IUCrData

ISSN 2414-3146

1,1-Dimethyl-3-[4-(trifluoromethyl)phenyl]urea

Gamal A. El-Hiti,[a]* Keith Smith,[b] Amany S. Hegazy,[b] Mohammed B. Alshammari[c] and Benson M. Kariuki[b]‡

[a]Cornea Research Chair, Department of Optometry, College of Applied Medical Sciences, King Saud University, PO Box 10219, Riyadh 11433, Saudi Arabia, [b]School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, Wales, and [c]Chemistry Department, College of Sciences and Humanities, Prince Sattam bin Abdulaziz University, PO Box 83, Al-Kharij 11942, Saudi Arabia. *Correspondence e-mail: gelhiti@ksu.edu.sa

Received 5 January 2018
Accepted 7 January 2018

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

‡ Additional corresponding author, e-mail: kariukib@cardiff.ac.uk.

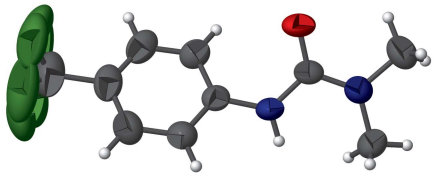
Keywords: crystal structure; disorder; hydrogen bond; urea.

CCDC reference: 1815173

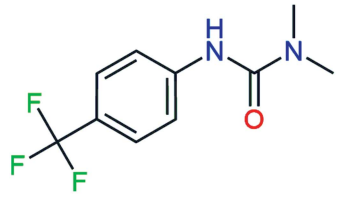
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{10}H_{11}F_3N_2O$, the dihedral angle between the dimethylurea and phenyl group planes is 37.49 (7)°. In the crystal, molecules are linked by N—H···O hydrogen bonds, generating chains propagating in the [010] direction. The trifluoromethyl group is disordered over two orientations in a 0.577 (12):0.423 (12) ratio.

3D view



Chemical scheme



Structure description

Various synthetic methods are known for the production of ureas (*e.g.*: Artuso *et al.*, 2007; Carnaroglio *et al.*, 2013). As part of our studies in this area, we now describe the synthesis and structure of the title compound (Fig. 1).

The angle between the planes through the non-hydrogen atoms of the dimethylurea and phenyl groups is 37.49 (7)°. In the crystal (Fig. 2), the molecules are linked by N—H···O hydrogen bonds (Table 1) to generate *C*(4) amide chains propagating in the [010] direction with adjacent molecules in the chain related by *b*-glide symmetry.

Synthesis and crystallization

4-Trifluoromethylaniline (10 mmol) and dimethylcarbamoyl chloride (11 mmol) in anhydrous dichloromethane containing triethylamine (15 mmol) were heated under reflux for 1 h. The mixture was allowed to cool down and poured into water. The layers were separated and the organic layer was dried (anhydrous magnesium sulfate) and evaporated under reduced pressure. The solid obtained was recrystallized from ethyl acetate solution to give colourless blocks of (I), m.p. 195–196°C (lit. 193–194°C; Hutchby, 2013).

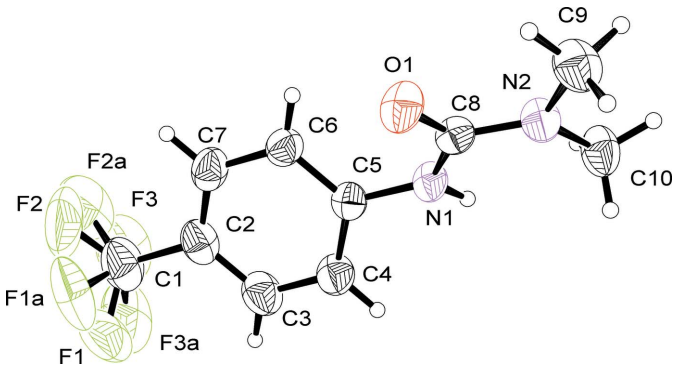


Figure 1
The molecular structure of the title compound, showing 50% displacement ellipsoids.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The trifluoromethyl group is disordered over two orientations in a 0.577 (12):0.423 (12) ratio.

Funding information

The project was supported by King Saud University, Deanship of Scientific Research, Research Chairs and Cardiff University.

References

Agilent (2014). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.

