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3-(4-Methoxyphenyl)-4-(5-phenyl-2,4-pentadienoyl)sydnone

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Key indicators

Single-crystal X-ray study

$T = 160$ K

Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å

R factor = 0.044

wR factor = 0.119

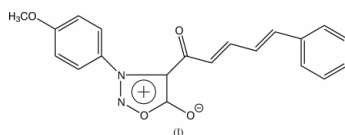
Data-to-parameter ratio = 7.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The dihedral angle between the sydnone moiety and methoxyphenyl ring of the title compound, $\text{C}_{20}\text{H}_{16}\text{N}_2\text{O}_4$, is $66.32(10)^\circ$. Electron conjugation is observed between the pentadienone group and the attached phenyl ring. In the crystal structure, symmetry-related molecules are linked by weak intermolecular $\text{C}-\text{H} \cdots \text{O}$ interactions to form a sheet-like structure, which lies parallel to the ab plane.

Comment

Sydnones are the product of dehydration of *N*-nitroso- α -amino acids and are named after the site of their discovery at the University of Sydney (Earl & Mackney, 1935). They have attained importance due, not only to their structural features and chemical properties, but also to their biological properties. Sydnones are less toxic (Pillai *et al.*, 1993), but potent porphyrinogenic (Sutherland *et al.*, 1986; Marks, 1987) and anti-inflammatory (Satyanarayana & Rao, 1995) compounds, and have the effect of scavenging free radicals (Narla & Rao, 1995). Sydnone halogen derivatives change their colour irreversibly under the influence of UV light ($\lambda < 400$ nm) (Hašek *et al.*, 1979). In view of its biological importance, the crystal structure analysis of the title sydnone, (I), was carried out.



The bond lengths in the sydnone ring of (I) agree with the corresponding average values given in Table 1 for other 3,4-disubstituted sydnones (Hašek *et al.*, 1978, 1979; Ueng *et al.*, 1987*a,b*, 1989; Ajees *et al.*, 2002). The dihedral angles between the planes of the sydnone ring and the attached phenyl ring in the 3,4-disubstituted sydnone derivatives (55 – 79°) are larger than those in the 3-substituted sydnone derivatives (2 – 39° ; Ueng *et al.*, 1987*a*). In (I), the dihedral angle between the planes of the sydnone moiety and the methoxyphenyl ring is $66.32(10)^\circ$. The carbonyl group ($\text{C}14=\text{O}14$) is in an *s-cis* conformation, as can be seen from the torsion angle $\text{C}16-\text{C}15-\text{C}14-\text{O}14$ of $-1.5(4)^\circ$. The pentadienone moiety is effectively planar and its mean plane is oriented at angles of $10.98(17)$ and $6.57(18)^\circ$ with respect to those of the sydnone and methoxyphenyl rings, respectively. The lengths of the $\text{C}19-\text{C}18$ [$1.462(4)$ Å], $\text{C}18-\text{C}17$ [$1.333(5)$ Å], $\text{C}17-\text{C}16$ [$1.435(4)$ Å], $\text{C}16-\text{C}15$ [$1.330(4)$ Å], $\text{C}15-\text{C}14$ [$1.471(3)$ Å], $\text{C}14-\text{O}14$ [$1.228(4)$ Å] and $\text{C}14-\text{C}4$ [$1.457(4)$ Å] bonds indicate conjugation. Atoms H15 and

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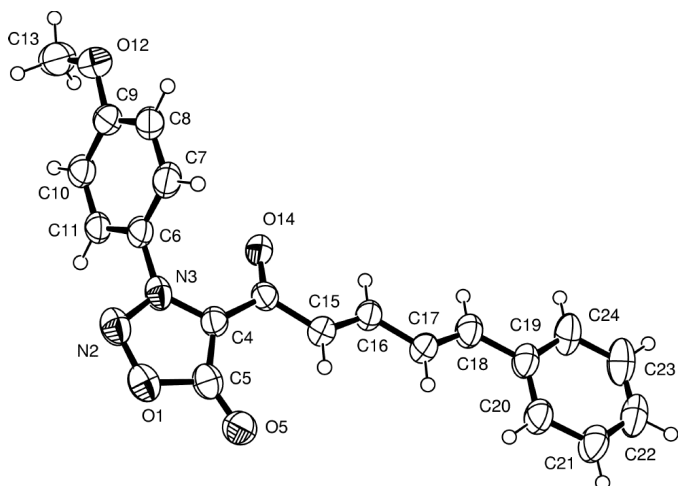


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary radii.

