Fig. 2. Stereoview of the crystal packing. Direction of projection *a* axis, the *c* axis is vertical. Only one carboxylate orientation is indicated for molecule *A*.

of the other mirror image conformer to form chains extending along the [010] direction, the chains being held together by van der Waals forces. For the hydrogen-bonding interactions, a carboxylate oxygen is the donor atom to the ring carbonyl oxygen of an adjacent molecule; the O(12)$\cdots$O(22), O'(13)$\cdots$O(22) and O(33)$\cdots$O(2) distances are 2·74, 2·68 and 2·644 (4) Å respectively, with the O(33)—H(O33) and H(O33)$\cdots$O(2) spacings 0·96 (6) and 1·72 (6) Å and the O(33)—H(O33)$\cdots$O(2) angle 161 (4)°.

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Structure of 3,6-Diphenylpyrrolo[3,4-*c*]pyrrole-1,4-dithione

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Abstract. The crystal structure of 3,6-diphenylpyrrolo[3,4-*c*]pyrrole-1,4-thione has been determined. There are two different crystal forms, both of which are monoclinic. The crystal data are: $C_{18}H_{12}N_2S_2$, $M_r = 320·426$; modification (I), $P2_1/n$, $a = 7·986$ (1), $b = 4·757$ (1), $c = 19·685$ (3) Å, $\beta = 99·18$ (2)°, $V = 738·2$ (5) Å^3, $Z = 2$, $D_x = 1·441$, $D_m = 1·432$ Mg m^{-3}, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å, $T = 293$ K, $F(000) = 332$, $\mu = 0·342$ mm^{-1}, $R = 0·064$ for 1208 reflexions; modification (II), $P2_1/n$, $a = 4·873$ (1), $b = 18·613$ (2), $c = 7·995$ (1) Å, $\beta = 95·76$ (2)°, $V = 721·5$ (5) Å^3, $Z = 2$, $D_x = 1·475$, $D_m = 1·465$ Mg m^{-3}, graphite-mono-chromated Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å, $T = 293$ K, $F(000) = 332$, $\mu = 0·350$ mm^{-1}, $R = 0·054$ for 877 reflexions. The crystal forms are similar but differ slightly in the following points: both phenyl rings are twisted out of the plane of the heterocyclic ring system by 13° in modification (I), and by only 6° in modification (II). In the projection along the stacking axis, all the molecules are arranged in parallel in modification (I), and in a zigzag arrangement in modification (II).

Introduction. 3,6-Diphenylpyrrolo[3,4-*c*]pyrrole-1,4-dithione (DPPT, Fig. 1) is a thionated derivative of

3,6-diphenylpyrrolo[3,4-c]pyrrole-1,4-dione (DPP). A chlorinated derivative of the latter (which is red) has been on the market since 1986 (Iqbal, Cassar, Rochat, Pfenninger & Wallquist, 1988).

It was previously reported that the solvent-vapor treatment of an evaporated film of DPPT induces a large spectral shift in the visible and near-IR region toward longer wavelengths (Mizuguchi & Rochat, 1988). Furthermore, this vapor treatment brings about a drastic increase in photoconductivity by two orders of magnitude. Because of the enhanced absorption and photoconduction in the near IR, DPPT was used as an excellent photoconductor for the electrophotographic photoreceptor for laser printers. In order to gain insight into the mechanism of the above effects, an X-ray diffraction study was carried out on the evaporated DPPT film. The diffraction peak at a 2θ of 25·20° was found to shift slightly toward higher angles by 0·25° due to the vapor treatment, which corresponds to a shortening of the intermolecular spacing by 0·03 Å. This indicates that the molecules are rearranged upon vapor treatment.

A more detailed investigation on the molecular rearrangement was then performed by means of Fourier transform infrared spectroscopy (FT-IR) and X-ray photoelectron spectroscopy (XPS) (Mizuguchi & Homma, 1989). These studies revealed that a large intermolecular interaction of a charge-transfer nature operates along the molecular stacking axis via π-electrons of C, N and S atoms. The solvent vapor causes a rearrangement of the molecules so that the donor part of the molecule and the acceptor part of the other lie directly above one another. This greatly facilitates an intermolecular electron transfer in the excited state.

