Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl 2-[4-(5-methyl-1,3,4-oxadiazol-2-yl)phenoxy]propanoate

Rui-Dian Ma,* Bao-Lei Wang, Zheng-Ming Li and Hai-Bin Song

State Key Laboratory Institute of Elemento-Organic Chemistry, Nankai University, Tianjin 300071, People's Republic of China
Correspondence e-mail: nkumrd@yahoo.com.cn

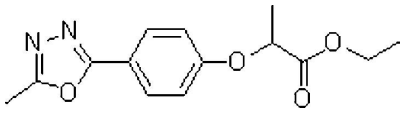
Received 25 April 2007; accepted 2 May 2007

Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.049; wR factor = 0.121; data-to-parameter ratio = 14.4.

The title compound, $C_{14}H_{16}N_2O_4$, is a herbicidal compound containing oxadiazole and benzene ring rings. X-ray analysis reveals weak C—H···N hydrogen-bonding interactions and the dihedral between oxadiazole ring and benzene ring is close to zero.

Related literature

For related literature, see: Wang *et al.* (2004); Wang *et al.* (2006).



Experimental

Crystal data

$C_{14}H_{16}N_2O_4$
M_r = 276.29
Monoclinic, $P2_1/c$
a = 15.7947 (15) Å
b = 7.8792 (5) Å
c = 10.8786 (12) Å
β = 91.190 (9)°
V = 1353.5 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 113 (2) K
0.32 × 0.24 × 0.20 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.969, T_{max} = 0.980
13948 measured reflections
2672 independent reflections
2314 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.121
S = 1.10
2672 reflections
186 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3A···N1^{i}	0.98	2.53	3.455 (2)	156

Symmetry code: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005).

This project was supported by the China 973 Program (2003CB114406).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2286).

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 doi:10.1107/S160053680702168X

supplementary materials

Acta Cryst. (2007). E**63**, o2897 [**doi:10.1107/S160053680702168X**]

Ethyl 2-[4-(5-methyl-1,3,4-oxadiazol-2-yl)phenoxy]propanoate

R.-D. Ma, B.-L. Wang, Z.-M. Li and H.-B. Song

Comment

Ketol-acid reductoisomerase (KARI) is a promising target for the design of herbicides because it is another essential enzyme for the synthesis of branched chain amino acids in plants and microorganisms yet absent in animals (Wang *et al.*, 2004). There are few literatures published about the molecular design of KARI inhibitors. In order to find new KARI inhibitors and novel herbicides, a series of 1,3,4-oxadiazole compounds containing ethyl phenoxylpropanate moiety were designed and synthesized based on the structures of monoamidines reported by (Wang *et al.*, 2006), and the bioassay results revealed that some of which showed favorable KARI inhibitory activities and herbicidal activities. The X-ray crystal structure determination of the title compound (I) was undertaken to investigate the relationship between structure and herbicidal activities.

The molecular structure of (I) is shown in Fig. 1. The X-ray analysis reveals that the conformation and weak C—H···N hydrogen-bonding interaction, and the dihedral between oxadiazole ring and benzene ring is approximately 180° suggesting that these two rings are almost at the same plane. The C1—C4 distance of 1.455 (2) Å is definitely bellow the normal C—C single-bond distance of 1.537 Å, which shows that C1—C4 is conjugated with the oxadiazole ring and benzene ring (Fig. 2 and Table 1).

Experimental

The title compound was synthesized by mixing ethyl 2-(4-(1*H*-tetrazol-5-yl) phenoxy) propanoate (0.53 g, 2 mmol) with acetic anhydride (20 ml). The mixture was stirred and refluxed for 3 h, and then water (20 ml) was poured into the reaction system and stirred for another 1 h at room temperature. After that, the mixture was extracted with dichloromethane (15 ml×3). The dichloromethane layer was dried over anhydrous sodium sulfate. Dichloromethane was then removed by distillation and the residue was crystallized from ethanol to give white crystals (m.p. 352 K, 0.29 g, 52.5% yield). Colorless single crystals of (I) suitable for X-ray diffraction analysis was obtained by once more recrystalliztion with ethyl acetate and petroleum ether. ^{1}H NMR ($CDCl_3$): δ 7.948 (d, *J*=8.7 Hz, 2H, Ph—H), 6.965 (d, *J*=8.7 Hz, Ph—H, 2H), 4.821 (q, *J*= 6.9 Hz, CH_2, H), 4.234 (q, *J*=6.9 Hz, CH, 2H), 2.593 (s, oxadiazole-CH_3, 3H), 1.658 (d, *J*=6.9 Hz, $CHCH_3$, 3H), 1.256 (t, *J*=6.9 Hz, CH_2CH_3, 3H); elemental analysis calculated for '$C_{14}H_{16}N_2O_4$': C 60.86, H 5.84, N 10.14%; found: C 60.93, H 5.79, N 10.15%.

