Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[4-(diphenylmethyleneamino)phenyl]-methanone

Sylvain Bernès,[a]* Guadalupe Hernández,[b] Roberto Portillo,[b] Sandra Cruz[c] and René Gutiérrez[b]

[a]DEP Facultad de Ciencias Químicas, UANL, Guerrero y Progreso S/N, Col. Treviño, 64570 Monterrey, NL, Mexico, [b]Laboratorio de Síntesis de Complejos, Facultad de Ciencias Químicas, Universidad Autónoma de Puebla, AP 1067, 72001 Puebla, Pue., Mexico, and [c]Departamento de Ingeniería Química, Universidad Politécnica de Tlaxcala, Calle 21, no. 611, Col. La Loma Xicohténcatl, Tlaxcala, Tlax., Mexico
Correspondence e-mail: sylvain_bernes@Hotmail.com

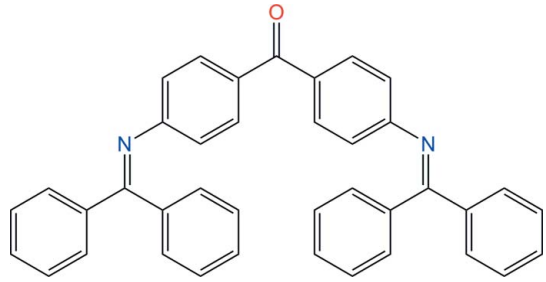
Received 23 April 2010; accepted 4 May 2010

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.113; data-to-parameter ratio = 15.4.

The title molecule, $C_{39}H_{28}N_2O$, is a well known dendron used in the synthesis of phenylazomethine dendrimers. The central benzophenone core is twisted, as expected, due to hindrance between H atoms: the dihedral angle between core benzene rings is 54.49 (5)°, identical to that of the stable polymorph of benzophenone (56°). For the same reason, phenyl groups substituting imine C atoms make a large dihedral angle, although similar for each imine: 71.83 (6) and 67.64 (5)°. The six aromatic rings in the molecule thus seem to be quite randomly oriented, and such an arrangement is not favorable for efficient stacking interactions in the crystal. The same behaviour is observed in the vast majority of diphenylimino-containing organics. The low triclinic crystal symmetry may be a consequence of these features.

Related literature

For the use of the title molecule in the synthesis of dendritic systems, see: Higuchi *et al.* (2001); Takanashi *et al.* (2004); Yamamoto & Higuchi (2004). For the structure of benzophenone, see: Fleischer *et al.* (1968); Kutzke *et al.* (2000). For related structures including the diphenylimino fragment, see: Appel *et al.* (1985); Buhmann *et al.* (1993). For geometrical analysis using the Cambridge Structural Database, see: Bruno *et al.* (2002).



Experimental

Crystal data

$C_{39}H_{28}N_2O$
M_r = 540.63
Triclinic, $P\overline{1}$
a = 11.1723 (10) Å
b = 11.3487 (13) Å
c = 13.2331 (15) Å
α = 103.121 (9)°
β = 105.170 (8)°
γ = 108.746 (8)°
V = 1441.9 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 296 K
0.55 × 0.28 × 0.24 mm

Data collection

Bruker P4 diffractometer
6892 measured reflections
5865 independent reflections
4471 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
3 standard reflections every 97 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.113
S = 1.03
5865 reflections
380 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* .

Partial support from VIEP-UAP (GUPJ-NAT08-G) is acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2303).

References

Appel, R., Knoch, F. & Zimmermann, R. (1985). *Chem. Ber.* **118**, 814–824.
Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* B**58**, 389–397.
Buhmann, M., Würthwein, E.-U. & Möller, M. H. (1993). *Chem. Ber.* **126**, 957–967.
Fleischer, E. B., Sung, N. & Hawkinson, S. (1968). *J. Phys. Chem.* **72**, 4311–4312.
Higuchi, M., Shiki, S., Ariga, K. & Yamamoto, K. (2001). *J. Am. Chem. Soc.* **123**, 4414–4420.
Kutzke, H., Klapper, H., Hammond, R. B. & Roberts, K. J. (2000). *Acta Cryst.* B**56**, 486–496.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *XSCANS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Takanashi, K., Chiba, H., Higuchi, M. & Yamamoto, K. (2004). *Org. Lett*. **6**, 1709–1712.
Yamamoto, K. & Higuchi, M. (2004). *Pure Appl. Chem*. **76**, 1399–1408.