Since the spectral shift as well as the photoelectrical properties change drastically even on a slight change of the crystal structure, the structure analysis of DPPT was carried out. In this study, two crystal modifications have been found, but neither of these coincides with that of the photoactive phase caused by the vapor treatment.

Experimental. DPPT was prepared from commercially available DPP by using a Lawesson's reagent as the thionation agent (Mizuguchi & Rochat, 1988). The product synthesized was identified by means of mass spectroscopy, NMR and elemental analysis. The blue-black DPPT powder was then recrystallized from N-methylpyrrolidone for purification. The powder was further purified four times by sublimation in an argon atmosphere using apparatus which has been described previously (Mizuguchi, 1981).

The single crystal was grown from the vapor phase using argon as the carrier gas in the same subli-

Table 1 *Positional and isotropic displacement parameters with e.s.d.'s in parentheses for modification* (I)

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2 \times 8\pi^2)$
S(1)	0·6117 (1)	0·3040 (3)	0·41959 (6)	4·29 (2)
N(2)	0·7639 (3)	0·6496 (7)	0·5170 (1)	2·51 (5)
C(3)	0·7796 (4)	0·4477 (8)	0·4677 (2)	2·66 (7)
C(4)	0·9591 (4)	0·4088 (7)	0·4726 (1)	2·13 (6)
C(5)	0·9157 (5)	0·7373 (6)	0·5538 (2)	2·25 (7)
C(6)	0·9207 (5)	0·9395 (7)	0·6085 (2)	2·38 (6)
C(7)	1·0677 (4)	0·982 (1)	0·6549 (2)	3·97 (9)
C(8)	1·0734 (6)	1·168 (1)	0·7090 (3)	4·9 (1)
C(9)	0·9312 (7)	1·315 (1)	0·7176 (3)	4·35 (9)
C(10)	0·7836 (6)	1·2806 (7)	0·6733 (2)	3·55 (8)
C(11)	0·7761 (4)	1·0937 (9)	0·6184 (2)	2·97 (8)
H(12)	0·657 (6)	0·699 (8)	0·528 (2)	1·5 (9)*
H(13)	1·151 (3)	0·93 (1)	0·647 (2)	1·8 (9)*
H(14)	1·171 (6)	1·164 (8)	0·742 (3)	3 (1)*
H(15)	0·955 (5)	1·446 (8)	0·750 (2)	0·2 (7)*
H(16)	0·665 (4)	1·39 (1)	0·675 (2)	1·3 (9)*
H(17)	0·680 (3)	1·077 (9)	0·590 (2)	1·1 (8)*

* Atoms refined isotropically.

Table 2 *Positional and isotropic displacement parameters with e.s.d.'s in parentheses for modification* (II)

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2 \times 8\pi^2)$
S(1)	0·2006 (4)	0·4172 (1)	0·3667 (2)	5·48 (3)
N(2)	−0·1463 (9)	0·5161 (2)	0·2345 (5)	3·54 (9)
C(3)	0·053 (1)	0·4674 (3)	0·2105 (5)	3·5 (1)
C(4)	0·091 (1)	0·4726 (3)	0·0339 (5)	2·9 (1)
C(5)	−0·242 (1)	0·5547 (3)	0·0918 (5)	2·94 (9)
C(6)	−0·447 (1)	0·6082 (3)	0·0987 (6)	3·1 (1)
C(7)	−0·530 (1)	0·6517 (3)	−0·0408 (7)	4·1 (1)
C(8)	−0·725 (1)	0·7040 (3)	−0·0330 (8)	4·7 (1)
C(9)	−0·848 (1)	0·7163 (3)	0·1118 (7)	4·3 (1)
C(10)	−0·771 (1)	0·6729 (4)	0·2518 (7)	4·4 (1)
C(11)	−0·577 (1)	0·6209 (3)	0·2448 (6)	3·8 (1)
H(12)	−0·22 (1)	0·523 (4)	0·340 (8)	5 (2)*
H(13)	−0·419 (9)	0·650 (2)	−0·123 (6)	2 (1)*
H(14)	−0·77 (1)	0·739 (3)	−0·121 (8)	5 (2)*
H(15)	−0·979 (7)	0·754 (2)	0·125 (4)	0·6 (7)*
H(16)	−0·90 (1)	0·678 (3)	0·331 (7)	2 (1)*
H(17)	−0·56 (1)	0·582 (3)	0·322 (7)	4 (1)*