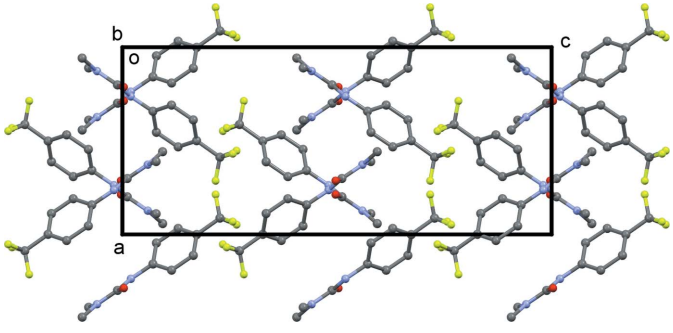


Figure 2
A view of the crystal packing down [100]. H atoms have been omitted for clarity.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.08	2.8939 (13)	157

Symmetry code: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{11}F_3N_2O$
M_r	232.21
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	293
a, b, c (Å)	9.8152 (3), 10.0783 (2), 22.5120 (7)
V (Å^3)	2226.90 (11)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.10
Crystal size (mm)	0.30 × 0.23 × 0.10
Data collection	
Diffractometer	Agilent SuperNova, Dual, Cu at zero, Atlas
Absorption correction	Gaussian (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.940, 0.974
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7168, 2217, 1708
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.623
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.139, 1.06
No. of reflections	2217
No. of parameters	176
No. of restraints	60
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.17

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS2013* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015>ORTEP-3 for Windows and WinGX (Farrugia, 2012) and CHEMDRAW Ultra (Cambridge Soft, 2001).

Artuso, E., Degani, I., Fochi, R. & Magistris, C. (2007). *Synthesis*, pp. 3497–3506.
Cambridge Soft (2001). *CHEMDRAW Ultra*, Cambridge Soft Corporation, Cambridge, Massachusetts, USA.
Carnaroglio, D., Martina, K., Palmisano, G., Penoni, A., Domini, C. & Cravotto, G. (2013). *Beilstein J. Org. Chem.* **9**, 2378–2386.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Hutchby, M. (2013). *Novel Synthetic Chemistry of Ureas and Amides.* Springer Thesis. Berlin Heidelberg: Springer-Verlag.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst*. C**71**, 3–8.

full crystallographic data

IUCrData (2018). **3**, x180040 [https://doi.org/10.1107/S2414314618000408]

1,1-Dimethyl-3-[4-(trifluoromethyl)phenyl]urea

Gamal A. El-Hiti, Keith Smith, Amany S. Hegazy, Mohammed B. Alshammari and Benson M. Kariuki

1,1-Dimethyl-3-[4-(trifluoromethyl)phenyl]urea

Crystal data

$C_{10}H_{11}F_3N_2O$
M_r = 232.21
Orthorhombic, *Pbca*
a = 9.8152 (3) Å
b = 10.0783 (2) Å
c = 22.5120 (7) Å
V = 2226.90 (11) Å^3
Z = 8
F(000) = 960
D_x = 1.385 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 2946 reflections
θ = 3.9–73.8°
μ = 1.10 mm^{-1}
T = 293 K
Block, colourless
0.30 × 0.23 × 0.10 mm

