

Fig. 3. Projections of (a) the  $\alpha$ -trans-UO<sub>2</sub>Cl<sub>2</sub>(tppo)<sub>2</sub> form and (b) the  $\beta$ -trans-UO<sub>2</sub>Cl<sub>2</sub>(tppo)<sub>2</sub> form along the Cl—U—Cl bonds.

Selected bond lengths and angles (Table 3) show that the distances about the U atom are equivalent within e.s.d.'s for the *cis* and  $\beta$ -*trans* structures. The distances are also in agreement with the  $\alpha$ -*trans* structure (Bombieri, Forsellini, Day & Azeez, 1978) and a search of the Cambridge Structural Database (Table 3).

The angle at the bridging O atom of the triphenyl phosphine oxide ligand is found to be lowest for the *cis* form, 144.5°, compared with values from 154 to 167° observed in the data search and is probably due to steric hindrance between the adjacent bulky ligands. The values observed in the  $\beta$ -*trans* structure of 151.7 and 166.2° are assumed to be different only because of packing constraints.

In the paper by Bombieri *et al.* (1978) on the  $\alpha$ -*trans*-UO<sub>2</sub>Cl<sub>2</sub>(tppo)<sub>2</sub> structure the probability of the *cis* form being determined was anticipated. The determination of the new  $\beta$ -*trans*-UO<sub>2</sub>Cl<sub>2</sub>(tppo)<sub>2</sub> structure suggests that there may be more structure forms dependent only upon conditions of crystallization.

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## Structure of Tetraphenylarsonium Bis(1,2-dithiooxalato-*S,S'*)palladate(II)

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**Abstract.** [As(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>]<sub>2</sub>[Pd(C<sub>2</sub>O<sub>2</sub>S<sub>2</sub>)<sub>2</sub>], *M<sub>r</sub>* = 1113.37, triclinic, *P* $\bar{1}$ , *a* = 11.101 (7), *b* = 13.316 (5), *c* = 18.485 (8) Å,  $\alpha$  = 110.90 (5),  $\beta$  = 77.74 (8),  $\gamma$  = 106.28 (8)°, *V* = 2431 (2) Å<sup>3</sup>, *Z* = 2, *D<sub>m</sub>* = 1.53 (1), *D<sub>x</sub>* = 1.52 g cm<sup>-3</sup>,  $\lambda$ (Mo *K* $\alpha$ ) = 0.71069 Å,  $\mu$  =

19.317 cm<sup>-1</sup>, *T* = 296 K, *F*(000) = 1120, *R* = 0.041, *wR* = 0.045 for 5034 observed reflections with *I* > 3 $\sigma$ (*I*). The asymmetric unit of the title compound contains one discrete complex [Pd(C<sub>2</sub>S<sub>2</sub>O<sub>2</sub>)<sub>2</sub>]<sup>2-</sup> anion and two [As(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>]<sup>+</sup> cations linked through electrostatic interactions and hydrogen contacts of type C—H...O.

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**Introduction.** In recent years many works have been published on the coordination chemistry and characterization of the dithiooxalate ligand showing some interesting points. Thus, the dithiooxalate has the highest ligand field strength of all geminal and vicinal ligands (Latham, Hascall & Gray, 1965). The presence of four donor atoms in the dithiooxalate dianion and the possibilities of charge delocalization on any two of these atoms result in a multifunctional ligand with unique coordination properties (Coucouvani, 1970). X-ray structure determinations demonstrate the ability of the dithiooxalate ligand to coordinate to the central metal ion by two S or two O donor atoms, respectively; furthermore, dithiooxalate may coordinate simultaneously to more than one metal ion in polynuclear complexes (Coucouvani, Baenzinger & Johnson, 1973; Gleizes & Verdager, 1984; Frasse, 1985). In this paper we describe the X-ray structure of  $[\text{As}(\text{C}_6\text{H}_5)_4]_2[\text{Pd}(\text{C}_2\text{S}_2\text{O}_2)_2]$ .