supplementary materials

Acta Cryst. (2010). E**66**, o1322-o1323 [**doi:10.1107/S1600536810016375**]

Bis[4-(diphenylmethyleneamino)phenyl]methanone

S. Bernès, G. Hernández, R. Portillo, S. Cruz and R. Gutiérrez

Comment

The title benzophenone derivative has been widely employed as a dendron in the synthesis of phenylazomethine dendrimers (DPAs), mostly in the group of Yamamoto at the Keio University (Higuchi *et al.*, 2001; Takanashi *et al.*, 2004; Yamamoto & Higuchi, 2004). This group and others reported on the preparation of a vast array of supramolecular entities with interesting properties. We became interested in preparing this dendron by using microwave heating, given that it is becoming an important method in laboratories worldwide: it is an environment-friendly technique for the efficient syntheses of organic molecules. The main advantages of microwave-assisted organic synthesis are shorter reaction times, minimum waste and generally higher yields, operational simplicity as well as reduction of thermal degradative byproducts along with cleaner work-up. As expected, better yields were obtained and we realized that, surprisingly, the crystal structure had not been reported so far.

The molecule (Fig. 1) crystallizes in the low symmetry space group $P\bar{1}$. The imine bond lengths, 1.2813 (18) and 1.2784 (19) Å, are as expected, however, N atoms significantly deviate from trigonality. Large C═N—C angles are observed, 127.61 (12) and 123.09 (13)°, probably because of the steric repulsion between the central benzophenone benzene rings and the diphenylmethylene groups. The central benzophenone displays a twisted conformation, the dihedral angle between benzene rings being 54.49 (5)°. This value is indeed close to that reported for benzophenone, 56° (orthorhombic phase, Fleischer *et al.*, 1968) or 64° (metastable monoclinic phase, Kutzke *et al.*, 2000). This conformation avoids any intramolecular H···H contacts. In the same way, diphenyl groups bonded to imine C atoms are twisted, by 71.83 (6)° (diphenyl group at C9) and 67.64 (5)° (diphenyl group at C29). These angles are common for diphenylimino-containing organics (range of angles retrieved from the CSD : 57 to 90°; CSD, version 5.31 with all updates; Bruno *et al.*, 2002).

As a whole, the six rings in the molecule seem to be randomly oriented. This chaotic arrangement is consistent with the low crystal symmetry, and does not favor π···π or C—H···π interactions in the crystal structure. For example, the shortest intermolecular separation between centroids of two rings is 4.45 Å. The calculated packing index is indeed low for this polyphenyl molecule, 0.672. A search in the CSD for organic molecules containing the Ph_2C═N fragment shows that more densely packed crystals in this class of compounds are scarce. For 151 hits, only two structures present symmetry-related diphenylimino groups with phenyl rings separated by less than 3.9 Å (Appel *et al.*, 1985; Buhmann *et al.*, 1993).

Experimental

A modified procedure for improved synthesis of the title compound was used. The Higuchi's route (Higuchi *et al.*, 2001; see compound 'dendron G2' in this paper) consists of the condensation between benzophenone and 4,4'-diaminobenzophenone in presence of DABCO (1,4-diazabicyclo[2.2.2]octane) and $TiCl_4$, in chlorobenzene. In the original synthesis, the mixture was heated at 398 K for 24 h to afford dendron G2 in 48% yield. In place of thermal activation, we performed a microwave-assisted synthesis in a monomode MIC-1 oven (Tekno-lab, S.A.) with maximum power output of 600 W. Irradiation was applied for 20 min., affording the title compound with an enhanced yield of 65% after silica gel column chromatography (ethyl acetate:hexane = 1:5). Single crystals were obtained by slow evaporation of the eluate at 298 K.

Refinement

All H atoms were placed in idealized positions and refined as riding to their carrier C atoms, with bond lengths fixed to 0.93 Å. Isotropic displacement parameters were calculated as U_{iso}(H) = 1.2U_{eq}(carrier C atom).

Figures

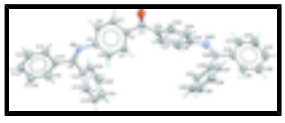


Fig. 1. Molecular structure of the title compound, with 50% probability level displacement ellipsoids for non-H atoms.