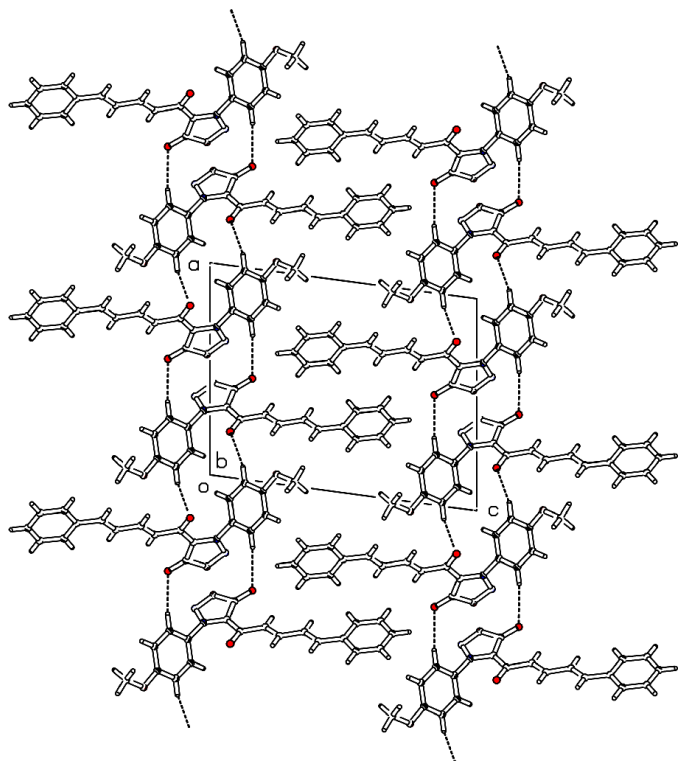


Figure 2
Packing diagram showing C—H...O interactions, viewed down the *b* axis.

H16; H17 and H18 are *trans* to each other. The bond angles C15—C16—C17 [124.3 (3)°], C16—C17—C18 [123.9 (3)°] and C17—C18—C19 [127.2 (3)°] are significantly larger than the normal value of 120°, and this may be due to steric repulsion between atoms H15 and H17 (2.46 Å), between atoms H16 and H18 (2.41 Å), and between atoms H17 and H20 (2.18 Å). A close intramolecular contact between O5 and H15 (2.43 Å), being a repulsive interaction, may have caused the significant opening of the C5—C4—C14 [129.7 (4)°] and C4—C5—O5 [137.3 (3)°].

In the crystal structure, atom C8 acts as a hydrogen-bond donor and forms a weak intermolecular C—H...O interaction with the carbonyl atom O14 of an adjacent molecule, thereby linking the molecules into extended chains which have a graph-set motif of *C*(8) (Bernstein *et al.*, 1995). Atom C11 acts as a donor for an intermolecular C—H...O interaction with atom O5 of the sydnone ring of another adjacent molecule. This weak intermolecular interaction also links the molecules into extended chains which have a graph-set motif of *C*(7) (Bernstein *et al.*, 1995). The above two weak intermolecular C—H...O interactions are combined to form a sheet-like structure which lies parallel to the *ab* plane (Fig. 2).

Experimental

4-Acetyl-3-(4-methoxy)phenylsydnone (2.34 g, 0.01 mol) was suspended in sodium hydroxide solution (0.5 g sodium hydroxide in 5 ml water and 5 ml ethanol) and benzaldehyde (1 g, 0.01 mol) was added. The reaction mixture was stirred for 30 min at room temperature and the resultant precipitate was collected immediately and washed with water. Recrystallization from an acetone–ethanol mixture afforded crystals of (I) suitable for X-ray analysis (m.p. 449–451 K).

Crystal data

C₂₀H₁₆N₂O₄
M_r = 348.35
 Monoclinic, *P*2₁
a = 12.0961 (8) Å
b = 4.7750 (4) Å
c = 15.3516 (12) Å
 β = 97.719 (3)°
V = 878.66 (12) Å³
Z = 2

D_x = 1.317 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 1742 reflections
 θ = 1.0–25.0°
 μ = 0.09 mm⁻¹
T = 160 (2) K
 Prism, yellow
 0.25 × 0.18 × 0.13 mm

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans with κ offsets
 Absorption correction: none
 1752 measured reflections
 1752 independent reflections
 1389 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.078
 θ_{\max} = 25.0°
h = 0 → 14
k = 0 → 5
l = -18 → 18

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.044
wR(*F*²) = 0.120
S = 1.05
 1752 reflections
 237 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0734P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.18 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.17 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.049 (11)

Table 1

Comparison of the bond lengths (Å) in the sydnone ring of (I) with the corresponding average values found in 3,4-disubstituted sydnone derivatives.

	(I)	Average value
O1—C5	1.424 (4)	1.407 (4)
O1—N2	1.369 (3)	1.379 (3)
N2—N3	1.303 (4)	1.310 (3)
N3—C4	1.364 (3)	1.351 (3)
C4—C5	1.411 (5)	1.413 (3)
C5—O5	1.210 (3)	1.206 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C8—H8 \cdots O14 ⁱ	0.95	2.38	3.281 (4)	159
C11—H11 \cdots O5 ⁱⁱ	0.95	2.47	3.410 (3)	170

Symmetry codes: (i) $2-x, y-\frac{1}{2}, 2-z$; (ii) $1-x, \frac{1}{2}+y, 2-z$.

The methyl H atoms were constrained to an ideal geometry (C—H = 0.98 Å), with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, but were allowed to rotate freely about the C—C bonds. All remaining H atoms were placed in geometrically idealized positions (C—H = 0.95 Å) and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Although the molecule is achiral, the structure possesses a polar axis. Owing to the absence of any significant anomalous scatterers in the compound, the absolute structure was assigned arbitrarily and Friedel pairs were merged before the final refinement. Reflections 001 and $\bar{1}01$ were partially obscured by the beam stop and were omitted.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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