Gamal A. El-Hiti*, Keith Smith, Mohammed B. Alshammari, Mohammad Hayal Alotaibi and Benson M. Kariuki

Crystal structure of 1,1-dimethyl-3-(2-phenylethyl)urea, $C_{11}H_{16}N_2O$

DOI 10.1515/ncrs-2016-0052

Received March 14, 2016; accepted May 8, 2016; available online May 21, 2016

Abstract

$C_{11}H_{16}N_2O$, orthorhombic, *Pbca* (no. 61), $a = 10.7388(6)$ Å, $b = 9.8449(5)$ Å, $c = 21.1259(14)$ Å, $V = 2233.5(2)$ Å³, $Z = 8$, $R_{gt}(F) = 0.0582$, $wR_{ref}(F^2) = 0.1795$, $T = 293$ K.

CCDC no.: 1478571

Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

1,1-Dimethyl-3-(2-phenylethyl)urea was synthesized from reaction of (2-phenylethyl)amine with dimethylcarbamoyl chloride (1.1 mole equivalents) in the presence of triethylamine (1.4 mole equivalents) in dichloromethane under reflux for 1 h. Recrystallization using a mixture of ethyl acetate

Table 1: Data collection and handling.

Crystal:	Colourless, block
Wavelength:	Size $0.37 \times 0.17 \times 0.10$ mm
μ :	Mo K_{α} radiation (0.71073 Å)
Diffractometer, scan mode:	0.8 cm^{-1}
$2\theta_{\max}$, completeness:	SuperNova, ω
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	59.4° , $>99\%$
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	7627, 2680, 0.025
$N(\text{param})_{\text{refined}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1669
Programs:	129
	SHELX [13], CrysAlis ^{PRO} [14],
	WinGX [15]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.48541(18)	0.06211(19)	0.35228(8)	0.0580(5)
C2	0.5322(2)	0.1652(2)	0.31509(11)	0.0743(6)
H2	0.6015	0.2135	0.3288	0.089*
C3	0.4777(3)	0.1979(3)	0.25766(12)	0.0921(8)
H3	0.5107	0.2678	0.2333	0.111*
C4	0.3765(3)	0.1288(3)	0.23680(13)	0.0993(8)
H4	0.3409	0.1505	0.1980	0.119*
C5	0.3273(2)	0.0284(3)	0.27259(13)	0.0975(8)
H5	0.2573	−0.0183	0.2586	0.117*
C6	0.3810(2)	−0.0053(2)	0.33001(11)	0.0784(6)
H6	0.3463	−0.0746	0.3542	0.094*
C7	0.54501(19)	0.0256(2)	0.41422(9)	0.0687(6)
H7A	0.4881	−0.0306	0.4384	0.082*
H7B	0.5599	0.1080	0.4382	0.082*
C8	0.66752(18)	−0.04998(19)	0.40590(9)	0.0618(5)
H8A	0.6514	−0.1347	0.3839	0.074*
H8B	0.7222	0.0041	0.3794	0.074*
C9	0.79739(16)	0.01667(16)	0.49494(10)	0.0565(5)
C10	0.8638(2)	−0.1564(2)	0.57266(11)	0.0833(7)
H10A	0.7839	−0.1822	0.5891	0.125*
H10B	0.9248	−0.1612	0.6058	0.125*
H10C	0.8868	−0.2170	0.5390	0.125*
C11	0.9276(2)	0.0821(2)	0.58375(12)	0.0848(7)
H11A	0.9358	0.1629	0.5587	0.127*
H11B	1.0088	0.0470	0.5934	0.127*
H11C	0.8846	0.1031	0.6224	0.127*
N1	0.73091(15)	−0.07966(14)	0.46471(7)	0.0604(4)
H1	0.7261	−0.1599	0.4806	0.072*
N2	0.85761(15)	−0.01881(16)	0.54863(8)	0.0675(5)
O1	0.80282(14)	0.13384(12)	0.47359(8)	0.0751(5)

*Corresponding author: Gamal A. El-Hiti, Cornea Research Chair, Department of Optometry, College of Applied Medical Sciences, King Saud University, P.O. Box 10219, Riyadh 11433, Saudi Arabia, e-mail: gelhiti@ksu.edu.sa

Keith Smith and Benson M. Kariuki: School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, UK

Mohammed B. Alshammari: Chemistry Department, College of Sciences and Humanities, Prince Sattam bin Abdulaziz University, P.O. Box 83, Al-Kharj 11942, Saudi Arabia

Mohammad Hayal Alotaibi: National Center for Petrochemicals Technology, King Abdulaziz City for Science and Technology, P.O. Box 6086, Riyadh 11442, Saudi Arabia

and diethyl ether (1:3 by volume) gave the title compound (~99%) as colourless crystals, mp 89–90 °C (lit. 88–90 °C [1]; 81–82 °C [2]).