Data collection

Agilent SuperNova, Dual, Cu at zero, Atlas diffractometer
ω scans
Absorption correction: gaussian (CrysAlis PRO; Agilent, 2014)
T_{min} = 0.940, T_{max} = 0.974
7168 measured reflections
2217 independent reflections
1708 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 74.0°, θ_{min} = 3.9°
h = −11→7
k = −12→10
l = −28→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.139
S = 1.06
2217 reflections
176 parameters
60 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0878P)^2 + 0.0492P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: SHELXL2013 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0102 (9)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All hydrogen atoms were placed in calculated positions and refined using a riding model. Methyl C—H bonds were fixed at 0.96 Å, with displacement parameters 1.5 times U_{eq}(C), and were allowed to spin about the C—N bond. The N—H bond was fixed at 0.86 Å and aromatic C—H distances were set to 0.93 Å and their U(iso) set to 1.2 times the U_{eq} for the atoms to which they are bonded.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.5872 (3)	0.0387 (3)	0.72719 (10)	0.1049 (8)	
C2	0.49935 (19)	0.02575 (19)	0.67302 (8)	0.0730 (5)	
C3	0.5321 (2)	−0.06604 (19)	0.62979 (9)	0.0800 (5)	
H3	0.6070	−0.1214	0.6349	0.096*	
C4	0.45445 (17)	−0.07598 (15)	0.57921 (8)	0.0678 (4)	
H4	0.4774	−0.1378	0.5502	0.081*	
C5	0.34195 (14)	0.00549 (12)	0.57108 (6)	0.0517 (3)	
C6	0.30912 (16)	0.09806 (14)	0.61453 (7)	0.0613 (4)	
H6	0.2341	0.1533	0.6096	0.074*	
C7	0.38819 (18)	0.10778 (17)	0.66514 (7)	0.0707 (5)	
H7	0.3664	0.1701	0.6941	0.085*	
C8	0.20108 (14)	0.08594 (11)	0.48853 (6)	0.0517 (3)	
C9	0.0580 (2)	0.15002 (18)	0.40623 (10)	0.0831 (5)	
H9A	0.0521	0.2304	0.4290	0.125*	
H9B	−0.0320	0.1199	0.3964	0.125*	
H9C	0.1082	0.1665	0.3704	0.125*	
C10	0.1105 (2)	−0.08604 (16)	0.42132 (9)	0.0778 (5)	
H10A	0.1922	−0.1151	0.4019	0.117*	
H10B	0.0354	−0.0912	0.3941	0.117*	
H10C	0.0928	−0.1418	0.4550	0.117*	
N1	0.26294 (13)	−0.01353 (10)	0.51968 (5)	0.0546 (3)	
H1	0.2529	−0.0934	0.5070	0.065*	
N2	0.12717 (14)	0.04922 (11)	0.44086 (6)	0.0622 (4)	
O1	0.21290 (14)	0.20262 (9)	0.50340 (5)	0.0728 (4)	
F1	0.7132 (6)	0.024 (2)	0.7171 (3)	0.169 (5)	0.423 (12)
F2	0.5686 (15)	0.1449 (10)	0.7571 (5)	0.170 (5)	0.423 (12)
F3	0.5525 (14)	−0.0492 (11)	0.7660 (4)	0.164 (4)	0.423 (12)
F1A	0.6752 (9)	0.1385 (8)	0.7205 (3)	0.148 (3)	0.577 (12)
F3A	0.6621 (13)	−0.0643 (6)	0.7389 (4)	0.179 (4)	0.577 (12)
F2A	0.5211 (7)	0.0711 (18)	0.7739 (2)	0.206 (6)	0.577 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.108 (2)	0.137 (2)	0.0695 (13)	−0.0207 (16)	−0.0197 (12)	0.0095 (13)
C2	0.0724 (11)	0.0874 (10)	0.0592 (9)	−0.0186 (8)	−0.0063 (7)	0.0109 (7)
C3	0.0773 (11)	0.0784 (10)	0.0841 (12)	0.0059 (8)	−0.0200 (9)	0.0042 (8)
C4	0.0750 (10)	0.0551 (7)	0.0734 (10)	0.0061 (6)	−0.0107 (8)	−0.0048 (6)
C5	0.0568 (8)	0.0428 (5)	0.0554 (7)	−0.0081 (5)	0.0007 (6)	0.0026 (5)
C6	0.0601 (9)	0.0642 (8)	0.0597 (8)	−0.0048 (6)	0.0068 (6)	−0.0068 (6)

