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2-Methoxy-4-[2-methyl-5-oxo-4,5-dihydro-1,3-oxazol-4-ylidene)methyl]phenyl acetate

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Key indicators

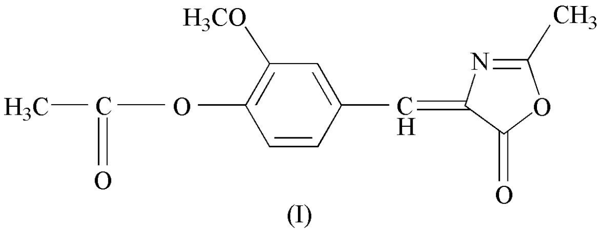
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.137
Data-to-parameter ratio = 17.6

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

In the title compound, $C_{14}H_{13}NO_5$, the C atom between the benzene and oxazolone rings displays a distorted trigonal bonding geometry. The oxazolone and aromatic rings are approximately coplanar.

Comment

Oxazolone compounds are important intermediates in the synthesis of 2-aryllactic acids. The title compound, (I), was prepared by the reaction of vanillin and acetylglycine in boiling acetic anhydride (Shaw *et al.*, 1958; Wong *et al.*, 1992). An X-ray crystal structure determination of (I) was carried out in order to elucidate the structure and the result is presented here.



(I)

The molecular structure of (I) is shown in Fig. 1. The oxazolone ring is almost planar, with atoms C1 and C2 deviating by 0.0085 (2) and −0.0084 (2) Å from the mean plane. The oxazolone and aromatic rings are approximately coplanar, forming a dihedral angle of 8.2 (3)°. The torsion angle N1—C2—C5—C6 is 1.8 (4)°, indicating these atoms are almost coplanar. Because of the steric hindrance of the oxazolone and aromatic rings, atom C5 has a distorted trigonal geometry, with the angle C2—C5—C6 [130.8 (2)°] deviating significantly from the ideal sp^2-hybridized value.

Experimental

To a mixture of vanillin (1.52 g, 10 mmol), acetylglycine (1.17 g, 10 mmol) and anhydrous sodium acetate (0.08 g, 10 mmol) was added boiling acetic anhydride (3 ml, 30 mmol). The mixture was maintained at 373 K for 7 h. After completion of the reaction, the mixture was allowed to cool to room temperature, then iced water (5 ml) was added. The resulting yellow precipitate was filtered, washed with 50% aqueous EtOH, dried under vacuum and recrystallized from acetone to give slightly yellow crystals (yield 69%); single crystals (m.p. 423–424 K) suitable for crystallographic analysis were obtained by slow evaporation of an acetone–toluene solution. IR (KBr, ν cm^{-1}): 3452, 3329, 1759, 1692, 1649, 1509, 1208, 915; ^{1}H NMR (CDCl$_3$): δ 7.97–7.86 (*m*, 3H), 7.23 (*s*, 1H), 3.82 (*s*, 3H), 2.40 (*s*, 3H), 2.29 (*s*, 3H). Analysis calculated for $C_{14}H_{13}NO_5$: C 61.09, H 4.76, N 5.08%; found: C 61.20, H 4.87, N 5.02%.

Crystal data

$C_{14}H_{13}NO_5$
$M_r = 275.25$
Monoclinic, $C2/c$
$a = 26.552\ (7)$ Å
$b = 7.3127\ (19)$ Å
$c = 15.933\ (4)$ Å
$\beta = 119.648\ (3)^\circ$
$V = 2688.6\ (12)$ Å^3
$Z = 8$
$D_x = 1.360$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1346 reflections
$\theta = 2.9$–21.4°
$\mu = 0.11$ mm^{-1}
$T = 293\ (2)$ K
Prism, colourless
$0.20 \times 0.18 \times 0.16$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.979$, $T_{\max} = 0.983$
8744 measured reflections
3293 independent reflections
1535 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 28.1^\circ$
$h = -32 \rightarrow 34$
$k = -9 \rightarrow 9$
$l = -21 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.137$
$S = 0.97$
3293 reflections
184 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0686P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

All H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined as riding. For the CH groups, $U_{\rm iso}$(H) values were set equal to $1.2U_{\rm eq}$(carrier atom) and for the methyl groups they were set equal to $1.5U_{\rm eq}$(carrier atom).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

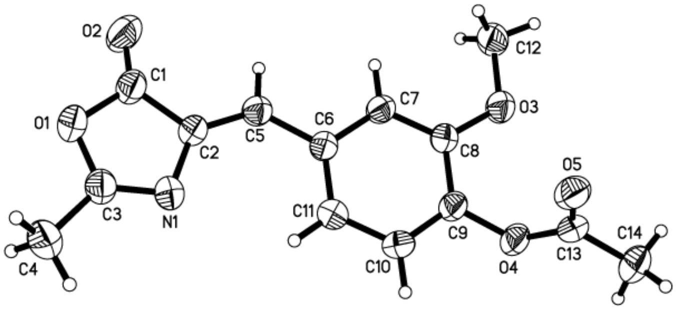


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.

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