Bis[4-(diphenylmethyleneamino)phenyl]methanone

Crystal data

$C_{39}H_{28}N_2O$	$Z = 2$
$M_r = 540.63$	$F(000) = 568$
Triclinic, $P\overline{1}$	$D_x = 1.245$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 11.1723$ (10) Å	Cell parameters from 82 reflections
$b = 11.3487$ (13) Å	$\theta = 4.6$–$12.5°$
$c = 13.2331$ (15) Å	$\mu = 0.08$ mm^{-1}
$\alpha = 103.121$ (9)°	$T = 296$ K
$\beta = 105.170$ (8)°	Prism, yellow
$\gamma = 108.746$ (8)°	0.55 × 0.28 × 0.24 mm
$V = 1441.9$ (3) Å^3	

Data collection

Bruker P4 diffractometer	$R_{int} = 0.018$
Radiation source: fine-focus sealed tube	$\theta_{max} = 26.4°$, $\theta_{min} = 2.0°$
graphite	$h = -13\rightarrow2$
2θ/ω scans	$k = -13\rightarrow13$
6892 measured reflections	$l = -16\rightarrow16$
5865 independent reflections	3 standard reflections every 97 reflections
4471 reflections with $I > 2\sigma(I)$	intensity decay: 1%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters constrained
$wR(F^2) = 0.113$	$w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 0.275P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
5865 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
380 parameters	$\Delta\rho_{min} = -0.14$ e Å^{-3}
0 restraints	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
0 constraints	Extinction coefficient: 0.0198 (18)
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.81457 (15)	0.23158 (15)	0.66331 (12)	0.0425 (3)
O1	0.92972 (12)	0.26828 (14)	0.66240 (11)	0.0658 (4)
C2	0.70743 (14)	0.24601 (14)	0.57791 (12)	0.0383 (3)
C3	0.72166 (15)	0.24315 (15)	0.47594 (12)	0.0425 (3)
H3A	0.7902	0.2224	0.4599	0.051*
C4	0.63532 (15)	0.27069 (15)	0.39873 (12)	0.0407 (3)
H4A	0.6459	0.2673	0.3309	0.049*
C5	0.53212 (14)	0.30355 (13)	0.42020 (11)	0.0373 (3)
C6	0.51690 (15)	0.30587 (15)	0.52197 (12)	0.0424 (3)
H6A	0.4486	0.3270	0.5381	0.051*
C7	0.60322 (15)	0.27683 (15)	0.59917 (12)	0.0416 (3)
H7A	0.5914	0.2779	0.6663	0.050*
N8	0.46589 (12)	0.34758 (12)	0.33981 (10)	0.0419 (3)
C9	0.34487 (14)	0.34189 (13)	0.31471 (11)	0.0374 (3)
C10	0.30371 (14)	0.40362 (14)	0.23057 (12)	0.0395 (3)
C11	0.39664 (17)	0.46820 (17)	0.18792 (13)	0.0505 (4)
H11A	0.4834	0.4696	0.2098	0.061*
C12	0.3613 (2)	0.5302 (2)	0.11344 (15)	0.0614 (5)
H12A	0.4245	0.5732	0.0858	0.074*
C13	0.23276 (19)	0.52870 (18)	0.07989 (14)	0.0585 (4)
H13A	0.2094	0.5712	0.0304	0.070*
C14	0.14043 (18)	0.46438 (19)	0.11986 (15)	0.0614 (5)
H14A	0.0534	0.4622	0.0968	0.074*
C15	0.17516 (16)	0.40203 (17)	0.19474 (14)	0.0536 (4)
H15A	0.1109	0.3585	0.2212	0.064*
C16	0.24182 (14)	0.27839 (14)	0.36114 (11)	0.0372 (3)
C17	0.20701 (16)	0.35573 (16)	0.43619 (13)	0.0473 (4)
H17A	0.2458	0.4474	0.4564	0.057*
C18	0.11442 (17)	0.29636 (18)	0.48108 (13)	0.0529 (4)
H18A	0.0925	0.3485	0.5321	0.064*
C19	0.05504 (17)	0.16072 (18)	0.45026 (14)	0.0547 (4)
H19A	−0.0065	0.1214	0.4808	0.066*
C20	0.08672 (18)	0.08337 (17)	0.37436 (16)	0.0575 (4)
H20A	0.0454	−0.0083	0.3527	0.069*
C21	0.18011 (16)	0.14185 (15)	0.33012 (14)	0.0477 (4)