* Atoms refined isotropically.

mation apparatus described above. DPPT sublimed at about 573 K, and the argon flow rate was controlled to 33 mm³ s⁻¹ using a mass flow meter (F-100/200, Bronkhorst BV) so as to transport DPPT at a rate of about 33 µg s⁻¹. After 24 h of vapor growth, a number of scale-like and tetragonal-shaped single crystals were obtained in the sublimation tube.

The dimensions of the crystals were 0·72 × 0·63 × 0·08 mm (scale-like crystal) for modification (I), and 1·1 × 0·63 × 0·3 mm (tetragonal-shaped) for modification (II). The crystal structures were analyzed at 293 K by using a four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation [Nonius CAD-4 with $\lambda = 0.71069$ Å for modification (I), and Philips PW1100 with $\lambda = 0.70926$ Å for modification (II)]. θ–2θ scans. Unit-cell dimensions: modification

(I), from 25 reflexions $(10 < \theta < 15°)$; modification (II), from 25 reflexions $(10 < \theta < 15°)$. 2450 independent reflexions obtained in the range $4 < 2\theta < 60°$ for modification (I), and 2110 in the range $6 < 2\theta < 60°$ for modification (II). Among these, 1208 and 877 reflexions with $I > 3\sigma(I)$ were used for structure refinement for modifications (I) and (II), respectively. Corrections for absorption and secondary extinction were not applied. Three standard reflexions monitored every 2 h during data collection showed no significant change in intensity. The densities of the single crystals were measured by flotation in a mixture of benzene and carbon tetrachloride. The crystallographic data are given in the Abstract.

Structure determination and refinement

The structures were determined by direct methods using *MULTAN82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The structures were refined on F by full-matrix least-squares calculations using the *SDP* package (Frenz, 1985) with anisotropic atomic displacement parameters, neglecting H atoms, up to $R = 0.074$ for modification (I) and $R = 0.068$ for modification (II). Six H atoms were then located in difference electron density maps. Final $R = 0.064$, $wR = 0.069$, $S = 1.14$ for modification (I) and $R = 0.054$, $wR = 0.063$, $S = 2.15$ for modification (II). Weights of each reflexion in refinement were $w = 1/\sigma^2(F_o)$, $\sigma(F_o)$ being the e.s.d. based on counting statistics. The maximum weights in the final difference electron density maps were 0.36 and 0.59 e $Å^{-3}$ for modifications (I) and (II), respectively. Atomic scattering factors for C, N and S were taken from *International Tables for X-ray Crystallography* (1974). The final atomic positional and equivalent isotropic displacement parameters appear in Tables 1 and 2.*

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53005 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
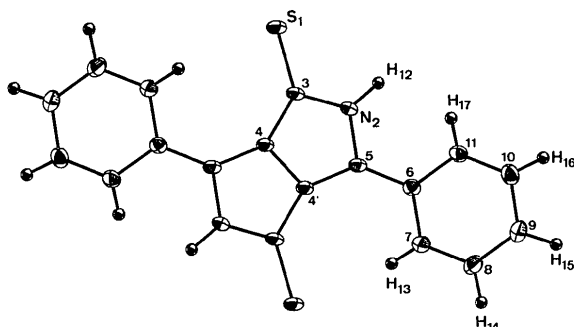

Fig. 1. Conformation of 3,6-diphenylpyrrolo[3,4-c]pyrrole-1,4-dithione.

