = -0L13^{\circ} & 1.599 (2) & P_{1} = P2 & 2.932 (2) \\ p_{3} = -0L13^{\circ} & 1.599 (2) & P_{1} = P2 & 2.932 (1) \\ p_{3} = -0L23 & 1.604 (2) & P_{2} = P_{3} & 2.926 (1) \\ N_{1} = C1 & 1.481 (3) & P_{1} = P3^{\circ} & 2.927 (1) \\ p_{4} = 24.96^{\circ} & 0E_{11} = P_{1} = -0L13 & 10.62 (10) & P_{2} = -0L23 & 98.31 (9) \\ 0E_{11} = P_{1} = -0L13 & 10.62 (10) & P_{2} = -0L23 & 98.31 (9) \\ 0E_{11} = P_{1} = -0L13 & 10.618 (9) & P_{2} = -0L23 = P3 & 132.71 (11) \\ None & l = 0 \rightarrow 10 & 0E_{12} = P_{1} = -0L12 & 10.85 (10) & 93^{\circ} = -0.13 = P1 & 132.11 (10) \\ 0E_{12} = P_{1} = -0L12 & 10.35 (10) & P_{2} = -P_{1} = P_{2} & 107.68 (4) \\ 0E_{33} \text{ independent reflections} & 1 \text{ standard reflection (031)} \\ 633 \text{ independent reflections} & 1 \text{ standard reflection (031)} \\ frequency: 120 \text{ min} \\ I[I > 2\sigma(I)] & frequency: 120 \text{ min} \\ Refinement \\ Refinement \text{ on } F^{2} & \Delta\rho_{\text{max}} = 0.299 \text{ e} \text{ Å}^{-3} \\ Refinement \text{ on } F^{2} & \Delta\rho_{\text{max}} = 0.299 \text{ e} \text{ Å}^{-3} \\ Refinement \text{ on } F^{2} & \Delta\rho_{\text{max}} = 0.299 \text{ e} \text{ Å}^{-3} \\ Refinement \\ Refinement \text{ on } F^{2} & \Delta\rho_{\text{max}} = 0.299 \text{ e} \text{ Å}^{-3} \\ Refinement \text{ on } F^{2} & \Delta\rho_{\text{max}} = 0.299 \text{ e} \text{ Å}^{-3} \\ Refinement \text{ on } F^{2} & \Delta\rho_{\text{max}} = 0.299 \text{ e} \text{ Å}^{-3} \\ Refinement \text{ on } F^{2} & \Delta\rho_{\text{max}} = 0.299 \text{ e} \text{ Å}^{-3} \\ Refinement & SHELXL93 (Sheldrick, \\ y = 0.955 & SHELXL93 (Sheldrick, \\ y = 0.955 & SHELXL93 (Sheldrick, \\ y = 1.93 \text{ of } 1023 \end{pmatrix} $	(toluene as nycnometric		P3—OE31	1.476 (2)	C8—C9 <sup>1</sup>	1.518 (4)
Inquid) $p_3 = OL13^i$ $1.599(2)$ $p_1 = P2$ $2.932(2)$ Data collection $p_3 = OL23$ $1.604(2)$ $p_2 = P3$ $2.926(1)$ NI = C1 $1.481(3)$ $p_1 = P3^i$ $2.927(1)$ Enraf-Nonius CAD-4 $R_{int} = 0.0066$ $OE11 = P1 = OE12$ $119.65(10)$ $OL13^i = P3 = OL23$ $98.31(9)$ $\omega/2\theta$ scans $h = -13 \rightarrow 13$ $OE11 = P1 = OL13$ $110.62(10)$ $P2 = OL23 = P3$ $132.71(11)$ $\omega/2\theta$ scans $h = -13 \rightarrow 12$ $OE11 = P1 = OL12$ $110.35(10)$ $P3 = OL13 = P1$ $132.11(10)$ $One$ $l = 0 \rightarrow 10$ $OL13 = P1 = OL12$ $99.36(9)$ $P1 = P2 = P3$ $125.55(4)$ $3885$ measured reflections $1$ standard reflection (031) $OE22 = P2 = OL23$ $116.61(10)$ $N1 = C1 = C2^{-1}$ $119.62(10)$ $P2 = P3 = -P1^{-1}$ $3088$ observed reflections $1$ standard reflection (031) $OE22 = P2 = OL23$ $116.61(10)$ $N1 = C1 = C2^{-1}$ $113.3(2)$ $3088$ observed reflections $1$ standard reflection (031) $OE22 = P2 = OL23$ $110.61(10)$ $N1 = C1 = C2^{-1}$ $111.9(2)$ $3088$ observed reflections $1$ standard reflection (031) $OE22 = P2 = OL23$ $110.61(10)$ $N1 = C1 = C2^{-1}$ $113.3(2)$ $3088$ observed reflections $1$ standard reflection (031) $OE22 = P2 = OL23$ $106.36(9)$ $C1^{-1} = C2 = C3$ $113.3(2)$ $2022 = P2 = OL23$ $10.63(9)$ $C1^{-1} = C2^{-1}$ $113.2(2)$ $OE31 = P3 = OL33$ <td>liquid)</td> <td></td> <td>P3—OE32</td> <td>1.488 (2)</td> <td>C9—C9<sup>v</sup></td> <td>1.506 (5)</td>	liquid)		P3—OE32	1.488 (2)	C9—C9 <sup>v</sup>	1.506 (5)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	nquia)		P3—OL13'	1.599 (2)	P1—P2	2.932 (2)
$\begin{array}{c c c c c c c c c c c c c c c c c c c $			P3—OL23	1.604 (2)	P2—P3	2.926 (1)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Data collection		N1-C1	1.481 (3)	P1—P3'	2.927 (1)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Enraf-Nonius CAD-4	$R_{\rm int} = 0.0066$	OE11-P1-OE12	119.65 (10)	OL13 <sup>i</sup> —P3—OL23	98.31 (9)
$\begin{aligned} & \omega/2\theta \text{ scans} & har = 13 \rightarrow 13 \\ \omega/2\theta \text{ scans} & h = -13 \rightarrow 13 \\ \text{Absorption correction:} & k = -13 \rightarrow 12 \\ \text{none} & l = 0 \rightarrow 10 \\ 3885 \text{ measured reflections} \\ 3633 \text{ independent reflections} \\ 3088 \text{ observed reflections} \\ 1 \text{ standard reflection} \\ 3088 \text{ observed reflections} \\ 1 \text{ standard reflection} \\ 1 \text{ standard reflection} \\ 1 \text{ standard reflection} \\ I = 2\sigma(I) \end{bmatrix} \\ \hline \\ Refinement \\ S = 0.955 \\ \hline \\ \\ \omega/2d \text{ scans} \\ h = -13 \rightarrow 12 \\ 1 = 13 \rightarrow 12 \\ h = -13 \rightarrow 12 \\ 0 = 12 - 13 \rightarrow 12 \\ 0 = 12 - 12 - 0L13 \\ 0 = 10 - 0L12 \\ 0 = 10 - 0L1$	diffractometer	$\theta_{\rm max} = 24.96^{\circ}$	OE11—P1—OL13	110.62 (10)	P2—OL12—P1	132.11 (10)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	4.1/24 scans	h = -13 + 13	OE12—P1—OL13	106.18 (9)	P2_OL23_P3	132.71 (11)