H21A	0.2015	0.0890	0.2792	0.057*
C22	0.78337 (15)	0.17280 (14)	0.74811 (12)	0.0404 (3)
C23	0.89013 (16)	0.20074 (16)	0.84510 (13)	0.0475 (4)
H23A	0.9765	0.2609	0.8578	0.057*
C24	0.87042 (17)	0.14101 (16)	0.92269 (13)	0.0513 (4)
H24A	0.9428	0.1621	0.9875	0.062*
C25	0.74229 (16)	0.04910 (14)	0.90414 (12)	0.0436 (3)
C26	0.63567 (16)	0.02012 (16)	0.80788 (13)	0.0465 (4)
H26A	0.5500	−0.0420	0.7944	0.056*
C27	0.65529 (16)	0.08267 (15)	0.73147 (12)	0.0448 (3)
H27A	0.5820	0.0642	0.6683	0.054*
N28	0.72437 (15)	−0.02708 (13)	0.97419 (11)	0.0495 (3)
C29	0.73136 (14)	0.02098 (14)	1.07383 (12)	0.0391 (3)
C30	0.72464 (14)	−0.06736 (14)	1.14219 (12)	0.0409 (3)
C31	0.7430 (2)	−0.18335 (17)	1.10693 (15)	0.0584 (4)
H31A	0.7576	−0.2060	1.0405	0.070*
C32	0.7397 (2)	−0.2648 (2)	1.17030 (18)	0.0717 (6)
H32A	0.7527	−0.3419	1.1465	0.086*
C33	0.7174 (2)	−0.23299 (19)	1.26838 (16)	0.0657 (5)
H33A	0.7156	−0.2883	1.3107	0.079*
C34	0.69800 (19)	−0.11972 (19)	1.30364 (15)	0.0598 (5)
H34A	0.6823	−0.0985	1.3697	0.072*
C35	0.70164 (16)	−0.03645 (16)	1.24098 (13)	0.0487 (4)
H35A	0.6886	0.0404	1.2654	0.058*
C36	0.74461 (14)	0.15897 (14)	1.12429 (12)	0.0386 (3)
C37	0.85587 (15)	0.24739 (15)	1.21870 (13)	0.0452 (3)
H37A	0.9195	0.2189	1.2534	0.054*
C38	0.8732 (2)	0.37737 (17)	1.26174 (15)	0.0577 (4)
H38A	0.9499	0.4367	1.3232	0.069*
C39	0.7766 (2)	0.41837 (19)	1.21333 (17)	0.0657 (5)
H39A	0.7879	0.5056	1.2423	0.079*
C40	0.6634 (2)	0.3310 (2)	1.12217 (16)	0.0655 (5)
H40A	0.5973	0.3588	1.0911	0.079*
C41	0.64726 (18)	0.20148 (18)	1.07617 (14)	0.0513 (4)
H41A	0.5716	0.1434	1.0134	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0422 (8)	0.0497 (9)	0.0425 (8)	0.0226 (7)	0.0182 (6)	0.0192 (7)
O1	0.0458 (7)	0.1042 (10)	0.0667 (8)	0.0347 (7)	0.0264 (6)	0.0504 (8)
C2	0.0402 (7)	0.0395 (7)	0.0392 (7)	0.0174 (6)	0.0164 (6)	0.0164 (6)
C3	0.0430 (8)	0.0527 (9)	0.0414 (8)	0.0253 (7)	0.0209 (7)	0.0179 (7)
C4	0.0414 (8)	0.0510 (8)	0.0347 (7)	0.0192 (7)	0.0181 (6)	0.0177 (6)
C5	0.0350 (7)	0.0376 (7)	0.0397 (7)	0.0130 (6)	0.0138 (6)	0.0161 (6)
C6	0.0424 (8)	0.0517 (8)	0.0437 (8)	0.0243 (7)	0.0220 (7)	0.0200 (7)
C7	0.0468 (8)	0.0499 (8)	0.0364 (7)	0.0229 (7)	0.0201 (6)	0.0186 (6)
N8	0.0394 (7)	0.0496 (7)	0.0434 (7)	0.0193 (6)	0.0172 (5)	0.0232 (6)

