C(31A)	0.6267 (3)	0.6144 (2)	0.2100 (2)	0.044 (1)
O(30A)	0.6866 (2)	0.7353 (2)	0.2522 (2)	0.062 (1)
O(31A)	0.4650 (2)	0.5708 (2)	0.1955 (2)	0.068 (1)
C(1B)	0.1412 (2)	0.2520 (2)	0.4240 (2)	0.035 (1)
C(2B)	0.1530 (3)	0.1295 (2)	0.3481 (2)	0.036 (1)
C(3B)	0.0036 (2)	0.0338 (2)	0.3145 (2)	0.035 (1)
N(3B)	0.0135 (3)	-0.0907 (2)	0.2275 (2)	0.042 (1)
C(4B)	-0.1549 (3)	0.0553 (2)	0.3562 (2)	0.041 (1)
C(5B)	-0.1649 (3)	0.1765 (2)	0.4338 (2)	0.045 (1)
C(6B)	-0.0171 (3)	0.2753 (2)	0.4669 (2)	0.041 (1)
O(10B)	0.2840 (2)	0.4812 (1)	0.5022 (2)	0.050 (1)
O(11B)	0.4443 (2)	0.3223 (2)	0.4372 (2)	0.054 (1)
C(11B)	0.2992 (3)	0.3597 (2)	0.4575 (2)	0.038 (1)
OW(1)	0.1904 (2)	0.0097 (2)	0.9851 (2)	0.050 (1)
OW(2)	0.3627 (3)	0.8709 (3)	0.2380 (3)	0.082 (1)

Table 4. Selected geometric parameters (Å, °) for (2)

N(1A) - C(6A)	1.323 (3)	C(31A)O(31A)	1.272 (3)
N(1A) - C(2A)	1.342 (2)	C(1B) - C(6B)	1.385 (3)
$C(2A) \rightarrow C(3A)$	1.403 (3)	C(1B) - C(2B)	1.392 (3)
$C(2A) \rightarrow C(21A)$	1.533 (3)	C(1B)—C(11B)	1.487 (3)
C(3A) - N(4A)	1.347 (2)	C(2B) - C(3B)	1.380 (3)
C(3A) - C(31A)	1.529 (3)	C(3B)C(4B)	1.377 (3)
N(4A) - C(5A)	1.323 (3)	C(3B)—N(3B)	1.466 (2)
$C(5A) \longrightarrow C(6A)$	1.381 (3)	C(4B)—C(5B)	1.387 (3)
C(21A)—O(21A)	1.204 (3)	C(5B)—C(6B)	1.385 (3)
C(21A)—O(20A)	1.267 (3)	O(10B)—C(11B)	1.252 (2)
C(31A)—O(30A)	1.219 (3)	O(11B) - C(11B)	1.277 (2)
C(6A)— $N(1A)$ — $C(2A)$	118.8 (2)	O(31A)—C(31A)—C(3A)	119.4 (2)
N(1A) - C(2A) - C(3A)	120.1 (2)	C(6B) - C(1B) - C(2B)	120.4 (2)
N(1A) - C(2A) - C(21A)	111.4 (2)	C(6B) - C(1B) - C(11B)	119.9 (2)
C(3A) - C(2A) - C(21A)	128.5 (2)	C(2B) - C(1B) - C(11B)	119.7 (2)
N(4A)— $C(3A)$ — $C(2A)$	120.2 (2)	C(3B)— $C(2B)$ — $C(1B)$	118.5 (2)
N(4A) - C(3A) - C(31A)	111.1 (2)	C(4B)— $C(3B)$ — $C(2B)$	121.9 (2)
C(2A) - C(3A) - C(31A)	128.7 (2)	C(4B)— $C(3B)$ — $N(3B)$	119.2 (2)
C(5A)— $N(4A)$ — $C(3A)$	118.2 (2)	C(2B)— $C(3B)$ — $N(3B)$	118.9 (2)
N(4A) - C(5A) - C(6A)	121.6 (2)	C(3B)— $C(4B)$ — $C(5B)$	119.2 (2)
N(1A) - C(6A) - C(5A)	120.9 (2)	$C(6B) \rightarrow C(5B) \rightarrow C(4B)$	120.1 (2)
O(21A)-C(21A)-O(20A) 122.2 (2)	C(1B) - C(6B) - C(5B)	120.0 (2)
O(21A)— $C(21A)$ — $C(2A)$	117.9 (2)	O(10B)—C(11B)—O(11B) 123.6 (2)
O(20A)— $C(21A)$ — $C(2A)$	119.9 (2)	O(10B)— $C(11B)$ — $C(1B)$	119.1 (2)
O(30A)-C(31A)-O(31A) 121.7 (2)	O(11B)— $C(11B)$ — $C(1B)$	117.3 (2)
O(30A) - C(31A) - C(3A)	118.9 (2)		

Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods and refined by fullmatrix least squares with anisotropic displacement parameters for all non-H atoms. H-atom positions were located by difference methods and included in the respective refinements with both positional and isotropic displacement parameters refined.

For both compounds, data collection: *MolEN* (Fair, 1990); cell refinement: *MolEN*; data reduction: *Xtal3.2* (Hall, Flack & Stewart, 1992); program(s) used to solve structures: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *PLA-TON92* (Spek, 1990).

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References

- Boone, C. D. G., Derissen, J. L & Schoone, J. C. (1977). Acta Cryst. B33, 3205–3206.
- Brown, C. J. (1968). Proc. R. Soc. A, 302, 185-199.
- Etter, M. C. (1990). Acc. Chem. Res. 23, 120-126.
- Etter, M. C. & Adsmond, D. A. (1990). J. Chem. Soc. Chem. Commun. pp. 589-591.
- Fair, C. K. (1990). MolEN. An Interactive Intelligent System for Crystal Structure Analysis. Enraf-Nonius, Delft, The Netherlands.
- Hall, S. R., Flack, H. D. & Stewart, S. J. (1992). *Xtal3.2 Reference Manual*. Univs. of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Hardy, G. E., Kaska, W. C., Chandra, B. P. & Zink, J. I. (1981). J. Am. Chem. Soc. 103, 1074–1079.
- Khan, M. Y. & Srivastava, P. (1968). Ind. J. Pure Appl. Phys. 6, 166-170.
- Leiserowitz, L. (1976). Acta Cryst. B32, 775-802.
- Lynch, D. E., Smith, G., Byriel, K. A., Kennard, C. H. L., Whittaker, A. K. (1994). Aust. J. Chem. 47, 309-319.
- Sheldrick, G. M. (1985). SHELXS86. Program for the Solution of Crystal Structures. Univ. of Göttingen, Germany.
- Sheldrick, G. M. (1993). SHELXL93. Program for the Refinement of Crystal Structures. Univ. of Göttingen, Germany.
- Smith, G., Gentner, J. M., Lynch, D. E., Byriel, K. A. & Kennard, C. H. L. (1995). Aust. J. Chem. 48, 1151–1161.
- Spek, A. L. (1990). Acta Cryst. A46, C-34.
- Takusagawa, F. & Shimada, A. (1973). Chem. Lett. pp. 1121-1122.
- Voogd, J., Verzijl, B. H. M. & Duisenberg, A. J. M. (1980). Acta Cryst. B36, 2805-2806.

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Novel Six- and Eight-Membered Heterocycles. A Trithiadiazaphosphorinane and a Pentathiadiazaphosphocine

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Abstract

We report the single-crystal X-ray structure determinations of two novel phosphorus-, nitrogen- and sulfurcontaining heterocycles, namely, 4,6-bis[2-(2-methyl)propyl]-5-oxo-5-phenyl-1,2,3,4,6,5-trithiadiazaphosphorinane, $C_{14}H_{23}N_2OPS_3$, and 6,8-bis[2-(2-methyl)propyl]-7-oxo-7-phenyl-1,2,3,4,5,6,8,7-pentathiadiazaphosphocine, $C_{14}H_{23}N_2OPS_5$. The phosphorinane compound is

Lists of structure factors, anisotropic displacement parameters, Hatom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1032). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

unusual in that it displays a high degree of steric compression in the S—S—S bond angle, which has a value of only 96.3°. The phosphocine compound has bond angles similar to those found in S₈ rings. One S—S— S—S torsion angle, however, is compressed to 30°. The phosphocine has two conformationally identical molecules in the asymmetric unit.

Comment

There has recently been some interest in sulfurcontaining cyclic systems, both for their biological activity (Behar & Danishefsky, 1993) and as precursors for diatomic sulfur (Salama, Steliou & Yu, 1992). We have been involved in attempts at generating new forms of sulfurated heterocycles of interest in these and other areas (Bottle et al., 1993). We now report the singlecrystal X-ray structure determinations of two unusual phosphorus-, nitrogen- and sulfur-containing heterocycles, one six-membered, 4,6-bis[2-(2-methyl)propyl]-5oxo-5-phenyl-1,2,3,4,6,5-trithiadiazaphosphorinane, (1), the other eight-membered, 6,8-bis[2-(2-methyl)propyl]-7-oxo-7-phenyl-1,2,3,4,5,6,8,7-pentathiadiazaphosphocine, (2). These unusual compounds were isolated as minor by-products of the reaction of sulfur monochloride with di-tert-butylphenylphosphonic diamide. Both compounds (1) and (2) were present as equilibrium components of a complex mixture of other phosphorus-, nitrogen- and sulfur-containing heterocycles, as well as elemental sulfur. The major component of the mixture was a seven-membered tetrathia heterocycle, (3), the structure of which has been published recently (Bottle et al., 1993).



The molecular of (1) (Fig. 1) displays a remarkably symmetrical chair arrangement with the bulky tertbutyl groups and phosphonic O atom occupying pseudoequatorial positions. This conformation allows the best extension of π bonding through the O—P—N—S system, which is supported by the torsion angles between these atoms $[175.1(1) \text{ and } -175.1(1)^{\circ}]$. In this regard, compound (1) differs from the seven-memberedring analogue (3) (Bottle et al., 1993) which has angles of -107.5(2) and $159.3(1)^{\circ}$. With respect to the bond lengths and the planarity around the N atom, however, the six-membered ring of compound (1) displays similar structural characteristics to the seven-membered analogue of (3). The geometry about each N atom is quite planar, indicating much sp^2 character arising from significant $d\pi - p\pi$ overlap with the P atom. The S—

N and P-N bond lengths in (1) are all within 1% of those in the analogous seven-membered system. There is minor variation of the S-S bond lengths, which, at 2.058(1) and 2.065(1)Å in (1), are intermediate between the values of 2.043(1) and 2.071(1) Å found for compound (3). The most significant difference relates to the S-S-S bond angle, which has been compressed substantially to $96.3 (4)^{\circ}$ from the usual value of 108° in S₈ (Abrahams, 1961) and of over 100° in the seven-membered-ring tetrasulfide (Bottle et al., 1993). In addition, the S—S—S—N torsion angles [66.3(1) and $-66.4(1)^{\circ}$ are small for such sulfur rings, e.g. in S_8 they are close to 100° (Abrahams, 1961) and in S_6 74.5° (Caron, Donohue & Goldfish, 1961). This compression would give rise to an increase of the overall strain in the molecule, resulting in compound (1) being less favoured thermodynamically.



