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(*E*)-1-Benzyl-2-(*p*-methoxystyryl)-3-(*p*-tolyl)-1,2,3,4-tetrahydroquinazolin-4-one

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.182
Data-to-parameter ratio = 15.3

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

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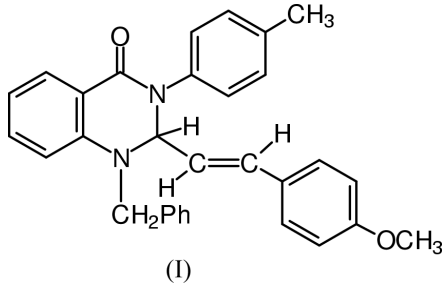
The title molecule, $C_{31}H_{28}N_2O_2$, consisting of a quinazoline moiety with benzyl, (*p*-methoxy)styryl and *p*-tolyl substituents at the 1, 2 and 3 positions, respectively, assumes an *E* configuration about the vinyl C=C bond. The six-membered heterocyclic ring adopts a distorted sofa conformation.

Comment

Substituted quinazolines display therapeutic activity and specific inhibitory effects on tyrosine kinase and dihydrofolate reductase (Jackman *et al.*, 1997; Bridges *et al.*, 1996; Rewcastle *et al.*, 1995). As part of our ongoing studies on the synthesis and characterization of new nitrogen-containing heterocyclic systems of biological importance (Maiti *et al.*, 2000; Mukherjee *et al.*, 1999) and to build up a hierarchy for such systems, the structure determination of 1-benzyl-2-[(*p*-methoxy)styryl]-3-(*p*-tolyl)-1,2,3,4-tetrahydroquinazoline-4-one, (I) (Fig. 1), was undertaken.



(I)

In (I), the vinyl C=C bond is in the *E* configuration [C15—C16—C17—C18 174.3 (2)°]. The six-membered heterocyclic ring (C1/C6/N1/C15/N2/C14) adopts a distorted sofa conformation with C15 deviating by −0.609 (2) Å from the least-squares plane (r.m.s deviation 0.038 Å) defined by the remaining endocyclic atoms. The benzyl and (*p*-methoxy)-styryl substituents are nearly perpendicular to the planar part of the quinazoline moiety with dihedral angles of 83.03 (4) and 83.14 (3)°, respectively. The *p*-tolyl group is oriented at an angle of 46.64 (6)°. The sum of the bond angles around N1 [349.1 (2)°] indicates a pyramidal configuration, whereas N2 [359.0 (2)°] is in the planar trigonal geometry. The bond lengths and angles in (I) are similar to those reported for substituted quinazoline structures (Kubicki *et al.*, 1997; Lindeman *et al.*, 1995). The *cis* orientation of C22—O2 with respect to C20—C21 about the O2—C21 bond [C22—O2—C21—C20 7.6 (3)°] results in repulsion between C20 and C22 and this causes the widening of C20—C21—O2 and narrowing

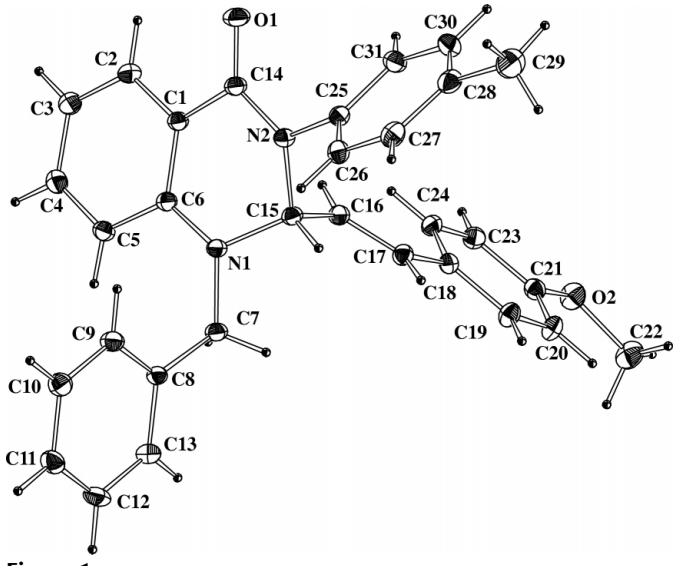


Figure 1
ZORTEP (Zsolnai, 1995) view (50% probability level) of the molecule (I).

of C23—C21—O2 from 120°. A similar observation has been reported for heterocyclic compounds with methoxyphenyl substituents (Mukherjee *et al.*, 2000).