C9	0.0381 (7)	0.0367 (7)	0.0374 (7)	0.0149 (6)	0.0144 (6)	0.0123 (6)
C10	0.0392 (8)	0.0399 (8)	0.0383 (7)	0.0154 (6)	0.0130 (6)	0.0137 (6)
C11	0.0499 (9)	0.0678 (11)	0.0520 (9)	0.0316 (8)	0.0268 (8)	0.0320 (8)
C12	0.0657 (11)	0.0802 (13)	0.0621 (11)	0.0352 (10)	0.0355 (9)	0.0438 (10)
C13	0.0653 (11)	0.0697 (11)	0.0510 (9)	0.0329 (9)	0.0185 (8)	0.0343 (9)
C14	0.0475 (9)	0.0747 (12)	0.0651 (11)	0.0275 (9)	0.0112 (8)	0.0360 (10)
C15	0.0381 (8)	0.0637 (10)	0.0615 (10)	0.0174 (8)	0.0157 (7)	0.0329 (9)
C16	0.0337 (7)	0.0412 (7)	0.0382 (7)	0.0161 (6)	0.0121 (6)	0.0160 (6)
C17	0.0423 (8)	0.0450 (8)	0.0481 (8)	0.0151 (7)	0.0154 (7)	0.0089 (7)
C18	0.0487 (9)	0.0704 (11)	0.0422 (8)	0.0269 (8)	0.0210 (7)	0.0142 (8)
C19	0.0466 (9)	0.0738 (12)	0.0572 (10)	0.0240 (8)	0.0268 (8)	0.0376 (9)
C20	0.0563 (10)	0.0488 (9)	0.0772 (12)	0.0199 (8)	0.0311 (9)	0.0328 (9)
C21	0.0514 (9)	0.0426 (8)	0.0591 (9)	0.0223 (7)	0.0282 (8)	0.0205 (7)
C22	0.0453 (8)	0.0446 (8)	0.0380 (7)	0.0234 (7)	0.0167 (6)	0.0168 (6)
C23	0.0447 (8)	0.0502 (9)	0.0439 (8)	0.0159 (7)	0.0120 (7)	0.0189 (7)
C24	0.0524 (9)	0.0550 (9)	0.0376 (8)	0.0181 (8)	0.0059 (7)	0.0176 (7)
C25	0.0571 (9)	0.0407 (8)	0.0372 (7)	0.0219 (7)	0.0195 (7)	0.0147 (6)
C26	0.0461 (8)	0.0487 (9)	0.0441 (8)	0.0167 (7)	0.0170 (7)	0.0176 (7)
C27	0.0437 (8)	0.0519 (9)	0.0389 (7)	0.0208 (7)	0.0119 (6)	0.0173 (7)
N28	0.0639 (9)	0.0438 (7)	0.0414 (7)	0.0212 (6)	0.0180 (6)	0.0181 (6)
C29	0.0350 (7)	0.0417 (8)	0.0405 (8)	0.0147 (6)	0.0114 (6)	0.0180 (6)
C30	0.0374 (7)	0.0428 (8)	0.0423 (8)	0.0152 (6)	0.0111 (6)	0.0194 (6)
C31	0.0796 (12)	0.0550 (10)	0.0570 (10)	0.0361 (9)	0.0311 (9)	0.0284 (8)
C32	0.0982 (16)	0.0598 (11)	0.0783 (13)	0.0440 (11)	0.0347 (12)	0.0407 (10)
C33	0.0734 (12)	0.0606 (11)	0.0667 (12)	0.0231 (10)	0.0186 (10)	0.0418 (10)
C34	0.0614 (11)	0.0696 (12)	0.0512 (9)	0.0185 (9)	0.0243 (8)	0.0328 (9)
C35	0.0497 (9)	0.0516 (9)	0.0509 (9)	0.0203 (7)	0.0217 (7)	0.0246 (7)
C36	0.0397 (7)	0.0448 (8)	0.0427 (7)	0.0204 (6)	0.0213 (6)	0.0230 (6)
C37	0.0415 (8)	0.0470 (8)	0.0523 (9)	0.0195 (7)	0.0203 (7)	0.0201 (7)
C38	0.0665 (11)	0.0458 (9)	0.0624 (10)	0.0193 (8)	0.0317 (9)	0.0165 (8)
C39	0.1079 (16)	0.0549 (10)	0.0672 (12)	0.0482 (11)	0.0528 (12)	0.0327 (9)
C40	0.0996 (15)	0.0870 (14)	0.0656 (12)	0.0726 (13)	0.0506 (12)	0.0506 (11)
C41	0.0556 (10)	0.0696 (11)	0.0486 (9)	0.0373 (9)	0.0239 (8)	0.0322 (8)