Absorption correction: $k = -13 \rightarrow 12$ $OE[2 - P] - OL[2$ $108.71 (10)$ $P2 - P] - P3'$ $107.68 (4)$ none $l = 0 \rightarrow 10$ $OL[3 - P] - OL[2$ $99.36 (9)$ $P1 - P2 - P3$ $125.55 (4)$ 3885 measured reflections1 standard reflection (031) $OL[3 - P1 - OL[2]$ $99.36 (9)$ $P1 - P2 - P3$ $125.55 (4)$ 3633 independent reflections1 standard reflection (031) $OE22 - P2 - OL23$ $111.61 (10)$ $N1 - C1 - C2^{ii}$ $111.9 (2)$ 3088 observed reflectionsintensity decay: $0.64\%$ $OE22 - P2 - OL23$ $106.36 (9)$ $C1^{iii} - C2 - C3$ $113.3 (2)$ $[I > 2\sigma(I)]$ intensity decay: $0.64\%$ $OE22 - P2 - OL23$ $10.63 (9)$ $C1^{iii} - C3 - C2$ $112.3 (2)$ $Refinement$ $Refinement$ $OE21 - P2 - OL12$ $108.81 (9)$ $C3^{iii} - C4 - C5^{ii}$ $115.4 (2)$ $Refinement$ $P_{max} = 0.299$ e Å <sup>-3</sup> $OE32 - P3 - OL13^{ii}$ $107.21 (10)$ $N3 - C7 - C8$ $113.1 (2)$ $OE31 - P3 - OL13^{ii}$ $107.21 (10)$ $N3 - C7 - C8$ $113.1 (2)$ $OE31 - P3 - OL13^{ii}$ $100.82 (9)$ $C7 - C8 - C9^{iii}$ $113.7 (3)$ $wR(F^2) = 0.0954$ Extinction correction: $SHELXL93$ (Sheldrick, $SHeltrick, 100.23$ $109.51 (10)$ $C9^{ii} - c3, 2 - y, 1 - z;$ $iii 1 - x, 1 - y, 1 - z;$ $ii 2 - x, 2 - y, 2 - z.$ $Signmetry codes: (i) 2 - x, 1 - y, 1 - z;$ $ii 2 - x, 2 - y, 2 - z.$ $ii - x, 1 - y, 1 - z;$ $ii 2 - x, 2 - y, 2 - z.$		$n = -13 \rightarrow 13$	OE11—P1—OL12	110.35 (10)	P3'-OL13-P1	132.11 (10)
none $l = 0 \rightarrow 10$ $OL[3=P]-OL[2$ $99.36 (9)$ $P]-P2=P3$ $125.55 (4)$ 3885 measured reflections1 standard reflection (031) $OE22=P2-OE2$ $118.37 (10)$ $P2=P3-P1'$ $117.65 (4)$ 3633 independent reflectionsfrequency: 120 min intensity decay: 0.64% $OE22=P2-OL23$ $111.61 (10)$ $N1-C1-C2^{ii}$ $111.9 (2)$ 3088 observed reflectionsintensity decay: 0.64% $OE22=P2-OL23$ $106.36 (9)$ $C1^{ii}-C2=C3$ $113.3 (2)$ $(I > 2\sigma(I)]$ $refinement$ $OE22=P2-OL23$ $106.36 (9)$ $C1^{ii}-C2=C3$ $113.3 (2)$ Refinement $P=2-P3-P1'$ $117.65 (4)$ $OE22=P2-OL23$ $106.36 (9)$ $C1^{ii}-C2=C3$ $113.3 (2)$ $Refinement$ $P=2-P3-P1'$ $113.6 (2)$ $OE22=P2-OL12$ $109.61 (0)$ $C4^{iii}-C3=C2$ $112.3 (2)$ $Refinement$ $P=2-P3-P1'$ $115.4 (2)$ $OE22=P2-OL12$ $108.81 (9)$ $C3^{iii}-C4=C5'$ $115.4 (2)$ $OE31=P3-OL3^{ii}$ $107.21 (10)$ $N3=C7=C8$ $113.1 (2)$ $OE31=P3-OL13^{ii}$ $107.21 (10)$ $N3=C7=C8$ $113.7 (3)$ $WR(F^2) = 0.0954$ Extinction correction: $SHELXL93$ (Sheldrick, $OE31=P3-OL23$ $109.51 (10)$ $C9^{-}C9=C8^{ii}$ $113.7 (3)$ $S=0.955$ $SHELXL93$ (Sheldrick, $1-x, 1-y, 1-z; (iv) 2-x, 2-y, 2-z; (v) 3-x, 2-y, 2-z.10.2, 2, 2, 2, 2, 2, 2, 2, 2, 2, 2, 2, 2, 2$	Absorption correction:	$k = -13 \rightarrow 12$	OE12—P1—OL12	108.71 (10)	P2—P1—P3'	107.68 (4)