The lack of π-bonding in the segments between phenyl rings precludes any possible π conjugation across the whole molecule. The aromatic nature of the rings is therefore localized within the rings and their direct substituents. Except for a weak C—H···O intermolecular interaction involving the carbonyl O atom (Table 2) the crystal packing is essentially stabilized by the van der Waals forces.

Experimental

A mixture of 2-{[*N*-benzyl-*N*-(prop-2′-ynyl)]amino}benzamide, *p*-methoxyiodobenzene, $(Ph_3P)_2PdCl_2$, CuI and triethylamine in acetonitrile was stirred for about 16 h at room temperature. After the usual work-up, the product was purified by column chromatography on silica gel (60–120 mesh) to yield a disubstituted alkyne which was then refluxed with NaOEt in ethanol for about 48 h. The crude product on chromatography through silica gel yielded the title compound, (I), on elution with $CHCl_3$. The m.p. of the title compound is 458 K. Single crystals suitable for X-ray analysis were obtained by slow crystallization from a dilute solution of (I) in a light petroleum (333–353 K)—chloroform mixture (3:1).

Crystal data

$C_{31}H_{28}N_2O_2$
M_r = 460.55
Monoclinic, $P2_1/c$
a = 13.281 (3) Å
b = 11.161 (2) Å
c = 17.756 (3) Å
β = 107.49 (1)°
V = 2510.3 (8) Å^3
Z = 4
D_x = 1.219 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 21 reflections
θ = 38.7–39.8°
μ = 0.60 mm^{-1}
T = 296 (2) K
Prismatic, colourless
0.50 × 0.37 × 0.25 mm

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: empirical (North *et al.*, 1968)
T_{min} = 0.754, T_{max} = 0.865
5062 measured reflections
4847 independent reflections
3442 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 77.3°
$h = -16 \rightarrow 16$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 21$
3 standard reflections
every 150 reflections
intensity decay: 0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.182
S = 1.04
4847 reflections
316 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.12P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C14	1.224 (2)	N2—C14	1.369 (2)
O2—C21	1.367 (2)	N2—C25	1.437 (3)
O2—C22	1.429 (3)	N2—C15	1.475 (2)
N1—C6	1.397 (2)	C15—C16	1.517 (3)
N1—C15	1.457 (3)	C16—C17	1.330 (3)
N1—C7	1.468 (2)	C17—C18	1.466 (3)
C6—N1—C15	115.5 (2)	C25—N2—C15	116.4 (2)
C6—N1—C7	119.4 (2)	C17—C16—C15	123.5 (2)
C15—N1—C7	114.2 (1)	C16—C17—C18	127.8 (2)
C14—N2—C25	122.8 (2)	O2—C21—C23	115.3 (2)
C14—N2—C15	119.8 (2)	O2—C21—C20	125.4 (2)
C6—N1—C7—C8	−84.1 (2)	C14—N2—C25—C31	−59.0 (3)
N1—C7—C8—C9	−6.6 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C22—H22A···O1^{i}	0.96	2.53	3.409 (3)	152

Symmetry code: (i) $1+x, \frac{1}{2}-y, \frac{1}{2}+z$.

H atoms were refined using a riding model and their isotropic displacement parameters were set to 1.2 times (1.5 times for CH_3 groups) the equivalent displacement parameters of their parent atoms.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1995); program(s) used to solve structure: *MULTAN*88 (Debaerdemaeker *et al.*, 1988); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1995); software used to prepare material for publication: *SHELXL*97.

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