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Key indicators

Single-crystal X-ray study
T = 295 K
Mean $\sigma(\text{C}-\text{C})$ = 0.003 Å
R factor = 0.044
wR factor = 0.124
Data-to-parameter ratio = 13.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Methyl [4-methoxy-3-(methanesulfonyloxy)benzoyl]formate

The crystal structure of the title compound, C₁₁H₁₂O₇S, confirms an earlier proposal concerning the regioselectivity of electrophilic substitution reactions of mesyl guaiacol.

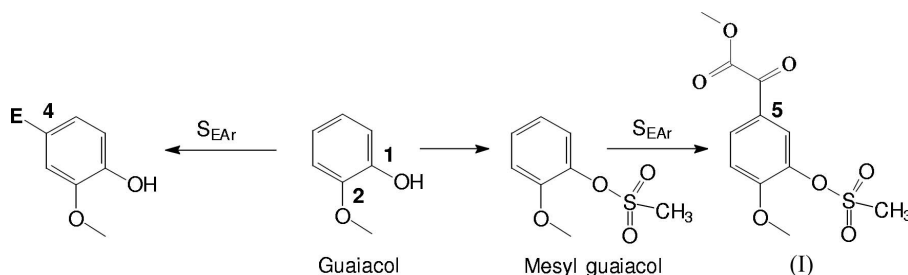
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Comment

The title compound, (I), was prepared in an approach to synthesize regioselective substituted guaiacol derivatives as intermediates for further steps. To address this goal, we started from mesyl guaiacol. 5-Substituted guaiacol derivatives are difficult to prepare, since the electrophilic substitution of guaiacol takes place predominantly at the 4-position while the corresponding mesylate masking the phenol into a deactivating group directs entry to the 5-position (see scheme; Benschel *et al.*, 2002). Subsequently, we performed Friedel–Crafts-type acylation of mesyl guaiacol with methyl glyoxalate acid chloride (see *Experimental*).



In the crystal structure, weak C—H···O interactions link molecules into centrosymmetric dimers. These dimers are, in turn, linked by further C—H···O interactions to form tapes in the *a*-axis direction (see Table 1 and Fig. 2).

Experimental

To a stirred mixture of AlCl₃ (7.0 g, 52 mmol) in CH₂Cl₂ at 273 K, methyl glyoxalate acid chloride (7.0 g, 57 mmol) was added dropwise. After the mixture had been stirred for 30 min, 1-methoxy-2-methylsulfonyloxybenzol (mesyl guaiacol) (10.0 g, 50 mmol) was added slowly. The mixture was stirred overnight at room temperature. After being quenched with ice and HCl, the organic layer was washed with water and brine. The organic phase was dried over Na₂SO₄ and concentrated to give the crude product as a brown oil. The oil was purified by silica-gel column chromatography using ethyl acetate/hexanes (1:1 *v/v*), yielding the product as a light-yellow oil which was dried to give 4.43 g (31%) of colourless crystals of (I). The title compound was obtained by slow crystallization of purified (I) from dichloromethane.

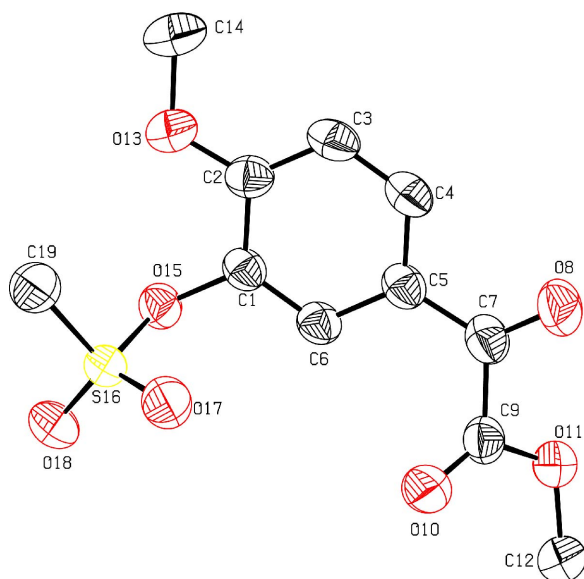


Figure 1
ORTEP view (Johnson, 1976) of (I). Displacement ellipsoids are shown at the 50% probability level. H atoms have been omitted for clarity.

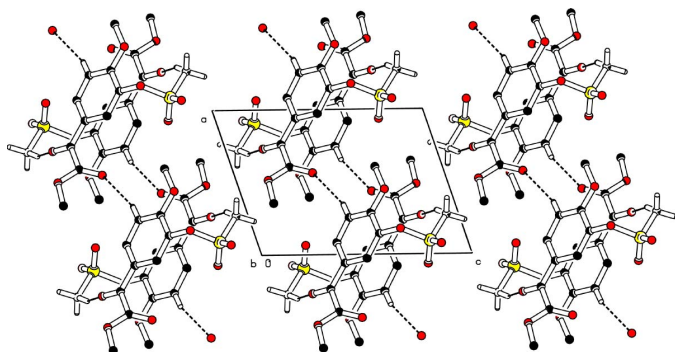


Figure 2
Packing diagram, viewed along the *b* axis, showing the hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonds have been omitted.

Crystal data

$C_{11}H_{12}O_7S$
 $M_r = 288.27$
 Triclinic, $P\bar{1}$
 $a = 8.4530$ (15) Å
 $b = 8.6560$ (12) Å
 $c = 10.7923$ (12) Å
 $\alpha = 105.221$ (10)°
 $\beta = 98.919$ (12)°
 $\gamma = 116.400$ (13)°
 $V = 647.9$ (2) Å³

$Z = 2$
 $D_x = 1.478$ Mg m⁻³
 Cu $K\alpha$ radiation
 Cell parameters from 24 reflections
 $\theta = 60\text{--}69^\circ$
 $\mu = 2.50$ mm⁻¹
 $T = 295$ (2) K
 Block, colourless
 0.32 × 0.22 × 0.22 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\theta/2\omega$ scans
 Absorption correction: ψ scan (CORINC; Dräger & Gattow, 1971)
 $T_{\min} = 0.379$, $T_{\max} = 0.577$
 2633 measured reflections
 2456 independent reflections

2297 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.055$
 $\theta_{\text{max}} = 69.9^\circ$
 $h = -10 \rightarrow 0$
 $k = -9 \rightarrow 10$
 $l = -12 \rightarrow 13$
 3 standard reflections
 frequency: 60 min
 intensity decay: 3%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.124$
 $S = 1.08$
 2456 reflections
 182 parameters
 Only H-atom U 's refined

$w = 1/[\sigma^2(F_o^2) + (0.0747P)^2 + 0.1755P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.47$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0122 (15)

Table 1

Hydrogen-bonding geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
C3—H3...O10 ⁱ	0.93	2.48	3.371 (3)	161
C19—H19A...O8 ⁱⁱ	0.96	2.49	3.399 (3)	158

Symmetry codes: (i) 1 + *x*, *y*, *z*; (ii) −*x*, −*y*, 1 − *z*.

H atoms were placed in calculated positions with C—H distances of 0.93 (phenyl) and 0.98 Å (methyl), and the isotropic displacement parameters were refined.

Data collection: CAD-4 EXPRESS (Nonius, 1998); cell refinement: CAD-4 EXPRESS; data reduction: CORINC (Dräger & Gattow, 1971); program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP (Johnson, 1976); software used to prepare material for publication: SHELXL97.

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