

C3—C2—C12	112.4 (2)	C2—C3—C4	113.8 (2)
C3—C4—C5	114.6 (3)	C3—C4—C5A	111 (1)
C5A—C6A—C7	108 (2)	C4—C5—C6	114.6 (4)
C6—C7—C8	116.1 (3)	C6A—C7—C8	115.2 (7)
C1—C8—C7	115.6 (3)	C11—C9—C1	114.5 (2)
C11—C9—C10	115.7 (2)	C4—C5A—C6A	110 (2)
C11—C9—C13	115.3 (2)	C1—C9—C10	107.5 (2)
C1—C9—C13	102.9 (2)	C10—C9—C13	99.1 (2)
C12—C10—C9	124.6 (2)	C2—C12—C11	108.0 (2)
C12—C10—C11	128.3 (3)	C2—C12—C13	103.2 (2)
C9—C10—C11	106.9 (3)	C11—C12—C13	99.4 (2)
C13—C11—C10	127.7 (3)	C15—C13—C16	107.4 (2)
C13—C11—C12	125.2 (2)	C15—C13—C9	113.7 (2)
C10—C11—C12	106.8 (2)	C15—C13—C12	113.7 (2)
C14—C12—C2	114.4 (2)	C16—C13—C9	114.1 (2)
C14—C12—C11	114.6 (2)	C16—C13—C12	115.6 (2)
C14—C12—C13	115.6 (2)	C9—C13—C12	92.0 (2)
C8—C1—C2—C3	2.3 (4)	C2—C1—C8—C7	-83.9 (3)
C6—C7—C8—C1	81.1 (4)	C5—C6—C7—C8	-73.9 (5)
C4—C5—C6—C7	106.2 (4)	C3—C4—C5—C6	-59.5 (5)
C2—C3—C4—C5	-51.9 (4)	C1—C2—C3—C4	89.3 (3)
C4—C5A—C6A—C7	-110.5 (16)	C3—C4—C5A—C6A	81.4 (17)

Atoms C5 and C6 are disordered over two sites with unequal occupancies. The major sites were assigned occupancies of 85%, based on earlier population-parameter refinement, and were refined anisotropically. The minor sites C5A and C6A were assigned 15% occupancy and were refined isotropically. H atoms were placed in calculated positions with C—H 0.95 Å and $B_{\text{iso}}(\text{H}) = 1.3B_{\text{eq}}(\text{C})$, except for those of the minor disorder sites which were not included.

Programs used were *MolEN* (Fair, 1990), *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: CR1147). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-(4-Methylphenyl)-1-(3-thienyl)-2-propen-1-one

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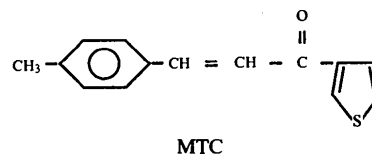
(Received 22 November 1993; accepted 15 July 1994)

Abstract

The title compound, $\text{C}_{14}\text{H}_{12}\text{OS}$, is roughly planar with a dihedral angle of $4.67(8)^\circ$ between the planes of the phenyl and thienyl rings. The O—C—C torsion angle in the central $\text{C}_2\text{H}_2\text{CO}$ group is $1.9(5)^\circ$.

Comment

Chalcone crystals have recently been developed as new and highly efficient organic non-linear optical materials. We have synthesized a series of substituted thiophene chalcone derivatives of which the title compound, 3-(4-methylphenyl)-1-(3-thienyl)-2-propen-1-one (MTC), is an example.



The crystal structure belongs to the centrosymmetric space group $P2_1/n$. The O(1)—C(10)—C(9)—C(8) torsion angle in the central $\text{C}_2\text{H}_2\text{CO}$ group is $1.9(5)^\circ$ and the dihedral angle between the planes of the phenyl and thienyl rings is $4.67(8)^\circ$. Thus, the molecule is roughly planar. These structural data may be compared with those of its isomer (Li & Su, 1993) which

was grown at 298 K (the crystal studied here was grown at 313 K). The isomer crystallized in the non-centrosymmetric space group *Fdd2*, with a torsion angle of the central C₂H₂CO group of 4(1)° and a dihedral angle of 7.85° between the ring planes. These differences may be due to the different crystallization conditions which have an effect on the molecular packing arrangements. Since the title crystal is centrosymmetric, no second harmonic generation (SHG) properties can be exhibited; this has been confirmed by SHG efficiency measurements on a powder sample using the method of Kurtz & Perry (1968). A strong signal was observed from the isomer for the same type of powder measurements. A *PLUTO* (Motherwell & Clegg, 1978) drawing of MTC is shown in Fig. 1.

