Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

10-(4-Methoxyphenyl)-3,7-dimethyl-1*H*,9*H*,10*H*-dipyrano[3,4-*e*:4′,3′-*b*]pyran-1,9-dione

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.116
Data-to-parameter ratio = 17.1

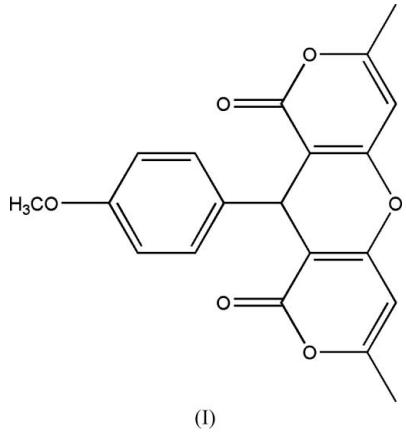
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 3 January 2006
Accepted 9 January 2006
Online 13 January 2006

The title compound, $C_{20}H_{16}O_6$, was synthesized by the reaction of 4-methoxybenzaldehyde with triacetic acid lactone in ethylene glycol catalyzed by KF–alumina. The pyran ring adopts a boat conformation and the benzene ring is perpendicular to the mean plane through the fused ring system.

Comment

The utility of fluoride salts as potential bases in a variety of synthetic reactions has been recognized in recent years (Ando & Yamawaki, 1981; Clark, 1980). The condensation reaction of carbonyl compounds with active methylene compounds is one of the most important synthetic methods leading to substituted alkenes (Yamawaki *et al.*, 1983). In our earlier paper (Wang *et al.*, 2003), we have reported that alumina coated with potassium fluoride is a versatile solid-supported reagent for the Knoevenagel reaction and Michael addition condensation. In the present paper, we report the crystal structure of the title compound, (I).



(I)

In (I), the pyranone rings are each essentially planar. The pyran ring adopts a boat conformation, with atoms O1 and C6 deviating from the C5/C1/C7/C11 plane by 0.076 (3) and 0.183 (3) Å, respectively (Fig. 1). The methoxy group is almost coplanar with the benzene ring, with a C20—O6—C17—C16 torsion angle of 8.8 (3)°. The C14–C19 benzene ring is perpendicular to the C5/C1/C7/C11 plane [dihedral angle 89.14 (7)°].

Experimental

Compound (I) was prepared by the reaction of 4-methoxybenzaldehyde (1 mmol) with triacetic acid lactone (2 mmol) in ethylene glycol catalyzed by KF–alumina. Single crystals of (I)

suitable for X-ray diffraction were obtained by slow evaporation of a 95% aqueous ethanol solution (yield 85%; m.p. 545–547 K). IR (cm^{-1}): 1735, 1636, 1604, 1381; ^1H NMR ($CDCl_3$): 2.24 (6H, *s*, $2CH_3$), 3.75 (3H, *s*, CH_3), 4.78 (1H, *s*, CH), 5.98 (2H, *s*, 2CH), 6.79 (2H, *d*, ArH), 7.25 (2H, *d*, ArH).

Crystal data

$C_{20}H_{16}O_6$	D_x = 1.390 Mg m^{-3}
M_r = 352.33	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 3166 reflections
a = 9.7018 (18) Å	θ = 2.3–27.8°
b = 11.090 (2) Å	μ = 0.10 mm^{-1}
c = 15.757 (3) Å	T = 298 (2) K
β = 96.839 (3)°	Block, white
V = 1683.2 (5) $Å^3$	0.49 × 0.47 × 0.43 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	4076 independent reflections
	2601 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.032
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	θ_{max} = 28.3°
	$h = -8 \rightarrow 12$
T_{min} = 0.951, T_{max} = 0.957	$k = -14 \rightarrow 14$
12075 measured reflections	$l = -20 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 0.4864P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.042	
$wR(F^2)$ = 0.116	
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.001
4076 reflections	$\Delta\rho_{max}$ = 0.22 e $Å^{-3}$
238 parameters	$\Delta\rho_{min}$ = −0.17 e $Å^{-3}$
H-atom parameters constrained	Extinction correction: *SHELXL97*
	Extinction coefficient: 0.0150 (14)

All H atoms were positioned geometrically and treated as riding, with C—H distances of 0.93–0.98 Å, and with $U_{iso}(H) = 1.5U_{eq}$ for methyl H atoms and $1.2U_{eq}(C)$ for others.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); 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, 1999); software used to prepare material for publication: *SHELXTL*.

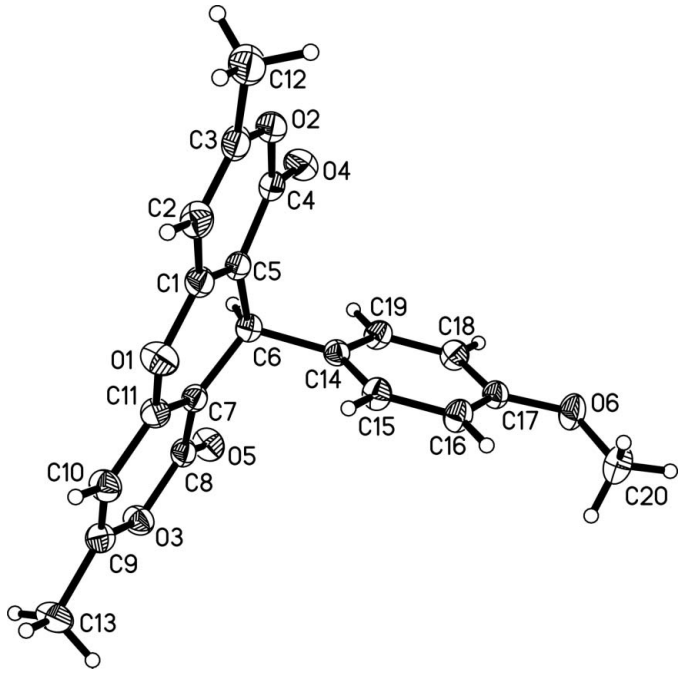


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids.

The authors thank the Natural Science Foundation of the Guangdong Province (grant No. 031792)

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