Fig. 1. Molecular configuration and atom-numbering scheme for compound (1) shown with 20% probability displacement ellipsoids.

For the eight-membered heterocyclic compound (2) (Fig. 2), there are two independent but conformationally identical molecules in the asymmetric unit. These two molecules are not related by any crystallographic symmetry. The geometries about the N atoms in these molecules are again quite planar, indicating similar $d\pi$ $p\pi$ overlap with the P atom. As expected, the N— S bond lengths (average 1.695 Å) are similar to those of related systems in which the N atom is sp^2 hybridized (Engelsein, Kalker, Lex & Linke, 1974). The S—S—S bond angles have an average value of 105.4° and are quite normal for S₈ rings (Abrahams, 1961). One of the S—S—S—S torsion angles [96.3 (2) (molecule 1) and $-96.1(2)^{\circ}$ (molecule 2)] is also as expected for eight-membered sulfur rings. The second S—S—S—S torsion angle, however, is unusually small $[-32.9(2) \text{ (molecule 1) and } -30.9(2)^{\circ} \text{ (molecule 2)]}.$

This compression increases the overall strain in the molecule and explains why compound (2) is a minor component in the reaction mixture.



Fig. 2. Molecular configuration and atom-numbering scheme for compound (2) shown with 20% probability displacement ellipsoids.

Experimental

The phosphorinane (1) and the phosphocine (2) were prepared by the action of sulfur monochloride (S₂Cl₂, 220 mg, 1.6 mmol) on di-tert-butylphenylphosphonic diamide (200 mg, 0.75 mmol), with pyridine (5 ml) being used as both solvent and base. Upon addition of the S₂Cl₂, a creamy precipitate of pyridinium hydrochloride formed (confirmed by NMR). The solvent was removed under high vacuum and worked up via standard extraction procedures. Flash chromatography (EtOAc, CHCl₃ and 60-80 hexanes, 1:1:2) was used to separate the products from elemental sulfur and other by-products. The compounds were further purified by reversed-phase MPLC (medium pressure liquid chromatography) using a Büchi system. This gave phosphorinane (1) (17 mg; 6.3% yield) as a white powder, which was recrystallized from MeOH to give needles (m.p. 435-436 K), and phosphocine (2) (26 mg; 8.2% yield) as a yellow/white powder, which on recrystallization from acetonitrile gave prisms (m.p. 400-402 K). Both original specimens used for X-ray analysis were cleaved from larger crystals.

Compound (1)

Crystal	data
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C14H23N2OPS3	Mo $K\alpha$ radiation
$M_r = 362.5$	$\lambda = 0.71073 \text{ Å}$
Monoclinic	Cell parameters from 25
$P2_1/a$	reflections
a = 14.002 (3) Å	$\theta = 6-14^{\circ}$
b = 9.0989 (6) Å	$\mu = 0.503 \text{ mm}^{-1}$
c = 15.413(4) Å	T = 298 (2) K
$\beta = 114.24(1)^{\circ}$	Prismatic
V = 1790.6 (6) Å ³	$0.35 \times 0.25 \times 0.12 \text{ mm}$
Z = 4	Colourless
$D_x = 1.345 \text{ Mg m}^{-3}$	

Data collection

Enraf–Nonius CAD-4 diffractometer $\omega/2\theta$ scans Absorption correction: none 3290 measured reflections 3146 independent reflections 2557 observed reflections $[I > 2\sigma(I)]$	$R_{int} = 0.049$ $\theta_{max} = 25^{\circ}$ $h = 0 \rightarrow 16$ $k = 0 \rightarrow 10$ $l = -18 \rightarrow 16$ 3 standard reflections monitored every 250 reflections intensity decay: 0.5%
Refinement Refinement on F^2 R(F) = 0.0367	$w = 1/[\sigma^2(F_o^2) + (0.0738P)^2 + 0.99P]$
$wR(F^2) = 0.1133$ S = 0.959	where $P = [\max(F_o^2, 0) + 2F_c^2]/3$
3146 reflections 282 parameters All H-atom parameters refined	$(\Delta/\sigma)_{\text{max}} = 0.1$ $\Delta\rho_{\text{max}} = 0.44 \text{ e} \text{ Å}^{-3}$ $\Delta\rho_{\text{min}} = -0.36 \text{ e} \text{ Å}^{-3}$ Atomic scattering factors
	from International Tables for X-ray Crystallography (1974, Vol. IV)

Table	1.	Fractie	onal	atomic	coordinates	and	equivalent
i	soti	ropic d	ispla	icement	parameters (Ų),	for (1)

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

			-	
	x	у	Ζ	U_{eq}
P(1)	0.7165(1)	0.0632(1)	0.2044(1)	0.035 (1)
O(1)	0.6275(1)	0.1626 (2)	0.1612(1)	0.048 (1)
N(2)	0.8042 (2)	0.1261 (2)	0.3106(1)	0.043 (1)
C(2)	0.7918 (2)	0.2477 (3)	0.3739 (2)	0.056(1)
C(21)	0.6919 (4)	0.3360 (5)	0.3241 (4)	0.096 (2)
C(22)	0.8870 (4)	0.3476 (6)	0.4000 (4)	0.097 (1)
C(23)	0.7926 (5)	0.1777 (6)	0.4644 (3)	0.094 (1)
S(3)	0.9144 (1)	0.0244 (1)	0.3608(1)	0.052(1)
S(4)	0.8605(1)	-0.1700(1)	0.3949(1)	0.065(1)
S(5)	0.7717(1)	-0.2353 (1)	0.2566 (1)	0.054 (1)
N(6)	0.6755 (1)	-0.1082 (2)	0.2159(1)	0.041(1)
C(6)	0.5663 (2)	-0.1431 (3)	0.2101 (2)	0.045 (1)
C(61)	0.5594 (3)	-0.3035 (4)	0.2342 (3)	0.065 (1)
C(62)	0.4885 (3)	-0.1162 (6)	0.1077 (3)	0.081 (1)
C(63)	0.5443 (4)	-0.0490 (5)	0.2813 (4)	0.084 (1)
C(11)	0.7895 (2)	0.0403 (3)	0.1324 (2)	0.036(1)
C(12)	0.8712 (2)	0.1338 (3)	0.1413 (2)	0.043 (1)
C(13)	0.9140 (2)	0.1312 (3)	0.0746 (2)	0.051 (1)
C(14)	0.8756 (2)	0.0356 (4)	-0.0008 (2)	0.055 (1)
C(15)	0.7950(2)	-0.0579 (4)	-0.0101(2)	0.058 (1)
C(16)	0.7521 (2)	-0.0561 (3)	0.0560 (2)	0.049 (1)

Table 2. Selected geometric parameters (Å, °) for (1)

P(1)O(1)	1.461 (2)	S(5)—N(6)	1.689 (2)
P(1)N(2)	1.693 (2)	N(6)—C(6)	1.529 (3)
P(1)—N(6)	1.696 (2)	C(6)—C(61)	1.519 (4)
P(1)—C(11)	1.803 (2)	C(6)—C(63)	1.520 (4)
N(2)C(2)	1.530 (3)	C(6)C(62)	1.524 (4)
N(2)—S(3)	1.700 (2)	C(11)—C(16)	1.387 (4)
C(2)—C(21)	1.520 (5)	C(11)—C(12)	1.387 (3)
C(2)—C(22)	1.524 (5)	C(12)—C(13)	1.386 (4)
C(2)—C(23)	1.530 (5)	C(13)—C(14)	1.373 (4)
S(3)—S(4)	2.0584 (11)	C(14)—C(15)	1.373 (4)
S(4)—S(5)	2.0651 (13)	C(15)—C(16)	1.380 (4)
O(1) - P(1) - N(2)	112.07 (10)	C(6)—N(6)—S(5)	120.4 (2)
O(1)—P(1)—N(6)	110.76 (10)	C(6) - N(6) - P(1)	123.9 (2)
N(2) - P(1) - N(6)	110.61 (10)	S(5)—N(6)—P(1)	114.56 (11)
O(1)-P(1)-C(11)	112.60 (10)	C(61)—C(6)—C(63)	108.2 (3)
N(2)—P(1)—C(11)	105.42 (10)	C(61)—C(6)—C(62)	108.5 (3)
N(6) - P(1) - C(11)	105.05 (10)	C(63)—C(6)—C(62)	112.8 (3)

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C14H23N2OPS3 AND C14H23N2OPS5

