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3-Methyl-5-(3-nitrophenyl)cyclohex-2-enone

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Key indicators

Single-crystal X-ray study
T = 113 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.045
wR factor = 0.129
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.

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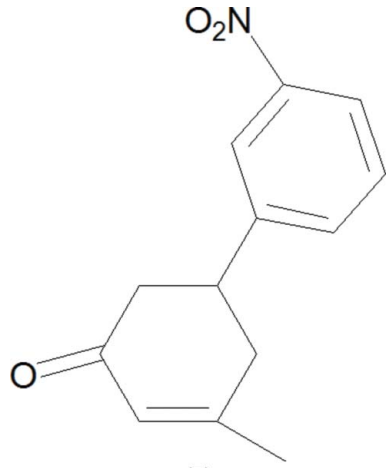
In the title compound, $C_{13}H_{13}N_1O_3$, the cyclohexene ring displays an envelope conformation, with the C atom attached to the benzene ring in the flap position. There is no hydrogen bonding in the crystal structure.

Comment

2,6-Dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylic acid monomethyl ester is an important intermediate for preparing dihydropyridine calcium antagonists (Yiu & Knaus, 1999; Goldmann & Stoltefuss, 1991). The title compound, 3-methyl-5-(3-nitrophenyl)cyclohex-2-enone, (I), is a by-product in the preparation of that intermediate (Fig. 1). The cyclohex-2-enone ring displays an envelope conformation, with atom C3 displaced from the mean plane through the other ring atoms by 0.660 (1) Å. The dihedral angle between the benzene ring and the C1/C2/C4/C5/C6 plane is 46.75 (1)°.



(I)

Experimental

The title compound was prepared from 3-*tert*-butyl 5-methyl 2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate, (II), and formic acid in aqueous ethanol. Compound (II) (900 mg, 2.32 mmol) was dissolved in aqueous ethanol (36 ml), and formic acid (12 ml) was added to the solution. The reaction mixture was stirred at refluxing temperature for a further 26 h. The solvent was removed by vacuum evaporation and the product was purified by chromatography on a silica-gel column (eluted by ethyl acetate and petroleum ether, 1:6 *v*/*v*) at room temperature. The product was obtained in a yield of 10%. Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a solution in ethyl acetate and methanol (1:1).

Crystal data

$C_{13}H_{13}NO_3$
M_r = 231.24
Monoclinic, $P2_1/c$
a = 10.5944 (14) Å
b = 11.2733 (15) Å
c = 9.5282 (10) Å
β = 97.703 (6)°
V = 1127.7 (2) Å^3
Z = 4
D_x = 1.362 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 113 (2) K
Block, colourless
0.18 × 0.16 × 0.14 mm

Data collection

Rigaku Saturn diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.980, T_{max} = 0.987
10401 measured reflections
2667 independent reflections
1760 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 27.9°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.129
S = 1.03
2667 reflections
156 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.069 (8)

H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C). The methyl H atoms are disordered and were refined over two sites, each constrained to ride on their parent C atom with 50% occupancy.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MSC, 2005); software used to prepare material for publication: *CrystalStructure*.

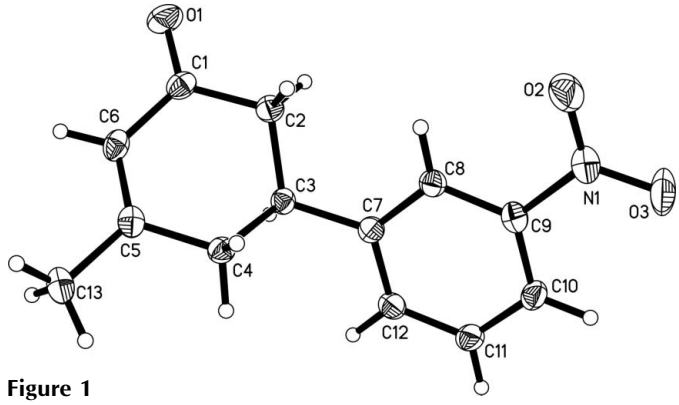


Figure 1
The molecular structure of the title compound, (I). Displacement ellipsoids are drawn at the 30% probability level. Only one disorder component is shown..

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References

Goldmann, S. & Stoltefuss, J. (1991). *Angew. Chem. Int. Ed. Engl.* **30**, 1559–1578.
Jacobson, R. (1998). Private communication to Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2005). *CrystalClear* (Version 1.36) and *CrystalStructure* (Version 3.7.0). Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Yiu, S. H. & Knaus, E. E. (1999). *Drug Dev. Res.* **48**, 26–37.