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(3*S**,4*S**)-1,3,4-Triphenylazetidin-2-one

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.077
Data-to-parameter ratio = 17.6

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

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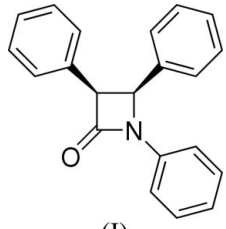
In the crystal structure of the title compound, $C_{21}H_{17}NO$, the nitrogen configuration is almost planar.

Comment

Fig. 1 shows the structure of the title compound, (I). Selected molecular parameters are listed in Table 1. The compound crystallizes in the monoclinic space group $P2_1/c$, with one molecule in the asymmetric unit. The nitrogen configuration is almost planar, as indicated by the large C16—N1—C2—C1 torsion angle [166.39 (9)°], which is different from the reported related structure (Deschamps *et al.*, 2003). This is due to the strong interactions between the nitrogen lone pair and the π electrons of the C═O double bond. There are no π–π stacking or other weak intermolecular interactions in (I), and the crystal packing (Fig. 2) is controlled by van der Waals forces.



(I)

Experimental

To a solution of phenylacetylene (102 mg, 1 mmol) in dimethylformamide (DMF, 3 ml) under argon at 273 K, triethylamine (101 mg, 1 mmol) was added and the mixture was stirred for 30 min. Cuprous iodide (190 mg, 1 mmol) was added and the solution was stirred for another 5 min, after which a DMF solution of the α,*N*-diphenylnitrone (197 mg, 1 mmol) was added slowly over a period of 5 min. After stirring for another 30 min, the reaction was stirred at room temperature overnight. The mixture was diluted with water and filtered through celite. The celite bed was washed with ethyl acetate (5 ml). The combined filtrate and washings were extracted with ethyl acetate (20 ml), which was washed three times with 10 ml aliquots of brine. After drying over anhydrous Na_2SO_4, the solvent was removed under reduced pressure and the resulting oil was purified by column chromatography to afford the product (I) (170 mg, yield 57%). Compound (I) was recrystallized from hexane as colorless crystals. ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.03 (*d*, 1H, *J* = 6.1 Hz), 5.48 (*d*, 1H, *J* = 6.1 Hz), 7.00–7.50 (*m*, 15H); 13 C NMR (125 MHz, $CDCl_3$): δ 60.53, 60.58, 117.47, 124.31, 127.37, 127.41, 128.11, 128.33, 128.47, 129.14, 129.34, 132.34, 134.61, 137.95.

Crystal data

$C_{21}H_{17}NO$	D_x = 1.241 Mg m^{-3}
M_r = 299.37	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 11258 reflections
a = 9.016 (3) Å	θ = 3.1–27.5°
b = 8.752 (3) Å	μ = 0.08 mm^{-1}
c = 20.664 (8) Å	T = 296 (1) K
β = 100.614 (14)°	Platelet, colorless
V = 1602.7 (10) Å^3	0.36 × 0.30 × 0.12 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID diffractometer	2161 reflections with $F^2 > 2\sigma(F^2)$
	R_{int} = 0.030
ω scans	θ_{max} = 27.5°
Absorption correction: none	$h = -11 \rightarrow 11$
15391 measured reflections	$k = -11 \rightarrow 11$
3669 independent reflections	$l = -26 \rightarrow 26$

Refinement

Refinement on F^2	$w = 1/[0.0001F_o^2 + 1.1\sigma(F_o^2)]/(4F_o^2)$
$R[F^2 > 2\sigma(F^2)]$ = 0.036	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.077	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
S = 1.00	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
3669 reflections	Extinction correction: Larson (1970)
209 parameters	
H-atom parameters constrained	Extinction coefficient: 5.2 (3) × 10^2

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.2122 (14)	N1—C3	1.3681 (14)
N1—C2	1.4773 (13)	N1—C16	1.4048 (14)
C2—N1—C3	94.64 (8)	C3—N1—C16	133.43 (8)
C2—N1—C16	129.93 (8)		

All H atoms were placed in calculated positions (C—H = 0.98 Å), with U_{iso}(H) = 1.2U_{eq} of the carrier atoms, and included in the final cycles of refinement using a riding model.

Data collection: *PROCESS-AUTO* (Rigaku,1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure* and *PLATON* (Spek, 2003).

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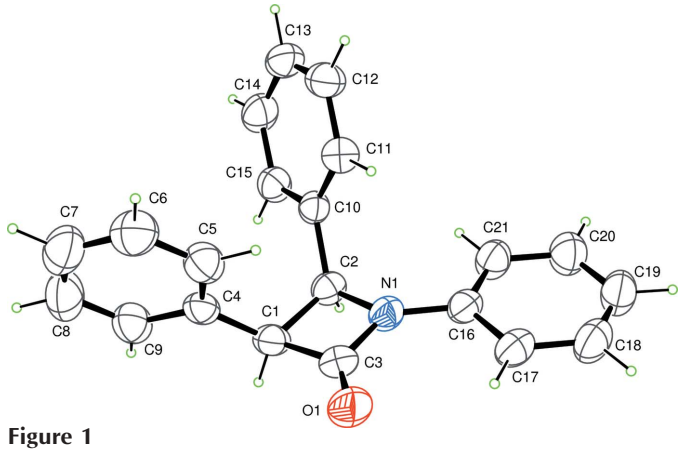

Figure 1
The molecule of (I). Displacement ellipsoids are drawn at the 40% probability level for non-H atoms.

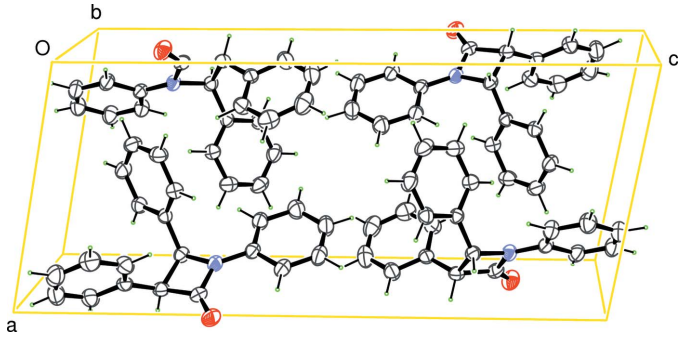

Figure 2
A packing diagram, viewed approximately along the *b* axis.

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