C(2) = N(2) = P(1)	129.4 (2)	C(61) - C(6) - N(6)	110.3 (2)	N(81)	0.6360 (3)	0.3204 (2	2) 0.0957 (2)	0.043 (1)
C(2) - N(2) - S(3)	115.5 (2)	C(63)—C(6)—N(6)	109.6 (2)	C(111)	0.4981 (4)	0.3156 (3	3) 0.2481 (2)	0.040(1)
P(1)—N(2)—S(3)	114.49 (12)	C(62)—C(6)—N(6)	107.4 (2)	C(121)	0.4417 (5)	0.2292 (3	3) 0.2850 (3)	0.049 (1)
C(21)—C(2)—C(22)	110.0 (4)	C(16)—C(11)—C(12)	118.8 (2)	C(131)	0.4545 (5)	0.2315 (4	4) 0.3732 (3)	0.064 (1)
C(21)—C(2)—C(23)	110.0 (4)	C(16) - C(11) - P(1)	118.9 (2)	C(141)	0.5223 (6)	0.3216 (2	(3) (3)	0.075 (2)
C(22) - C(2) - C(23)	109.2 (4)	C(12) - C(11) - P(1)	121.4 (2)	C(151)	0.5789(6)	0.4075 (4	+) 0.3887 (3) 4) 0.2009 (3)	0.074(2)
C(21) - C(2) - N(2)	112.4 (3)	C(13) = C(12) = C(11)	120.4 (2)	C(101)	0.3680(3)	0.4039 (4	(3) 0.3008(3)	0.037(1)
C(22) = C(2) = N(2)	106.5 (3)	C(14) - C(13) - C(12) C(13) - C(15)	120.1 (3)	C(21)	0.1303(4)	0.0865 (4	0.0000(5)	0.079(2)
V(23) = V(2) = N(2) N(2) = S(3) = S(4)	108.0 (3)	C(13) = C(14) = C(15)	120.0(3)	C(221)	0.1531(7)	0.2081 (2	-0.0053(4)	0.088(2)
S(3) = S(4) = S(5)	96 31 (4)	C(14) = C(15) = C(10)	120.5(3)	C(231)	0.1576 (6)	0.2669 (6	5) 0.1481 (6)	0.099 (3)
N(6) = S(5) = S(4)	103 71 (8)	C(13) - C(10) - C(11)	120.5 (5)	C(81)	0.6857 (5)	0.3663 (3	3) 0.0135 (3)	0.054(1)
14(0) 5(5) 5(4)	105.71 (07			C(811)	0.7857 (8)	0.3143 (0	6) -0.0286 (5)	0.087 (2)
Compound (2)				C(821)	0.5577 (7)	0.3503 (0	6) -0.0496 (4)	0.076 (2)
Compound (2)				C(831)	0.7607 (8)	0.4774 (5) 0.0389 (5)	0.081 (2)
Crystal data				P(12)	0.9558 (1)	0.6981 (1) 0.3848 (1)	0.039 (1)
C U N ODS		Ma Ka radiation		O(12)	0.8170 (3)	0.6266 (2	2) 0.3960 (2)	0.064 (1)
C14H23IN2OF35		NO K α radiation		S(32)	1.1056 (1)	0.8947 (1) 0.4792 (1)	0.065 (1)
$M_r = 426.6$		$\lambda = 0./10/3$ A		S(42)	1.3206 (2)	0.9358 (1) 0.4820 (2)	0.125 (1)
Triclinic		Cell parameters from	om 25	S(52)	1.3707 (2)	0.9188 (2	2) 0.3525 (2)	0.149 (1)
$P\overline{1}$		reflections		S(62)	1.2109 (3)	0.9471 (2	2) 0.2874 (2)	0.147 (1)
a = 9.836(9) Å		$\theta = 6 - 14^{\circ}$		S(72)	1.0623 (2)	0.8081 (1) 0.2444 (1)	0.101 (1)
h = 14.01(1) Å		$u = 0.640 \text{ mm}^{-1}$		N(22)	1.0382 (3)	0.7698 (2) 0.4771 (2)	0.043 (1)
v = 14.01(1) A		$\mu = 0.049 \text{ mm}$		N(82)	0.9405 (4)	0.7791 (3) 0.3172 (2)	0.055(1)
c = 15.733 (8) A		I = 298 (2) K		C(112)	1.0762 (4)	0.6359 (3) 0.3390 (2)	0.042(1)
$\alpha = 96.89 (5)^{\circ}$		Prismatic		C(122)	1.0214 (5)	0.5596 (3) 0.2704 (3)	0.056(1)
$\beta = 91.95 (5)^{\circ}$		$0.30 \times 0.25 \times 0.1$	12 mm	C(132)	1.1085 (6)	0.5088 (4) 0.2324 (3)	0.066(1)
$\alpha = 107.70(4)^{\circ}$		Colourless		C(142)	1.2483 (6)	0.5319 (-	4) 0.2615 (3)	0.068 (1)
$\gamma = 107.70(4)$		Colouriess		C(152)	1.3018 (6)	0.6054 (-	4) 0.3306 (4)	0.072(2)
v = 2045 (3) A				C(162)	1.2108 (5)	0.0570 ($\begin{array}{ccc} 4) & 0.3097(3) \\ 5) & 0.5635(2) \\ \end{array}$	0.050(1)
Z = 4				C(22)	1.0185 (0)	0.7272 ((3) 0.5035(3)	0.074(2)
$D_x = 1.386 \text{ Mg m}^-$	3			C(212)	0.0208 (10)	0.0197 (0.007(0)	0.107(3)
				C(222)	0.9208(19) 0.8642(14)	0.7754 (10) 0.5004(7)	0.056(3)
Data collection				C(232)	1 1389 (11)	0.7234 (0.5504(7) 8) 0.6278(5)	0.030(2) 0.125(3)
				C(82)	0.8063 (6)	0.8121 ((3) = 0.3091(3)	0.076(2)
Enraf–Nonius CAL	9-4	$R_{\rm int} = 0.020$		C(812)	0.7421 (8)	0.8186 (0.3957(5)	0.089(2)
diffractometer		$\theta_{\rm max} = 25^{\circ}$		C(822)	0.8450 (12)	0.9149 (7) $0.2797(7)$	0.128 (3)
				-(0=-)			,	
$\omega/2\theta$ scans		$h = 0 \rightarrow 11$		C(832)	0.7006 (9)	0.7352 (7) 0.2434 (5)	0.110(3)
$\omega/2\theta$ scans	on.	$\begin{array}{l} h = 0 \rightarrow 11 \\ k = -16 \rightarrow 15 \end{array}$		C(832)	0.7006 (9)	0.7352 (7) 0.2434 (5)	0.110(3)
$\omega/2\theta$ scans Absorption correction	on:	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $k = -18 \rightarrow 18$		C(832)	0.7006 (9) 4. Selecteo	0.7352 (I geometric	7) 0.2434 (5) c parameters (Å, °)	0.110(3)
$\omega/2\theta$ scans Absorption correcting none	on:	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$		C(832) Table	0.7006 (9) 4. Selected	0.7352 (l geometric	7) 0.2434 (5) c parameters (Å, °)	0.110(3)
 ω/2θ scans Absorption correcting none 7659 measured refile 	on: ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflections	ons	C(832) Table	0.7006 (9) 4. Selected	0.7352 (<i>geometric</i> 1.466 (3)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82)	0.110 (3) 1.681 (4)
 ω/2θ scans Absorption correcting none 7659 measured refl 7200 independent r 	on: ections eflections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every	ons y 250	C(832) Table P(11)-O(1 P(11)-N(8	0.7006 (9) 4. Selectea (1) 31)	0.7352 (1 geometric 1.466 (3) 1.685 (3)	7) 0.2434 (5) <i>c parameters</i> (Å, °) P(12)—N(82) P(12)—N(22) P(12)—N(22)	0.110 (3)) for (2) 1.681 (4) 1.688 (3)
 ω/2θ scans Absorption correcting 7659 measured refl 7200 independent reflet 4847 observed reflet 	on: ections eflections ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflections monitored every reflections	ons y 250	C(832) Table - P(11)O(1 P(11)N(8 P(11)N(2	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (d geometric 1.466 (3) 1.685 (3) 1.693 (3) 1.705 (4)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) P(12)—C(112)	0.110 (3)) for (2) 1.681 (4) 1.688 (3) 1.794 (4)
$\omega/2\theta$ scans Absorption correctinone 7659 measured refl 7200 independent r 4847 observed refle $[L > 2\sigma(D)]$	on: ections eflections ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every reflections intensity decay:	ons y 250	C(832) Table - P(11)O(1 P(11)N(8 P(11)N(2 P(11)C(1 S(21)C(1)	0.7006 (9) 4. Selected (1) (1) (1) (1) (11) (1) (1) (1	0.7352 (1 geometric 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.698 (2)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—N(22) S(32)—S(42)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.012 (2)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle $[I > 2\sigma(I)]$	on: ections eflections ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every reflections intensity decay:	ons y 250 1.5%	C(832) Table - P(11)O(1 P(11)N(8 P(11)N(2 P(11)C(1 S(31)N(2 S(31)N(2 S(31)N(2) S(31)N(2) S(31)N(3)N(3) S(31)N(3)N(3) S(31)N(3	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (2) (1) (2) (2) (2) (2) (2) (2) (3) (3) (4) (5) (4) (5) (5) (6) (7) (6) (7) (7) (7) (7) (7) (7) (7) (7	0.7352 (1 geometric 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—N(22) S(32)—S(42) S(52)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle $[I > 2\sigma(I)]$	on: ections eflections ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every reflections intensity decay:	ons y 250 1.5%	C(832) Table - P(11)O(1 P(11)N(8 P(11)N(2 P(11)C(1 S(31)N(2 S(31)S(4 S(41)-S(5 S(41)-S(5 S(41)-S(5 S(41)-S(5 S(41)-S(5 S(4)-S(5)-S(5)-S(5)-S(5)-S(5)-S(5)-S(5)-S(5	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.114 (3)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—N(22) S(32)—S(42) S(42)—S(52) S(62)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle $[I > 2\sigma(I)]$ <i>Refinement</i>	on: ections eflections ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every reflections intensity decay:	ons y 250 1.5%	C(832) Table $-$ P(11)-O(1 P(11)-N(8 P(11)-N(2 P(11)-C(1 S(31)-N(2 S(31)-S(4 S(41)-S(5) S(51)-S(6)	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (<i>geometric</i> 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.114 (3) 2.021 (2)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—N(22) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(52)—S(62) S(52)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.011 (4)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle $[I > 2\sigma(I)]$ <i>Refinement</i> Refinement on F^2	on: ections eflections ections	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (f_o^2)]$	ons y 250 1.5% 0.0454P) ²	C(832) Table $-$ P(11)-O(1 P(11)-N(8 P(11)-N(2 P(11)-N(2 S(31)-S(4 S(31)-S(4 S(41)-S(5 S(51)-S(6 S(51)-S(6) S(61)-S(7)	0.7006 (9) 4. Selected (1) (1) (1) (21) (1) (1) (21) (1) (1) (1) (1) (2) (2) (2) (3) (4) (5) (4) (5) (5) (5) (5) (5) (5) (5) (5	0.7352 (<i>geometric</i> 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.114 (3) 2.021 (2) 2.067 (2)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(52)—S(62) S(62)—S(72) N(82)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle $[I > 2\sigma(I)]$ <i>Refinement</i> Refinement on F^2	on: ections eflections ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (\frac{1}{2} + 324P)]$	ons y 250 1.5% 0.0454P) ²	C(832) Table $-$ P(11)—O(1 P(11)—N(8 P(11)—N(2 P(11)—N(2 S(31)—S(4 S(31)—S(4 S(41)—S(5 S(51)—S(6 S(61)—S(7)—N(8)	0.7006 (9) 4. Selected (1) (1) (1) (21) (1) (1) (1) (1) (1) (1) (1) (0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.114 (3) 2.021 (2) 2.067 (2) 1.678 (3)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—N(22) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(52)—S(62) S(62)—S(72) S(72)—N(82) N(22)—C(22)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle $[I > 2\sigma(I)]$ <i>Refinement</i> Refinement on F^2 R(F) = 0.0475	on: ections eflections ections	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (\frac{1}{2} + \frac{1}{2})]$ where P = [mag]	ons y 250 1.5% $0.0454P)^2$ $0x(E^2 0)$	C(832) Table - P(11) \rightarrow O(1) P(11) \rightarrow N(8 P(11) \rightarrow N(2 P(11) \rightarrow C(1 S(31) \rightarrow N(2 S(31) \rightarrow S(5 S(51) \rightarrow S(6 S(51) \rightarrow S(7 S(71) \rightarrow N(8 N(2) \rightarrow C(2)	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.114 (3) 2.021 (2) 2.067 (2) 1.678 (3) 1.527 (5)	7) 0.2434 (5) c parameters (Å, °) P(12)-N(82) P(12)-N(22) P(12)-C(112) S(32)-N(22) S(32)-S(42) S(42)-S(52) S(52)-S(62) S(62)-S(72) S(72)-N(82) N(22)-C(22) N(82)-C(82)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.531 (6)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle $[I > 2\sigma(I)]$ <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$	on: ections eflections ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (\frac{1}{2})^2 + (\frac$	ons y 250 1.5% $(0.0454P)^2$ $(F_0^2, 0)$	C(832) Table $-$ P(11)—O(1) P(11)—N(8 P(11)—N(2 P(11)—C(1) S(31)—N(2 S(31)—S(4 S(51)—S(6) S(51)—S(6) S(51)—S(7) N(81)—C(8) N(81)—C(8)	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (<i>geometric</i> 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.014 (3) 2.021 (2) 2.067 (2) 1.678 (3) 1.572 (5)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—N(22) S(32)—S(42) S(42)—S(52) S(62)—S(62) S(62)—S(72) S(72)—N(82) N(22)—C(22) N(82)—C(22) N(82)—C(22) N(82)—C(22) N(82)—C(12)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.571 (6)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent of 4847 observed refle [$I > 2\sigma(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045	on: ections eflections ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3.24P]]$ where $P = [max + 2P]$	ons y 250 1.5% $0.0454P)^2$ $x(F_o^2, 0)$ $r_c^2]/3$	C(832) Table - P(11) - O(1) P(11) - N(8 P(11) - N(2 P(11) - N(2 P(11) - N(2 S(31) - S(4 S(31) - S(4 S(51) - S(6 S(51) - S(7) S(61) - S(7) N(21) - C(2 N(81) - C(3) N(21) - C(3) N(3) - C(3) N	0.7006 (9) 4. Selected (1) (1) (1) (1) (2) (1) (1) (1) (1) (1) (1) (1) (1	0.7352 (2 geometric 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.016 (2) 2.017 (2) 2.021 (2) 2.027 (2) 1.678 (3) 1.527 (5) 1.542 (5) 1.375 (6)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—N(22) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(62)—S(72) N(82)—C(82) N(22)—C(82) C(112)—C(162) C(112)—C(122)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.571 (6) 1.379 (6) 1.389 (6)
ω/2θ scans Absorption correctinon 7659 measured refl 7200 independent of 4847 observed refle [$I > 2σ(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections	on: ections eflections ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3.24P]]$ where $P = [max + 2P]$ $(\Delta/\sigma)_{max} = 0.1$	ons y 250 1.5% $0.0454P)^2$ $1x(F_o^2,0)$ $7_c^2]/3$	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - N(2) \\ S(31) - N(2) \\ S(31) - S(4) \\ S(31) - S(4) \\ S(51) - S(6) \\ S(51) - S(6) \\ S(51) - S(6) \\ S(71) - N(8) \\ N(21) - C(2) \\ N(81) - C(2) \\ C(111) - C(2) \\ C(11) - $	0.7006 (9) 4. Selected (1) (1) (1) (21) (1) (1) (1) (1) (1) (1) (1) (0.7352 (2 geometric 1.466 (3) 1.685 (3) 1.693 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.016 (2) 2.017 (2) 1.678 (3) 1.527 (5) 1.542 (5) 1.375 (6) 1.393 (6)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—S(42) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(62)—S(72) S(72)—N(82) N(22)—C(22) N(82)—C(82) C(112)—C(162) C(112)—C(122) C(122)—C(132)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.017 (4) 1.680 (4) 1.543 (5) 1.531 (6) 1.379 (6)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle [$I > 2\sigma(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters	on: ections eflections ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (1 + 3.24P)]$ where $P = [max_{ont} + 2P]$ $(\Delta/\sigma)_{max} = 0.1$ $\Delta\rho_{max} = 0.83 \text{ e} \text{ Å}$	ons y 250 1.5% $0.0454P)^2$ $\exp(F_o^2, 0)$ 7_c^2]/3 -3	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - N(2) \\ S(31) - N(2) \\ S(31) - N(2) \\ S(31) - S(4) \\ S(41) - S(5) \\ S(51) - S(6) \\ S(61) - S(7) \\ S(71) - N(8) \\ N(21) - C(2) \\ N(81) - C(2) \\ N(81) - C(2) \\ C(111) - C(2) \\ C(11) - C$	0.7006 (9) 4. Selected (1) (1) (21) (1) (21) (1) (1) (1) (1) (1) (1) (21) (1) (21) (121) (161) (131)	0.7352 (<i>geometric</i> 1.466 (3) 1.685 (3) 1.693 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.114 (3) 2.016 (2) 2.017 (2) 1.678 (3) 1.527 (5) 1.542 (5) 1.375 (6) 1.385 (6)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—S(42) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(52)—S(62) S(72)—N(82) N(22)—C(22) N(82)—C(82) C(112)—C(162) C(112)—C(122) C(122) C(122)—C(122) C(12)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.017 (4) 1.680 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.379 (6) 1.362 (7)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle [$I > 2\sigma(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameters	on: ections effections ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (1 + 3.24P)]$ where $P = [max + 2P]$ $(\Delta/\sigma)_{max} = 0.1$ $\Delta\rho_{max} = 0.83 \text{ e Å}$ $\Delta\rho_{min} = -0.72 \text{ e f}$	ons y 250 1.5% $0.0454P)^2$ $\exp(F_c^2, 0)$ $F_c^2]/3$ -3 Å $^{-3}$	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(2) \\ P(11) - N(2) \\ S(31) - S(4) \\ S(31) - S(4) \\ S(31) - S(5) \\ S(51) - S(6) \\ S(61) - S(7) \\ S(71) - N(8) \\ N(21) - C(2) \\ N(81) - C(3) \\ C(111) - C(3) \\ C(111) - C(3) \\ C(121) - C(3) \\$	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.016 (2) 2.017 (2) 2.067 (2) 1.678 (3) 1.527 (5) 1.542 (5) 1.375 (6) 1.385 (6) 1.385 (6)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(62)—S(72) S(72)—N(82) N(22)—C(22) N(82)—C(82) C(112)—C(162) C(112)—C(132) C(132)—C(142) C(142)—C(152)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.580 (4) 1.531 (6) 1.379 (6) 1.389 (6) 1.379 (7)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle [$I > 2\sigma(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameter	on: ections effections ections	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3.24P)]$ where P = [man + 2F (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Δtomic scattering	ons y 250 1.5% $(0.0454P)^2$ $(x(F_o^2, 0))$ $(x_c^2)/3$ $(x_c^2)/3$ $(x_c^2)/3$ $(x_c^2)/3$	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) \longrightarrow (11) \longrightarrow (12) \\ P(11) \longrightarrow (12) \\ P(1$	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.016 (2) 2.017 (2) 1.678 (3) 1.527 (5) 1.527 (5) 1.527 (5) 1.375 (6) 1.393 (6) 1.385 (6) 1.375 (8)	7) 0.2434 (5) 7) 0.2434 (5) 7) $P(12) - N(82)$ P(12) - N(22) P(12) - C(112) S(32) - N(22) S(32) - S(42) S(52) - S(52) S(52) - S(52) S(52) - S(62) S(62) - S(72) N(82) - C(82) C(112) - C(162) C(112) - C(162) C(112) - C(122) C(122) - C(132) C(132) - C(142) C(142) - C(152) C(152) - C(162)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.531 (6) 1.379 (6) 1.379 (6) 1.379 (7) 1.371 (7) 1.383 (6)