**Experimental.** Tetraphenylarsonium bis(dithiooxalato)palladate(II) was obtained by mixing aqueous solutions of potassium bis(dithiooxalato)palladate(II) and tetraphenylarsonium chloride. Yellow prismatic crystals were grown by slow evaporation of a dichloromethane solution of the complex. A crystal with approximate dimensions  $0.10 \times 0.12 \times 0.20$  mm was analyzed at 296 K with an Enraf-Nonius CAD-4 diffractometer, utilizing graphite-monochromated Mo  $K\alpha$  radiation.

Unit-cell parameters were obtained from a least-squares fitting of the setting angles for 25 reflections with  $19 \leq 2\theta \leq 21^\circ$ . Reflections were scanned in the  $\omega/2\theta$  mode,  $2\theta_{\text{max}} = 50^\circ$ , reflections with indices  $h \leq 12$ ,  $k \leq 15$ ,  $l \leq 21$  collected. During the data collection, two reference reflections ( $\bar{1}48$ ,  $\bar{1}17$ ) were monitored every 100 reflections. No crystal decay was observed. Data were corrected for Lorentz-polarization. 8383 unique reflections ( $R_{\text{int}} = 0.018$ ), 5034 with  $I > 3\sigma(I)$  were used in the refinement. The positions of Pd and As atoms were located on a Patterson map, the remaining non-H atoms of the structure being located on successive Fourier syntheses. An empirical absorption ( $\mu = 19.317 \text{ cm}^{-1}$ ) correction following the *DIFABS* procedure (Walker & Stuart, 1983) was applied to isotropically refined data. The structure was then refined anisotropically. H atoms were located from difference Fourier syntheses and were isotropically refined. A weighting scheme of type  $w = w_1 w_2$  with  $w_1 = k_1/(a + b|F_o| + c|F_o|^2)$  and  $w_2 = k_2/(d + e \sin\theta/\lambda + f \sin^2\theta/\lambda^2)$  was used to obtain flat dependence in  $\langle w\Delta^2 F \rangle$  vs  $\langle F_o \rangle$  and vs  $\langle \sin\theta/\lambda \rangle$  (Martínez-Ripoll & Cano, 1975); the coefficients used are  $k_1 = 0.607$ ,  $k_2 = 1.157$ ,  $a = 2.989$ ,  $b = 0.066$ ,  $c = 0.000$  for  $|F_o| \leq 11.63$ ;  $a = 6.790$ ,  $b = -0.330$ ,  $c = 0.005$  for  $11.63 < |F_o| \leq 41.43$ ;  $a = 1.391$ ,  $b = -0.003$ ,  $c = 0.000$  for  $|F_o| > 41.43$ ;  $d =$

Table 1. Fractional atomic coordinates and equivalent isotropic temperature parameters ( $\text{\AA}^2$ ) with *e.s.d.*'s in parentheses

$$U_{\text{eq}} = \frac{1}{3} \sum [U_{ij} a_i^* a_j^* a_i a_j \cos(\mathbf{a}_i, \mathbf{a}_j)].$$