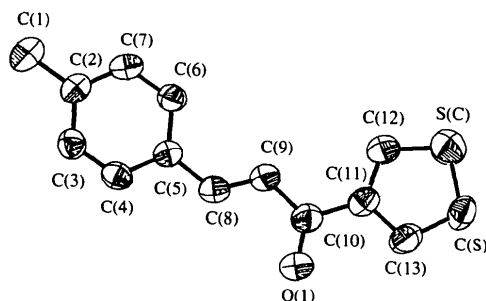


Fig. 1. Molecular structure and atomic numbering of MTC. The displacement ellipsoids are drawn at the 50% probability level.

Refinement

Refinement on *F*
R = 0.058
wR = 0.079
S = 2.40
 1366 reflections
 146 parameters
 H-atom parameters not refined

$w = 1/\sigma^2(F)$
 $(\Delta/\sigma)_{\max} = 0.0038$
 $\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$B_{\text{eq}} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
S(C)†	-0.3769 (1)	0.0044 (3)	0.6073 (1)	7.42 (8)
O(1)	-0.0879 (2)	-0.4891 (4)	0.6586 (2)	7.3 (1)
C(S)‡	-0.3946 (1)	-0.2487 (3)	0.5544 (1)	5.35 (9)
C(1)	0.3897 (3)	0.3454 (6)	0.9723 (2)	6.0 (2)
C(2)	0.3026 (2)	0.2101 (5)	0.9172 (2)	4.3 (1)
C(3)	0.3166 (2)	-0.0049 (5)	0.8825 (2)	4.4 (1)
C(4)	0.2365 (2)	-0.1287 (5)	0.8308 (2)	4.1 (1)
C(5)	0.1398 (2)	-0.0461 (4)	0.8117 (2)	3.6 (1)
C(6)	0.1259 (2)	0.1704 (5)	0.8465 (2)	4.2 (1)
C(7)	0.2066 (2)	0.2934 (5)	0.8982 (2)	4.4 (1)
C(8)	0.0579 (2)	-0.1848 (5)	0.7589 (2)	4.2 (1)
C(9)	-0.0368 (2)	-0.1277 (5)	0.7236 (2)	4.4 (1)
C(10)	-0.1106 (2)	-0.2923 (5)	0.6728 (2)	4.8 (1)
C(11)	-0.2158 (2)	-0.2191 (5)	0.6377 (2)	4.5 (1)
C(12)	-0.2576 (2)	-0.0145 (6)	0.6565 (2)	5.5 (2)
C(13)	-0.2895 (2)	-0.3581 (6)	0.5808 (2)	5.6 (1)

† S(C) = 0.57S + 0.43C.

‡ C(S) = 0.57C + 0.43S.

Experimental

Single crystals of MTC were grown from anhydrous ethanol and recrystallized at 313 K.

Crystal data

C₁₄H₁₂OS
M_r = 228.31
 Monoclinic
*P*2₁/*n*
a = 13.944 (4) Å
b = 5.823 (2) Å
c = 15.172 (5) Å
 β = 105.86 (2)°
V = 1185.0 (7) Å³
Z = 4
D_x = 1.28 Mg m⁻³

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 20 reflections
 θ = 5–8°
 μ = 0.236 mm⁻¹
T = 296 K
 Prismatic
 0.6 × 0.3 × 0.2 mm
 Yellow

Data collection

Rigaku AFC-5R diffractometer
 ω -2 θ scans
 Absorption correction: empirical
 $T_{\min} = 0.93$, $T_{\max} = 1.00$
 2399 measured reflections
 2302 independent reflections
 1366 observed reflections
 [*I* > 3σ(*I*)]

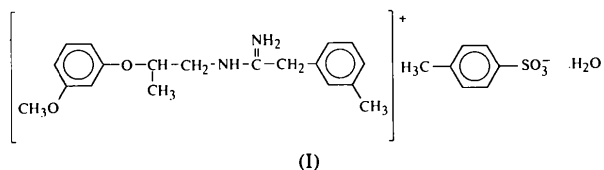
*R*_{int} = 0.025
 $\theta_{\max} = 25^\circ$
h = 0 → 17
k = 0 → 7
l = -18 → 18
 3 standard reflections
 frequency: 66 min
 intensity decay: 1.3%

Table 2. Selected geometric parameters (Å, °)