ω/2θ scans Absorption correctinon 7659 measured refi 7200 independent of 4847 observed refie [$I > 2σ(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameter refined	on: ections eflections ections	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3.24P]]$ where P = [man + 2F (Δ/σ)max = 0.1 Δρmax = 0.83 e Å Δρmin = -0.72 e Atomic scattering form Latornatio	ons y 250 1.5% $(0.0454P)^2$ $(x(F_o^2, 0))$ $(x^2_c)/3$ $(x^2_c)/3$ $(x^2_c)/3$ $(x^2_c)/3$	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(8) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - N(2) \\ S(31) - N(2) \\ S(31) - S(4) \\ S(31) - S(4) \\ S(51) - S(6) \\ S(51) - S(6) \\ S(61) - S(7) \\ S(71) - N(8) \\ N(21) - C(2) \\ N(81) - C(8) \\ C(111) - C(2) \\ C(11) - C(2) \\ C$	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (<i>geometric</i> 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.014 (3) 2.021 (2) 2.021 (2) 2.027 (5) 1.572 (5) 1.572 (5) 1.375 (6) 1.393 (6) 1.385 (8) 1.380 (7)	7) 0.2434 (5) c parameters (Å, °) P(12)N(82) P(12)N(22) P(12)C(12) S(32)N(22) S(32)S(42) S(42)S(52) S(52)S(62) S(62)S(72) S(72)N(82) N(22)C(22) N(82)C(122) C(112)C(162) C(112)C(152) C(152)C(152) C(152)C(162) C(22)C(22)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.371 (7) 1.383 (6) 1.45 (2)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle [$I > 2\sigma(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameter refined	on: ections eflections ections	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3.24P]]$ where $P = [max + 2P]$ ($\Delta/\sigma)_{max} = 0.1$ $\Delta\rho_{max} = 0.83$ e Å $\Delta\rho_{min} = -0.72$ e Atomic scattering from Internation	ons y 250 1.5% $0.0454P)^2$ $x(F_o^2,0)$ $r_o^2]/3$ -3 Å -3 factors nal Tables	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(8) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - N(2) \\ S(31) - S(4) \\ S(31) - S(4) \\ S(51) - S(6) \\ S(51) - S(6) \\ S(61) - S(7) \\ S(71) - N(8) \\ N(21) - C(2) \\ N(81) - C(2) \\ N(81) - C(2) \\ C(111) - C(2) \\ C(21) - $	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (2 geometric 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.016 (2) 2.017 (2) 2.021 (2) 2.021 (2) 2.027 (5) 1.542 (5) 1.375 (6) 1.393 (6) 1.375 (7) 1.355 (8) 1.380 (7) 1.506 (7)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—N(22) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(62)—S(72) N(82)—C(82) N(22)—C(22) N(82)—C(82) C(112)—C(162) C(112)—C(162) C(122)—C(132) C(132)—C(142) C(152)—C(152) C(152)—C(162) C(22)—C(22) C(22)—C(212)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.379 (6) 1.379 (6) 1.362 (7) 1.383 (6) 1.45 (2) 1.483 (9)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent refl 847 observed refle [$I > 2\sigma(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameter refined	on: ections effections ections	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: w = 1/[σ ² (F _o ²) + (+ 3.24P] where P = [ma + 2P (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Atomic scattering from Internation for X-ray Crysta	ons y 250 1.5% $0.0454P)^2$ $1x(F_o^2,0)$ $r_o^2]/3$ r_a^3 factors nal Tables allography	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - N(2) \\ S(31) - N(2) \\ S(31) - S(4) \\ S(31) - S(4) \\ S(31) - S(5) \\ S(51) - S(6) \\ S(51) - S(6) \\ S(71) - N(8) \\ N(21) - C(2) \\ N(81) - C(2) \\ C(111) - C(2) \\ C(21) - $	0.7006 (9) 4. Selected (1) (1) (1) (21) (1) (1) (1) (1) (1) (1) (1) (0.7352 (<i>geometric</i> 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.017 (2) 2.021 (2) 2.021 (2) 2.021 (2) 2.027 (2) 1.575 (6) 1.375 (6) 1.375 (6) 1.372 (7) 1.355 (8) 1.380 (7) 1.506 (7) 1.504 (7) 1.504 (7)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—S(42) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(62)—S(72) S(72)—N(82) N(22)—C(22) N(82)—C(82) C(112)—C(162) C(112)—C(162) C(122)—C(132) C(132)—C(142) C(142)—C(152) C(152)—C(162) C(122)—C(162) C(122)—C(162) C(22)—C(22) C(22)—C(22) C(22)—C(22) C(22)—C(232)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.017 (4) 1.680 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.383 (6) 1.379 (6) 1.383 (6) 1.45 (2) 1.483 (9) 1.482 (10)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle [$I > 2\sigma(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameter refined	on: ections effections ections	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: w = 1/[σ ² (F _o ²) + (+ 3.24P] where P = [ma + 2F (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Atomic scattering from Internation for X-ray Crysta (1974, Vol. IV)	ons y 250 1.5% $0.0454P)^2$ $1x(F_o^2,0)$ $7_c^2]/3$ -3 factors nal Tables allography	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - N(2) \\ S(31) - N(2) \\ S(31) - N(2) \\ S(31) - N(3) \\ S(31) - S(5) \\ S(51) - S(6) \\ S(51) - S(6) \\ S(71) - N(8) \\ N(21) - C(2) \\ N(81) - C(2) \\ N(81) - C(2) \\ C(111) - C(2) \\ C(21) - $	0.7006 (9) 4. Selected (1) (1) (21) (21) (1) (21) (1) (1) (1) (1) (1) (1) (1) (0.7352 (<i>geometric</i> 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.114 (3) 2.016 (2) 2.017 (2) 1.678 (3) 1.527 (5) 1.542 (5) 1.375 (6) 1.393 (6) 1.385 (6) 1.372 (7) 1.350 (7) 1.506 (7) 1.504 (7) 1.506 (7)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—S(42) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(62)—S(72) S(72)—N(82) N(22)—C(22) N(82)—C(82) C(112)—C(162) C(112)—C(162) C(112)—C(162) C(122)—C(122) C(122)—C(122) C(122)—C(122) C(122)—C(122) C(122)—C(122) C(22)—C(22) C(22)—C(22) C	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.382 (7) 1.383 (6) 1.482 (10) 1.483 (9) 1.483 (12)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent r 4847 observed refle [$I > 2\sigma(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameters Refined	on: ections effections ections	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: w = 1/[σ ² (F _o ²) + (+ 3.24P] where P = [ma + 2F (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Atomic scattering from Internation for X-ray Crysta (1974, Vol. IV)	ons y 250 1.5% $0.0454P)^2$ $\exp(F_o^2, 0)$ $7_c^2]/3$ -3 Å -3 factors nal Tables allography	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) \longrightarrow (11) \longrightarrow (12) \\ P(11) \longrightarrow (12) \\ P(1$	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.016 (2) 2.017 (2) 2.067 (2) 1.678 (3) 1.527 (5) 1.542 (5) 1.375 (6) 1.375 (6) 1.375 (6) 1.375 (8) 1.380 (7) 1.506 (7) 1.507 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.507 (7) 1	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—N(22) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(52)—S(62) S(62)—S(72) N(82)—C(82) C(112)—C(162) C(112)—C(162) C(112)—C(162) C(112)—C(142) C(142)—C(142) C(142)—C(142) C(142)—C(142) C(142)—C(152) C(152)—C(162) C(22)—C(22) C(22)—C(22) C(22)—C(22) C(22)—C(22)' C(22)-C(22)' C(22)/ C(22)—C(22)' C(22)/ C(2)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.371 (7) 1.383 (6) 1.45 (2) 1.483 (9) 1.482 (10) 1.583 (12) 0.72 (2) 1.599 (10)
ω/2θ scans Absorption correctinon 7659 measured refi 7200 independent of 4847 observed refie [$I > 2σ(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameter refined	on: ections effections ections eters <i>nal atomic</i>	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3.24P)]$ where P = [ma + 2F (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Atomic scattering from Internation for X-ray Crystal (1974, Vol. IV) coordinates and decay	ons y 250 1.5% $(0.0454P)^2$ $(x(F_o^2, 0))$ $(x^2)/3$	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - C(1) \\ S(31) - N(2) \\ S(31) - N(2) \\ S(31) - S(3) \\ S(51) - S(5) \\ S(51) - S(6) \\ S(61) - S(7) \\ S(71) - N(8) \\ N(21) - C(1) \\ C(111) - C(2) \\ C(11) - $	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.016 (2) 2.017 (2) 1.678 (3) 1.527 (5) 1.572 (5) 1.375 (6) 1.393 (6) 1.385 (6) 1.385 (6) 1.385 (6) 1.380 (7) 1.506 (7) 1.506 (7) 1.503 (7) 1.517 (7) 1	7) 0.2434 (5) 7) 0.2434 (5) 7) $P(12)-N(82)$ P(12)-N(22) P(12)-C(112) S(32)-N(22) S(32)-S(42) S(52)-S(52) S(52)-S(52) S(62)-S(72) N(82)-C(12) N(12)-C(122) C(112)-C(162) C(112)-C(162) C(112)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(22)-C(222) C(22)-C(222) C(22)-C(222') C(22)-C(22') C(22)-C(22') C(22)-C(22') C(22)-C(22') C(22)-C(22') C(22)-C(22') C(22)-C(22')	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.531 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.383 (6) 1.45 (2) 1.483 (9) 1.483 (9) 1.483 (12) 0.72 (2) 1.508 (10) 1.513 (0)
ω/2θ scans Absorption correctinon 7659 measured refi 7200 independent of 4847 observed refie [$I > 2σ(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameter refined Table 3. <i>Fraction</i> <i>isotropic dis</i>	on: ections effections ections eters eters end atomic placement	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3.24P]]$ where P = [ma + 2F (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Atomic scattering from Internation for X-ray Crystal (1974, Vol. IV) coordinates and apparameters (Å ²) for	ons y 250 1.5% $(0.0454P)^2$ $(x(F_o^2,0))$ $(x^2_c)/3$ $(x^2_c)/$	C(832) Table $-$ P(11)—O(1) P(11)—N(8 P(11)—N(2 P(11)—N(2 P(11)—C(1 S(31)—N(2 S(31)—S(4 S(31)—S(4 S(31)—S(5 S(51)—S(6 S(61)—S(7 S(71)—N(8 N(21)—C(2 C(11)—C(2 C(11)—C(2 C(11)—C(2 C(11)—C(2 C(11)—C(2 C(11)—C(2 C(21)—C(2)=C(2)=C(2)=C(2)=C(2)=C(2)=C(2)=C(2)=	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (<i>geometric</i> 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.011 (2) 2.021 (2) 2.021 (2) 2.027 (5) 1.542 (5) 1.375 (6) 1.393 (6) 1.385 (6) 1.385 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.507 (7) 1.507 (7) 1.502 (7) 1.517 (7) 1.520 (7)	7) 0.2434 (5) c parameters (Å, °) P(12)N(82) P(12)N(22) S(32)N(22) S(32)N(22) S(32)S(42) S(42)S(52) S(52)S(62) S(62)S(72) S(72)N(82) N(22)C(22) N(82)C(122) C(112)C(142) C(112)C(142) C(122)C(132) C(122)C(132) C(122)C(132) C(122)C(142) C(122)C(152) C(122)C(152) C(22)C(22) C(22)C(22) C(22)C	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.017 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.017 (4) 1.680 (4) 1.543 (5) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.383 (6) 1.45 (2) 1.483 (9) 1.483 (9) 1.483 (12) 0.72 (2) 1.508 (10) 1.513 (9)
ω/2θ scans Absorption correctinon 7659 measured refi 7200 independent of 4847 observed refie [$I > 2σ(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameter refined	on: ections effections ections eters eters eal atomic placement	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: w = 1/[σ ² (F _o ²) + (+ 3.24P] where P = [ma + 2F (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Atomic scattering from Internation for X-ray Crystat (1974, Vol. IV) coordinates and a parameters (Å ²) for	ons y 250 1.5% $0.0454P)^2$ $x(F_o^2,0)$ $r_o^2]/3$ -3 Å -3 factors nal Tables allography equivalent or (2)	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(8 \\ P(11) - N(2 \\ P(11) - N(2 \\ P(11) - N(2 \\ S(31) - S(4 \\ S(31) - S(4 \\ S(31) - S(4 \\ S(51) - S(6 \\ S(61) - S(7 \\ S(71) - N(8 \\ N(21) - C(2 \\ N(81) - C(8 \\ O(11) - C(2 \\ C(111) - C(2 \\ C(11) - C($	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (<i>geometric</i> 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.017 (2) 2.021 (2) 2.021 (2) 2.027 (5) 1.572 (5) 1.572 (5) 1.375 (6) 1.375 (6) 1.375 (6) 1.375 (7) 1.504 (7) 1.504 (7) 1.504 (7) 1.504 (7) 1.504 (7) 1.504 (7) 1.503 (7) 1.517 (7) 1.520 (7) 1.458 (3)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—N(22) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(62)—S(72) N(82)—C(82) N(22)—C(22) N(82)—C(82) C(112)—C(162) C(112)—C(162) C(122)—C(152) C(152)—C(162) C(152)—C(162) C(152)—C(162) C(22)—C(22) C(22)—C(22) C(22)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.383 (12) 1.483 (9) 1.482 (10) 1.583 (12) 0.72 (2) 1.508 (10) 1.513 (9) 1.525 (9)
ω/2θ scans Absorption correctinon 7659 measured refi 7200 independent of 4847 observed refic [$I > 2σ(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameter refined Table 3. <i>Fraction</i> <i>isotropic dis</i> U	on: ections effections ections etters eal atomic placement $q = (1/3)\Sigma_{1}^{2}$	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: w = 1/[σ ² (F _o ²) + (+ 3.24P] where P = [ma + 2P (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Atomic scattering from Internation for X-ray Crysta (1974, Vol. IV) coordinates and a parameters (Å ²) for Σ _j U _{ij} a [*] _i a [*] _i a _i .	ons y 250 1.5% $(0.0454P)^2$ $(x(F_o^2, 0))$ $r_o^2]/3$ -3 Å -3 factors nal Tables allography equivalent or (2)	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(8) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - N(2) \\ S(31) - S(4) \\ S(31) - S(4) \\ S(31) - S(4) \\ S(51) - S(6) \\ S(51) - S(6) \\ S(51) - S(6) \\ N(21) - C(2) \\ N(81) - C(2) \\ N(81) - C(2) \\ C(111) - C(2) \\ C(21) - C(2$	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (2 geometric 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.017 (2) 2.067 (2) 1.678 (3) 1.527 (5) 1.547 (5) 1.393 (6) 1.375 (8) 1.385 (6) 1.385 (6) 1.372 (7) 1.506 (7) 1.504 (7) 1.502 (7) 1.517 (7) 1.520 (7) 1.458 (3) 112.2 (2)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(62)—S(72) S(72)—N(82) N(22)—C(22) N(82)—C(82) C(112)—C(162) C(112)—C(162) C(122)—C(132) C(132)—C(142) C(142)—C(152) C(152)—C(162) C(122)—C(122) C(122)—C(122) C(22)—C(22) C(22)—C(22) C(2)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.379 (6) 1.379 (6) 1.362 (7) 1.383 (6) 1.452 (2) 1.483 (9) 1.482 (10) 1.583 (12) 0.72 (2) 1.508 (10) 1.513 (9) 1.525 (9) 112.1 (2)
ω/2θ scans Absorption correctinon 7659 measured refl 7200 independent of 4847 observed refle [$I > 2σ(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameters All H-atom parameters refined	on: ections effections ections eters eters end atomic placement $q = (1/3) \sum_{i}^{n} 2^{n}$	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: w = 1/[σ ² (F _o ²) + (+ 3.24P] where P = [ma + 2P (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Atomic scattering from Internation for X-ray Crysta (1974, Vol. IV) coordinates and a parameters (Å ²) for Σ _j U _{ij} a _i *a _j *a _i .a _j .	ons y 250 1.5% $(0.0454P)^2$ $(F_o^2, 0)$ $(T_o^2)/3$ -3 (A^{-3}) factors nal Tables allography equivalent or (2)	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - N(2) \\ S(31) - N(2) \\ S(31) - S(4) \\ S(31) - S(4) \\ S(31) - S(5) \\ S(51) - S(6) \\ S(51) - S(6) \\ S(51) - S(7) \\ N(21) - C(2) \\ N(21) - C(2) \\ C(111) - C(2) \\ C(2) \\ C(2) - C(2) \\ C(2) - C(2) \\ C(2) \\ C(2) \\ C(2) - C($	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.016 (2) 2.017 (2) 2.067 (2) 1.542 (5) 1.542 (5) 1.542 (5) 1.375 (6) 1.375 (6) 1.375 (8) 1.385 (6) 1.375 (8) 1.380 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.507 (7) 1.506 (7) 1.507 (7) 1.507 (7) 1.507 (7) 1.517 (7) 1.520 (7) 1.521 (7) 1.522 (2) 112.9 (2) 12.9 (2) 1.50 (2) 1.50 (2) 1.50 (2) 1.50 (2) 1.50 (2) 1.522 (2) 1.50 (2) 1.50 (2) 1.522 (2) 1.50 (2)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—S(42) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(62)—S(72) S(72)—N(82) N(22)—C(22) N(82)—C(82) C(112)—C(162) C(112)—C(162) C(122)—C(132) C(132)—C(142) C(142)—C(152) C(152)—C(162) C(122)—C(122) C(22)—C(222) C(22)—C(222) C(22)—C(222') C(22)—C(222') C(82)—C(822) C(82)—C(812) O(12)—P(12)—C(112) N(8)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.383 (6) 1.482 (10) 1.583 (12) 0.72 (2) 1.508 (10) 1.513 (9) 1.525 (9) 112.1 (2) 105.6 (2)