3885 measured reflections1 standard reflection (031) $OE22-P2-OE21$ $118.37(10)$ $P2-P3-P1^{-}$ $117.65(4)$ 3633 independent reflectionsfrequency: 120 min intensity decay: 0.64% $OE22-P2-OL23$ $111.61(10)$ $N1-C1-C2^{ii}$ $111.9(2)$ 3088 observed reflectionsintensity decay: 0.64% $OE22-P2-OL23$ $106.36(9)$ $C1^{ii}-C2-C3$ $113.3(2)$ $[I > 2\sigma(I)]$ $Refinement$ $OE22-P2-OL23$ $106.36(9)$ $C1^{ii}-C2-C3$ $113.3(2)$ Refinement on $F^2$ $\Delta \rho_{max} = 0.299$ e Å <sup>-3</sup> $OE21-P2-OL12$ $108.81(9)$ $C3^{iii}-C4-C5^{ii}$ $111.4(2)$ $OE31-P3-OE32$ $118.15(11)$ $N2-C6-C5$ $111.5(2)$ $OE31-P3-OL3^{ii}$ $107.21(10)$ $N3-C7-C8$ $113.1(2)$ $OE31-P3-OL3^{ii}$ $109.51(10)$ $C9^{ii}-C9-C8^{iii}$ $113.7(3)$ $WR(F^2) = 0.0954$ Extinction correction: $SHELXL93$ (Sheldrick, $100.30$ ) $Symmetry$ codes: (i) $2 - x, 1 - y, 1 - z$ ; (ii) $2 - x, 2 - y, 1 - z$ ; (iii) $1 - x, 1 - y, 1 - z$ ; (iv) $2 - x, 2 - y, 2 - z$ .	none	$l = 0 \rightarrow 10$	OL13-P1-OL12	99.36 (9)	P1—P2—P3	125.55 (4)
$\begin{array}{llllllllllllllllllllllllllllllllllll$	3885 measured reflections	1 standard reflection (031)	OE22—P2—OE21	118.37 (10)	P2	117.65 (4)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	3633 independent reflections	frequency: 120 min	0E22_P2_0L23	111.61 (10)	NI-CI-C2"	111.9 (2)
$ \begin{array}{c} [I > 2\sigma(I)] \\ \hline [I > 2\sigma(I)] \\ \hline Refinement \\ Refinement \\ Refinement on F^2 \\ R[F^2 > 2\sigma(F^2)] = 0.0282 \\ mr(F^2) = 0.0954 \\ S = 0.955 \\ S = 0$	2009 charmed reflections	intensity decay 0.64%	0E21—P2—0L23	106.36 (9)	$C1^{-}-C2-C3$	113.3 (2)
$[I > 2\sigma(I)]$ Refinement Refinement on $F^2$ $\Delta \rho_{max} = 0.299 \text{ e} \text{ Å}^{-3}$ $R[F^2 > 2\sigma(F^2)] = 0.0282$ $M^{(F^2)} = 0.0954$ $M^{(F^2)} = 0.0954$ SHELXL93 (Sheldrick, 1000 SHELXE)) SHELXEPS (SHELXEPS SHELXEPS SHELXEP	SUSS Observed Tenections	intensity decay: 0.04%	OE22 - P2 - OL12	110.96 (10)	$C4^{}C3C2$	112.3 (2)
RefinementRefinement on $F^2$ $\Delta \rho_{max} = 0.299 \text{ e} \text{ Å}^{-3}$ $OE31 - P3 - OE32$ $118.15(11)$ $N2 - C6 - C5$ $111.4(2)$ $R[F^2 > 2\sigma(F^2)] = 0.0282$ $\Delta \rho_{min} = -0.351 \text{ e} \text{ Å}^{-3}$ $OE31 - P3 - OE32$ $118.15(11)$ $N3 - C7 - C8$ $113.1(2)$ $wR(F^2) = 0.0954$ Extinction correction: $S = 0.955$ $SHELXL93$ (Sheldrick, $OE31 - P3 - OL13^{\circ}$ $10.82(9)$ $C7 - C8 - C9^{\circ\circ}$ $113.7(3)$ South contraction:SHELXL93 (Sheldrick, $1003$	$[I > 2\sigma(I)]$		0E21-P2-0L12	108.81 (9)		115.4 (2)