Experimental details

All H atoms were placed in calculated positions and refined using a riding model. For the methyl groups, C–H bonds were fixed at 0.96 Å and $U_{\text{iso}}(\text{H})$ set to 1.5 $U_{\text{eq}}(\text{C})$ with free rotation around the C–C bond (HFIX 137 in SHELX [13]). For the rest of the hydrogens, $U_{\text{iso}}(\text{H})$ was set to 1.2 $U_{\text{eq}}(\text{C})$ with aromatic C–H and N–H distances of 0.93 and 0.86 Å, respectively.

Discussion

The synthesis of substituted ureas is of great interest for both academia and industry since ureas represent various biologically active compounds [2–4]. Urea derivatives can be synthesized using various efficient procedures [5–7]. Regioselective lithiation of aromatic ureas with lithium reagents followed by reaction with electrophiles at low temperatures is considered to be one of the most clean, efficient and convenient processes for the production of a large number of substituted derivatives in high yields [8–11].

The asymmetric unit (Figure) of the crystal structure consists of a molecule of C₁₁H₁₆N₂O. In the crystal, the amide group is involved in N–H···O hydrogen bonding (N···O distance = 2.850(2) Å, N–H···O angle = 152.4°) leading to the formation of C(4) chains [12] along the [010] direction.

Acknowledgements: The authors extend their appreciation to the College of Applied Medical Sciences Research Centre and the Deanship of Scientific Research at King Saud University for their funding of this research.

References

- Smith, K.; El-Hiti, G. A.; Alshammari, M. B.: Lithiation and substitution of *N'*-(ω -phenylalkyl)-*N,N*-dimethylureas. *Synthesis* **44** (2012) 20139–2022.
- Wilson, A. A.; Garcia, A.; Houle, S.; Sadovski, O.; Vasdev, N.: Synthesis and application of isocyanates radiolabeled with carbon-11. *Chem. Eur. J.* **17** (2011) 259–264.
- Gennäs, G. B.; Mologni, L.; Ahmed, S.; Rajaratnam, M.; Marin, O.; Lindholm, N.; Viltadi, M.; Gambacorti-Passerini, C.; Scapozza, L.; Yli-Kauhaluoma, J.: Design, synthesis, and biological activity of urea derivatives as anaplastic lymphoma kinase inhibitors. *Chem. Med. Chem.* **6** (2011) 1680–1692.
- Kocyigit-Kaymakcioglu, B.; Celen, A. O.; Tabanca, N.; Ali, A.; Khan, S. I.; Khan, I. A.; Wedge, D. E.: Synthesis and biological activity of substituted urea and thiourea derivatives containing 1,2,4-triazole moieties. *Molecules* **18** (2013) 3562–3576.
- Artuso, E.; Degani, I.; Fochi, R.; Magistris, C.: Preparation of mono-, di-, and trisubstituted ureas by carbonylation of aliphatic amines with *S,S*-dimethyl dithiocarbonate. *Synthesis* **22** (2007) 3497–3506.
- Mizuno, T.; Nakai, T.; Mihara, M.: Synthesis of unsymmetrical ureas by sulfur-assisted carbonylation with carbon monoxide and oxidation with molecular oxygen under mild conditions. *Synthesis* **15** (2009) 2492–2496.
- Thalluri, K.; Manne, S. R.; Dev, D.; Mandal, B.: Ethyl 2-cyano-2-(4-nitrophenylsulfonyloxymino)acetate-mediated Lossen rearrangement: single-pot racemization-free synthesis of hydroxamic acids and ureas from carboxylic acids. *J. Org. Chem.* **79** (2014) 3765–3775.
- Smith, K.; El-Hiti, G. A.; Alshammari, M. B.: Directed lithiation of *N'*-(2-(4-methoxyphenyl)ethyl)-*N,N*-dimethylurea and *tert*-butyl 2-(4-methoxyphenyl)ethylcarbamate. *Synthesis* **46** (2014) 394–402.
- Smith, K.; El-Hiti, G. A.; Alshammari, M. B.: Control of site of lithiation of 3-(aminomethyl)pyridine derivatives. *Synthesis* **45** (2013) 3426–3434.
- Smith, K.; El-Hiti, G. A.; Alshammari, M. B.: Variation in the site of lithiation of 2-(2-methylphenyl)ethanamine derivatives. *J. Org. Chem.* **77** (2012) 11210–11215.
- Smith, K.; El-Hiti, G. A.; Hegazy, A. S.: Lateral lithiation of *N'*-(2-methylbenzyl)-*N,N*-dimethylurea and *N*-(2-methylbenzyl)pivalamide: synthesis of tetrahydroisoquinolines. *Synthesis* **8** (2010) 1371–1380.
- Grell, J.; Bernstein, J.; Tinhofer, G.: Investigation of hydrogen bond patterns: a review of mathematical tools for the graph set approach. *Crystallogr. Rev.* **8** (2002) 1–56.
- Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112–122.
- Agilent. CrysAlis^{PRO}. Agilent Technologies, Yarnton, England, 2014.
- Farrugia, L. J.: WinGX and ORTEP for Windows: an update. *J. Appl. Crystallogr.* **45** (2012) 849–854.