	x	y	z	$U_{\text{eq}}$
Pd	0.03440 (5)	0.15638 (4)	-0.24119 (3)	0.042 (3)
As(1)	0.36891 (6)	0.23569 (5)	-0.04459 (4)	0.039 (3)
As(2)	0.75978 (6)	0.32053 (5)	-0.44146 (4)	0.038 (3)
S(1)	-0.05268 (21)	-0.02985 (16)	-0.27477 (14)	0.071 (1)
S(2)	-0.14512 (18)	0.18860 (17)	-0.15597 (13)	0.067 (9)
S(3)	0.11921 (18)	0.34255 (15)	-0.21571 (11)	0.058 (8)
S(4)	0.21213 (20)	0.12178 (16)	-0.32699 (13)	0.067 (9)
O(1)	-0.2611 (6)	-0.1329 (6)	-0.2148 (5)	0.106 (4)
O(2)	-0.3463 (5)	0.0471 (5)	-0.1224 (4)	0.078 (3)
O(3)	0.2903 (6)	0.4358 (5)	-0.3057 (4)	0.079 (3)
O(4)	0.3814 (7)	0.2557 (5)	-0.3883 (4)	0.105 (4)
C(1)	-0.1927 (7)	-0.0429 (7)	-0.2144 (5)	0.061 (4)
C(2)	-0.2414 (7)	0.0593 (7)	-0.1596 (4)	0.056 (4)
C(3)	0.2384 (7)	0.3492 (6)	-0.2916 (4)	0.052 (3)
C(4)	0.2863 (8)	0.2458 (7)	-0.3405 (4)	0.065 (4)
C(111)	0.2423 (6)	0.3205 (5)	0.0006 (4)	0.041 (3)
C(112)	0.2765 (6)	0.4255 (6)	0.0528 (4)	0.051 (3)
C(113)	0.1877 (8)	0.4912 (6)	0.0834 (4)	0.055 (3)
C(114)	0.0679 (7)	0.4528 (7)	0.0613 (4)	0.053 (3)
C(115)	0.0368 (7)	0.3500 (8)	0.0096 (5)	0.066 (4)
C(116)	0.1214 (6)	0.2817 (6)	-0.0208 (4)	0.054 (3)
C(121)	0.4574 (6)	0.2776 (5)	-0.1339 (4)	0.042 (3)
C(122)	0.4380 (6)	0.3662 (6)	-0.1478 (4)	0.049 (3)
C(123)	0.5068 (8)	0.3974 (6)	-0.2107 (5)	0.057 (3)
C(124)	0.5946 (8)	0.3409 (7)	-0.2597 (5)	0.062 (4)
C(125)	0.6139 (9)	0.2520 (8)	-0.2450 (5)	0.086 (5)
C(126)	0.5448 (8)	0.2195 (7)	-0.1827 (5)	0.070 (4)
C(131)	0.4805 (6)	0.2774 (5)	0.0327 (4)	0.039 (3)
C(132)	0.4335 (7)	0.2626 (6)	0.1042 (4)	0.055 (3)
C(133)	0.5115 (10)	0.2974 (7)	0.1626 (5)	0.072 (4)
C(134)	0.6359 (9)	0.3536 (7)	0.1485 (6)	0.072 (4)
C(135)	0.6808 (7)	0.3667 (7)	0.0782 (6)	0.066 (4)
C(136)	0.6051 (6)	0.3302 (6)	0.0187 (5)	0.055 (3)
C(141)	0.2949 (6)	0.0822 (5)	-0.0743 (4)	0.042 (3)
C(142)	0.2195 (8)	0.0357 (6)	-0.1354 (4)	0.059 (3)
C(143)	0.1601 (9)	-0.0742 (7)	-0.1529 (5)	0.074 (4)
C(114)	0.1741 (10)	-0.1371 (6)	-0.1122 (6)	0.083 (4)
C(145)	0.2499 (11)	-0.0928 (7)	-0.0534 (6)	0.088 (5)
C(146)	0.3089 (9)	0.0177 (6)	-0.0330 (5)	0.069 (4)
C(211)	0.8918 (6)	0.3479 (5)	0.3613 (4)	0.042 (3)
C(212)	0.8771 (7)	0.4052 (6)	0.3145 (4)	0.052 (3)
C(213)	0.9716 (8)	0.4230 (6)	0.2559 (4)	0.061 (4)
C(214)	1.0799 (9)	0.3844 (7)	0.2442 (5)	0.071 (4)
C(215)	1.0951 (8)	0.3282 (7)	0.2899 (5)	0.070 (4)
C(216)	0.9996 (7)	0.3096 (6)	0.3484 (4)	0.052 (3)
C(221)	0.8243 (6)	0.2639 (5)	0.5043 (4)	0.040 (3)
C(222)	0.8682 (7)	0.3321 (6)	0.5736 (4)	0.050 (3)
C(223)	0.9185 (8)	0.2877 (7)	0.6151 (4)	0.063 (4)
C(224)	0.9251 (8)	0.1789 (7)	0.5874 (5)	0.068 (4)
C(225)	0.8814 (9)	0.1122 (7)	0.5185 (5)	0.068 (4)
C(226)	0.8274 (7)	0.1535 (6)	0.4760 (4)	0.056 (3)
C(231)	0.6209 (6)	0.2099 (5)	0.3964 (4)	0.045 (3)
C(232)	0.5284 (8)	0.1597 (6)	0.4441 (5)	0.062 (4)
C(233)	0.4265 (9)	0.0812 (7)	0.4124 (6)	0.074 (4)
C(234)	0.4146 (9)	0.0520 (7)	0.3328 (6)	0.070 (4)
C(235)	0.5070 (8)	0.1007 (7)	0.2871 (6)	0.067 (4)
C(236)	0.6109 (7)	0.1797 (6)	0.3174 (4)	0.053 (3)
C(241)	0.7097 (6)	0.4556 (5)	0.5040 (4)	0.044 (3)
C(242)	0.5985 (7)	0.4513 (7)	0.5558 (4)	0.052 (3)
C(243)	0.5649 (8)	0.5491 (7)	0.6025 (5)	0.064 (4)
C(244)	0.6380 (8)	0.6499 (7)	0.5961 (5)	0.065 (4)
C(245)	0.7435 (9)	0.6510 (7)	0.5427 (5)	0.069 (4)
C(246)	0.7809 (8)	0.5555 (6)	0.4964 (5)	0.057 (3)

