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5-Methoxy-2,3-dimethylindole

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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.095
Data-to-parameter ratio = 9.3

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

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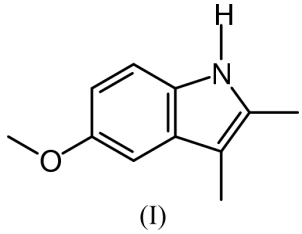
The attempted sublimation of the anti-inflammatory drug indomethacin gave a decomposition reaction from which crystals of 5-methoxy-2,3-dimethylindole, $C_{11}H_{13}NO$, were isolated. The crystal structure includes a close intermolecular approach of the amine H atom to the face of an indole ring.

Comment

During the course of studies on the polymorphic nature of the anti-inflammatory and antipyretic drug indomethacin [1-(*p*-chlorobenzoyl)-5-methoxy-2-methlindolylacetic acid; Shen *et al.*, 1963; Winter *et al.*, 1963], we attempted to grow single crystals by sublimation. Unexpectedly, the compound decomposed and only colourless, acicular crystals of 5-methoxy-2,3-dimethylindole, (I), and *para*-chlorobenzoic acid could be isolated.



(I)

As expected, the molecular structure of (I) is flat with no non-H atom lying more than 0.035 Å from the molecular plane (Fig. 1). The bond lengths and angles are also unexceptional, although the C2—C3 distance of 1.441 (4) Å does indicate a

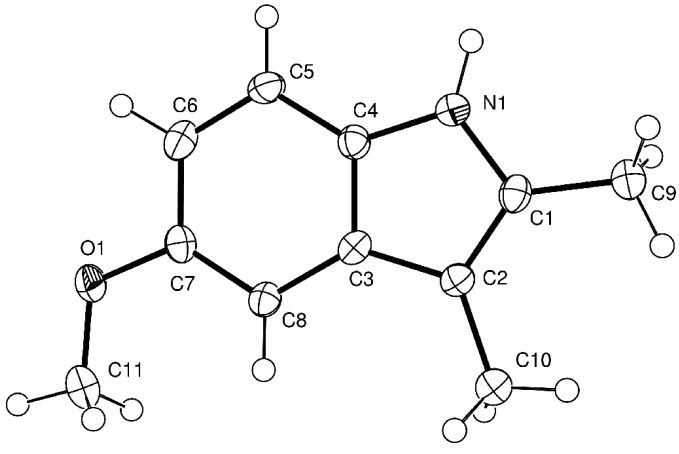


Figure 1
Displacement ellipsoid plot of (I). Non-H atoms are shown as 50% probability ellipsoids and H atoms as small spheres of arbitary size.

lesser degree of conjugation than is general. The most interesting feature is the intermolecular contacts of the amine H1 atom. It approaches the face of a neighbouring five-membered ring [the angle between molecular planes is 73.33 (5)°] in an offset manner, such that the shortest contacts are not with nitrogen but with the C1 and C2 atoms. [Distances from H1 to N1^{i}, C1^{i}, C2^{i}, C3^{i} and C4^{i} are 2.68 (4), 2.51 (3), 2.50 (3), 2.66 (3) and 2.77 (4) Å, respectively; symmetry code: (i) $1-x$, $\frac{1}{2}+y, 2-z$]. This is reminiscent of the preference of cations to interact with the heteroaromatic ring of indoles, discussed by Gokel *et al.* (2000) with respect to biological systems.

Experimental

Indomethacin (α-polymorph) was placed at the bottom of an evacuated sublimation tube and heated to 425 K. Crystals of (I) grew at the furthest end of the tube, where the temperature was approximately 295 K.

Crystal data

$C_{11}H_{13}NO$
$M_r = 175.22$
Monoclinic, $P2_1$
$a = 7.7769$ (4) Å
$b = 6.2492$ (3) Å
$c = 9.6263$ (5) Å
$\beta = 92.749$ (2)°
$V = 467.29$ (4) Å^3
$Z = 2$
$D_x = 1.245$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1140 reflections
$\theta = 1.0$–27.5°
$\mu = 0.08$ mm^{-1}
$T = 123$ (2) K
Needle, colourless
0.60 × 0.10 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
6426 measured reflections
1163 independent reflections
810 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.5°$
$h = -9 \rightarrow 10$
$k = -8 \rightarrow 8$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.095$
$S = 1.03$
1163 reflections
125 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

H1, the atom bonded to N1, was refined isotropically, but all other H atoms were placed in calculated positions with riding modes. The methyl groups were allowed to rotate but not to tip.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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