C7	0.0772 (11)	0.0825 (10)	0.0523 (8)	−0.0172 (8)	0.0104 (7)	−0.0088 (7)
C8	0.0574 (8)	0.0389 (5)	0.0587 (7)	−0.0017 (5)	0.0043 (6)	0.0007 (5)
C9	0.0924 (13)	0.0718 (10)	0.0850 (12)	0.0069 (8)	−0.0206 (10)	0.0156 (8)
C10	0.0931 (13)	0.0589 (8)	0.0814 (11)	−0.0074 (7)	−0.0245 (9)	−0.0080 (7)
N1	0.0669 (7)	0.0348 (5)	0.0620 (7)	−0.0007 (4)	−0.0077 (5)	−0.0041 (4)
N2	0.0704 (8)	0.0499 (6)	0.0664 (7)	0.0012 (5)	−0.0119 (6)	0.0021 (5)
O1	0.1053 (10)	0.0351 (5)	0.0781 (7)	0.0012 (4)	−0.0095 (6)	−0.0024 (4)
F1	0.086 (3)	0.322 (15)	0.099 (4)	−0.005 (5)	−0.029 (2)	−0.024 (6)
F2	0.213 (12)	0.172 (6)	0.124 (7)	0.007 (5)	−0.083 (8)	−0.061 (5)
F3	0.203 (9)	0.210 (7)	0.078 (4)	−0.017 (6)	−0.043 (4)	0.056 (4)
F1A	0.148 (5)	0.178 (5)	0.116 (4)	−0.073 (4)	−0.067 (3)	0.023 (3)
F3A	0.217 (8)	0.168 (4)	0.152 (6)	0.037 (5)	−0.117 (6)	0.009 (3)
F2A	0.140 (4)	0.421 (18)	0.0560 (18)	−0.029 (8)	−0.0078 (18)	−0.026 (5)

Geometric parameters (Å, º)

C1—F1	1.267 (6)	C6—C7	1.382 (2)
C1—F2	1.277 (7)	C6—H6	0.9300
C1—F2A	1.277 (7)	C7—H7	0.9300
C1—F3	1.290 (7)	C8—O1	1.2281 (16)
C1—F3A	1.300 (6)	C8—N2	1.3471 (19)
C1—F1A	1.334 (5)	C8—N1	1.3658 (17)
C1—C2	1.499 (3)	C9—N2	1.449 (2)
C2—C7	1.380 (3)	C9—H9A	0.9600
C2—C3	1.381 (3)	C9—H9B	0.9600
C3—C4	1.374 (3)	C9—H9C	0.9600
C3—H3	0.9300	C10—N2	1.4417 (19)
C4—C5	1.388 (2)	C10—H10A	0.9600
C4—H4	0.9300	C10—H10B	0.9600
C5—C6	1.390 (2)	C10—H10C	0.9600
C5—N1	1.4060 (18)	N1—H1	0.8600
F1—C1—F2	109.5 (6)	C5—C6—H6	120.1
F1—C1—F3	107.3 (7)	C2—C7—C6	120.49 (16)
F2—C1—F3	100.4 (6)	C2—C7—H7	119.8
F2A—C1—F3A	108.9 (6)	C6—C7—H7	119.8
F2A—C1—F1A	103.3 (6)	O1—C8—N2	122.07 (12)
F3A—C1—F1A	105.0 (5)	O1—C8—N1	121.39 (13)
F1—C1—C2	113.9 (3)	N2—C8—N1	116.54 (11)
F2—C1—C2	114.9 (4)	N2—C9—H9A	109.5
F2A—C1—C2	113.6 (4)	N2—C9—H9B	109.5
F3—C1—C2	109.8 (4)	H9A—C9—H9B	109.5
F3A—C1—C2	114.9 (3)	N2—C9—H9C	109.5
F1A—C1—C2	110.3 (2)	H9A—C9—H9C	109.5
C7—C2—C3	119.65 (16)	H9B—C9—H9C	109.5
C7—C2—C1	120.4 (2)	N2—C10—H10A	109.5
C3—C2—C1	119.9 (2)	N2—C10—H10B	109.5
C4—C3—C2	120.26 (17)	H10A—C10—H10B	109.5

C4—C3—H3	119.9	N2—C10—H10C	109.5
C2—C3—H3	119.9	H10A—C10—H10C	109.5
C3—C4—C5	120.50 (16)	H10B—C10—H10C	109.5
C3—C4—H4	119.8	C8—N1—C5	124.59 (10)
C5—C4—H4	119.8	C8—N1—H1	117.7
C4—C5—C6	119.25 (14)	C5—N1—H1	117.7
C4—C5—N1	117.81 (13)	C8—N2—C10	124.32 (12)
C6—C5—N1	122.89 (13)	C8—N2—C9	119.22 (13)
C7—C6—C5	119.85 (15)	C10—N2—C9	116.46 (14)
C7—C6—H6	120.1		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.08	2.8939 (13)	157

Symmetry code: (i) −*x*+1/2, *y*−1/2, *z*.