Table 3. *Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses*

	Modification (I)	Modification (II)
S(1)—C(3)	1·660 (3)	1·664 (5)
N(2)—C(3)	1·386 (5)	1·357 (7)
N(2)—C(5)	1·375 (4)	1·389 (6)
C(2)—H(12)	0·94 (5)	0·96 (7)
C(3)—C(4)	1·434 (4)	1·445 (6)
C(4)—C(4')	1·456 (5)	1·422 (7)
C(4)—C(5)	1·385 (5)	1·399 (7)
C(5)—C(6)	1·438 (5)	1·416 (7)
C(6)—C(7)	1·382 (5)	1·405 (7)
C(6)—C(11)	1·408 (5)	1·404 (7)
C(7)—C(8)	1·379 (5)	1·367 (9)
C(7)—H(13)	0·76 (3)	0·89 (5)
C(8)—C(9)	1·367 (7)	1·375 (9)
C(8)—H(14)	0·93 (5)	0·96 (6)
C(9)—C(10)	1·360 (6)	1·401 (8)
C(9)—H(15)	0·89 (4)	0·96 (4)
C(10)—C(11)	1·393 (6)	1·358 (9)
C(10)—H(16)	1·10 (4)	0·94 (6)
C(11)—H(17)	0·88 (3)	0·95 (6)
C(3)—N(2)—C(5)	114·1 (3)	114·5 (4)
C(3)—N(2)—H(12)	122· (2)	123 (4)
C(5)—N(2)—H(12)	124· (2)	123 (4)
S(1)—C(3)—N(2)	122·0 (3)	122·0 (3)
S(1)—C(3)—C(4)	133·9 (3)	133·1 (4)
N(2)—C(3)—C(4)	104·1 (2)	104·9 (5)
C(3)—C(4)—C(4')	107·4 (2)	106·5 (4)
C(3)—C(4)—C(5')	144·4 (3)	143·6 (5)
C(4)—C(4')—C(5)	108·2 (2)	109·8 (4)
N(2)—C(5)—C(4')	106·2 (3)	104·2 (4)
N(2)—C(5)—C(6)	120·9 (3)	120·7 (4)
C(4)—C(5)—C(6)	132·9 (4)	135·1 (4)
C(5)—C(6)—C(7)	120·8 (3)	121·5 (5)
C(5)—C(6)—C(11)	121·8 (3)	122·2 (4)
C(7)—C(6)—C(11)	117·4 (4)	116·4 (5)
C(6)—C(7)—C(8)	121·6 (4)	121·4 (5)
C(6)—C(7)—H(13)	120 (3)	114 (3)
C(8)—C(7)—H(13)	118 (3)	123 (3)
C(7)—C(8)—C(9)	119·9 (4)	121·4 (5)
C(7)—C(8)—H(14)	116 (3)	123 (4)
C(9)—C(8)—H(14)	123 (3)	115 (5)
C(8)—C(9)—C(10)	120·8 (5)	118·2 (6)
C(8)—C(9)—H(15)	111 (2)	124 (2)
C(10)—C(9)—H(15)	127 (2)	118 (2)
C(9)—C(10)—C(11)	120·0 (4)	120·6 (5)
C(9)—C(10)—H(16)	126 (2)	109 (3)
C(11)—C(10)—H(16)	114 (2)	128 (3)
C(6)—C(11)—C(10)	120·3 (3)	122·0 (5)
C(6)—C(11)—H(17)	122 (3)	114 (3)
C(10)—C(11)—H(17)	118 (3)	121 (3)

Discussion.

Molecular geometry

The conformation of DPPT is given in Fig. 1, where the atoms are represented by displacement ellipsoids according to *ORTEP* (Johnson, 1971). The interatomic distances and bond angles together with their e.s.d.'s are listed for both modifications in Table 3. The DPPT molecule is not entirely planar, and the phenyl rings are twisted out of plane of the planar heterocyclic system by 13 and 7° in modifications (I) and (II), respectively. The phenyl rings are, however, parallel in both modifications because of the C_i molecular symmetry.

A slight difference in bond lengths is noted between the two modifications; in particular, the

C(4)—C(4') bond is longer in modification (I) than in modification (II) by 0·034 Å, which is larger than the standard deviation. A certain deviation of nearly the same order is also seen in the C(9)—C(10) and C(10)—C(11) bonds. On the other hand, no noticeable disagreement is found in bond angles which involve only non-H atoms. The bond parameters associated with H atoms vary greatly owing to the weak scattering power of hydrogen.