Geometric parameters (Å, °)

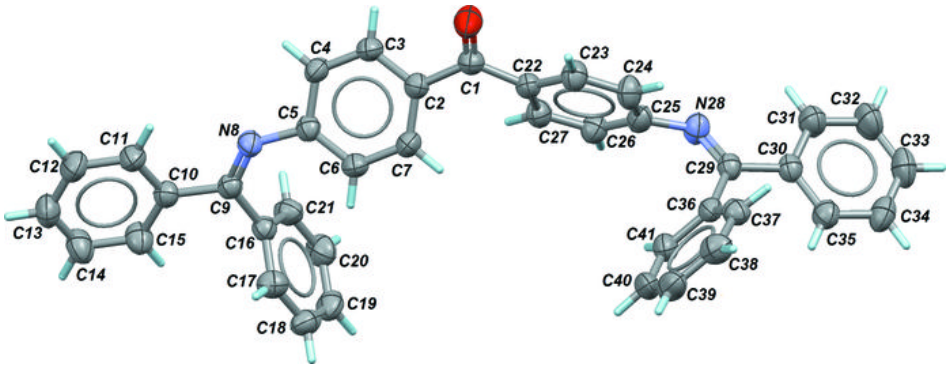
C1—O1	1.2225 (18)	C21—H21A	0.9300
C1—C22	1.490 (2)	C22—C27	1.391 (2)
C1—C2	1.496 (2)	C22—C23	1.393 (2)
C2—C7	1.395 (2)	C23—C24	1.379 (2)
C2—C3	1.394 (2)	C23—H23A	0.9300
C3—C4	1.377 (2)	C24—C25	1.392 (2)
C3—H3A	0.9300	C24—H24A	0.9300
C4—C5	1.396 (2)	C25—C26	1.383 (2)
C4—H4A	0.9300	C25—N28	1.4130 (19)
C5—C6	1.3964 (19)	C26—C27	1.384 (2)
C5—N8	1.4127 (18)	C26—H26A	0.9300
C6—C7	1.387 (2)	C27—H27A	0.9300
C6—H6A	0.9300	N28—C29	1.2784 (19)

C7—H7A	0.9300	C29—C30	1.4926 (19)
N8—C9	1.2813 (18)	C29—C36	1.498 (2)
C9—C10	1.4950 (19)	C30—C35	1.387 (2)
C9—C16	1.5033 (19)	C30—C31	1.392 (2)
C10—C15	1.382 (2)	C31—C32	1.380 (2)
C10—C11	1.393 (2)	C31—H31A	0.9300
C11—C12	1.383 (2)	C32—C33	1.375 (3)
C11—H11A	0.9300	C32—H32A	0.9300
C12—C13	1.381 (3)	C33—C34	1.370 (3)
C12—H12A	0.9300	C33—H33A	0.9300
C13—C14	1.361 (2)	C34—C35	1.390 (2)
C13—H13A	0.9300	C34—H34A	0.9300
C14—C15	1.388 (2)	C35—H35A	0.9300
C14—H14A	0.9300	C36—C37	1.389 (2)
C15—H15A	0.9300	C36—C41	1.391 (2)
C16—C21	1.386 (2)	C37—C38	1.384 (2)
C16—C17	1.391 (2)	C37—H37A	0.9300
C17—C18	1.390 (2)	C38—C39	1.373 (3)
C17—H17A	0.9300	C38—H38A	0.9300
C18—C19	1.376 (2)	C39—C40	1.375 (3)
C18—H18A	0.9300	C39—H39A	0.9300
C19—C20	1.375 (2)	C40—C41	1.389 (3)
C19—H19A	0.9300	C40—H40A	0.9300
C20—C21	1.387 (2)	C41—H41A	0.9300
C20—H20A	0.9300		
O1—C1—C22	119.75 (13)	C20—C21—H21A	119.7
O1—C1—C2	118.82 (13)	C27—C22—C23	118.10 (13)
C22—C1—C2	121.43 (13)	C27—C22—C1	123.37 (13)
C7—C2—C3	118.23 (13)	C23—C22—C1	118.30 (14)
C7—C2—C1	123.51 (13)	C24—C23—C22	121.35 (15)
C3—C2—C1	117.86 (13)	C24—C23—H23A	119.3
C4—C3—C2	120.68 (13)	C22—C23—H23A	119.3
C4—C3—H3A	119.7	C23—C24—C25	120.03 (14)
C2—C3—H3A	119.7	C23—C24—H24A	120.0
C3—C4—C5	121.35 (13)	C25—C24—H24A	120.0
C3—C4—H4A	119.3	C26—C25—C24	119.11 (14)
C5—C4—H4A	119.3	C26—C25—N28	119.71 (14)
C4—C5—C6	118.22 (13)	C24—C25—N28	120.61 (14)
C4—C5—N8	114.03 (12)	C25—C26—C27	120.64 (15)
C6—C5—N8	127.22 (13)	C25—C26—H26A	119.7
C7—C6—C5	120.31 (13)	C27—C26—H26A	119.7
C7—C6—H6A	119.8	C26—C27—C22	120.73 (14)
C5—C6—H6A	119.8	C26—C27—H27A	119.6
C6—C7—C2	121.20 (13)	C22—C27—H27A	119.6
C6—C7—H7A	119.4	C29—N28—C25	123.09 (13)
C2—C7—H7A	119.4	N28—C29—C30	117.26 (13)
C9—N8—C5	127.61 (12)	N28—C29—C36	123.77 (13)
N8—C9—C10	116.13 (13)	C30—C29—C36	118.97 (12)
N8—C9—C16	126.07 (13)	C35—C30—C31	118.81 (14)