Refinement $DE31-F3-OE32$ $118.15(11)$ $N2-C6-C53$ $111.5(2)$ Refinement on $F^2$ $\Delta \rho_{max} = 0.299$ e Å <sup>-3</sup> $OE31-F3-OL3^3$ $107.21(10)$ $N3-C7-C8$ $113.1(2)$ $R[F^2 > 2\sigma(F^2)] = 0.0282$ $\Delta \rho_{min} = -0.351$ e Å <sup>-3</sup> $OE31-P3-OL13^3$ $107.21(10)$ $N3-C7-C8-C9^{v}$ $110.2(2)$ $wR(F^2) = 0.0954$ Extinction correction: $SHELXL93$ (Sheldrick, $OE31-P3-OL23$ $109.51(10)$ $C9^{\circ}-C9-C8^{v}$ $113.7(3)$ $S = 0.955$ $SHELXL93$ (Sheldrick, $100.31$ $Symmetry$ codes: (i) $2 - x, 1 - y, 1 - z$ ; (ii) $2 - x, 2 - y, 1 - z$ ; (iii) $1 - x, 1 - y, 1 - z$ ; (iv) $2 - x, 2 - y, 2 - z$ .			0L23—P20L12	98.89 (9)	10C3C4 N2 - C4 - C5	111.4 (2)
Control Contrelation Control Control Control Control Control Cont	Refinement		0E31P30E32	118.15 (11)	N2-C6-C3	111.3(2)
Refinement on $F^2$ $\Delta \rho_{max} = 0.299 \text{ e A}^{-3}$ $OE32=F3=OE13$ $110.82(9)$ $C7=C6=C9$ $110.2(2)$ $R[F^2 > 2\sigma(F^2)] = 0.0282$ $\Delta \rho_{min} = -0.351 \text{ e A}^{-3}$ $OE31=P3=OL23$ $109.51(10)$ $C9^{\circ}=C9=C8^{10}$ $113.7(3)$ $wR(F^2) = 0.0954$ Extinction correction:SHELXL93 (Sheldrick, $SHELXL93$ (Sheldrick, $1003$ ) $Symmetry$ codes: (i) $2 - x, 1 - y, 1 - z$ ; (ii) $2 - x, 2 - y, 1 - z$ ; (iii)		A 0.000 Å−3	$OE31 - P3 - OL13^{1}$	107.21 (10)	$N_{3} - C_{7} - C_{8}$	113.1(2)
$\begin{array}{l} R[F^2 > 2\sigma(F^2)] = 0.0282 \\ wR(F^2) = 0.0954 \\ S = 0.955 \\ $	Refinement on F <sup>2</sup>	$\Delta \rho_{\rm max} = 0.299 \ {\rm e} \ {\rm A}_{\odot}$	0E32P3	100.51 (10)	$C_{1} = C_{0} = C_{0}$	110.2(2)
$wR(F^2) = 0.0954$ Extinction correction:       Superior Structure       Superior Structure       Superior Structure $S = 0.955$ SHELXL93 (Sheldrick, 1003)       Superior Structure       Superior Structure       Superior Structure	$R[F^2 > 2\sigma(F^2)] = 0.0282$	$\Delta \rho_{\rm min} = -0.351 \ {\rm e} \ {\rm A}^{-3}$	OE31-P3-OL23	110.97 (10)	() -()-(0	113.7 (3)
S = 0.955 SHELXL93 (Sheldrick, 1003) $SHELXL93 (Sheldrick, 1003)$ $Symmetry codes: (1) 2 - x, 1 - y, 1 - z; (11) 2 - x, 2 - y, 1 - z; (11) 1 - x, 1 - y, 1 - z; (11) 2 - x, 2 - y, 2 - z; (11) 2 - x, 2 - y; (11) 2 - x; (11) 2$	$wR(F^2) = 0.0954$	Extinction correction:	0232-13-0223	110.57 (10)		
1 - x, 1 - y, 1 - z; (iv) 2 - x, 2 - y, 2 - z; (v) 3 - x, 2 - y, 2 - z.	S = 0.955	SHELXL93 (Sheldrick	Symmetry codes: (1)	2 - x, 1 - y,	1 - z; (1) $2 - x, 2 - y$	, 1 - z; (11)
	3633 reflections	1003)	1 - x, 1 - y, 1 - z;	(1v) 2 - x, 2 -	y, 2 - z; (v) 3 - x, 2 -	y, 2 - z.