Arrangement of molecules

Figs. 2 and 3 show the stereodiagrams of the crystal structures of modifications (I) and (II), respectively. The overlap of the two molecules along the stacking axis is also shown in Fig. 4. The intermolecular distance between the heterocyclic ring systems is 3·46 Å in modification (I) and 3·35 Å in modification (II).

Modifications (I) and (II) are very similar in structure, except for the arrangement of molecules on the ac plane in Fig. 2 and the bc plane in Fig. 3. In these projections, all molecules are parallel in modification (I), whereas they are arranged in a zigzag fashion in modification (II).
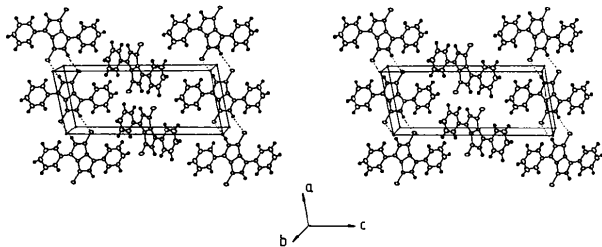

Fig. 2. Stereoview of the crystal structure of modification (I). The angle of the NH···S interaction is 162 (3)° and the distances between the S/H, N/H and N/S interactions are 2·53 (5), 0·94 (5) and 3·345 (3) Å, respectively. The intermolecular hydrogen bond is represented by dotted lines.

Hydrogen bonding

For the pigment application, the intermolecular hydrogen bond is extremely important to inhibit the invasion of solvent between the molecules. A strong intermolecular hydrogen bond is an indication of the low solubility of a pigment in organic solvents.

In Figs. 2 and 3, the hydrogen bonds based on NH···S are shown in dotted lines. The angles of the NH/S and the distances between S/H, N/H and N/S are as follows: modification (I): NH···S = 162°, S/H = 2·53, N/H = 0·94 and N/S = 3·435 Å and modification (II): NH···S = 150°, S/H = 2·59, N/H = 0·95 and N/S = 3·440 Å.

The hydrogen bonds in the above modifications are assumed to be rather weak for the following reasons: the NH···S hydrogen bond is known to be much smaller than NH···O because the S atom is less electronegative (Pauling, 1960). A strong intermolecular NH···O bond is generally linear (to within 10°) on the same axis, and the nitrogen–oxygen distance is about 2·8 Å. In addition, the hydrogen-bond-forming power decreases greatly as the atoms deviate from linearity (about 0·1 kcal mol^{-1} per 6°). The hydrogen bond in the present case is due to the NH···S interaction [3·435 and 3·440 Å in modifications (I) and (II), respectively], and the N, H and S atoms are neither on the same axis nor in the plane of the heterocyclic system. In fact, the solubility of DPPT in organic solvents is significantly higher than that of DPP based on NH···O, where the hydrogen-bonding atoms are in the plane of the heterocyclic system, and the NH···O length is 2·79 Å (Iqbal, Cassar, Rochat, Pfenninger & Wallquist, 1988).

Photoactive phase

The photoactive phase appears only as a result of the solvent-vapor treatment which accompanies the enhanced absorption and photoconduction in the near IR. An attempt was made to assign the diffraction patterns of the evaporated films before and after
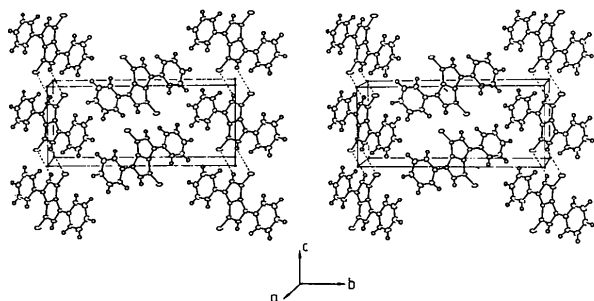