Refinement

All H atoms were placed in calculated positions [C—H = 0.95, 0.98, 0.99 and 1.00Å for phenyl, methyl, methylene and methine H atoms, respectively] and included in the refinement using a riding model, with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(methyl C).

Figures

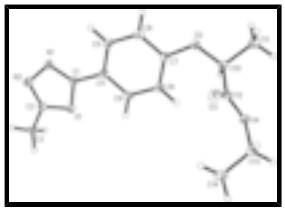

Fig. 1. View of the title compound (I), with displacement ellipsoids drawn at the 30% probability level.

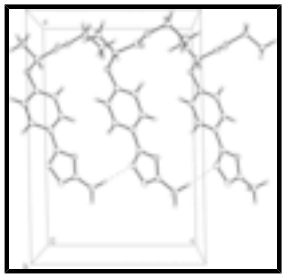

Fig. 2. View of the hydrogen bonding interactions shown with dashed lines of (I) in the unit-cell.

Fig. 3. Reaction scheme.

Ethyl 2-[4-(5-methyl-1,3,4-oxadiazol-2-yl)phenoxy]propanoate

Crystal data

$C_{14}H_{16}N_2O_4$	F_{000} = 584
M_r = 276.29	D_x = 1.356 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71070 Å
Hall symbol: -P 2ybc	Cell parameters from 3238 reflections
a = 15.7947 (15) Å	θ = 2.6–25.0º
b = 7.8792 (5) Å	μ = 0.10 mm^{-1}
c = 10.8786 (12) Å	T = 113 (2) K
β = 91.190 (9)º	Prism, colourless
V = 1353.5 (2) Å^3	0.32 × 0.24 × 0.20 mm
Z = 4	

Data collection

Rigaku Saturn diffractometer	2672 independent reflections
Radiation source: rotating anode	2314 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.046
T = 113(2) K	θ_{max} = 26.0º
ω scans	θ_{min} = 2.6º
Absorption correction: multi-scan (Jacobson, 1998)	h = −19→19
T_{min} = 0.969, T_{max} = 0.980	k = −9→9
13948 measured reflections	l = −13→13

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.049$

$wR(F^2) = 0.121$

$S = 1.10$

2672 reflections

186 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2 + 0.2367P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.19$ e Å^{-3}

$\Delta\rho_{min} = -0.18$ e Å^{-3}

Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.013 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.41602 (7)	0.58798 (14)	0.23101 (10)	0.0254 (3)
O2	0.77137 (7)	0.62012 (14)	−0.04248 (10)	0.0258 (3)
O3	0.87322 (8)	0.71131 (15)	0.15754 (11)	0.0326 (3)
O4	0.90040 (7)	0.43446 (14)	0.19355 (10)	0.0265 (3)
N1	0.38260 (9)	0.78071 (19)	0.09044 (13)	0.0287 (4)
N2	0.30974 (9)	0.74429 (19)	0.15997 (13)	0.0285 (4)
C1	0.44251 (10)	0.6862 (2)	0.13514 (14)	0.0234 (4)
C2	0.33238 (10)	0.6316 (2)	0.23984 (15)	0.0258 (4)
C3	0.28137 (11)	0.5489 (2)	0.33429 (16)	0.0326 (4)
H3A	0.3095	0.5619	0.4149	0.049*
H3B	0.2753	0.4280	0.3150	0.049*
H3C	0.2252	0.6018	0.3359	0.049*
C4	0.52943 (10)	0.6709 (2)	0.09470 (14)	0.0228 (4)
C5	0.55561 (11)	0.7610 (2)	−0.00833 (15)	0.0266 (4)
H5	0.5173	0.8355	−0.0497	0.032*