ω/2θ scans Absorption correctinon 7659 measured refl 7200 independent of 4847 observed refle [$I > 2σ(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameters All H-atom parameters Call the state of F^2 U_{e}	on: ections effections ections eters enal atomic placement $q = (1/3)\Sigma_{12}^{12}$	$h = 0 \rightarrow 11$ $k = -16 \rightarrow 15$ $l = -18 \rightarrow 18$ 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3,24P]]$ where P = [max + 2F (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Atomic scattering from Internation for X-ray Crysta (1974, Vol. IV) coordinates and e parameters (Å ²) for Σ _j U _{ij} a [*] _i a [*] _j a _i .a _j .	ons y 250 1.5% $(0.0454P)^2$ $(F_o^2, 0)$ $(T_c^2)/3$ (A^{-3}) factors nal Tables allography equivalent or (2)	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) \longrightarrow (11) \longrightarrow (12) \\ P(11) \longrightarrow (12) \\ P(12) \longrightarrow (12) \\ P(1$	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.016 (2) 2.017 (2) 2.067 (2) 1.678 (3) 1.527 (5) 1.542 (5) 1.375 (6) 1.393 (6) 1.385 (6) 1.385 (6) 1.385 (6) 1.385 (6) 1.385 (6) 1.385 (6) 1.385 (6) 1.385 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.503 (7) 1.507 (7) 1.503 (7) 1.517 (7) 1.520 (7) 1	7) 0.2434 (5) 7) 0.2434 (5) 7) $P(12)-N(82)$ P(12)-N(22) P(12)-C(112) S(32)-N(22) S(32)-S(42) S(42)-S(52) S(52)-S(62) S(62)-S(72) N(82)-C(82) C(112)-C(162) C(112)-C(162) C(112)-C(122) C(122)-C(132) C(132)-C(142) C(142)-C(142) C(142)-C(142) C(142)-C(142) C(142)-C(142) C(22)-C(22)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (3) 2.115 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.371 (7) 1.383 (6) 1.45 (2) 1.553 (12) 0.72 (2) 1.508 (10) 1.525 (9) 112.1 (2) 105.8 (2) 107.5 (2)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refi 7200 independent refi 7200 independent refi 7200 independent refi 847 observed refie [$I > 2\sigma(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameters All H-atom parameters All H-atom parameters All H-atom parameters <i>All H-atom parameters</i> <i>All H-atom parameters <i>All H-atom parameters</i> <i>All H-atom parameters</i> <i>All H-atom</i></i>	on: ections effections ections eters mal atomic placement $_{q} = (1/3) \sum_{i,2}^{v}$	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3.24P)]$ where P = [ma + 2F (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Atomic scattering from Internation for X-ray Crysta (1974, Vol. IV) coordinates and C parameters (Å ²) for E _j U _{ij} a [*] _i a [*] _j a _i .a _j .	ons y 250 1.5% $(0.0454P)^2$ $(F_o^2, 0)$ $(r_c^2)/3$ (A^{-3}) factors nal Tables allography equivalent or (2) U_{eq} (0.039(1))	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(8) \\ P(11) - N(2) \\ P(11) - C(1) \\ S(31) - N(2) \\ S(31) - N(4) \\ S(31) - S(5) \\ S(51) - S(6) \\ S(51) - S(7) \\ S(71) - N(8) \\ N(21) - C(2) \\ C(111) - C(1) \\ C(11) - P(1) \\ O(11) - P(1) \\ O(1) \\ O(1) - P(1) \\ O(1) \\ O(1) - P(1) \\ $	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.016 (2) 2.017 (2) 1.678 (3) 1.527 (5) 1.527 (5) 1.375 (6) 1.393 (6) 1.385 (6) 1.385 (6) 1.380 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.507 (7) 1.608 (2) 1.608 (2) 1	7) 0.2434 (5) 7) 0.2434 (5) 7) $P(12)-N(82)$ P(12)-N(22) P(12)-C(112) S(32)-N(22) S(32)-S(42) S(52)-S(52) S(52)-S(52) S(52)-S(52) S(62)-S(72) N(82)-C(12) N(12)-C(122) C(112)-C(122) C(112)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(22)-C(22) C(22)-C(2) C(22)-C(2) C(0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.371 (7) 1.383 (6) 1.45 (2) 1.483 (9) 1.482 (10) 1.583 (12) 0.72 (2) 1.508 (10) 1.513 (9) 1.525 (9) 112.1 (2) 105.8 (2) 107.5 (2) 110.12 (14) 105.8 (2) 107.5 (2) 110.12 (14) 105.8 (2) 107.5 (2) 110.12 (14) 105.8 (2) 105.8 (2) 105.
ω/2θ scans Absorption correctinon 7659 measured refi 7200 independent of 4847 observed refie [$I > 2σ(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameters(II) 0.4787(1) $0.4384(3)$	on: ections effections ections eters enal atomic placement $_{q} = (1/3)\sum_{i,2}^{v}$	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3.24P]]$ where P = [ma + 2F (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Atomic scattering from Internation for X-ray Crystal (1974, Vol. IV) coordinates and apparameters (Å ²) for Σ _j U _{ij} a [*] _i a [*] _j a _i .a _j . (1) 0.1349 (1) (2) 0.1175 (2) (1) 0.0345 (1)	ons y 250 1.5% $(0.0454P)^2$ $(x(F_o^2, 0))$ $(r_o^2)/3$ $(r_o^3)^2$ $(r_o^3)^3$ factors <i>nal Tables</i> <i>allography</i> <i>equivalent</i> <i>or</i> (2) U_{eq} (0.039(1)) (0.056(1)) (0.025(1))	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) - O(1) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - N(2) \\ P(11) - C(1) \\ S(31) - N(2) \\ S(31) - S(4) \\ S(31) - S(4) \\ S(51) - S(6) \\ S(51) - S(6) \\ S(61) - S(7) \\ S(71) - N(8) \\ N(21) - C(2) \\ C(111) - C(1) \\ C(111) - C(1) \\ C(111) - C(1) \\ C(111) - C(1) \\ C(111) - C(2) \\ C(11) - C(2) \\ C(1$	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.017 (2) 2.067 (2) 1.678 (3) 1.527 (5) 1.375 (6) 1.393 (6) 1.393 (6) 1.393 (6) 1.393 (6) 1.393 (6) 1.393 (6) 1.393 (7) 1.506 (7) 1.507 (7) 1.506 (7) 1.506 (7) 1.507 (7) 1.506 (7) 1.508 (3) 112.9 (2) 106.0 (2) 111.6 (2) 106.6 (2) 106.7 (2) 107.2 (2)	7) 0.2434 (5) 7) 0.2434 (5) 7) $P(12)-N(82)$ P(12)-N(22) P(12)-C(12) S(32)-S(42) S(32)-S(42) S(52)-S(62) S(62)-S(72) S(62)-S(72) N(22)-C(12) N(22)-C(12) C(112)-C(162) C(112)-C(162) C(12)-C(132) C(132)-C(142) C(142)-C(152) C(122)-C(122) C(22)-C(22) C(32)-C(32) C(32)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.017 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.017 (4) 1.680 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.379 (6) 1.383 (6) 1.45 (2) 1.483 (9) 1.483 (9) 1.483 (9) 1.483 (12) 0.72 (2) 1.508 (10) 1.513 (9) 1.525 (9) 112.1 (2) 105.8 (2) 107.5 (2) 110.12 (14) 103.86 (14)
ω/2θ scans Absorption correctinon 7659 measured refi 7200 independent of 4847 observed refic [$I > 2σ(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameters All H-atom parameters All H-atom parameters ($II = 0.4787(I)$) U_{C} V	on: ections effections ections eters eal atomic placement $_{q} = (1/3)\Sigma_{1}^{2}$	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: w = 1/[$\sigma^2(F_o^2)$ + (+ 3.24P] where P = [ma + 2F (Δ/σ) _{max} = 0.1 $\Delta\rho_{max}$ = 0.83 e Å $\Delta\rho_{min}$ = -0.72 e Atomic scattering from Internation for X-ray Crystat (1974, Vol. IV) coordinates and of parameters (Å ²) for $\Sigma_j U_{ij}a_i^*a_j^*a_i.a_j.$	ons y 250 1.5% $(0.0454P)^2$ $(F_o^2, 0)$ $F_c^2]/3$ -3 Å -3 factors nal Tables allography equivalent or (2) U_{eq} (0.039(1)) (0.056(1)) (0.042(1))	C(832) Table - P(11) $-O(1)$ P(11) $-N(8$ P(11) $-N(2$ P(11) $-N(2$ P(11) $-N(2$ P(11) $-N(2$ P(11) $-N(2$ S(31) $-N(2$ S(31) $-S(4$ S(31) $-S(4$ S(31) $-S(4$ S(31) $-S(4$ S(31) $-S(4$ S(31) $-S(6$ S(61) $-S(7)$ S(71) $-N(8$ N(21) $-C(2$ C(11) $-C(2$ C(11) $-C(2$ C(11) $-C(2$ C(11) $-C(2$ C(11) $-C(2$ C(11) $-C(2$ C(11) $-C(2$ C(11) $-C(2$ C(11) $-C(2$ C(21) $-C(2)$ C(21) $-C($	0.7006 (9) 4. Selected (1) (1) (1) (1) (2) (1) (2) (1) (2) (1) (1) (1) (1) (1) (1) (1) (1	0.7352 (<i>geometric</i> 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.011 (2) 2.021 (2) 2.067 (2) 1.678 (3) 1.527 (5) 1.375 (6) 1.393 (6) 1.375 (6) 1.393 (6) 1.375 (7) 1.506 (7) 1.504 (7) 1.502 (7) 1.517 (7) 1.520 (7) 1.458 (3) 112.9 (2) 106.0 (2) 111.6 (2) 106.6 (2) 107.0 (13)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—N(22) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(62)—S(72) N(82)—C(82) C(112)—C(162) C(112)—C(162) C(112)—C(162) C(122)—C(152) C(152)—C(162) C(152)—C(162) C(22)—C(22) C(22)—C	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.383 (12) 0.72 (2) 1.508 (10) 1.513 (9) 1.525 (9) 112.1 (2) 105.8 (2) 107.5 (2) 110.12 (14) 105.95 (13) 103.86 (14) 1.41 (12) 1.138 (14) 1.151 (14) 1.151 (14) 1.151 (15) 1.151 (14) 1.151 (15) 1.151 (15
ω/2θ scans Absorption correctinon 7659 measured refi 7200 independent of 4847 observed refic [$I > 2σ(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameters All H-atom parameters All H-atom parameters ($II = 0.4787$ (on: ections effections ections etters al atomic blacement $u_q = (1/3)\Sigma_{1}^{2}$ 0.1222 0.00800 0.0544	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3.24P]]$ where P = [marrow + 2P] (Δ/σ)max = 0.1 Δρmax = 0.83 e Å Δρmin = -0.72 e Atomic scattering from Internation for X-ray Crysta (1974, Vol. IV) coordinates and of parameters (Å ²) for E _j U _{ij} a _i *a _j *a _i .a _j . 2 (1) 0.1349 (1) (2) 0.1175 (2) (1) 0.0848 (1)	ons y 250 1.5% $(0.0454P)^2$ $(x(F_o^2, 0))^2$ $(x(F_o^2, 0))^2$	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) = O(1) \\ P(11) = N(8) \\ P(11) = N(2) \\ P(11) = N(2) \\ P(11) = N(2) \\ P(11) = N(2) \\ S(31) = S(3) \\ S(31) = S(3) \\ S(31) = S(3) \\ S(31) = S(3) \\ S(31) = C(2) \\ C(11) = C(2) \\ C(21) = C($	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (<i>geometric</i> 1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.017 (2) 2.021 (2) 2.021 (2) 2.027 (2) 1.578 (3) 1.527 (5) 1.393 (6) 1.375 (6) 1.393 (6) 1.375 (6) 1.375 (8) 1.375 (7) 1.504 (7) 1.502 (7) 1.517 (7) 1.520 (7) 1.458 (3) 112.2 (2) 112.9 (2) 106.6 (2) 107.2 (2) 106.7 (13) 106.87 (8)	7) 0.2434 (5) c parameters (Å, °) P(12)—N(82) P(12)—N(22) P(12)—C(112) S(32)—S(42) S(42)—S(52) S(52)—S(62) S(62)—S(72) S(72)—N(82) N(22)—C(22) N(82)—C(82) C(112)—C(162) C(112)—C(162) C(122)—C(122) C(122)—C(122) C(122)—C(122) C(122)—C(122) C(122)—C(122) C(122)—C(122) C(122)—C(122) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(222) C(22)—C(112) N(22)—P(12)—C(112) N(22)—P(12)—C(112) N(22)—P(12)—C(112) N(22)—S(42)—S(42) S(52)—S(42)—S(52) S(52)—S(42) S(52)—S(42)	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.648 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.383 (6) 1.379 (6) 1.383 (6) 1.379 (6) 1.383 (6) 1.379 (6) 1.383 (6) 1.353 (12) 0.72 (2) 1.508 (10) 1.513 (9) 1.525 (9) 112.1 (2) 105.8 (2) 107.5 (2) 110.12 (14) 105.95 (13) 103.86 (14) 106.31 (13) 109.2 (2)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent of 4847 observed refle [$I > 2\sigma(I)$] <i>Refinement</i> Refinement on F^2 R(F) = 0.0475 $wR(F^2) = 0.1306$ S = 1.045 7200 reflections 582 parameters All H-atom parameters All H-atom parameters All H-atom parameters <i>All H-atom parameters</i> <i>All H-atom parameters</i> <i>Refined</i> Table 3. <i>Fraction</i> <i>isotropic dis</i> <i>Ua</i> <i>x</i> P(11) 0.4384 (3) S(31) 0.4174 (1) S(41) 0.403 (1) S(51) 0.7599 (1)	on: ections effections cons effections cons effections eff	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3.24P])$ where P = [max + 2P (Δ/σ) _{max} = 0.1 Δρ _{max} = 0.83 e Å Δρ _{min} = -0.72 e Atomic scattering from Internation for X-ray Cryster (1974, Vol. IV) coordinates and of parameters (Å ²) for $\Sigma_j U_{ij}a_i^*a_j^*a_i.a_j.$	ons y 250 1.5% $(0.0454P)^2$ $(F_o^2, 0)$ $(r_o^2)/3$ $(r_o^2)/3$ -3 Å -3 factors nal Tables allography equivalent or (2) U_{eq} (0.39(1)) (0.056(1)) (0.054(1)) (0.068(1)) (0.068(1)) (0.068(1))	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) \longrightarrow (11) \longrightarrow (12) \\ P(11) \longrightarrow (12) \\ P(12) \longrightarrow (12) \\ P(1$	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.016 (2) 2.017 (2) 2.067 (2) 1.678 (3) 1.527 (5) 1.542 (5) 1.375 (6) 1.393 (6) 1.385 (6) 1.385 (6) 1.385 (6) 1.385 (6) 1.385 (6) 1.380 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.503 (7) 1.507 (7) 1.503 (7) 1.517 (7) 1.520 (7) 1.520 (7) 1.517 (7) 1.520 (7) 1.520 (7) 1.520 (7) 1.520 (7) 1.520 (7) 1.520 (7) 1.520 (7) 1.520 (7) 1.503 (7) 1.517 (7) 1.520 (7) 1	7) 0.2434 (5) 7) 0.2434 (5) 7) $P(12)-N(82)$ P(12)-N(22) P(12)-C(112) S(32)-N(22) S(32)-S(42) S(42)-S(52) S(52)-S(52) S(52)-S(52) S(52)-S(52) N(82)-C(82) C(112)-C(162) C(112)-C(162) C(112)-C(122) C(132)-C(132) C(132)-C(142) C(142)-C(152) C(152)-C(162) C(22)-C(222) C(22)-C(22) C(2)-C(2)-C(22) C(2)-C(2)-C(2) C(2)-C(2)-C(2) C(2)-C(2)-C(2) C(2)-C(2)-C(2)-C(2) C(2)-C(2)-C(2)-C(2) C(2)-C(2)-C(2)-C(2) C(2)-C(2)-C(2)-C(2)-C(2) C(2)-C(2)-C(2)-C(2)-C(2)-C(2) C(2)-C(2)-C(2)-C(2)-C(2)-C(2)-C(2)-C(0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.389 (6) 1.379 (6) 1.383 (6) 1.482 (7) 1.383 (6) 1.452 (2) 1.525 (9) 112.1 (2) 105.8 (2) 107.5 (2) 110.12 (14) 105.95 (13) 103.86 (14) 106.31 (13) 109.2 (2) 118.2 (3)
$\omega/2\theta$ scans Absorption correctinon 7659 measured refl 7200 independent refl 7200 independent refl 7200 independent refl 7200 independent refl 7200 reflections 7200 reflection	on: ections effections ections etters eal atomic placement $q = (1/3) \sum_{i=1}^{i}$ y 0.3204 0.4091 0.1412 0.0887	h = 0 → 11 k = -16 → 15 l = -18 → 18 3 standard reflecting monitored every reflections intensity decay: $w = 1/[\sigma^2(F_o^2) + (+3.24P] + (+3.24P])$ where P = [may + 24P] where P = [may (\Delta/\sigma)_{max} = 0.1 \Delta\rho_{max} = 0.83 e Å \Delta\rho_{min} = -0.72 e Atomic scattering from Internation for X-ray Crysta (1974, Vol. IV) coordinates and of parameters (Å ²) for $\Sigma_j U_{ij}a_i^*a_j^*a_i.a_j.$ (1) 0.1349 (1) (2) 0.1175 (2) (1) 0.0345 (1) (1) 0.1581 (1) (1) 0.1571 (1)	ons y 250 1.5% $(0.0454P)^2$ $(F_o^2, 0)$ $(r_o^2)/3$ $(r_o^2)/$	$\begin{array}{c} C(832) \\ \hline Table \\ P(11) = O(1) \\ P(11) = N(8) \\ P(11) = N(2) \\ P(11) = C(1) \\ S(31) = N(2) \\ S(31) = N(2) \\ S(31) = S(3) \\ S(31) = S(3) \\ S(31) = S(3) \\ S(31) = C(3) \\ C(111) = C(3) \\ C(11) = C($	0.7006 (9) 4. Selected (1) (1) (1) (1) (1) (1) (1) (1)	0.7352 (1.466 (3) 1.685 (3) 1.693 (3) 1.796 (4) 1.688 (3) 2.016 (2) 2.016 (2) 2.017 (2) 2.067 (2) 1.678 (3) 1.527 (5) 1.542 (5) 1.375 (6) 1.393 (6) 1.385 (6) 1.385 (6) 1.385 (6) 1.380 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.506 (7) 1.507 (7) 1.508 (3) 11.2 (2) 11.6 (2) 106.6 (2) 107.2 (2) 110.79 (13) 105.80 (9)	7) 0.2434 (5) 7) 0.2434 (5) 7) $P(12)-N(82)$ P(12)-N(22) P(12)-C(112) S(32)-S(42) S(32)-S(42) S(42)-S(52) S(52)-S(52) S(52)-S(52) S(62)-S(72) N(82)-C(12) N(12)-C(122) C(112)-C(122) C(112)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(122)-C(122) C(22)-C(22) C(22)-C(22)-C(22) C(22)-C(22)-C(22) C(22)-C(22)-C(22) C(22)-C(22)-C(22) C(22)-C(22)-C(22) C(22)-C(22)-C(22) C(22)-C(22)-C(22)-C(22) C(22)-C(22)-C(22)-C(22)-C(22) C(22)-C(2	0.110 (3) 1.681 (4) 1.688 (3) 1.794 (4) 1.668 (3) 2.115 (4) 2.013 (3) 2.115 (4) 2.011 (4) 2.067 (4) 1.680 (4) 1.543 (5) 1.531 (6) 1.379 (6) 1.371 (7) 1.383 (6) 1.45 (2) 1.483 (9) 1.482 (10) 1.583 (12) 0.72 (2) 1.508 (10) 1.513 (9) 1.525 (9) 112.1 (2) 105.8 (2) 107.5 (2) 110.12 (14) 105.95 (13) 103.86 (14) 106.31 (13) 109.2 (2) 118.2 (3) 120.9 (3)

