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(*Z*)-3-(4-Methoxybenzylidene)-2-methylisoindolin-1-one

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.116
Data-to-parameter ratio = 11.8

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

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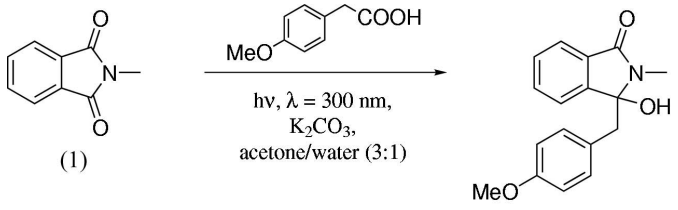
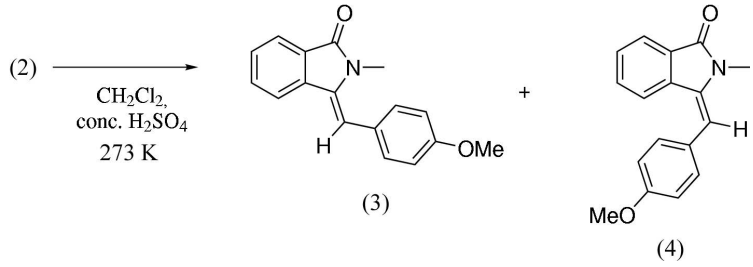
The title compound, $C_{17}H_{15}NO_2$, consists of two planar sub-units, *viz*. a lactam and a 4-methoxybenzyl moiety. The double bond connecting the sub-units has a *Z* configuration.

Comment

We have recently described a photochemical route to precursors of aristolactams, phenanthrene lactam alkaloids with antitumor activity. The crucial step in our approach is the photodecarboxylative radical benzylation of 2-methylisoindoline-1,3-dione, (1), with aryl acetates upon irradiation at λ = 300 nm in aqueous solution. With 2-(4-methoxyphenyl)acetic acid, photoinduced electron transfer to the electronically excited triplet manifold of (1) furnishes 3-(4-methoxybenzyl)-3-hydroxy-2-methylindolin-1-one, (2), in high yields. Rapid (5 min) dehydration of (2) with concentrated sulfuric acid in dichloromethane at 273 K gives the the title compound, (3), and the corresponding *E*-isomer (4) in a 1:8 ratio. The chemical and structural properties of (2) and (4) have been published previously (Griesbeck *et al.*, 2004).





Compound (3) consists of two planar sub-units connected by a (*Z*)-substituted double bond, which is only slightly twisted with a torsion angle for N4—C2—C10—C11 of 5.4° (Fig. 1). The dihedral angle between the lactam ring system (atoms N4/C1–C8) and the benzene ring of the 4-methoxybenzyl unit (atoms C11–C16) is 67.3 (1)°. Compound (3) exhibits a remarkably short centroid–centroid distance of 3.689 (1) Å between the six-membered ring and the five-membered ring of the lactam subunit, together with an interplanar distance of 3.567 (2) Å. These values strongly suggest the occurrence of π–π stacking (Fig. 2).

Experimental

A solution of 10 mmol of 2-methylisoindoline-1,3-dione (1), 12.5 mmol of 2-(4-methoxyphenyl)acetic acid and 6 mmol of potassium carbonate in a mixture of acetone (100 ml) and water (100 ml) was irradiated at λ = 300 nm in a Rayonet photoreactor at room temperature for 6 h. Removal of the acetone on a rotary evaporator yielded a white solid, which was filtered off and dried. Subsequent crystallization from ethanol furnished colorless needles of 3-hydroxy-3-(4-methoxybenzyl)-2-methylindolin-1-one, (2), in a 90% yield. To a cooled solution (273 K) of 3.5 mmol (2) in dichloromethane (100 ml), 5 drops of concentrated sulfuric acid were added. After 5 min, solid sodium hydrogen carbonate was added to the deep yellow solution. Filtration and evaporation yielded a deep yellow oil, from which compound (3) and the *E*-isomer (4) were isolated in a 1:8 ratio with a total yield of 85% by column chromatography on silica using cyclohexane/ethyl acetate as eluent. Recrystallization from benzene gave material of melting point 359 K suitable for X-ray diffraction.

Crystal data

$C_{17}H_{15}NO_2$	D_x = 1.329 Mg m^{-3}
M_r = 265.30	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 9520 reflections
a = 9.9750 (5) Å	θ = 2.4–27.0°
b = 14.6856 (7) Å	μ = 0.09 mm^{-1}
c = 10.1388 (5) Å	T = 100 (2) K
β = 116.812 (2)°	Prism, colorless
V = 1325.55 (11) Å^3	0.30 × 0.25 × 0.20 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.053
φ and ω scans	θ_{max} = 27.0°
Absorption correction: none	$h = -12 \rightarrow 11$
9520 measured reflections	$k = -18 \rightarrow 18$
2851 independent reflections	$l = -12 \rightarrow 12$
2066 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)]$ = 0.045	$w = 1/[\sigma^2(F_o^2) + (0.0674P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.116	$(\Delta/\sigma)_{max} < 0.001$
S = 1.04	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
2851 reflections	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
241 parameters	

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SCHAKAL99* (Keller, 1999); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

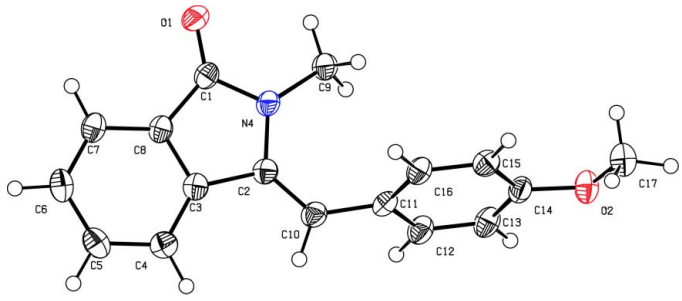


Figure 1
View of (3), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

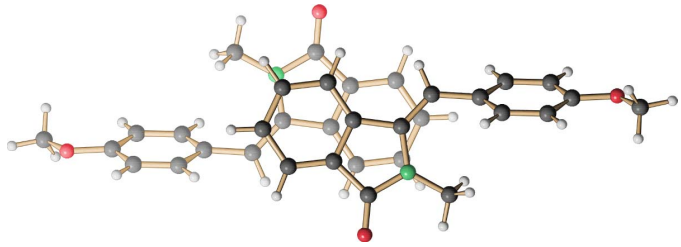


Figure 2
Close contact (3.69 Å) between the six- and five-membered ring of the lactam sub-units in a pair of molecules of (3).

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