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(2*E*)-1-(3-Methyl-2-thienyl)-3-(3-nitrophenyl)prop-2-en-1-one

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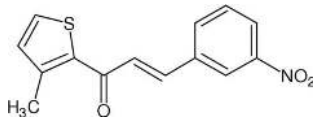
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.004 Å; R factor = 0.059; wR factor = 0.161; data-to-parameter ratio = 13.0.

Chalcones are a major class of natural products having interesting pharmaceutical activities. The title compound, $C_{14}H_{11}NO_3S$, is of interest as a potential bioactive agent. The central acyclic C=C double bond is *trans* configured. All non-H atoms lie in a common plane (r.m.s. deviation 0.075 Å). In the crystal structure, the molecules form a herringbone pattern.

Related literature

For related structures, see: Yathirajan *et al.* (2006); Yathirajan, Mayekar, Narayana *et al.* (2007); Fischer *et al.* (2007); Yathirajan, Mayekar, Sarojini *et al.* (2007); Sarojini *et al.* (2007). For pharmacological activities, see: Di Carlo *et al.* (1999); for bioactvities of chalcones, see: Dimmock *et al.* (1999); Go *et al.* (2005); for anti-infective and anti-inflammatory activities, see: Nowakowska (2007); for cancer chemopreventive agents, see: Won *et al.* (2005); for HIV-1 integrase inhibitors, see: Deng *et al.* (2007); for potent tyrosinase inhibitors, see: Khatib *et al.* (2005); for the excellent blue light transmittance and good crystallizability of chalcones, see: Fichou *et al.* (1988); Goto *et al.* (1991); Sarojini *et al.* (2006).



Experimental

Crystal data

$C_{14}H_{11}NO_3S$
M_r = 273.30
Monoclinic, $P2_1/c$
a = 13.9340 (18) Å
b = 5.4166 (4) Å
c = 17.789 (2) Å
β = 108.130 (10)°
V = 1276.0 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 173 (2) K
0.49 × 0.48 × 0.48 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.885, T_{max} = 0.887
6347 measured reflections
2252 independent reflections
1989 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.161
S = 1.04
2252 reflections
173 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2146).

References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Deng, J., Dayam, R., Al-Mawsawi, L. Q. & Neamati, N. (2007). *Curr. Pharm. Des.* **13**, 129–141.
Di Carlo, G., Mascolo, N., Izzo, A. A. & Capasso, F. (1999). *Life Sci.* **65**, 337–353.
Dimmock, J. R., Elias, D. W., Beazely, M. A. & Kandepu, N. M. (1999). *Curr. Med. Chem.* **6**, 1125–1149.
Fichou, D., Watanabe, T., Takeda, T., Miyata, S., Goto, Y. & Nakayama, M. (1988). *Jpn. J. Appl. Phys.* **27**, 429–430.
Fischer, A., Yathirajan, H. S., Ashalatha, B. V., Narayana, B. & Sarojini, B. K. (2007). *Acta Cryst.* E**63**, o1351–o1352.
Go, M. L., Wu, X. & Liu, X. L. (2005). *Curr. Med. Chem.* **12**, 483–499.
Goto, Y., Hayashi, A., Kimura, Y. & Nakayama, M. (1991). *J. Cryst. Growth*, **108**, 688–698.
Khatib, S., Nerya, O., Musa, R., Shmuel, M., Tamir, S. & Vaya, J. (2005). *Bioorg. Med. Chem.* **13**, 433–441.
Nowakowska, Z. (2007). *Eur. J. Med. Chem.* **42**, 125–137.
Sarojini, B. K., Narayana, B., Ashalatha, B. V., Indira, J. & Lobo, K. G. (2006). *J. Cryst. Growth*, **295**, 54–59.
Sarojini, B. K., Yathirajan, H. S., Lakshmana, K., Narayana, B. & Bolte, M. (2007). *Acta Cryst.* E**63**, o3211.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
Won, S. J., Liu, C. T., Tsao, L. T., Weng, J. R., Ko, H. H., Wang, J. P. & Lin, C. N. (2005). *Eur. J. Med. Chem.* **40**, 103–112.
Yathirajan, H. S., Ashalatha, B., Narayana, B., Bindya, S. & Bolte, M. (2006). *Acta Cryst.* E**62**, o4551–o4553.
Yathirajan, H. S., Mayekar, A. N., Narayana, B., Sarojini, B. K. & Bolte, M. (2007). *Acta Cryst.* E**63**, o2200–o2201.
Yathirajan, H. S., Mayekar, A. N., Sarojini, B. K., Narayana, B. & Bolte, M. (2007). *Acta Cryst.* E**63**, o424–o425.