C10—C9—C16	117.80 (12)	C35—C30—C29	121.70 (14)
C15—C10—C11	117.84 (14)	C31—C30—C29	119.49 (14)
C15—C10—C9	121.89 (13)	C32—C31—C30	120.19 (17)
C11—C10—C9	120.25 (13)	C32—C31—H31A	119.9
C12—C11—C10	120.72 (15)	C30—C31—H31A	119.9
C12—C11—H11A	119.6	C33—C32—C31	120.56 (18)
C10—C11—H11A	119.6	C33—C32—H32A	119.7
C13—C12—C11	120.39 (16)	C31—C32—H32A	119.7
C13—C12—H12A	119.8	C34—C33—C32	119.89 (16)
C11—C12—H12A	119.8	C34—C33—H33A	120.1
C14—C13—C12	119.44 (15)	C32—C33—H33A	120.1
C14—C13—H13A	120.3	C33—C34—C35	120.21 (17)
C12—C13—H13A	120.3	C33—C34—H34A	119.9
C13—C14—C15	120.52 (16)	C35—C34—H34A	119.9
C13—C14—H14A	119.7	C30—C35—C34	120.34 (16)
C15—C14—H14A	119.7	C30—C35—H35A	119.8
C10—C15—C14	121.07 (15)	C34—C35—H35A	119.8
C10—C15—H15A	119.5	C37—C36—C41	118.92 (14)
C14—C15—H15A	119.5	C37—C36—C29	120.45 (13)
C21—C16—C17	118.85 (14)	C41—C36—C29	120.62 (14)
C21—C16—C9	120.62 (13)	C38—C37—C36	120.79 (15)
C17—C16—C9	120.53 (13)	C38—C37—H37A	119.6
C16—C17—C18	120.17 (15)	C36—C37—H37A	119.6
C16—C17—H17A	119.9	C39—C38—C37	119.74 (18)
C18—C17—H17A	119.9	C39—C38—H38A	120.1
C19—C18—C17	120.25 (15)	C37—C38—H38A	120.1
C19—C18—H18A	119.9	C38—C39—C40	120.28 (17)
C17—C18—H18A	119.9	C38—C39—H39A	119.9
C20—C19—C18	119.98 (15)	C40—C39—H39A	119.9
C20—C19—H19A	120.0	C39—C40—C41	120.41 (17)
C18—C19—H19A	120.0	C39—C40—H40A	119.8
C19—C20—C21	120.12 (16)	C41—C40—H40A	119.8
C19—C20—H20A	119.9	C40—C41—C36	119.79 (17)
C21—C20—H20A	119.9	C40—C41—H41A	120.1
C16—C21—C20	120.60 (15)	C36—C41—H41A	120.1
C16—C21—H21A	119.7		
O1—C1—C2—C7	−144.52 (16)	O1—C1—C22—C27	−152.96 (16)
C22—C1—C2—C7	36.0 (2)	C2—C1—C22—C27	26.6 (2)
O1—C1—C2—C3	28.0 (2)	O1—C1—C22—C23	21.4 (2)
C22—C1—C2—C3	−151.51 (14)	C2—C1—C22—C23	−159.09 (14)
C7—C2—C3—C4	0.3 (2)	C27—C22—C23—C24	0.2 (2)
C1—C2—C3—C4	−172.64 (14)	C1—C22—C23—C24	−174.49 (14)
C2—C3—C4—C5	0.7 (2)	C22—C23—C24—C25	1.0 (3)
C3—C4—C5—C6	−1.0 (2)	C23—C24—C25—C26	−0.7 (2)
C3—C4—C5—N8	171.19 (13)	C23—C24—C25—N28	170.63 (14)
C4—C5—C6—C7	0.4 (2)	C24—C25—C26—C27	−0.8 (2)
N8—C5—C6—C7	−170.67 (14)	N28—C25—C26—C27	−172.23 (14)
C5—C6—C7—C2	0.6 (2)	C25—C26—C27—C22	2.0 (2)
C3—C2—C7—C6	−0.9 (2)	C23—C22—C27—C26	−1.7 (2)

