Crystal structure of 2-[(3,4-dimethoxy)-phenethyl-3-(3,4-dimethoxy)-phenethylamino]-2,3-dihydro-isoindol-1-one, C₂₈H₃₂N₂O₅

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Abstract

C₂₈H₃₂N₂O₅, monoclinic, *P*12₁/*a*1 (No. 14), *a* = 9.5162(6) Å, *b* = 16.0013(9) Å, *c* = 17.8001(8) Å, β = 102.883(6)°, *V* = 2642.2 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.051, *wR*_{ref}(*F*²) = 0.171, *T* = 293 K.

Source of material

A solution of phthalaldehydic acid (1 g, 6.67 mmol) in 30 mL of toluene was treated with homoveratrylamine (2.25 mL, 13.3 mmol) and refluxed under a Dean-Stark trap with constant stirring for 2 hours. After concentrating to dryness, the title product was purified by crystallization in cyclohexane affording white needles (yield 95%).

Experimental details

The hydrogen atom H10B (linked to N10) was located on a difference electron-density map and refined. The other hydrogen atoms were included in idealised positions with isotropic displacement parameters.

Discussion

The isoindolone moiety is planar and lies parallel to the phenethylamine linked to N2, having a torsion angle C25–C24–C23–N2 of 175.4(2)°. All methoxyl group carbon atoms are coplanar with the respective aromatic rings. However, the phenethylamine linked to C3 is twisted with respect to the isoindolone moiety forming a dihedral angle C3–N10–C11–C12 of –161.5(2)°.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.04 × 0.16 × 0.4 mm
Wavelength:	Cu <i>K</i> _α radiation (1.5418 Å)
μ :	6.67 cm ^{–1}
Diffractometer, scan mode:	Enraf Nonius CAD4, $\omega/2\theta$
$2\theta_{\max}$:	145.88°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	5440, 5271
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 2589
<i>N</i> (<i>param</i>) _{refined} :	325
Programs:	SIR-97 [1], SHELXL-97 [2], ORTEP-3 [3], WinGX [4]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(3)	4e	1.0462	0.1461	0.0404	0.066
H(6)	4e	1.0267	0.0808	–0.1198	0.083
H(7)	4e	0.8939	–0.0237	–0.1913	0.097
H(8)	4e	0.6889	–0.0755	–0.1598	0.092
H(9)	4e	0.6085	–0.0211	–0.0557	0.076
H(11A)	4e	0.8009	0.3045	–0.0622	0.082
H(11B)	4e	0.8049	0.2290	–0.1180	0.082
H(12A)	4e	0.9877	0.3014	–0.1638	0.130
H(12B)	4e	0.9592	0.3787	–0.1152	0.130
H(14)	4e	0.7665	0.4736	–0.1718	0.115

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Table 2. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(15)	4e	0.5825	0.5106	−0.2733	0.130
H(18)	4e	0.7895	0.2520	−0.2699	0.101
H(20A)	4e	0.7484	0.1983	−0.3884	0.254
H(20B)	4e	0.6032	0.1662	−0.4404	0.254
H(20C)	4e	0.6263	0.1640	−0.3504	0.254
H(22A)	4e	0.3777	0.5114	−0.3503	0.403
H(22B)	4e	0.3100	0.4985	−0.4382	0.403
H(22C)	4e	0.4589	0.5439	−0.4121	0.403
H(23A)	4e	0.7583	0.2476	0.1194	0.071
H(23B)	4e	0.8947	0.2857	0.0966	0.071
H(24A)	4e	0.9026	0.1597	0.2090	0.075