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supplementary materials

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(2*E*)-1-(3-Methyl-2-thienyl)-3-(3-nitrophenyl)prop-2-en-1-one

B. K. Sarojini, B. Narayana, A. M. Vijesh, H. S. Yathirajan and M. Bolte

Comment

Chalcones are one of the major classes of natural products with widespread distribution in fruits, vegetables, spices, tea and soy based foodstuff have been recently subjects of great interest for their interesting pharmacological activities. A vast number of naturally occurring chalcones are polyhydroxylated in the aryl rings. The radical quenching properties of the phenolic groups present in many chalcones have raised interest in using the compounds or chalcone rich plant extracts as drugs or food preservatives. Reviews on the bioactivities of varieties of chalcones are given by Dimmock *et al.* (1999) and Go *et al.* (2005). Recently, it has been noted that, among many organic compounds reported for their second harmonic generation, chalcone derivatives are known for their excellent blue light transmittance and good crystallizability. Thiophene analogs of antiviral chalcones have been reported. In continuation of our work on chalcones, the present paper reports the crystal structure of a newly synthesized chalcone.

Geometric parameters of the title compound (Fig. 2) are in the usual ranges. All non-H atoms lie in a common plane (r.m.s. deviation 0.075 Å). In the crystal, the molecules crystallize in a herringbone pattern.

Experimental

To a thoroughly stirred solution of 1-(3-methyl-2-thienyl)ethanone (1.40 g, 0.01 mol) and 3-nitrobenzaldehyde (1.51 g, 0.01 mol) in 25 ml me thanol, 5 ml of 40% KOH solution was added, stirred overnight and filtered. The product was crystallized from acetone (m.p.: 395–397 K). Analysis for $C_{14}H_{11}NO_3S$: Found (Calculated): C: 61.47 (61.52); H: 4.00 (4.06); S: 11.62% (11.73%).

Refinement

H atoms were found in a difference map, but they were refined using a riding model with C—H = 0.95Å and U_{iso}(H) = 1.2U_{eq}(C) [C—H = 0.98Å and U_{iso}(H) = 1.5U_{eq}(C) for the methyl group, which was allowed to rotate but not to tip].

Figures

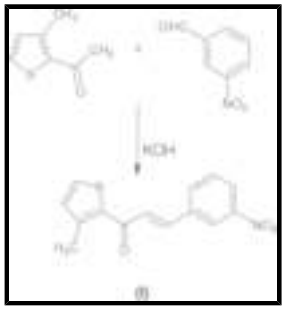


Fig. 1. Reaction scheme.

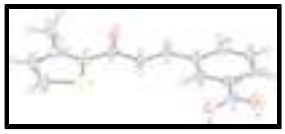


Fig. 2. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.

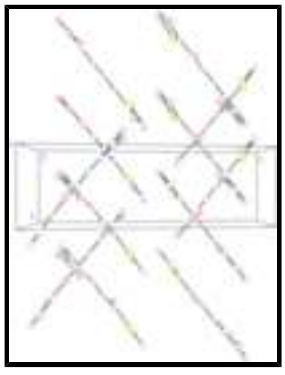


Fig. 3. Packing diagram of the title compound with view onto the *bc* plane.