C(21)—N(21)—S(31)	117.3 (2)	C(82)—N(82)—S(72)	118.6 (3)
C(21)—N(21)—P(11)	123.3 (2)	C(82)—N(82)—P(12)	122.0 (3)
S(31)—N(21)—P(11)	118.4 (2)	S(72)—N(82)—P(12)	118.6 (2)
C(81)—N(81)—S(71)	118.5 (3)	C(162) - C(112) - C(122)	119.1 (4)
C(81)—N(81)—P(11)	122.0 (3)	C(162) - C(112) - P(12)	123.7 (3)
S(71)—N(81)—P(11)	118.6 (2)	C(122) - C(112) - P(12)	117.2 (3)
C(121)—C(111)—C(161)	119.0 (4)	C(132)— $C(122)$ — $C(112)$	120.0 (5)
C(121)—C(111)—P(11)	123.5 (3)	C(142)-C(132)-C(122)	120.9 (5)
C(161)—C(111)—P(11)	117.4 (3)	C(132)—C(142)—C(152)	119.4 (5)
C(111)—C(121)—C(131)	120.7 (4)	C(142)-C(152)-C(162)	120.9 (5)
C(141)—C(131)—C(121)	119.2 (5)	C(112)—C(162)—C(152)	119.7 (4)
C(151)-C(141)-C(131)	121.0 (5)	C(222)—C(22)—C(212)	126.8 (9)
C(141)-C(151)-C(161)	120.3 (5)	C(222)—C(22)—C(232)	91.7 (9)
C(151)—C(161)—C(111)	119.8 (5)	C(212)-C(22)-C(232)	107.0 (6)
C(211)—C(21)—C(231)	109.6 (5)	C(222)—C(22)—N(22)	107.6 (8)
C(211)—C(21)—C(221)	108.8 (5)	C(212)—C(22)—N(22)	110.3 (4)
C(231)C(21)C(221)	108.9 (6)	C(232)—C(22)—N(22)	111.5 (5)
C(211)—C(21)—N(21)	110.0 (3)	C(222) - C(22) - C(222')	27.2 (7)
C(231)—C(21)—N(21)	110.8 (3)	C(212) - C(22) - C(222')	104.8 (7)
C(221)—C(21)—N(21)	108.6 (4)	C(232)—C(22)—C(222')	115.9 (7)
C(831)—C(81)—C(811)	110.9 (5)	N(22)—C(22)—C(222')	107.0 (5)
C(831)—C(81)—C(821)	110.2 (5)	C(822)—C(82)—C(832)	109.8 (6)
C(811)—C(81)—C(821)	108.4 (5)	C(822)—C(82)—C(812)	108.6 (6)
C(831)—C(81)—N(81)	107.7 (4)	C(832)—C(82)—C(812)	110.7 (6)
C(811)—C(81)—N(81)	109.5 (4)	C(822)—C(82)—N(82)	109.6 (6)
C(821)—C(81)—N(81)	110.1 (4)	C(832)—C(82)—N(82)	107.8 (5)
O(12)—P(12)—N(82)	111.4 (2)	C(812)—C(82)—N(82)	110.4 (4)
O(12)—P(12)—N(22)	113.4 (2)		