Table 2. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(24B)	4e	1.0412	0.1906	0.1834	0.075
H(26)	4e	0.8335	0.2296	0.3138	0.074
H(29)	4e	1.1285	0.4528	0.2948	0.076
H(30)	4e	1.1003	0.3472	0.2051	0.072
H(32A)	4e	1.2072	0.5047	0.4181	0.165
H(32B)	4e	1.1168	0.5641	0.4591	0.165
H(32C)	4e	1.0849	0.5603	0.3689	0.165
H(34A)	4e	0.6850	0.2610	0.3950	0.197
H(34B)	4e	0.7282	0.2779	0.4840	0.197
H(34C)	4e	0.8251	0.2206	0.4450	0.197
H(10B)	4e	1.025(4)	0.282(2)	0.003(2)	0.13(1)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(1)	4e	0.7403(2)	0.1141(2)	0.0367(1)	0.050(1)	0.055(1)	0.048(1)	0.006(1)	0.010(1)	0.007(1)
N(2)	4e	0.8449(2)	0.1724(1)	0.0535(1)	0.054(1)	0.058(1)	0.046(1)	0.0003(9)	0.0113(8)	−0.0075(9)
O(1B)	4e	0.6370(2)	0.1085(1)	0.0679(1)	0.065(1)	0.077(1)	0.073(1)	0.0014(9)	0.0310(9)	0.0047(9)
C(3)	4e	0.9556(2)	0.1645(2)	0.0066(1)	0.047(1)	0.068(2)	0.049(1)	−0.000(1)	0.009(1)	−0.006(1)
C(4)	4e	0.8947(2)	0.0931(1)	−0.0456(1)	0.048(1)	0.057(1)	0.048(1)	0.004(1)	0.007(1)	−0.004(1)
C(5)	4e	0.7702(2)	0.0635(1)	−0.0271(1)	0.049(1)	0.046(1)	0.052(1)	0.004(1)	0.006(1)	−0.001(1)
C(6)	4e	0.9431(3)	0.0608(2)	−0.1072(2)	0.072(2)	0.075(2)	0.065(2)	0.001(1)	0.026(1)	−0.015(1)
C(7)	4e	0.8639(4)	−0.0016(2)	−0.1492(2)	0.103(2)	0.070(2)	0.073(2)	0.010(2)	0.028(2)	−0.021(2)
C(8)	4e	0.7399(3)	−0.0326(2)	−0.1305(2)	0.092(2)	0.053(2)	0.077(2)	0.001(2)	0.002(2)	−0.016(1)
C(9)	4e	0.6913(3)	−0.0005(2)	−0.0687(2)	0.064(2)	0.051(1)	0.073(2)	−0.001(1)	0.010(1)	0.001(1)
N(10)	4e	0.9833(2)	0.2375(2)	−0.0335(1)	0.056(1)	0.076(2)	0.062(1)	−0.018(1)	0.011(1)	−0.005(1)
C(11)	4e	0.8620(3)	0.2735(2)	−0.0892(2)	0.065(2)	0.073(2)	0.066(2)	−0.012(1)	0.016(1)	0.008(1)
C(12)	4e	0.9145(4)	0.3302(2)	−0.1435(2)	0.093(2)	0.145(3)	0.077(2)	−0.057(2)	−0.001(2)	0.034(2)
C(13)	4e	0.7959(3)	0.3592(2)	−0.2102(2)	0.086(2)	0.096(2)	0.069(2)	−0.030(2)	0.021(2)	0.019(2)
C(14)	4e	0.7346(4)	0.4361(2)	−0.2119(2)	0.121(3)	0.097(3)	0.079(2)	−0.030(2)	0.041(2)	0.009(2)
C(15)	4e	0.6250(5)	0.4583(3)	−0.2732(3)	0.123(3)	0.106(3)	0.111(3)	0.014(2)	0.061(3)	0.025(3)
C(16)	4e	0.5775(4)	0.4062(3)	−0.3333(2)	0.093(3)	0.143(4)	0.087(3)	0.019(3)	0.021(2)	0.033(3)
C(17)	4e	0.6387(4)	0.3269(3)	−0.3325(2)	0.094(2)	0.117(3)	0.067(2)	−0.011(2)	0.009(2)	0.012(2)
C(18)	4e	0.7477(3)	0.3046(2)	−0.2708(2)	0.085(2)	0.090(2)	0.076(2)	−0.006(2)	0.013(2)	0.017(2)
