2-[(2-Phenyl-1,3-oxazol-4-yl)methyl]-2*H*-1,2,3-benzo-triazole

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Key indicators

Single-crystal X-ray study

$T = 180$ K

Mean σ(C–C) = 0.003 Å

R factor = 0.040

wR factor = 0.082

Data-to-parameter ratio = 8.3

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

The title compound, $C_{16}H_{12}N_4O$, was obtained through a two-step process. There are two molecules in the asymmetric unit with essentially equal bond lengths. The phenyl and oxazole rings are essentially coplanar. Steric interactions between the two halves of the molecule produce a wide angle at the central C atom.

Comment

The title compound, (I), was obtained through a two-step process. By reaction of 5-bromo-4-(bromomethyl)-2-phenyl-1,3-oxazole and a mixture of *N*-(1-cyanoethyl)benzene-sulfonamide and benzotriazole in the presence of a base and small traces of DMSO, the *N*-alkylation took place with the more reactive benzotriazole. A halogen–lithium exchange reaction was conducted on the obtained product resulting in the formation of (I).
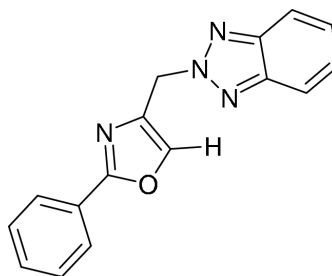

(I)

There are two molecules in the asymmetric unit. All corresponding bond lengths are equal within 2σ and in accordance with the chemical diagram. In both molecules, the dihedral angle between the phenyl and oxazole rings (less than 5°) indicates communicating π systems. The steric demand of the ring systems on the two sides of the molecule seems to have the effects (i) that the tetrahedral angle at the centre of the molecule formed by the non-H atoms is significantly enlarged to 112.0 (3) and 111.0 (3)° in the two molecules, respectively, and (ii) that the halves of the molecule are inclined to each other by about 43 and 33° in the two molecules.

Experimental

Following the method of Lesbosquain & Decruix (1993), 5-bromo-4-(bromomethyl)-2-phenyl-1,3-oxazole, K_2CO_3 and a mixture of *N*-(1-cyanoethyl)benzenesulfonamide and benzotriazole in the presence of traces of DMSO were refluxed in acetonitrile for 1 h. The resulting suspension was cooled, filtered and concentrated under reduced pressure, and the obtained compound was purified by column chro-
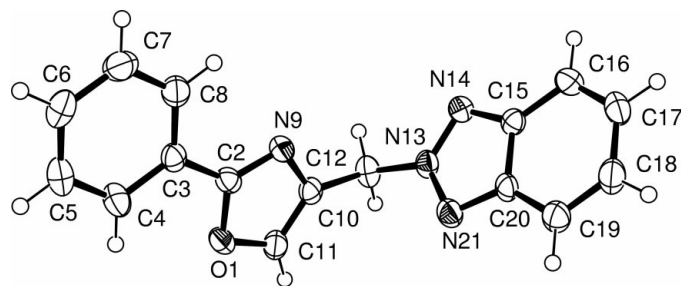
Figure 1
The structure of one molecule of (I) showing 50% probability ellipsoids.

matography. Br–Li exchange reaction was further carried out on the purified compound at 173 K for 2 h, as already described by Swaleh & Liebscher (1999). The compound isolated by subsequent column chromatography was recrystallized from absolute ethanol, affording (I).

Crystal data

$C_{16}H_{12}N_4O$	$D_x = 1.355$ Mg m^{-3}
$M_r = 276.30$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 4003
$a = 14.880$ (2) Å	reflections
$b = 4.6251$ (5) Å	$\theta = 3.1$–24.3°
$c = 20.043$ (3) Å	$\mu = 0.09$ mm^{-1}
$\beta = 100.963$ (19)°	$T = 180$ (2) K
$V = 1354.2$ (3) Å^3	Prism, colorless
$Z = 4$	$0.52 \times 0.24 \times 0.12$ mm

Data collection

Stoe IPDS diffractometer	$R_{int} = 0.068$
Scans: φ-rotation, φ-incr. = 1.2°, 183	$\theta_{max} = 25.9°$
exposures	$h = -18 \rightarrow 18$
9269 measured reflections	$k = -5 \rightarrow 5$
2943 independent reflections	$l = -24 \rightarrow 24$
2202 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.040$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.082$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.95$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
2943 reflections	$\Delta\rho_{min} = -0.16$ e Å^{-3}
356 parameters	Extinction correction: $SHELXL$97
H-atom parameters not refined	Extinction coefficient: 0.009 (2)

To save variable parameters, both monosubstituted benzene rings were refined as rigid ideal hexagons. This seems to be justified, because every ring is bonded to a C atom; and in such a case, according to Domenicano *et al.* (1975), the ideal symmetry of the C_6-hexagon is maintained. 2235 Friedel pairs were measured, *i.e.* 75.2% of those possible, and they were merged in the data set.

Data collection: *IPDS*-2.87 (Stoe & Cie, 1997); cell refinement: *IPDS*-2.87; data reduction: *IPDS*-2.87; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *OPTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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