For both compounds, data collection: *MolEN* (Fair, 1990); cell refinement: *MolEN*; data reduction: *Xtal*3.2 (Hall, Flack & Stewart, 1992); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *PLA-TON*92 (Spek, 1990).

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Lists of structure factors, anisotropic displacement parameters, Hatom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1044). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Abrahams, S. C. (1961). Acta Cryst. 14, 311-312.
- Behar, V. & Danishefsky, X. (1993). J. Am. Chem. Soc. 115, 7017-7018.
- Bottle, S. E., Bott, R. E., Jenkins, I. D., Kennard, C. H. L., Smith, G. & Wells, A. P. (1993). J. Chem. Soc. Chem. Commun. pp. 1684– 1685.
- Caron, A., Donohue, J. & Goldfish, E. (1961). J. Am. Chem. Soc. 83, 3748–3750.
- Engelsein, B., Kalker, H. G., Lex, J. & Linke, K. H. (1974). Z. Naturforsch. Teil B, 29, 130-131.
- Fair, C. K. (1990). MolEN. An Interactive Intelligent System for Crystal Structure Analysis. Enraf-Nonius, Delft, The Netherlands.
- Hall, S. R., Flack, H. D. & Stewart, J. M. (1992). Editors. *Xtal3.2 Reference Manual*. Univs. of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Salama, P., Steliou, K. & Yu, X. (1992). J. Am. Chem. Soc. 114, 1456-1461.
- Sheldrick, G. M. (1985). SHELXS86. Program for the Solution of Crystal Structures. Univ. of Göttingen, Germany.