O(19)	4e	0.5872(3)	0.2763(2)	−0.3944(2)	0.136(2)	0.166(3)	0.083(2)	−0.013(2)	−0.007(2)	−0.008(2)
C(20)	4e	0.6460(7)	0.1946(3)	−0.3933(3)	0.252(7)	0.130(4)	0.114(4)	−0.050(4)	0.017(4)	−0.025(3)
O(21)	4e	0.4708(4)	0.4221(3)	−0.3975(2)	0.143(3)	0.236(4)	0.121(2)	0.061(3)	−0.001(2)	0.055(3)
C(22)	4e	0.3985(8)	0.5004(5)	−0.3997(4)	0.263(8)	0.305(9)	0.214(7)	0.178(7)	0.005(6)	0.077(6)
C(23)	4e	0.8546(3)	0.2349(2)	0.1130(1)	0.068(2)	0.059(1)	0.048(1)	0.008(1)	0.009(1)	−0.007(1)
C(24)	4e	0.9470(3)	0.2076(2)	0.1903(1)	0.073(2)	0.064(2)	0.048(1)	0.010(1)	0.008(1)	−0.004(1)
C(25)	4e	0.9651(3)	0.2768(2)	0.2497(1)	0.056(1)	0.060(2)	0.042(1)	0.008(1)	0.007(1)	−0.003(1)
C(26)	4e	0.8937(3)	0.2745(2)	0.3100(1)	0.062(2)	0.072(2)	0.051(1)	−0.009(1)	0.012(1)	−0.008(1)
C(27)	4e	0.9103(3)	0.3369(2)	0.3637(1)	0.060(1)	0.084(2)	0.047(1)	−0.007(1)	0.016(1)	−0.013(1)
C(28)	4e	1.0001(3)	0.4046(2)	0.3592(1)	0.060(1)	0.075(2)	0.048(1)	−0.005(1)	0.014(1)	−0.014(1)
C(29)	4e	1.0694(3)	0.4076(2)	0.2991(1)	0.066(2)	0.069(2)	0.057(1)	−0.006(1)	0.020(1)	−0.006(1)
C(30)	4e	1.0522(3)	0.3442(2)	0.2451(1)	0.063(2)	0.074(2)	0.046(1)	0.004(1)	0.019(1)	−0.006(1)
O(31)	4e	1.0122(2)	0.4631(1)	0.4158(1)	0.091(1)	0.094(2)	0.069(1)	−0.021(1)	0.031(1)	−0.036(1)
C(32)	4e	1.1135(4)	0.5283(2)	0.4155(2)	0.134(3)	0.100(3)	0.102(3)	−0.040(2)	0.039(2)	−0.046(2)
O(33)	4e	0.8447(2)	0.3396(1)	0.4252(1)	0.096(1)	0.121(2)	0.062(1)	−0.036(1)	0.041(1)	−0.029(1)
C(34)	4e	0.7643(5)	0.2691(3)	0.4383(2)	0.149(3)	0.175(4)	0.092(2)	−0.090(3)	0.071(2)	−0.043(3)

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References

- Altomare, A.; Burla, M. C.; Camalli, M.; Carrozzini, B.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Rizzi, R.: Sir97: A new tool for crystal structure determination and refinement. *J. Appl. Crystallogr.* **32** (1999) 115–119.
- Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
- Farrugia, L. J.: ORTEP-3 for Windows - a version of ORTEP-III with Graphical User Interface (GUI). *J. Appl. Crystallogr.* **30** (1997) 565.
- Farrugia, L. J.: WinGX - Version 1.63.02. An integrated System of Windows Programs for the Solution, Refinement and Analysis of Single Crystals X-Ray Diffraction Data. *J. Appl. Crystallogr.* **32** (1999) 837–838.