# Table 3. Hydrogen-bonding geometry (Å, °)

D—H···A	D—H	H···A	$D \cdot \cdot \cdot A$	$D - H \cdots A$
$N1 - H1N1 \cdots OE21^{i}$	0.89 (3)	2.09 (3)	2.946 (3)	159 (3)
N1—H2N1···OW1	0.93 (4)	1.84 (4)	2.715 (4)	157 (3)
N1—H3N1···OE21	0.91 (4)	1.96 (4)	2.862 (3)	171 (3)
N2—H1N2· · · OE32 <sup>ii</sup>	0.85 (3)	2.02 (3)	2.865 (3)	170 (3)
N2—H2N2···OE32 <sup>iii</sup>	0.87 (4)	2.10 (4)	2.953 (3)	166 (3)
N2—H3N2···OE21	0.88 (4)	2.00 (4)	2.876 (3)	172 (3)
N3—H1N3· · ·O₩3 <sup>iv</sup>	0.89(3)	1.96 (3)	2.840(3)	175 (2)
N3—H2N3···OE12 <sup>v</sup>	0.88 (3)	2.01 (3)	2.787 (3)	147 (3)
N3—H3N3···OE12	0.91 (3)	1.92 (3)	2.801 (3)	163 (3)
OW1—H1W1···OE31	0.77 (4)	2.02 (5)	2.784 (4)	173 (4)
$OW1 - H2W1 \cdot \cdot \cdot OE22^{v_1}$	0.70 (5)	2.26 (5)	2.930(4)	163 (5)
OW2—H1 $W2$ ···OE22 <sup>V1</sup>	0.79 (4)	2.23 (4)	3.019(3)	178 (3)
OW2—H2W2···OE31	0.81 (4)	2.01 (5)	2.814 (3)	171 (4)
OW3—H1W3· · ·OE11	0.73 (4)	2.04 (4)	2.758 (3)	168 (4)
O₩3—H2₩3· · ·OE32 <sup>iii</sup>	0.81 (4)	2.19 (4)	2.943 (3)	156 (4)
Symmetry codes: (i) 2	- x. 2 - ·	$v_{1} = z_{1}$ (ii)	$2 - x \cdot 1 - y$	1 - 7; (iii)
x y z = 1; (iv) $x y 1 = 1$	7: (v) 1 -	$r_{1} = v_{1} = v_{1}$	$(v_i)_{2}^{-1} = x_{2}^{-1}$	$-v^{2} - z$
$x_1, y_2 \in [1, (1), x_2, y_2] \in T$	2, (*) 1	4, I — y, I —	(2, (1)) = 2, 2	-y, z - z.

Data collection: CAD-4 Software (Enraf-Nonius, 1989). Cell refinement: CAD-4 Software. Data reduction: MolEN (Fair, 1990). Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Software used to prepare material for publication: SHELXL93.

The authors would like to express their gratitude to Dr T. Jouini, Département de Chimie, Faculté des Sciences, Tunisia, for the X-ray data collection.

Lists of structure factors, anisotropic displacement parameters, Hatom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1153). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

 $\begin{array}{l} \mu = \mu_{[0} (x_{0}) + (\cos(2\pi)) \\ + 1.2088P] \\ \text{where } P = (F_{o}^{2} + 2F_{c}^{2})/3 \\ (\Delta/\sigma)_{\text{max}} < 0.001 \end{array}$ Vol. C, Tables 4.2.6.8 and 6.1.1.4) Table 1. Fractional atomic coordinates and equivalent

# isotropic displacement parameters ( $Å^2$ )

Extinction coefficient:

Atomic scattering factors

from International Tables

for Crystallography (1992,

0.0199 (21)