C1—C2—C7—C6	171.58 (14)	C1—C22—C27—C26	172.67 (14)
C4—C5—N8—C9	154.35 (15)	C26—C25—N28—C29	−115.96 (18)
C6—C5—N8—C9	−34.3 (2)	C24—C25—N28—C29	72.8 (2)
C5—N8—C9—C10	176.47 (13)	C25—N28—C29—C30	−173.82 (14)
C5—N8—C9—C16	−4.1 (2)	C25—N28—C29—C36	6.7 (2)
N8—C9—C10—C15	178.78 (15)	N28—C29—C30—C35	−165.71 (15)
C16—C9—C10—C15	−0.7 (2)	C36—C29—C30—C35	13.8 (2)
N8—C9—C10—C11	−3.0 (2)	N28—C29—C30—C31	14.9 (2)
C16—C9—C10—C11	177.57 (14)	C36—C29—C30—C31	−165.62 (15)
C15—C10—C11—C12	1.0 (2)	C35—C30—C31—C32	−0.7 (3)
C9—C10—C11—C12	−177.38 (15)	C29—C30—C31—C32	178.75 (17)
C10—C11—C12—C13	−0.2 (3)	C30—C31—C32—C33	0.4 (3)
C11—C12—C13—C14	−0.7 (3)	C31—C32—C33—C34	0.2 (3)
C12—C13—C14—C15	0.7 (3)	C32—C33—C34—C35	−0.4 (3)
C11—C10—C15—C14	−0.9 (3)	C31—C30—C35—C34	0.4 (2)
C9—C10—C15—C14	177.43 (16)	C29—C30—C35—C34	−179.01 (15)
C13—C14—C15—C10	0.0 (3)	C33—C34—C35—C30	0.1 (3)
N8—C9—C16—C21	−72.1 (2)	N28—C29—C36—C37	−120.14 (17)
C10—C9—C16—C21	107.31 (16)	C30—C29—C36—C37	60.38 (18)
N8—C9—C16—C17	107.75 (18)	N28—C29—C36—C41	58.6 (2)
C10—C9—C16—C17	−72.83 (18)	C30—C29—C36—C41	−120.93 (15)
C21—C16—C17—C18	1.6 (2)	C41—C36—C37—C38	−2.8 (2)
C9—C16—C17—C18	−178.23 (14)	C29—C36—C37—C38	175.92 (14)
C16—C17—C18—C19	−1.0 (2)	C36—C37—C38—C39	2.6 (2)
C17—C18—C19—C20	−0.3 (3)	C37—C38—C39—C40	−0.3 (3)
C18—C19—C20—C21	1.0 (3)	C38—C39—C40—C41	−1.8 (3)
C17—C16—C21—C20	−0.9 (2)	C39—C40—C41—C36	1.6 (3)
C9—C16—C21—C20	178.93 (15)	C37—C36—C41—C40	0.7 (2)
C19—C20—C21—C16	−0.4 (3)	C29—C36—C41—C40	−178.01 (14)

Fig. 1


C4
C3
C1
C22
C23
C24
C5
C2
C25
N28
C32
C31
C11
N8
C27
C26
C33
C12
C10
C6
C7
C30
C29
C9
C21
C36
C37
C34
C13
C15
C16
C20
C41
C35
C14
C17
C38
C40
C19
C39
C18