Fig. 3. Stereoview of the crystal structure of modification (II). The angle of the NH···S interaction is 150 (4)° and the distances between S/H, N/H and N/S interactions are 2·59 (5), 0·95 (5) and 3·440 (5) Å, respectively. The intermolecular hydrogen bond is represented by dotted lines.
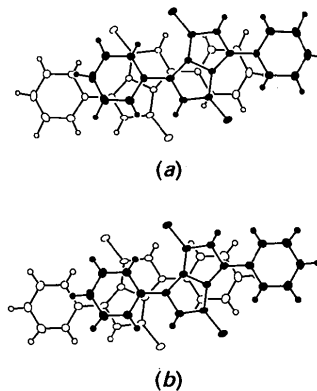

(a)

(b)

Fig. 4. Overlap of the two molecules along the stacking axis: (a) modification (I) and (b) modification (II).

the vapor treatment to the crystal parameters obtained here for modifications (I) and (II). However, neither of these modifications coincides with that of the photoactive phase caused by the vapor treatment.

The authors would like to express their sincere thanks to Messrs G. Giller, E. Baeriswyl and H. R. Walter for technical assistance.

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Structure of *trans*-2β-(4-Chlorophenyl)-1,3-dithiadecalin

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Abstract. 3-(4-Chlorophenyl)-2,4-dithiabicyclo[4.4.0]-decane, $C_{14}H_{17}ClS_2$, $M_r = 284 \cdot 9$, monoclinic, $P2_1/n$, $a = 6 \cdot 774$ (1), $b = 24 \cdot 307$ (2), $c = 8 \cdot 864$ (1) Å, $\beta = 108 \cdot 78$ (1)°, $V = 1381 \cdot 8$ (3) Å, $Z = 4$, $D_m = 1 \cdot 36$ (flotation in aqueous KI solution), $D_x = 1 \cdot 369$ Mg m^{-3}, λ(Cu $K\alpha$) = $1 \cdot 54178$ Å, $\mu = 5 \cdot 06$ mm^{-1}, $F(000) = 600$, $T = 291$ K, final $R = 0 \cdot 049$ ($wR = 0 \cdot 050$) for 1554 observed reflections. Both the cyclohexane and the dithiane ring of the bicyclic system have chair conformations. The equatorially attached phenyl ring is nearly eclipsed with respect to one of the C—S bonds of the dithiane ring with a corresponding torsion angle S—C(dithiane)—C(phenyl)—C(phenyl) of $-14 \cdot 6$ (6)°.

Introduction. This study is part of a series of structural investigations on the crystalline phases of mesogenic materials. The title compound itself does not form mesomorphic phases, but serves as a model structure for conformational analysis of a new class of liquid-crystalline heteroalicyclic substances containing the 2-(4-substituted phenyl)-1,3-dithiadecalin moiety (Tschierske, Joachimi, Vorbrodt, Zaschke, Wiegeleben, Hauser & Demus, 1989). The X-ray

crystal structure analysis reported in this paper was performed to obtain accurate geometrical parameters of the molecular structure for comparison with NMR measurements and a starting point for theoretical calculations, the results of which will be published elsewhere (Tschierske, Kleinpeter, Köhler & Zaschke, 1990).

Experimental. The title compound was synthesized by Tschierske (Tschierske, Joachimi, Vorbrodt, Zaschke, Wiegeleben, Hauser & Demus, 1989). A colourless optically clear crystal was cut to the dimensions $0 \cdot 34 \times 0 \cdot 34 \times 0 \cdot 13$ mm and mounted on a Syntex $P2_1$ diffractometer. Measurements were carried out with graphite-monochromatized Cu $K\alpha$ radiation (monochromator angle 26·57°), unit-cell parameters were determined by a least-squares treatment of the setting angles for 15 reflections in the 2θ range 20–30°. 1645 unique intensity data were measured for reflections with $2\theta < 115°$ and h, k, l ranging from $-7,0,0$ to 7,26,9. The intensities of the check reflections varied by $\pm 1 \cdot 8\%$ for $\bar{1}42$ and by $\pm 2 \cdot 1\%$ for $\bar{1}22$. 1558 reflections had intensities $I > 1 \cdot 96\sigma(I)$ and were considered observed. Lp correction was applied during data reduction.

The structure was solved by direct methods and refined by full-matrix least-squares methods on F with atomic scattering factors from *SHELX*76

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