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An unexpected *N*-substituted oxindole: 1-(3,3,5-trimethyl-2-phenyl-1,2-oxazolidin-5-yl)-1*H*-indol-3(2*H*)-one

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean *σ*(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.108
Data-to-parameter ratio = 17.4

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

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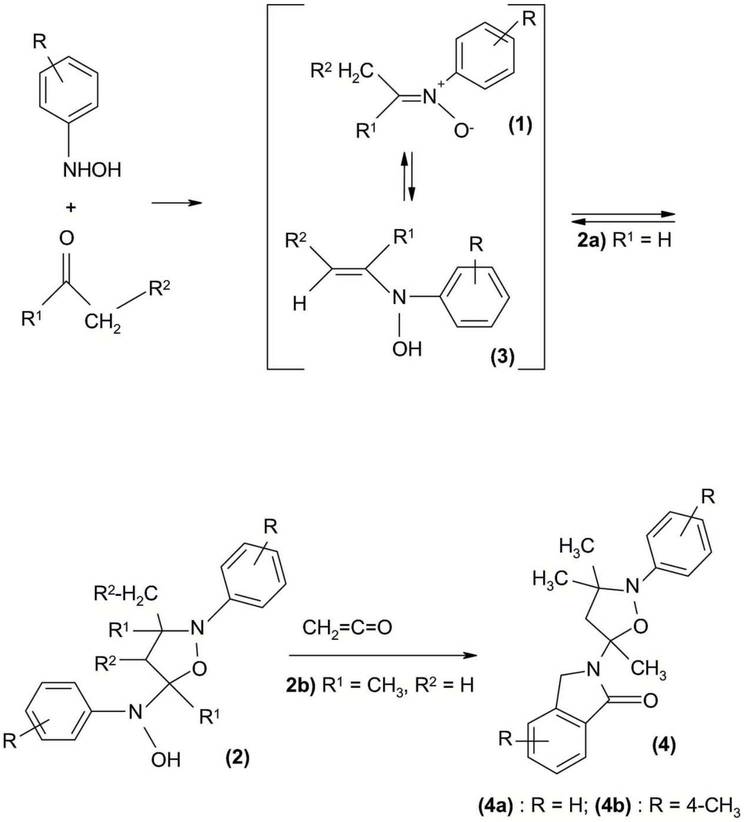
The molecule of the title compound, $C_{20}H_{22}N_2O_2$, an *N*-substituted oxindole obtained unexpectedly, is composed of a heterocyclic ring, which adopts an *N*-envelope conformation, and an isoindole group which is essentially planar.

Comment

Reactions of aldehydes and ketones with hydroxylamines yield the corresponding nitrones. However, it has been demonstrated that, in the case of nitrones with *α*-H atoms (1), the actual products are not the nitrones but their cyclodimers, isoxazolidine derivatives, (2) (Foster *et al.*, 1974; Aurich *et al.*, 1986).



We have already reported that, by the use of 1,3-dipolar cycloaddition reaction, aldonitrone cyclodimers (2*a*) undergo cycloreversion reactions in solution, and are in equilibrium with their corresponding nitrones (1) and enamines (3), but this equilibrium does not occur in the case of ketonitrone cyclodimers (Bigdeli & Mehrdad, 1993). When acetone cyclodimers (2*b*) were reacted at room temperature with ketene gas, the unexpected *N*-substituted oxindols (4) were obtained. In this paper, we report the structure of the *N*-substituted oxindol (4*a*).

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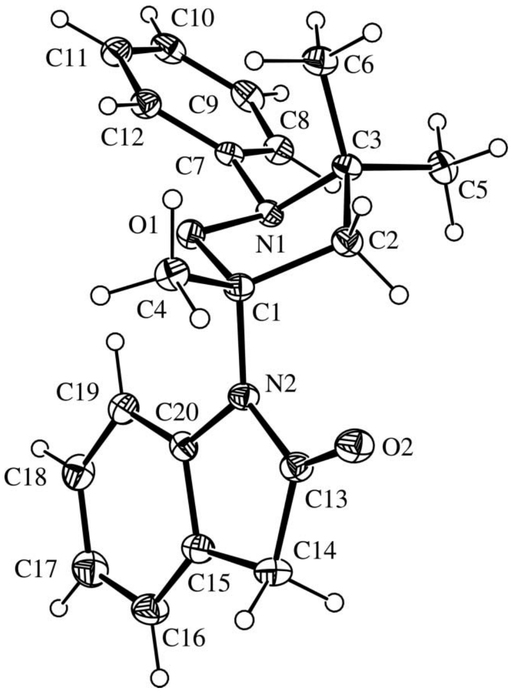

Figure 1
The molecular structure of (4*a*), with displacement ellipsoids drawn at the 30% probability level.

The molecular structure of (4*a*) (Fig. 1) is composed of a trimethyl- and phenyl-substituted isoxazolidine unit wherein the heterocyclic ring adopts an envelope conformation, with N1 lying 0.714 (2) Å out of the plane formed by the other atoms in the ring, and an isoindole group that is essentially planar to within 0.0278 (11) Å and is substituted by a ketonic function, with O2 displaced 0.1111 (14) Å from this group. There are no classical hydrogen bonds in the structure.

A search of the Cambridge Structural Database (CSD, 2006 Release, Version 5.27; Allen, 2002) for a 5-amino-3,3,5-trimethyl-2-phenylisoxazolidine fragment yielded only three hits [CSD refcodes: PMAAN01, PMAAN10 and QQQBIY (Foster *et al.*, 1974)] reporting a 5-hydroxy(phenyl)amino compound. The crystal structure of (4*b*) is reported in the following paper (Parvez *et al.*, 2007).

Experimental

Acetone cyclodimer (2*b*), used as a starting material, was prepared by previously reported methods (Foster *et al.*, 1974). Acetone cyclodimer (2*b*) (0.67 mmol) in Et_2O (50 ml) was treated with ketene gas (10 mmol); thin-layer chromatographic monitoring revealed the complete conversion of the dimer into (4) after 20–25 min. The solution was then evaporated *in vacuo*. A single recrystallization from petroleum ether (b.p. 310–330 K) in an ice box gave pure *N*-substituted oxindoles (4*a*) (Mehrdad *et al.*, 2007).

Crystal data

$C_{20}H_{22}N_2O_2$
$M_r = 322.40$
Monoclinic, $P2_1/c$
$a = 10.321$ (3) Å
$b = 14.446$ (4) Å
$c = 11.378$ (4) Å
$\beta = 98.554$ (12)°
$V = 1677.6$ (9) Å^3
$Z = 4$
$D_x = 1.277$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 173$ (2) K
Prism, colorless
0.28 × 0.20 × 0.16 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{min} = 0.977$, $T_{max} = 0.987$
6053 measured reflections
3825 independent reflections
2632 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.108$
$S = 1.02$
3825 reflections
220 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.104P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

H atoms were located in difference Fourier maps and were included in the refinements at geometrically idealized positions, with C—H = 0.95, 0.98 and 0.99 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

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