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Crystal structure of 3-(2-(4-chlorophenyl)-3-hydroxy-3,3-diphenylpropyl)-1,1-dimethylurea, $C_{24}H_{25}ClN_2O_2$

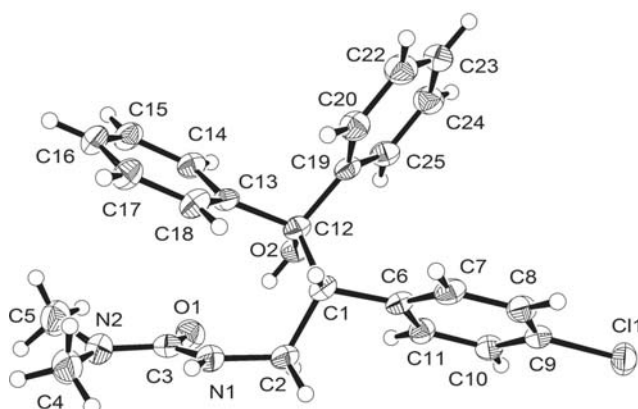


Table 1: Data collection and handling.

Crystal:	Colourless plate
	Size 0.21 × 0.10 × 0.03 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ :	18.4 cm ⁻¹
Diffractometer, scan mode:	SuperNova, ω -scans
$2\theta_{\max}$, completeness:	147.6°, >98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7255, 3968, 0.020
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3613
$N(\text{param})_{\text{refined}}$:	265
Programs:	CrysAlis ^{PRO} [13], SHELX [14], WinGX [15], CHEMDRAW [16]

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Abstract

$C_{24}H_{25}ClN_2O_2$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 5.9066(2)$ Å, $b = 15.7928(3)$ Å, $c = 21.7829(6)$ Å, $V = 2031.95(10)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0319$, $wR_{\text{ref}}(F^2) = 0.0838$, $T = 150(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.5633(4)	0.96117(14)	1.08808(11)	0.0266(5)
H1	0.7236	0.9809	1.0918	0.032*
C2	0.5183(5)	0.89733(14)	1.14050(11)	0.0329(5)
H2A	0.3610	0.8765	1.1363	0.039*
H2B	0.6200	0.8482	1.1345	0.039*
C3	0.3692(5)	0.95721(16)	1.23638(12)	0.0334(6)
C4	0.6414(6)	0.9787(2)	1.32070(13)	0.0448(7)
H4A	0.7101	0.9242	1.3101	0.067*
H4B	0.6320	0.9840	1.3655	0.067*
H4C	0.7345	1.0247	1.3042	0.067*
C5	0.2545(6)	1.0375(2)	1.32561(14)	0.0477(7)
H5A	0.3153	1.0951	1.3282	0.072*
H5B	0.2271	1.0157	1.3671	0.072*
H5C	0.1119	1.0382	1.3026	0.072*
C6	0.5425(4)	0.91177(14)	1.02844(11)	0.0262(5)
C7	0.7246(4)	0.90766(15)	0.98796(12)	0.0296(5)
H7	0.8580	0.9390	0.9968	0.035*
C8	0.7159(4)	0.85859(15)	0.93468(12)	0.0307(5)
H8	0.8417	0.8563	0.9076	0.037*
C9	0.5217(5)	0.81369(14)	0.92218(10)	0.0276(5)
C10	0.3370(4)	0.81533(15)	0.96129(11)	0.0274(5)
H10	0.2049	0.7833	0.9523	0.033*
C11	0.3487(4)	0.86494(15)	1.01423(11)	0.0275(5)
H11	0.2223	0.8669	1.0412	0.033*
C12	0.4074(4)	1.04095(14)	1.08983(11)	0.0259(5)
C13	0.4692(4)	1.10383(13)	1.14145(11)	0.0261(5)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C14	0.3042(4)	1.16086(15)	1.16002(12)	0.0302(5)
H14	0.1584	1.1586	1.1417	0.036*
C15	0.3481(5)	1.22138(16)	1.20493(12)	0.0350(6)
H15	0.2332	1.2601	1.2169	0.042*
C16	0.5594(5)	1.22496(16)	1.23214(12)	0.0352(6)
H16	0.5894	1.2654	1.2634	0.042*
C17	0.7270(5)	1.16927(16)	1.21362(12)	0.0343(6)
H17	0.8726	1.1717	1.2320	0.041*
C18	0.6824(4)	1.10974(15)	1.16810(12)	0.0314(5)
H18	0.7995	1.0726	1.1550	0.038*
C19	0.4242(4)	1.08905(14)	1.02868(11)	0.0262(5)
C20	0.6204(5)	1.13290(17)	1.01401(12)	0.0337(5)
H20	0.7448	1.1322	1.0417	0.040*
C22	0.6375(5)	1.17801(18)	0.95920(14)	0.0385(6)
H22	0.7729	1.2077	0.9497	0.046*
C23	0.4576(5)	1.17962(16)	0.91871(12)	0.0373(6)
H23	0.4696	1.2097	0.8811	0.045*
C24	0.2603(5)	1.13720(17)	0.93318(13)	0.0370(6)
H24	0.1353	1.1389	0.9057	0.044*
C25	0.2438(4)	1.09192(16)	0.98791(12)	0.0324(5)
H25	0.1077	1.0627	0.9974	0.039*
N1	0.5478(4)	0.92760(13)	1.20296(10)	0.0337(5)
H1A	0.6836	0.9268	1.2196	0.040*
N2	0.4156(4)	0.98348(15)	1.29458(11)	0.0380(5)
O1	0.1736(3)	0.95873(12)	1.21521(8)	0.0381(4)
O2	0.1771(3)	1.01443(10)	1.09648(8)	0.0274(4)
H2	0.1622	0.9878	1.1296	0.041*
Cl1	0.51034(11)	0.75092(4)	0.85623(2)	0.03654(15)