$6.859$ ,  $e = -34.435$ ,  $f = 48.541$  for  $\sin\theta/\lambda \leq 0.42$ ,  $d = 21.875$ ,  $e = -87.906$ ,  $f = 93.344$  for  $0.42 < \sin\theta/\lambda \leq 0.54$ ;  $d = 1.654$ ,  $e = -1.084$ ,  $f = 0.000$  for  $\sin\theta/\lambda > 0.54$ . Final refinement gave the discrepancy indices  $R = 0.041$ ,  $wR = 0.045$ . Max.  $\Delta/\sigma = 0.69$ ,  $\Delta\rho = 2.5 \text{ e \AA}^{-3}$  near Pd atom. Neutral-atom scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 72–98). Most calculations

Table 2. Main interatomic distances (Å) and bond angles (°) in [As(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>]<sub>2</sub>[Pd(C<sub>2</sub>S<sub>2</sub>O<sub>2</sub>)<sub>2</sub>]

## Bis(dithiooxalato)palladate(II) anion

Pd—S(1)	2.298 (3)	S(4)—C(4)	1.707 (9)
Pd—S(2)	2.302 (3)	C(1)—O(1)	1.22 (1)
Pd—S(3)	2.302 (3)	C(2)—O(2)	1.220 (9)
Pd—S(4)	2.298 (3)	C(3)—O(3)	1.23 (1)
S(1)—C(1)	1.715 (8)	C(4)—O(4)	1.23 (1)
S(2)—C(2)	1.740 (8)	C(1)—C(2)	1.54 (1)
S(3)—C(3)	1.721 (7)	C(3)—C(4)	1.52 (1)

S(1)—Pd—S(2)	89.5 (3)	S(2)—C(2)—O(2)	122.7 (8)
S(1)—Pd—S(3)	176.1 (2)	S(3)—C(3)—O(3)	122.3 (7)
S(1)—Pd—S(4)	89.7 (2)	S(4)—C(4)—O(4)	121.8 (8)
S(2)—Pd—S(3)	91.6 (2)	S(1)—C(1)—C(2)	121.0 (8)
S(2)—Pd—S(4)	179.2 (3)	S(2)—C(2)—C(1)	117.9 (7)
S(3)—Pd—S(4)	89.1 (2)	S(3)—C(3)—C(4)	119.1 (7)
Pd—S(1)—C(1)	105.3 (4)	S(4)—C(4)—C(3)	120.4 (7)
Pd—S(2)—C(2)	106.0 (4)	O(1)—C(1)—C(2)	116.7 (9)
Pd—S(3)—C(3)	103.7 (4)	O(2)—C(2)—C(1)	119.4 (9)
Pd—S(4)—C(4)	104.2 (4)	O(3)—C(3)—C(4)	118.5 (8)
S(1)—C(1)—O(1)	122.2 (8)	O(4)—C(4)—C(3)	117.7 (9)

## Tetraphenylarsonium cations

	X = 1	X = 2		X = 1	X = 2
As(X)—C(X11)	1.915 (7)	1.909 (7)	As(X)—C(X31)	1.920 (7)	1.908 (6)
As(X)—C(X21)	1.914 (7)	1.927 (8)	As(X)—C(X41)	1.898 (6)	1.919 (7)