S(C)—C(12)	1.630 (4)	C(5)—C(6)	1.401 (4)
S(C)—C(S)	1.665 (2)	C(5)—C(8)	1.448 (4)
O(1)—C(10)	1.224 (3)	C(6)—C(7)	1.383 (4)
C(S)—C(13)	1.547 (4)	C(8)—C(9)	1.324 (4)
C(1)—C(2)	1.497 (4)	C(9)—C(10)	1.462 (4)
C(2)—C(7)	1.379 (4)	C(10)—C(11)	1.480 (4)
C(2)—C(3)	1.392 (4)	C(11)—C(12)	1.389 (4)
C(3)—C(4)	1.380 (4)	C(11)—C(13)	1.404 (4)
C(4)—C(5)	1.386 (4)		
C(12)—S(C)—C(S)	99.2 (1)	C(2)—C(7)—C(6)	122.0 (3)
C(13)—C(S)—S(C)	103.6 (2)	C(9)—C(8)—C(5)	129.2 (3)
C(7)—C(2)—C(3)	117.8 (3)	C(8)—C(9)—C(10)	122.0 (3)
C(7)—C(2)—C(1)	121.6 (3)	O(1)—C(10)—C(9)	121.8 (3)
C(3)—C(2)—C(1)	120.6 (3)	O(1)—C(10)—C(11)	119.0 (3)
C(4)—C(3)—C(2)	120.6 (3)	C(9)—C(10)—C(11)	119.1 (3)
C(3)—C(4)—C(5)	121.9 (3)	C(12)—C(11)—C(13)	110.1 (3)
C(4)—C(5)—C(6)	117.4 (3)	C(12)—C(11)—C(10)	127.1 (3)
C(4)—C(5)—C(8)	119.8 (3)	C(13)—C(11)—C(10)	122.8 (3)
C(6)—C(5)—C(8)	122.8 (2)	C(11)—C(12)—S(C)	113.4 (3)
C(7)—C(6)—C(5)	120.4 (3)	C(11)—C(13)—C(S)	113.8 (3)

Data were collected with a scan width of (0.95 + 0.35tanθ)° using *CONTROL* software (Molecular Structure Corporation, 1989). The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983) and *DIRDIF* (Beurskens, 1984). The C, O and S atoms were located on an *E* map. The C(S) and S(C) atoms are distributed statistically. H atoms were placed in geometrically calculated positions (C—H = 0.95 Å) but were not included in the refinement. Anomalous-dispersion corrections were not applied. All calculations were performed on a MicroVAXII computer using the *TEXSAN* (Molecular Structure Corporation, 1987) program package.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and least-squares-planes data have been deposited with the IUCr (Reference: AB1139). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.



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Acta Cryst. (1995). **C51**, 991–993

N-[2-(3-Methoxyphenoxy)propyl]-*m*-tolyl-acetamidinium *p*-Toluenesulfonate Monohydrate (Xylamidine)[†]

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Abstract

The crystal structure of xylamidine, $C_{19}H_{25}N_2O_2^+ \cdot C_7H_7O_3S^- \cdot H_2O$, a peripheral 5-HT₂ receptor antagonist, is reported. The xylamidinium cation is folded with an almost parallel arrangement of the aromatic rings.

Comment

The crystal structure determination of xylamidine, (I), a peripheral 5-HT₂ receptor antagonist, forms part of our continuing work on serotonin antagonists (Blaton,

[†] Internal code of the Janssen Research Foundation: R13489.

Peeters & De Ranter, 1985, 1995, and references cited therein). The crystal determination showed rotational disorder of the *m*-tolyl moiety. The partial occupancies refined to 0.538 (7) for atoms C17–C23 and to 0.462 (7) for C117–C123, hereafter called conformations *A* and *B*, respectively. Fig. 1 shows the xylamidinium cation in the *A* conformation together with the water molecule and the *p*-toluenesulfonate anion. Neither the cation nor the anion exhibit any unusual bond lengths or angles. The xylamidinium cation is folded with an almost parallel arrangement of the aromatic rings [dihedral angles between the least-squares planes 4.0 (4) and 7.5 (4)° for conformations *A* and *B*, respectively]. The molecules are involved in a network of hydrogen bonds (Table 3) resulting in a double interconnected infinite chain of xylamidinium, H₂O and *p*-toluenesulfonate in the *a* direction.

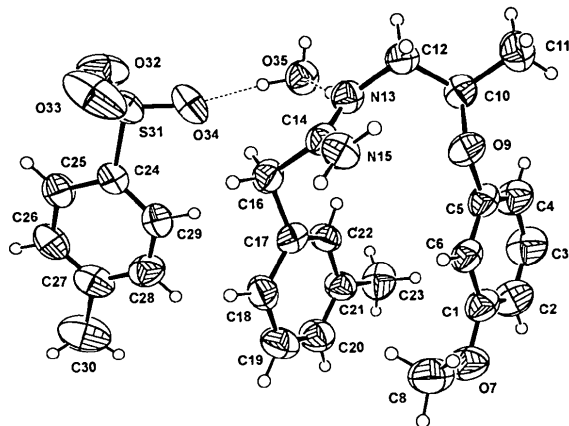


Fig. 1. Perspective view of the title compound in the *A* conformation with atomic numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

Experimental

Crystal data

$C_{19}H_{25}N_2O_2^+ \cdot C_7H_7O_3S^- \cdot H_2O$
 $M_r = 502.61$
 Monoclinic
 $P2_1/n$
 $a = 8.217 (7) \text{ \AA}$
 $b = 31.54 (1) \text{ \AA}$
 $c = 10.384 (4) \text{ \AA}$
 $\beta = 98.09 (4)^\circ$

Cu $K\alpha$ radiation
 $\lambda = 1.54184 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 14\text{--}28^\circ$
 $\mu = 1.430 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Prism