(2E)-1-(3-Methyl-2-thienyl)-3-(3-nitrophenyl)prop-2-en-1-one

Crystal data

$C_{14}H_{11}NO_3S$	F_{000} = 568
M_r = 273.30	D_x = 1.423 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 6618 reflections
a = 13.9340 (18) Å	θ = 4.0–25.3º
b = 5.4166 (4) Å	μ = 0.26 mm^{-1}
c = 17.789 (2) Å	T = 173 (2) K
β = 108.130 (10)º	Block, colourless
V = 1276.0 (2) Å^3	0.49 × 0.48 × 0.48 mm
Z = 4	

Data collection

STOE IPDS II two-circle diffractometer	2252 independent reflections
Radiation source: fine-focus sealed tube	1989 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.060
T = 173(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 4.0º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −16→15
T_{min} = 0.885, T_{max} = 0.887	k = −6→5
6347 measured reflections	l = −21→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.059$	H-atom parameters constrained
$wR(F^2) = 0.161$	$w = 1/[\sigma^2(F_o^2) + (0.0815P)^2 + 1.859P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
2252 reflections	$\Delta\rho_{max} = 0.65$ e Å^{-3}
173 parameters	$\Delta\rho_{min} = -0.50$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > 2$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}
S1	0.65303 (5)	0.90494 (15)	0.09287 (4)	0.0336 (3)
N1	0.53891 (18)	0.0052 (5)	0.33857 (14)	0.0337 (6)
O1	0.92587 (15)	0.6119 (4)	0.17453 (14)	0.0389 (6)
O2	0.51014 (18)	−0.1315 (6)	0.38191 (15)	0.0560 (7)
O3	0.48346 (16)	0.1519 (5)	0.29313 (14)	0.0430 (6)
C1	0.8357 (2)	0.6517 (5)	0.16477 (16)	0.0262 (6)
C2	0.7792 (2)	0.4971 (5)	0.20648 (15)	0.0246 (6)
H2	0.7098	0.5291	0.1991	0.030*
C3	0.8258 (2)	0.3148 (5)	0.25392 (15)	0.0240 (6)
H3	0.8950	0.2903	0.2588	0.029*
C11	0.7828 (2)	0.8523 (5)	0.11302 (16)	0.0264 (6)
C12	0.8250 (2)	1.0284 (5)	0.07533 (16)	0.0312 (7)
C13	0.7517 (2)	1.1975 (6)	0.03129 (16)	0.0345 (7)
H13	0.7670	1.3292	0.0017	0.041*
C14	0.6564 (3)	1.1539 (6)	0.03523 (17)	0.0357 (7)
H14	0.5990	1.2511	0.0089	0.043*
C15	0.9313 (3)	1.0447 (6)	0.08332 (18)	0.0397 (8)
H15A	0.9700	1.0738	0.1389	0.060*
H15B	0.9425	1.1815	0.0510	0.060*
H15C	0.9536	0.8899	0.0655	0.060*
C21	0.78249 (19)	0.1471 (5)	0.29973 (15)	0.0227 (6)
C22	0.6811 (2)	0.1600 (5)	0.29780 (15)	0.0242 (6)

H22	0.6373	0.2830	0.2674	0.029*
C23	0.6465 (2)	−0.0106 (5)	0.34115 (15)	0.0250 (6)
C24	0.7056 (2)	−0.1942 (5)	0.38641 (16)	0.0284 (6)
H24	0.6786	−0.3086	0.4150	0.034*
C25	0.8057 (2)	−0.2053 (6)	0.38861 (16)	0.0309 (7)
H25	0.8486	−0.3295	0.4191	0.037*
C26	0.8442 (2)	−0.0355 (5)	0.34632 (16)	0.0274 (6)
H26	0.9133	−0.0441	0.3492	0.033*

Atomic displacement parameters (Å^2)