## Hydrogen contacts

C—H...O	C—H	H...O	C...O	∠C—H...O	
C(126)—H(126)...O(2)	i	0.83 (8)	2.64 (8)	3.45 (1)	169 (6)
C(132)—H(132)...O(1)	ii	0.88 (6)	2.53 (7)	3.14 (1)	127 (6)
C(133)—H(133)...O(1)	ii	0.99 (7)	2.56 (6)	3.23 (1)	124 (5)
C(212)—H(212)...O(3)	iii	0.99 (7)	2.34 (8)	3.25 (1)	152 (5)
C(215)—H(215)...O(1)	iv	0.9 (1)	2.5 (1)	3.33 (1)	142 (8)
C(232)—H(232)...O(4)	v	1.06 (8)	2.48 (7)	3.18 (1)	122 (5)
C(242)—H(242)...O(4)	v	0.9 (1)	2.52 (9)	3.32 (1)	142 (8)
C(243)—H(243)...O(3)	v	0.95 (9)	2.54 (9)	3.39 (1)	149 (8)

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

were carried using the XRAY76 System (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) running on a MicroVAX II computer.

**Discussion.** Atomic coordinates and equivalent isotropic temperature factors of non-H atoms are given in Table 1.\* Bond lengths and angles are listed in Table 2. The atomic arrangement comprises one non-centrosymmetric [Pd(C<sub>2</sub>S<sub>2</sub>O<sub>2</sub>)<sub>2</sub>]<sup>2-</sup> anion and two crystallographically independent [As(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>]<sup>+</sup> cations. Fig. 1 shows the atomic numbering scheme used for the bis(dithiooxalato)palladate(II) anion. A unit-cell diagram is presented in Fig. 2.

The Pd atom is coordinated to two dithiooxalate ligands through the S atoms located at the corners of a quasi-square parallelogram. The angles between the plane determined by Pd and S atoms and the dithiooxalate ligand planes are 1.8 and 17.5°; this last value indicates that the anion deviates significantly from planarity. This deviation is due to the hydrogen contacts of type C—H...O (Taylor & Kennard, 1982)

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53311 (61 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

between three rings attached to As(2) and the O atoms O(3) and O(4); the contacts are: C(212)...O(3), C(232)...O(4), C(242)...O(4), and C(243)...O(3) (Table 2). So C(212) and C(232) atoms are placed at 2.92 and 1.61 Å below the mean plane defined by the Pd, S(1), S(2), S(3), S(4), C(1), C(2), C(3), C(4), O(1) and O(2) anion atoms, respectively. Meanwhile, O(3) and O(4) are in the same plane defined by the ring containing atoms C(242) and C(243). The study of an indium dithiooxalate complex with tetraphenylarsonium as cation also shows a strong distortion in the dithiooxalate ligands (Golic, Bulc & Dietzsch, 1982).

We have observed in related salts of nickel dithiooxalate with organic planar cations (Enjalbert, Gleizes, Trombe, Gutiérrez-Campo & Román, 1985) that packing of anions and cations follows the sequences *acac* or *acca* (where *a* = anion, *c* = cation). In the present compound anions and cations are disposed in a different way due to the non-planarity and high volume of these cations.

The generous support given by Iberduero, SA is gratefully acknowledged.

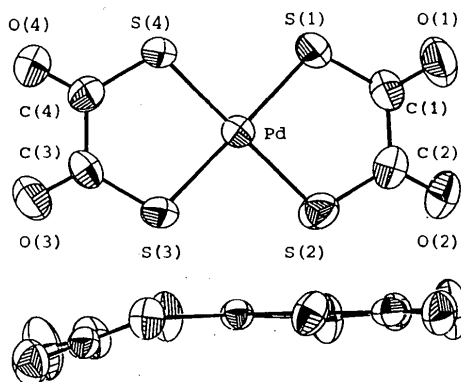


Fig. 1. Front and side views of the bis(dithiooxalato)palladate(II) anion with atom labelling.