Sheldrick, G. M. (1993). SHELXL93. Program for the Refinement of Crystal Structures. Univ. of Göttingen, Germany.

Spek, A. L. (1990). Acta Cryst. A46, C-34.

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5-Acetoxy-6-(1,1-dimethyl-2-propenyl)-2*H*-furo[2,3-*h*][1]benzopyran-2-one

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Abstract

The title ester {6-(1,1-dimethyl-2-propenyl)-2-oxo-2*H*-furo[2,3-*h*][1]benzopyran-5-yl acetate, $C_{18}H_{16}O_5$ }, is a furanocoumarin. The pyrone, phenyl and furan rings are planar. Molecules are held together by C—H···O hydrogen bonds.

Comment

Many coumarin derivatives are of biological importance (Michel & Durant, 1976; Schmalle, Jarchow, Hausen & Schulz, 1982). The crystal structure of the title compound has been determined as part of our programme on the crystal structure analysis of these derivatives. The title compound, (I), was prepared by the acetylation of 5-hydroxy-6-(1,1-dimethyl-2-propenyl)-2*H*-furo[2,3-*h*][1]-benzopyran-2-one, a furanocoumarin isolated from the roots of *Heracleum thomsoni*, a herb growing wild in the Ladakh region of Jammu and Kashmir State, India (Banerjee, Gupta & Atal, 1980).



An ORTEPII (Johnson, 1976) drawing of the molecule with the atomic numbering scheme is shown in Fig. 1. The furocoumarin atoms are almost coplanar as in other furocoumarin derivatives (Bideau, Bravic & Desvergne, 1979; Bravic & Bideau, 1978; Dall'Acqua, Benetollo & Bombieri, 1981). The deviations from the mean plane of the three rings range from -0.058 (3) to 0.040 (2) Å, indicating a reasonably planar system. Variations in the bond lengths and angles in the phenyl ring, which are due to the fusion of the pyrone and