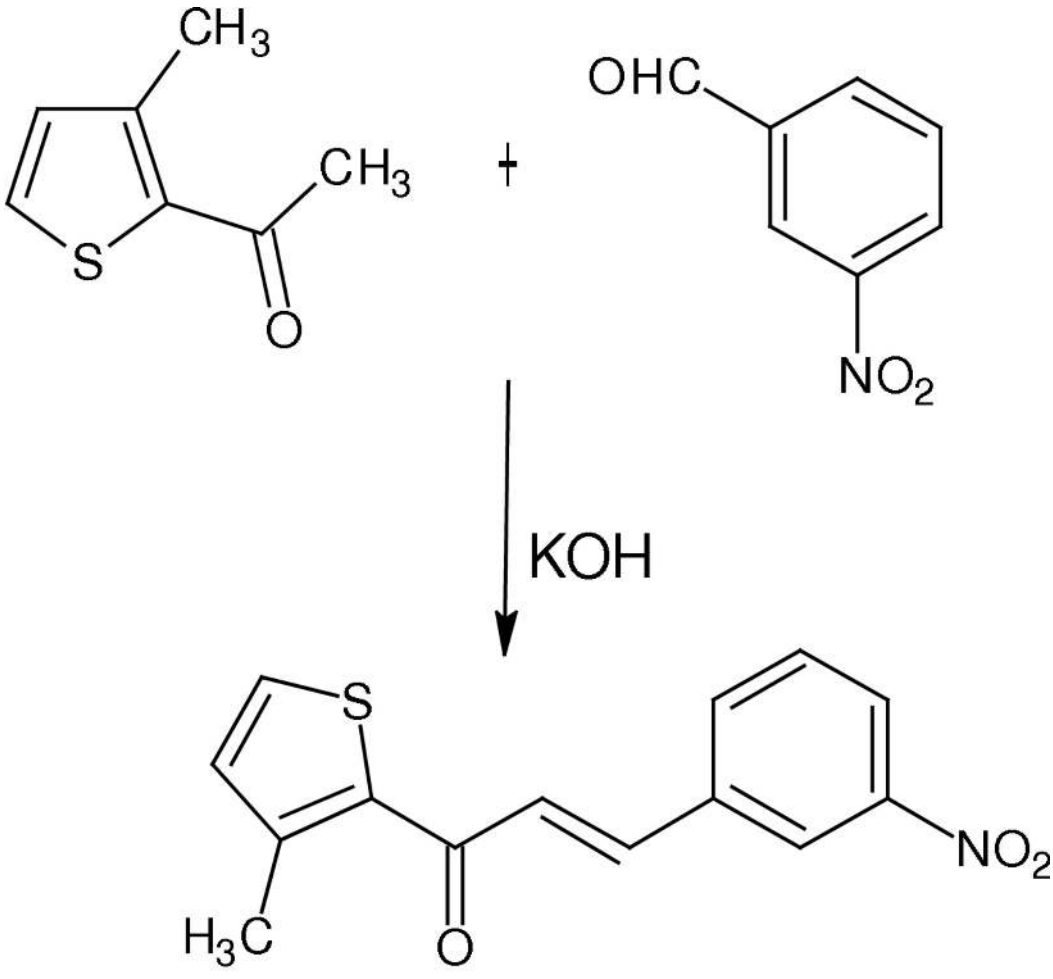
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0270 (4)	0.0387 (5)	0.0377 (4)	0.0044 (3)	0.0138 (3)	0.0058 (3)
N1	0.0249 (13)	0.0450 (15)	0.0345 (13)	−0.0067 (12)	0.0139 (10)	0.0022 (12)
O1	0.0269 (11)	0.0392 (12)	0.0567 (14)	−0.0010 (9)	0.0221 (10)	0.0088 (10)
O2	0.0361 (13)	0.0800 (19)	0.0592 (15)	−0.0079 (13)	0.0256 (12)	0.0275 (14)
O3	0.0255 (11)	0.0508 (14)	0.0569 (14)	0.0044 (10)	0.0190 (10)	0.0136 (12)
C1	0.0250 (14)	0.0273 (14)	0.0302 (13)	−0.0047 (11)	0.0144 (11)	−0.0038 (11)
C2	0.0200 (13)	0.0282 (14)	0.0291 (13)	−0.0029 (11)	0.0128 (11)	−0.0011 (11)
C3	0.0195 (12)	0.0270 (14)	0.0282 (13)	−0.0033 (11)	0.0115 (11)	−0.0042 (11)
C11	0.0303 (14)	0.0261 (14)	0.0269 (13)	−0.0043 (12)	0.0150 (11)	−0.0046 (11)
C12	0.0460 (17)	0.0263 (14)	0.0249 (13)	−0.0047 (13)	0.0165 (12)	−0.0045 (11)
C13	0.0502 (19)	0.0293 (15)	0.0250 (14)	0.0011 (14)	0.0130 (13)	0.0020 (12)
C14	0.0436 (17)	0.0346 (16)	0.0301 (15)	0.0052 (14)	0.0134 (13)	0.0028 (13)
C15	0.052 (2)	0.0311 (16)	0.0302 (15)	0.0027 (15)	0.0044 (14)	0.0014 (13)
C21	0.0211 (13)	0.0250 (13)	0.0230 (12)	−0.0022 (11)	0.0083 (10)	−0.0038 (10)
C22	0.0219 (13)	0.0272 (14)	0.0242 (12)	−0.0017 (11)	0.0082 (10)	−0.0004 (11)
C23	0.0223 (13)	0.0301 (14)	0.0250 (13)	−0.0055 (11)	0.0109 (11)	−0.0036 (11)
C24	0.0338 (15)	0.0289 (15)	0.0251 (13)	−0.0053 (12)	0.0131 (11)	−0.0004 (11)
C25	0.0338 (16)	0.0291 (15)	0.0302 (14)	0.0068 (12)	0.0106 (12)	0.0037 (12)
C26	0.0252 (14)	0.0306 (15)	0.0287 (14)	0.0010 (12)	0.0114 (11)	0.0004 (11)

