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4-*tert*-Butyl-2,6-bis(piperidinomethyl)phenol

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.057
wR factor = 0.176
Data-to-parameter ratio = 16.7

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

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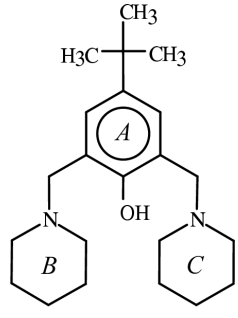
The title compound, $C_{22}H_{36}N_2O$, contains piperidine as a major constituent. The two piperidine rings attached to the phenyl ring are in chair conformations. The structure is stabilized by van der Waals forces as well as O—H···N intramolecular hydrogen bonds.

Comment

Piperidine derivatives are found to possess pharmacological activities and generally form an essential part of the molecular structure of important drugs. For example, the piperidine ring is a feature of antihistaminic agents, oral anesthetics (McElvain, 1927) and narcotic analgesics (Lu *et al.*, 1991). Clebopride, a 1,4-disubstituted piperidine, is used clinically to prevent post-operative vomiting, to speed up gastric emptying before anesthesia or to facilitate radiological evaluation and to correct a variety of disturbances of gastrointestinal function (Robinson, 1973). Several 2,6-disubstituted piperidines are found to be useful as tranquilizers (Bochringer & Soehne, 1961) and possess hypotensive activity. In addition, it has also a combination of stimulant and depressant effects on the central nervous system (Ganellin & Spickett, 1965), as well as bactericidal, fungicidal and herbicidal activities (Mobio *et al.*, 1990). Many piperidine derivatives also form the skeleton of several alkaloids (Hootele *et al.*, 1980). This compound also acts as a bridging molecule in polymetallic complexes. This property has application in the design of novel magnetic and electronic solid-state materials. Also, the complexes adopt the role of polymetallic sites in biological processes (Willet *et al.*, 1985; Marcus & Sutin, 1985). Synthetic binuclear ligands and their complexes can serve as suitable models for the natural binuclear metal centers when they mimic some physical and



(I)

chemical properties of the protein site and thereby provide an improved understanding of the biological analogue (Fenton *et al.*, 1982). In view of this, the title compound, (I) (Fig. 1), was synthesized and characterized by X-ray diffraction analysis.

The piperidine ring system offers a wide variety of conformational flexibility such as chair, boat and twist conformation (Hofer, 1976; Potapov, 1979), though the chair or slightly distorted chair conformations have been found to be the most favoured (Mulekar & Berlin, 1989). As in the related compound 2,6-bis(*N*-methylenepiperidino)-4-nitrophenol (Shanmuga Sundra Raj *et al.*, 1994), the piperidine rings *B* and *C* adopt the chair conformation [in the notation of Cremer & Pople (1975): Q_T = 0.574 (2) Å, q_2 = 0.014 (2), q_3 = −0.573 (2) and φ_2 = −128 (9)° for ring *B*; Q_T = 0.561 (2) Å, q_2 = 0.027 (2), q_3 = 0.560 (2) and φ_2 = 28 (5)° for ring *C*]. The mean bond length for the phenyl ring is 1.392 Å. In the *tert*-butyl system, C24 is in a (−)synclinal position with respect to the C4—C5 bond. There is a an O—H···N intramolecular hydrogen bond [H21···N15 = 1.76 (2) Å, O21···N15 = 2.649 (2) Å and angle at H21 = 156 (2)°].

Experimental

4-*tert*-Butylphenol (7.5 g, 0.05 mol) in ethanol (150 ml) and piperidine (8.7 g, 0.1 mol) were added to a 250 ml round-bottomed flask. The solution was stirred as formaldehyde (3.0 g, 0.1 mol) was added slowly. The solution was refluxed for 24 h, during which time 2.5 ml formaldehyde was added at time intervals of 8 h. The ethanol was evaporated under vacuum, the resulting oil washed with sodium carbonate solution, extracted with dimethyl ether and evaporated to yield a colourless solid (m.p. 393 K). Crystals were obtained by the slow evaporation of an acetone solution of the compound.

Crystal data

$C_{22}H_{36}N_2O$	Z = 2
M_r = 344.53	D_x = 1.070 Mg m^{-3}
Triclinic, $P\overline{1}$	Cu $K\alpha$ radiation
a = 10.540 (2) Å	Cell parameters from 25 reflections
b = 12.153 (3) Å	θ = 15–30°
c = 9.786 (2) Å	μ = 0.50 mm^{-1}
α = 106.20 (3)°	T = 293 (2) K
β = 116.89 (3)°	Needle, colourless
γ = 87.18 (4)°	0.40 × 0.20 × 0.20 mm
V = 1069.2 (4) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 69.9°
ω–2θ scans	h = −12 → 11
4237 measured reflections	k = −14 → 14
3904 independent reflections	l = 0 → 11
3680 reflections with $I > 2\sigma(I)$	3 standard reflections
R_{int} = 0.017	frequency: 120 min
	intensity decay: <1%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0939P)^2 + 0.2502P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.057	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.176	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
S = 1.09	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
3904 reflections	Extinction correction: *SHELXL*97
234 parameters	Extinction coefficient: 0.022 (4)
H atoms treated by a mixture of independent and constrained refinement	

The phenol H atom was located from a difference map and refined freely. H atoms bonded to C atoms were placed in calculated positions, refined using a riding model and given an isotropic displacement parameter equal to 1.2 times the equivalent isotropic displacement parameter of the CH and CH_2 groups, and 1.5 times the equivalent isotropic displacement parameter of the CH_3 groups.

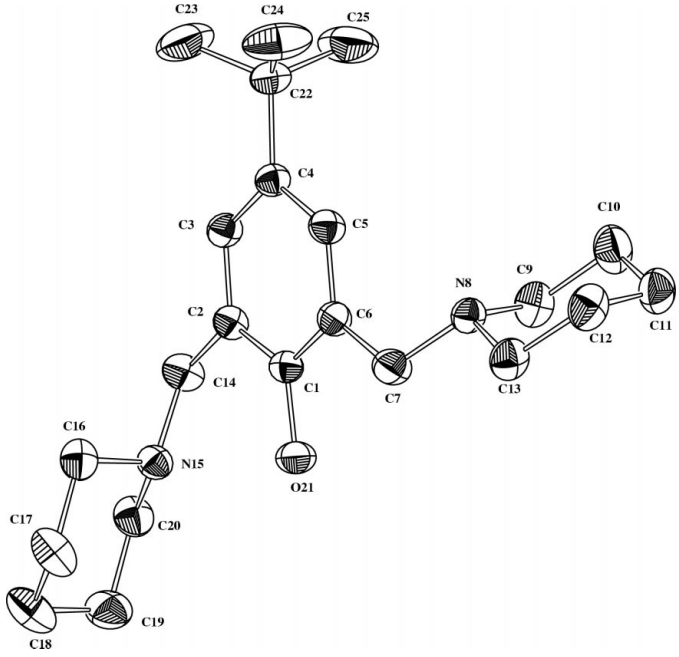


Figure 1
The molecular structure of (I) showing displacement ellipsoids at the 30% probability level. H atoms have been omitted for clarity.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *SDP* (Frenz, 1978); data reduction: *CAD*-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *PARST*97 (Nardelli, 1995).

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