C6	0.63673 (11)	0.7423 (2)	−0.05021 (15)	0.0268 (4)
H6	0.6541	0.8040	−0.1203	0.032*
C7	0.69356 (10)	0.6328 (2)	0.00992 (14)	0.0223 (4)
C8	0.66890 (10)	0.5461 (2)	0.11445 (15)	0.0242 (4)
H8	0.7079	0.4746	0.1573	0.029*
C9	0.58680 (10)	0.5646 (2)	0.15578 (15)	0.0235 (4)
H9	0.5696	0.5041	0.2265	0.028*
C10	0.82978 (10)	0.4994 (2)	0.00736 (15)	0.0246 (4)
H10	0.8006	0.3886	0.0212	0.030*
C11	0.89808 (11)	0.4781 (2)	−0.08708 (16)	0.0313 (4)
H11A	0.8729	0.4329	−0.1634	0.047*
H11B	0.9413	0.3991	−0.0555	0.047*
H11C	0.9242	0.5884	−0.1034	0.047*
C12	0.86873 (10)	0.5645 (2)	0.12807 (15)	0.0237 (4)
C13	0.94262 (11)	0.4775 (2)	0.31044 (15)	0.0284 (4)
H13A	0.9739	0.5855	0.3016	0.034*
H13B	0.9840	0.3876	0.3328	0.034*
C14	0.87892 (11)	0.4950 (2)	0.41083 (16)	0.0312 (4)
H14A	0.9087	0.5080	0.4902	0.047*
H14B	0.8432	0.3933	0.4126	0.047*
H14C	0.8435	0.5950	0.3949	0.047*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0245 (6)	0.0287 (7)	0.0229 (6)	0.0020 (5)	−0.0024 (5)	−0.0003 (5)
O2	0.0232 (6)	0.0299 (7)	0.0243 (6)	0.0000 (5)	−0.0027 (5)	0.0044 (5)
O3	0.0395 (7)	0.0255 (7)	0.0325 (7)	−0.0021 (5)	−0.0083 (6)	0.0001 (5)
O4	0.0286 (6)	0.0278 (7)	0.0228 (6)	0.0025 (5)	−0.0036 (5)	0.0001 (5)
N1	0.0257 (8)	0.0307 (9)	0.0295 (8)	0.0028 (6)	−0.0032 (6)	0.0004 (6)
N2	0.0250 (8)	0.0325 (9)	0.0280 (8)	0.0013 (6)	−0.0028 (6)	−0.0024 (6)
C1	0.0280 (9)	0.0213 (9)	0.0208 (8)	−0.0008 (7)	−0.0047 (7)	−0.0015 (6)
C2	0.0246 (9)	0.0289 (10)	0.0237 (9)	0.0011 (7)	−0.0057 (7)	−0.0070 (7)
C3	0.0283 (10)	0.0426 (12)	0.0270 (10)	−0.0020 (8)	−0.0015 (8)	−0.0025 (8)
C4	0.0263 (9)	0.0213 (9)	0.0207 (8)	0.0000 (6)	−0.0053 (7)	−0.0036 (6)
C5	0.0287 (9)	0.0257 (9)	0.0250 (9)	0.0026 (7)	−0.0053 (7)	0.0021 (7)
C6	0.0311 (10)	0.0279 (10)	0.0212 (9)	−0.0022 (7)	−0.0035 (7)	0.0040 (7)
C7	0.0227 (9)	0.0227 (9)	0.0214 (8)	−0.0023 (6)	−0.0028 (6)	−0.0025 (6)
C8	0.0259 (9)	0.0231 (9)	0.0235 (9)	0.0007 (7)	−0.0054 (7)	0.0002 (7)
C9	0.0262 (9)	0.0245 (9)	0.0196 (8)	−0.0014 (7)	−0.0029 (7)	−0.0003 (6)
C10	0.0219 (9)	0.0257 (9)	0.0260 (9)	0.0003 (7)	−0.0033 (7)	0.0008 (7)
C11	0.0286 (9)	0.0381 (11)	0.0272 (9)	0.0004 (8)	−0.0020 (7)	−0.0027 (8)
C12	0.0206 (8)	0.0272 (10)	0.0232 (9)	0.0000 (6)	0.0003 (7)	0.0020 (7)
C13	0.0262 (9)	0.0351 (10)	0.0238 (9)	0.0006 (7)	−0.0054 (7)	−0.0003 (7)
C14	0.0336 (10)	0.0346 (10)	0.0254 (9)	0.0005 (8)	0.0000 (8)	−0.0021 (8)