Geometric parameters (Å, °)

S1—C14	1.703 (3)	C13—H13	0.9500
S1—C11	1.754 (3)	C14—H14	0.9500
N1—O3	1.222 (3)	C15—H15A	0.9800
N1—O2	1.223 (3)	C15—H15B	0.9800
N1—C23	1.488 (3)	C15—H15C	0.9800
O1—C1	1.233 (3)	C21—C26	1.400 (4)
C1—C11	1.466 (4)	C21—C22	1.404 (4)
C1—C2	1.496 (4)	C22—C23	1.383 (4)
C2—C3	1.330 (4)	C22—H22	0.9500
C2—H2	0.9500	C23—C24	1.379 (4)
C3—C21	1.470 (4)	C24—C25	1.385 (4)
C3—H3	0.9500	C24—H24	0.9500
C11—C12	1.396 (4)	C25—C26	1.396 (4)
C12—C13	1.414 (4)	C25—H25	0.9500
C12—C15	1.446 (5)	C26—H26	0.9500

C13—C14	1.372 (5)		
C14—S1—C11	91.85 (15)	C12—C15—H15A	109.5
O3—N1—O2	123.2 (2)	C12—C15—H15B	109.5
O3—N1—C23	118.8 (2)	H15A—C15—H15B	109.5
O2—N1—C23	118.1 (3)	C12—C15—H15C	109.5
O1—C1—C11	120.6 (2)	H15A—C15—H15C	109.5
O1—C1—C2	120.2 (3)	H15B—C15—H15C	109.5
C11—C1—C2	119.2 (2)	C26—C21—C22	118.5 (2)
C3—C2—C1	120.1 (2)	C26—C21—C3	118.8 (2)
C3—C2—H2	120.0	C22—C21—C3	122.6 (2)
C1—C2—H2	120.0	C23—C22—C21	118.4 (3)
C2—C3—C21	127.3 (2)	C23—C22—H22	120.8
C2—C3—H3	116.3	C21—C22—H22	120.8
C21—C3—H3	116.3	C24—C23—C22	124.0 (3)
C12—C11—C1	127.1 (3)	C24—C23—N1	118.2 (2)
C12—C11—S1	110.6 (2)	C22—C23—N1	117.8 (2)
C1—C11—S1	122.3 (2)	C23—C24—C25	117.4 (3)
