# 3-[4-(Methylsulfanyl)phenyl]-1-(4-nitrophenyl)-prop-2-en-1-one

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The geometrical parameters for the title compound, $C_{16}H_{13}NO_2S$, are normal. The non-centrosymmetric crystal packing, which is consistent with the non-zero second harmonic generation response, may be influenced by a weak intermolecular C—H···O interaction.

## Comment

The title compound, (I) (Fig. 1), was prepared as part of our ongoing studies (Harrison *et al.*, 2005, 2006) of the non-linear optical (NLO) properties and crystal structures of chalcone derivatives. It is known that substitution at either benzene ring of the chalcone skeleton substantially affects the optical response (Uchida *et al.*, 1998) and we are now exploring the role of the methylsulfanyl ($H_3CS–$) substituent (Butcher *et al.*, 2006) in this process.



(I)
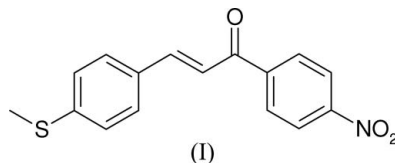
The non-centrosymmetric polar space group of (I) is consistent with its significant second harmonic generation (SHG) response of 0.6 times that of urea (Watson *et al.*, 1993). The geometrical parameters for (I) fall within their expected ranges (Allen *et al.*, 1987). The molecule of (I) is distinctly twisted about the C6—C7 and the C9—C10 bonds (Table 1). The dihedral angle between the benzene ring mean planes (C1–C6 and C10–C15) in (I) is 45.84 (4)°, which is significantly smaller than the equivalent value of 68.15 (6)° in 2-bromo-1-(4-methylphenyl)-3-[4-(methylsulfanyl)phenyl]prop-2-en-1-one (Butcher *et al.*, 2006). The nitro group in (I) is well ordered and makes a dihedral angle of 12.94 (15)° with respect to the C10–C15 benzene ring. The C16 methyl group is almost in the plane of the C1–C6 benzene ring [deviation = 0.049 (4) Å].

A *PLATON* (Spek, 2003) analysis of the crystal structure of (I) indicates a possible intermolecular C—H···O interaction (Table 2) that might help to establish the crystal packing (Fig. 2). The C—H···O interaction forms extended chains of molecules propagating along [001]. Adjacent chains form pseudo-layers in (100), with all the molecules oriented in the same sense with respect to the polar axis.

## Experimental

To a mixture of 4-(methylsulfanyl)benzaldehyde (1.52 g, 0.01 mol) and 4-nitroacetophenone (1.65 g, 0.01 mol) in ethanol (5 ml), a

solution of potassium hydroxide (5%, 5 ml) was added slowly with stirring. The mixture was stirred at room temperature for 24 h. The precipitated solid was filtered off, washed with water, dried and recrystallized from an acetone–toluene (1:1 $v/v$) solvent mixture (yield 86%; m.p. 409 K). Analysis for $C_{16}H_{13}NO_3S$ found (calculated) (%): C 64.15 (64.20), H 4.32 (4.38), N 4.66 (4.68).

*Crystal data*

| | |
|---|---|
| $C_{16}H_{13}NO_3S$ | $Z = 8$ |
| $M_r = 299.33$ | $D_x = 1.448$ Mg m$^{-3}$ |
| Orthorhombic, $Aba2$ | Mo $K\alpha$ radiation |
| $a = 13.7388 (4)$ Å | $\mu = 0.25$ mm$^{-1}$ |
| $b = 33.5802 (8)$ Å | $T = 120 (2)$ K |
| $c = 5.9538 (2)$ Å | Plate, yellow |
| $V = 2746.80 (14)$ Å$^3$ | $0.28 \times 0.24 \times 0.03$ mm |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | 27858 measured reflections |
| $\omega$ and $\varphi$ scans | 3173 independent reflections |
| Absorption correction: multi-scan | 2811 reflections with $I > 2\sigma(I)$ |
| ($SADABS$; Bruker, 2003) | $R_{int} = 0.063$ |
| $T_{min} = 0.936$, $T_{max} = 0.993$ | $\theta_{max} = 27.6°$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0338P)^2$ |
| $R[F^2 > 2\sigma(F^2)] = 0.035$ | $+ 1.763P]$ |
| $wR(F^2) = 0.081$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.06$ | $(\Delta/\sigma)_{max} = 0.001$ |
| 3173 reflections | $\Delta\rho_{max} = 0.29$ e Å$^{-3}$ |
| 191 parameters | $\Delta\rho_{min} = -0.21$ e Å$^{-3}$ |
| H-atom parameters constrained | Absolute structure: Flack (1983), |
| | 1416 Friedel pairs |
| | Flack parameter: 0.04 (8) |

**Table 1**
Selected torsion angles (°).

| | | | |
|---|---|---|---|
| C5—C6—C7—C8 | 10.3 (3) | O1—C9—C10—C15 | 21.2 (3) |
| C7—C8—C9—O1 | 11.4 (3) | | |

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| C11—H11$\cdots$O1$^i$ | 0.95 | 2.60 | 3.278 (3) | 129 |

Symmetry code: (i) $x, y, z + 1$.

A handful of reflections weakly violated the $h00$ ($h \neq 2n$) systematic absence condition for the space group $Aba2$. Attempts to develop a model in lower-symmetry space groups were not successful. The H atoms were positioned geometrically (C—H = 0.95–0.98 Å) and refined as riding, with $U_{iso}$(H) = $1.2U_{eq}$(carrier) or $1.5U_{eq}$(methyl). The methyl group was allowed to rotate but not to tip to best fit the electron density.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997), and *SORTAV* (Blessing, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.
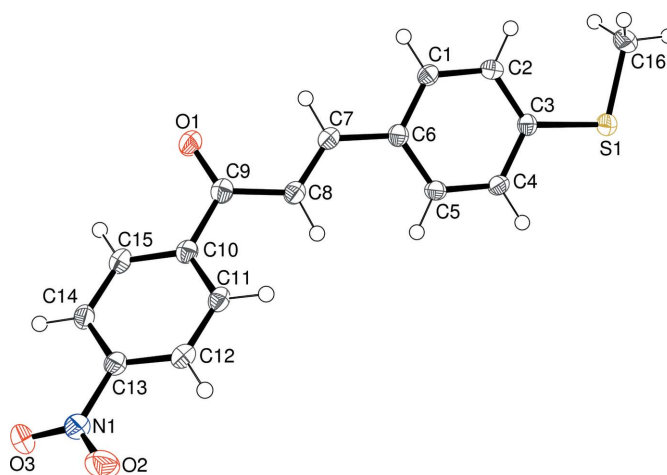


**Figure 1**
Molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).
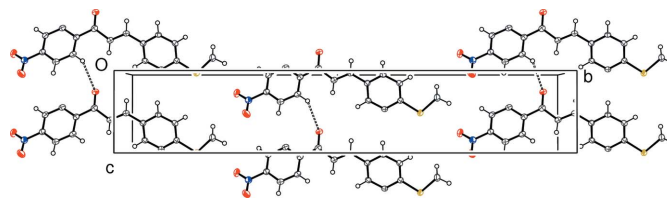


**Figure 2**
Part of a (100) sheet of molecules in (I) with C—H$\cdots$O interactions shown as dashed lines.

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