Geometric parameters (Å, °)

O1—C2	1.3704 (19)	C6—C7	1.397 (2)

O1—C1	1.3710 (19)	C6—H6	0.9500
O2—C7	1.3691 (19)	C7—C8	1.389 (2)
O2—C10	1.4246 (19)	C8—C9	1.389 (2)
O3—C12	1.202 (2)	C8—H8	0.9500
O4—C12	1.3391 (19)	C9—H9	0.9500
O4—C13	1.4633 (19)	C10—C11	1.514 (2)
N1—C1	1.291 (2)	C10—C12	1.527 (2)
N1—N2	1.419 (2)	C10—H10	1.0000
N2—C2	1.288 (2)	C11—H11A	0.9800
C1—C4	1.455 (2)	C11—H11B	0.9800
C2—C3	1.471 (2)	C11—H11C	0.9800
C3—H3A	0.9800	C13—C14	1.506 (2)
C3—H3B	0.9800	C13—H13A	0.9900
C3—H3C	0.9800	C13—H13B	0.9900
C4—C9	1.393 (2)	C14—H14A	0.9800
C4—C5	1.396 (2)	C14—H14B	0.9800
C5—C6	1.377 (2)	C14—H14C	0.9800
C5—H5	0.9500		
C2—O1—C1	102.80 (13)	C7—C8—H8	120.2
C7—O2—C10	118.05 (12)	C8—C9—C4	120.75 (15)
C12—O4—C13	116.32 (13)	C8—C9—H9	119.6
C1—N1—N2	106.17 (14)	C4—C9—H9	119.6
C2—N2—N1	106.39 (13)	O2—C10—C11	106.30 (13)
N1—C1—O1	112.28 (15)	O2—C10—C12	110.42 (13)
N1—C1—C4	128.48 (15)	C11—C10—C12	109.92 (13)
O1—C1—C4	119.21 (14)	O2—C10—H10	110.0
N2—C2—O1	112.35 (15)	C11—C10—H10	110.0
N2—C2—C3	128.93 (16)	C12—C10—H10	110.0
O1—C2—C3	118.73 (15)	C10—C11—H11A	109.5
C2—C3—H3A	109.5	C10—C11—H11B	109.5
C2—C3—H3B	109.5	H11A—C11—H11B	109.5
H3A—C3—H3B	109.5	C10—C11—H11C	109.5
C2—C3—H3C	109.5	H11A—C11—H11C	109.5
H3A—C3—H3C	109.5	H11B—C11—H11C	109.5
H3B—C3—H3C	109.5	O3—C12—O4	125.11 (15)
C9—C4—C5	119.22 (15)	O3—C12—C10	124.98 (15)
C9—C4—C1	120.93 (15)	O4—C12—C10	109.85 (14)
C5—C4—C1	119.84 (14)	O4—C13—C14	110.61 (14)
C6—C5—C4	120.28 (15)	O4—C13—H13A	109.5
C6—C5—H5	119.9	C14—C13—H13A	109.5
C4—C5—H5	119.9	O4—C13—H13B	109.5
C5—C6—C7	120.30 (15)	C14—C13—H13B	109.5
C5—C6—H6	119.9	H13A—C13—H13B	108.1
C7—C6—H6	119.9	C13—C14—H14A	109.5
O2—C7—C8	125.03 (14)	C13—C14—H14B	109.5
O2—C7—C6	115.07 (14)	H14A—C14—H14B	109.5
C8—C7—C6	119.89 (15)	C13—C14—H14C	109.5
C9—C8—C7	119.51 (15)	H14A—C14—H14C	109.5
C9—C8—H8	120.2	H14B—C14—H14C	109.5