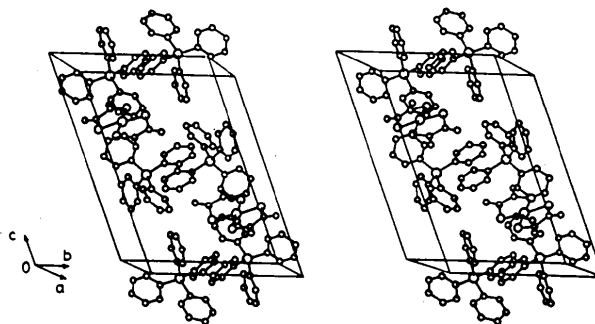


Fig. 2. Stereoview packing diagram for [As(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>]<sub>2</sub>[Pd(C<sub>2</sub>S<sub>2</sub>O<sub>2</sub>)<sub>2</sub>].

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## Structure of Bis(tetraethylammonium) Hexaiododimercurate(II) Diiodomercury(II)

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**Abstract.**  $[\text{N}(\text{C}_2\text{H}_5)_4]_2[\text{Hg}_2\text{I}_6] \cdot \text{HgI}_2$ ,  $M_r = 1781.53$ , tetragonal,  $P4_2/m$ ,  $a = 10.391(1)$ ,  $c = 17.051(1)$  Å,  $V = 1841.4$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 3.386$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 195.23$  cm<sup>-1</sup>,  $F(000) = 1628$ ,  $T = 293$  K,  $R = 0.0468$  for 1201 observed reflections. The structure consists of two crystallographically independent tetraethylammonium cations, a linear centrosymmetric  $\text{HgI}_2$  molecule [ $\text{Hg—I}$  bond length is  $2.574(1)$  Å] and a dimeric anion  $[\text{Hg}_2\text{I}_6]^{2-}$ . There are two van der Waals contacts between the Hg atom of  $\text{HgI}_2$  and two iodines from  $[\text{Hg}_2\text{I}_6]^{2-}$  [ $3.547(2)$  Å] and two I—I van der Waals contacts [ $3.769(2)$  Å] between iodines of  $\text{HgI}_2$  and  $[\text{Hg}_2\text{I}_6]^{2-}$ . The latter are considerably shorter than the van der Waals I...I distance ( $4.3$  Å).

**Introduction.** Previous studies have shown that the characteristic coordination of mercury by I atoms (Grdenić, 1965), which is in most cases distorted, is digonal, (rarely) trigonal and tetrahedral. In addition, it has often been observed that Hg atoms tend to be surrounded by further more-distant iodines. These iodines together with those involved in the characteristic coordination usually surround Hg in the form of a distorted regular object, such as an octahedron or a trigonal bipyramid. Structures

which contain Hg which is tetrahedrally coordinated by iodine may be separated into three groups: (a) with isolated molecules  $\text{HgI}_4^-$ , (b) with dimeric molecules  $\text{Hg}_2\text{I}_6^{2-}$  which consist of two edge-shared coordination tetrahedra and (c) Hg—I polymers with corner-shared tetrahedra. The aim of the present work was to establish the structure of the title compound and to compare it with related ones.

**Experimental.** The title compound was one of the products of the reaction of tetraethylammonium iodide with mercury(II) iodide in petroleum ether. Yellow crystals were prepared by subsequent recrystallization from propanone.

Sphere-ground crystal ( $R = 0.162$  mm),  $\mu R = 3.173$ , Enraf–Nonius CAD-4F diffractometer, 50 kV/30 mA, graphite-monochromated Mo  $K\alpha$ . Lattice parameters from least-squares refinement of 25 reflections ( $15.30 < \theta < 17.30^\circ$ ).  $\omega/2\theta$  scan mode,  $\omega = (1.30 + 1.05 \tan \theta)^\circ$ , vertical aperture 4 mm. The intensity-standard reflection (068) was monitored ever 1800 s; intensity variation ( $\pm 5\%$ ) during the data collection. The prescan speed was  $10^\circ \text{ min}^{-1}$ . The final scan was not applied for reflections where  $\sigma(I)/I$ , determined after the prescan, lay outside the interval (0.05, 1). The maximum final-scan measurement time was 30 s.

3057 reflections were measured up to  $\theta = 30^\circ$  ( $0 \leq h \leq 14$ ,  $0 \leq k \leq 14$ ,  $0 \leq l \leq 23$ ); *i.e.* 2776 unique

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