C11—C12—C13	111.7 (3)	C23—C24—H24	121.3
C11—C12—C15	124.5 (3)	C25—C24—H24	121.3
C13—C12—C15	123.8 (3)	C24—C25—C26	120.6 (3)
C14—C13—C12	113.8 (3)	C24—C25—H25	119.7
C14—C13—H13	123.1	C26—C25—H25	119.7
C12—C13—H13	123.1	C25—C26—C21	121.1 (3)
C13—C14—S1	112.0 (2)	C25—C26—H26	119.5
C13—C14—H14	124.0	C21—C26—H26	119.5
S1—C14—H14	124.0		
O1—C1—C2—C3	−0.4 (4)	C2—C3—C21—C26	179.6 (3)
C11—C1—C2—C3	−179.7 (2)	C2—C3—C21—C22	0.6 (4)
C1—C2—C3—C21	179.5 (2)	C26—C21—C22—C23	−0.9 (4)
O1—C1—C11—C12	−5.6 (4)	C3—C21—C22—C23	178.1 (2)
C2—C1—C11—C12	173.7 (3)	C21—C22—C23—C24	−0.1 (4)
O1—C1—C11—S1	176.3 (2)	C21—C22—C23—N1	180.0 (2)
C2—C1—C11—S1	−4.4 (4)	O3—N1—C23—C24	−173.3 (3)
C14—S1—C11—C12	0.4 (2)	O2—N1—C23—C24	6.1 (4)
C14—S1—C11—C1	178.8 (2)	O3—N1—C23—C22	6.6 (4)
C1—C11—C12—C13	−178.7 (3)	O2—N1—C23—C22	−173.9 (3)
S1—C11—C12—C13	−0.4 (3)	C22—C23—C24—C25	0.5 (4)
C1—C11—C12—C15	−1.7 (5)	N1—C23—C24—C25	−179.6 (2)
S1—C11—C12—C15	176.6 (2)	C23—C24—C25—C26	0.1 (4)
C11—C12—C13—C14	0.3 (4)	C24—C25—C26—C21	−1.1 (4)
C15—C12—C13—C14	−176.8 (3)	C22—C21—C26—C25	1.5 (4)
C12—C13—C14—S1	0.0 (3)	C3—C21—C26—C25	−177.6 (2)
C11—S1—C14—C13	−0.2 (2)		

Fig. 1



(I)

Fig. 2

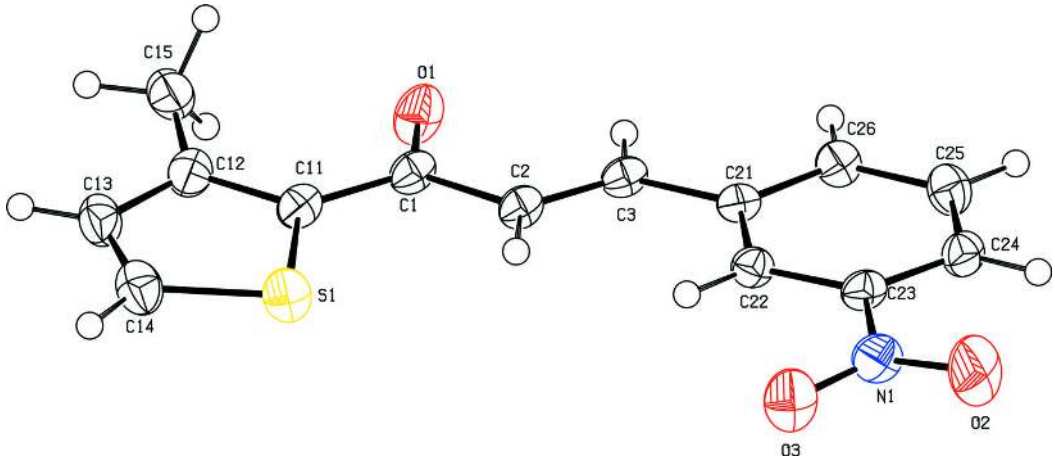


Fig. 3