Source of material

3-(2-(4-Chlorophenyl)-3-hydroxy-3,3-diphenylpropyl)-1,1-dimethylurea was synthesized *via* double lithiation of 3-(2-(4-chlorophenyl)ethyl)-1,1-dimethylurea with excess *tert*-butyllithium (3.3 mole equivalents) at -60 °C in anhydrous tetrahydrofuran (THF) under an inert atmosphere. The dilithium reagent produced *in-situ* was allowed to react with a solution of benzophenone (2.2 mole equivalents) in THF, added *via* a syringe. The reaction mixture was stirred and allowed to warm up to room temperature over 2 h. Following work-up, the crude product was purified by column chromatography (silica gel) using a mixture of diethyl ether and hexane (1:3 by volume) to give the title compound (87%). Crystallization from a mixture of diethyl ether and ethyl acetate (4:1 by volume) gave colorless crystals, Mp 164–165 °C.

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model. Methyl C–H bonds were fixed at 0.98 Å and the groups were allowed to spin about the C–C bond with displacement parameters 1.5 times $U_{eq}(C)$. Aromatic C–H distances were set to 0.95 Å and N–H set to

0.88 Å with U_{iso} set to 1.2 times the U_{eq} for the atoms to which they are bonded.

Discussion

Urea derivatives show various biological activities [1–4]. They can be synthesized efficiently in high yields using simple procedures [4–7]. Aromatic substituted ureas can be further easily substituted *via* lithiation followed by reactions with electrophiles [8–12].

In the crystal structure, the asymmetric unit comprises one molecule of C₂₄H₂₅ClN₂O₂. An intramolecular O–H···O hydrogen bond is observed in the molecule with an O2···O1 distance of 2.732(3) Å and O2–H2···O1 angle of 161.7°. Two weak intermolecular C–H···Cl interactions occur in the crystal structure (with geometry: C2···Cl1 = 3.806(3) Å, C2–H2a···Cl1 = 153.9° and C2···Cl1 = 3.733(3) Å, C2–H2b···Cl1 = 158.4°) leading to the formation of molecular chains along [100]. The structure is racemically twinned.

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