# $U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_i^* \mathbf{a}_i \cdot \mathbf{a}_j.$

x	у	z	$U_{eq}$
0.77327 (5)	0.51804 (5)	0.32682 (6)	0.0200 (2
0.94537 (5)	0.72793 (5)	0.58775 (6)	0.0198 (2
1.11003 (5)	0.69600 (5)	0.80720 (6)	0.0207 (2
0.9848 (2)	0.80500 (15)	0.4864 (2)	0.0286 (4
1.1673 (2)	0.8253 (2)	0.9318 (2)	0.0365 (4
0.8081 (2)	0.5705 (2)	0.2019 (2)	0.0343 (4
0.65042 (15)	0.5213 (2)	0.3982 (2)	0.0310 (4
0.88159 (15)	0.58147 (14)	0.4713 (2)	0.0259 (4
0.8679 (2)	0.7745 (2)	0.7235 (2)	0.0336 (4
1.0066 (2)	0.6084 (2)	0.8524 (2)	0.0318 (4
1.06986 (14)	0.7092 (2)	0.6464 (2)	0.0275 (4
0.77927 (14)	0.37080(14)	0.2615 (2)	0.0262 (4
1.1497 (3)	1.0606 (3)	0.9380 (3)	0.0673 (8
1.1266 (3)	0.9602 (3)	1.2572 (3)	0.0528 (6
0.7579(2)	0.6400(2)	-0.0444(3)	0.0454 (5
1.1498 (2)	1.0582(2)	0.6350(3)	0.0316 (5
1.0765 (2)	0.6564 (2)	0.1892 (3)	0.0265 (4
0.5907 (2)	0.6337 (2)	0.7192 (3)	0.0289 (4
1.2811 (2)	1.0553 (3)	0.6018 (3)	0.0340 (6
0.6307 (2)	0.8117 (2)	0.3332 (3)	0.0318 (5
0.4944 (2)	0.8117(2)	0.3578 (3)	0.0321 (5
0.5904 (2)	0.3232 (2)	0.6849 (3)	0.0332 (6
1.2706(2)	0.6682 (3)	0.3294 (3)	0.0298 (5
1.2148 (2)	0.6845 (3)	0.1897 (3)	0.0285 (5
0.5736(3)	0.7609 (3)	0.7409 (3)	0.0333 (6
0.5312 (3)	0.8302 (3)	0.9036 (3)	0.0354 (6
1.4834 (3)	1.0403 (3)	1.0825 (3)	0.0398 (6
	x 0.77327 (5) 0.94537 (5) 1.1103 (5) 0.9848 (2) 1.1673 (2) 0.8081 (2) 0.65042 (15) 0.88159 (15) 0.88159 (15) 0.88159 (2) 1.0066 (2) 1.06986 (14) 0.77927 (14) 1.1497 (3) 1.1266 (3) 0.7579 (2) 1.1498 (2) 1.0765 (2) 0.5907 (2) 1.2811 (2) 0.6307 (2) 1.2811 (2) 0.6307 (2) 1.2811 (2) 0.5904 (2) 1.2706 (2) 1.2148 (2) 0.5736 (3) 0.5312 (3) 1.4834 (3)	$\begin{array}{cccc} x & y \\ 0.77327 (5) & 0.51804 (5) \\ 0.94537 (5) & 0.72793 (5) \\ 1.11003 (5) & 0.69600 (5) \\ 0.9848 (2) & 0.80500 (15) \\ 1.1673 (2) & 0.8253 (2) \\ 0.8081 (2) & 0.5705 (2) \\ 0.65042 (15) & 0.5213 (2) \\ 0.88159 (15) & 0.58147 (14) \\ 0.8679 (2) & 0.7745 (2) \\ 1.0066 (2) & 0.6084 (2) \\ 1.06986 (14) & 0.7092 (2) \\ 0.77927 (14) & 0.37080 (14) \\ 1.1497 (3) & 1.0606 (3) \\ 1.1266 (3) & 0.9602 (3) \\ 0.7579 (2) & 0.64400 (2) \\ 1.07579 (2) & 0.65042 (2) \\ 1.0757 (2) & 0.6504 (2) \\ 1.0755 (2) & 0.6564 (2) \\ 0.5907 (2) & 0.6337 (2) \\ 1.2811 (2) & 1.0553 (3) \\ 0.6307 (2) & 0.8117 (2) \\ 0.4944 (2) & 0.8117 (2) \\ 0.4944 (2) & 0.8117 (2) \\ 0.4944 (2) & 0.8117 (2) \\ 0.5904 (2) & 0.5232 (2) \\ 1.2706 (2) & 0.6682 (3) \\ 1.2148 (2) & 0.6845 (3) \\ 0.5736 (3) & 0.7609 (3) \\ 0.5312 (3) & 0.8302 (3) \\ 1.4834 (3) & 1.0403 (3) \\ \end{array}$	xyz $0.77327$ (5) $0.51804$ (5) $0.32682$ (6) $0.94537$ (5) $0.72793$ (5) $0.58775$ (6) $1.11003$ (5) $0.69600$ (5) $0.80720$ (6) $0.9848$ (2) $0.80500$ (15) $0.4864$ (2) $1.1673$ (2) $0.8253$ (2) $0.9318$ (2) $0.8081$ (2) $0.5705$ (2) $0.2019$ (2) $0.65042$ (15) $0.5213$ (2) $0.3982$ (2) $0.8659$ (2) $0.7745$ (2) $0.7235$ (2) $1.0666$ (2) $0.6084$ (2) $0.8524$ (2) $1.06986$ (14) $0.7092$ (2) $0.6464$ (2) $0.77927$ (14) $0.37080$ (14) $0.2615$ (2) $1.1497$ (3) $1.0666$ (3) $0.9380$ (3) $1.1266$ (3) $0.9602$ (3) $1.2572$ (3) $0.7579$ (2) $0.6400$ (2) $-0.0444$ (3) $1.0765$ (2) $0.6564$ (2) $0.618$ (3) $0.6307$ (2) $0.6541$ (2) $0.3322$ (3) $0.5907$ (2) $0.66402$ (3) $0.3232$ (3) $0.6307$ (2) $0.6337$ (2) $0.7192$ (3) $1.2811$ (2) $1.0553$ (3) $0.6018$ (3) $0.6307$ (2) $0.6845$ (3) $0.3294$ (3) $1.2706$ (2) $0.6845$ (3) $0.3294$ (3) $1.2148$ (2) $0.6845$ (3) $0.7409$ (3) $0.5736$ (3) $0.7609$ (3) $0.7409$ (3) $0.5736$ (3) $0.7609$ (3) $0.7409$ (3) $0.5736$ (3) $0.7609$ (3) $0.7409$ (3) $0.5736$ (3) $0.7609$ (3) $0.7409$ (3) $0.5736$ (3) $0.7609$ (3) $0.7409$ (3) $0.5736$ (3) $0.76$