C1—N1—N2—C2	0.00 (17)	C10—O2—C7—C6	−174.42 (14)
N2—N1—C1—O1	−0.33 (18)	C5—C6—C7—O2	177.83 (14)
N2—N1—C1—C4	178.06 (15)	C5—C6—C7—C8	−1.8 (2)
C2—O1—C1—N1	0.51 (17)	O2—C7—C8—C9	−177.37 (14)
C2—O1—C1—C4	−178.05 (13)	C6—C7—C8—C9	2.2 (2)
N1—N2—C2—O1	0.32 (18)	C7—C8—C9—C4	−0.9 (2)
N1—N2—C2—C3	−179.63 (16)	C5—C4—C9—C8	−0.8 (2)
C1—O1—C2—N2	−0.50 (17)	C1—C4—C9—C8	177.84 (14)
C1—O1—C2—C3	179.46 (14)	C7—O2—C10—C11	164.65 (13)
N1—C1—C4—C9	178.51 (16)	C7—O2—C10—C12	−76.17 (17)
O1—C1—C4—C9	−3.2 (2)	C13—O4—C12—O3	0.7 (2)
N1—C1—C4—C5	−2.9 (3)	C13—O4—C12—C10	178.02 (12)
O1—C1—C4—C5	175.41 (14)	O2—C10—C12—O3	−23.0 (2)
C9—C4—C5—C6	1.2 (2)	C11—C10—C12—O3	94.0 (2)
C1—C4—C5—C6	−177.42 (15)	O2—C10—C12—O4	159.74 (13)
C4—C5—C6—C7	0.1 (2)	C11—C10—C12—O4	−83.30 (17)
C10—O2—C7—C8	5.2 (2)	C12—O4—C13—C14	83.97 (17)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3A···N1^{i}	0.98	2.53	3.455 (2)	156

Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2.

Fig. 1

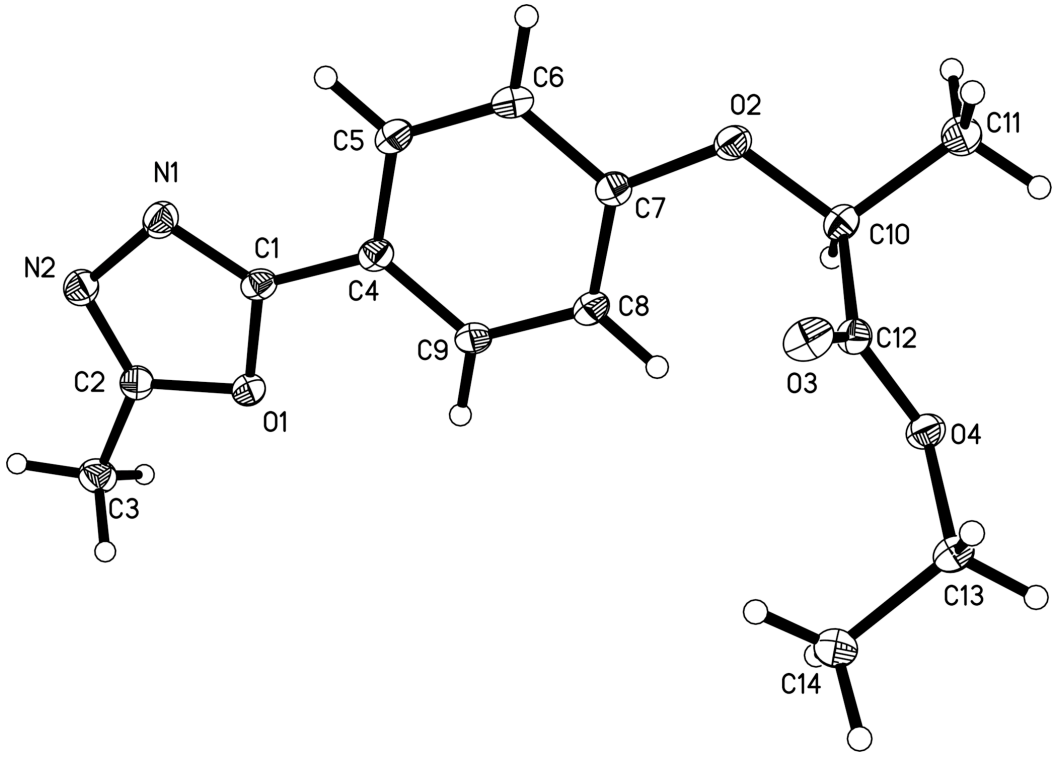

Fig. 2

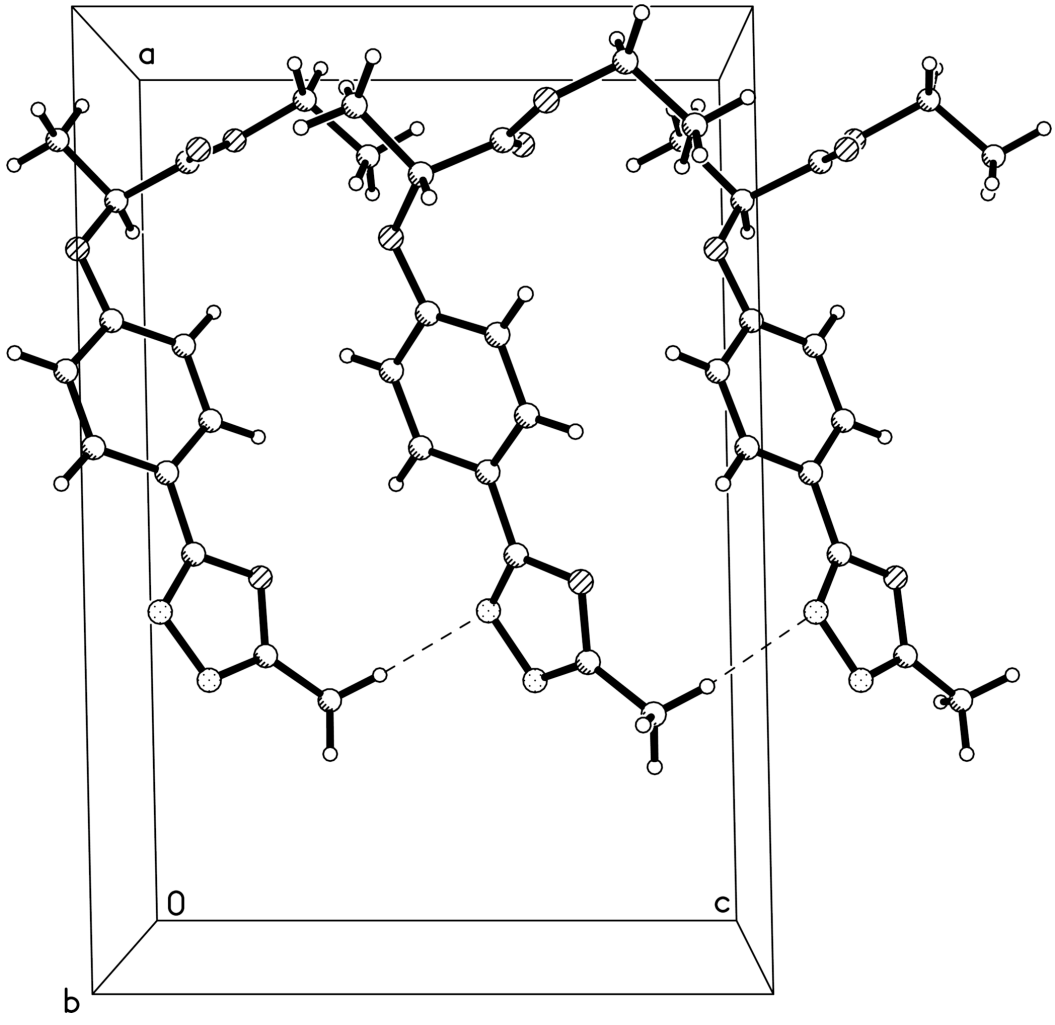

Fig. 3