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# A Dihydrooxazinone

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# Abstract

(3RS,6SR,1'RS)-6-tert-Butyl-3,6-dihydro-5-methoxy-3-methyl-3-(3-oxocyclohexyl)-2H-1,4-oxazin-2-one,  $C_{16}H_{25}NO_4$ , is an intermediate in the stereoselective synthesis of  $\alpha$ -methylated amino acids. Its structure agrees well with those of other dihydrooxazinones. The heterocyclic ring is almost planar while the side chains exhibit the expected staggered conformations.

## Comment

The stereospecific synthesis of dihydrooxazinones is a versatile synthetic route to enantiomerically pure  $\alpha$ methylated amino acids formed upon hydrolysis (Maywald, 1987). In order to study the influence of the size and nature of the substituents on the conformation of the heterocyclic ring, several dihydrooxazinones with an isopropyl group at C(6) have been investigated (Bolte, 1995). We have determined the structure of a racemic derivative, (I), with a tertiary butyl group replacing the isopropyl moiety.



The bond lengths and angles agree well with the other dihydrooxazinones studied so far (Bolte, 1995). In contrast to lactides (1,4-dioxane-2,5-diones) in which N(4) is replaced by an O atom (Bolte, Beck, Nieger & Egert, 1994), the heterocyclic ring is almost planar  $(\sigma = 0.04 \text{ Å})$ . The cyclohexanone ring adopts a chair conformation and is oriented such that the five-atom chain from C(2) to C(5') is fully extended.



Fig. 1. Molecular structure of (I) showing 40% probability displacement ellipsoids.

## Experimental

The compound was provided by Professor U. Schöllkopf (University of Göttingen) and recrystallized from cyclohexane.

### Crystal data

C <sub>16</sub> H <sub>25</sub> NO <sub>4</sub>	Mo $K\alpha$ radiation
$M_r = 295.4$	$\lambda = 0.71073 \text{ Å}$
Triclinic	Cell parameters from 50
PĪ	reflections
a = 6.806(1)  Å	$\theta = 10 - 12.5^{\circ}$
b = 8.166(1) Å	$\mu = 0.08 \text{ mm}^{-1}$
c = 15.568 (2) Å	T = 293  K
$\alpha = 100.69 (1)^{\circ}$	Block
$\beta = 94.99(2)^{\circ}$	$0.6 \times 0.4 \times 0.1 \text{ mm}$
$\gamma = 103.29 (1)^{\circ}$	Colourless
$V = 819.8 \text{ Å}^3$	
Z = 2	
$D_x = 1.197 \text{ Mg m}^{-3}$	
$D_m$ not measured	

### Data collection

Stoe–Siemens four-circle	$R_{\rm int} =$
diffractometer	$\theta_{max} =$
$\omega/2\theta$ scans	h = -
Absorption correction:	k = -
none	l = -
3763 measured reflections	3 stan
2882 independent reflections	mo
1947 observed reflections	r
$[F > 3\sigma(F)]$	inte

0.025 = 25°  $8 \rightarrow 7$  $9 \rightarrow 9$  $4 \rightarrow 18$ dard reflections nitored every 100 reflections ensity decay: none