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Crystal structure of *N*-(2-(trifluoromethyl)phenyl)hexanamide, $C_{13}H_{16}F_3NO$

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Abstract

$C_{13}H_{16}F_3NO$, triclinic, $P\bar{1}$ (no. 2), $a = 4.7889(4)$ Å, $b = 11.7867(10)$ Å, $c = 12.5323(11)$ Å, $\alpha = 100.762(5)^\circ$, $\beta = 100.232(4)^\circ$, $\gamma = 100.683(4)^\circ$, $V = 666.19(10)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0531$, $wR_{ref}(F^2) = 0.1760$, $T = 200$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The compound was synthesized upon reacting *ortho*-trifluoromethylaniline with an equimolar amount of hexanoylchloride in toluene. Crystals were obtained upon free evaporation of the solvent at room temperature.

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Table 1: Data collection and handling.

Crystal:	Yellow rods
Size:	$0.52 \times 0.17 \times 0.15$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.11 mm^{-1}
Diffraction, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	28.4° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11441, 3290, 0.026
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2248
$N(\text{param})_{\text{refined}}$:	168
Programs:	Bruker [1, 2], SHELX [3], WinGX/ORTEP [4], Mercury [5], PLATON [6]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	0.5693 (4)	0.99393 (11)	0.67521 (13)	0.0829 (5)
F2	0.3480 (3)	0.83849 (11)	0.71363 (11)	0.0579 (4)
F3	0.7950 (3)	0.90673 (13)	0.78576 (10)	0.0677 (4)
O1	1.0355 (3)	0.57740 (13)	0.71629 (12)	0.0468 (4)
N1	0.6219 (3)	0.64221 (13)	0.67946 (12)	0.0313 (3)
C1	0.5987 (4)	0.88805 (16)	0.69172 (16)	0.0413 (4)
C2	0.7965 (3)	0.58452 (15)	0.73483 (14)	0.0318 (4)
C3	0.6799 (4)	0.52981 (16)	0.82187 (15)	0.0349 (4)
H3A	0.465268	0.518276	0.805753	0.042*
H3B	0.758916	0.584990	0.895602	0.042*
C4	0.7583 (4)	0.41162 (15)	0.82640 (14)	0.0344 (4)
H4A	0.973060	0.423667	0.843630	0.041*
H4B	0.682652	0.357210	0.752120	0.041*
C5	0.6383 (4)	0.35383 (17)	0.91227 (16)	0.0391 (4)
H5A	0.715535	0.407846	0.986675	0.047*
H5B	0.423650	0.342488	0.895502	0.047*
C6	0.7143 (5)	0.23541 (19)	0.91577 (19)	0.0552 (5)
H6A	0.928651	0.246373	0.929609	0.066*
H6B	0.630783	0.180630	0.842075	0.066*
C7	0.6048 (7)	0.1789 (3)	1.0041 (3)	0.0845 (9)
H7A	0.391362	0.159790	0.986658	0.127*
H7B	0.677578	0.234194	1.076956	0.127*
H7C	0.674783	0.106046	1.005868	0.127*
C11	0.6950 (3)	0.69789 (14)	0.59371 (13)	0.0291 (4)
C12	0.6767 (3)	0.81467 (15)	0.59592 (14)	0.0325 (4)
C13	0.7455 (4)	0.86563 (17)	0.50970 (16)	0.0429 (4)
H13	0.729028	0.944776	0.510025	0.051*
C14	0.8370 (5)	0.80259 (19)	0.42404 (17)	0.0503 (5)
H14	0.885008	0.838344	0.365764	0.060*
C15	0.8591 (4)	0.68748 (18)	0.42268 (16)	0.0456 (5)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H15	0.925129	0.644236	0.364020	0.055 [*]
C16	0.7855 (4)	0.63497 (16)	0.50648 (15)	0.0364 (4)
H16	0.797021	0.554994	0.504312	0.044 [*]
H1	0.463 (4)	0.6438 (17)	0.6963 (16)	0.035 (5) [*]

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å for aromatic carbon atoms, C–H 0.99 Å for methylene groups) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2*U*_{eq}(C). The H atoms of the methyl group were allowed to rotate with a fixed angle around the C–C bond to best fit the experimental electron density (HFIX 137 in the *SHELX* program [3]), with *U*(H) set to 1.5*U*_{eq}(C). The nitrogen-bound H atom was refined freely.

Comment

The importance of amide-type bonding between amino and carboxyl groups for the structural appearance and its subsequent influence on the biological functionality of peptides in living organisms is well-established and has long entered fundamental textbooks of biochemistry. In continuation of our interest in the structures of carboxylic acids and their derivatives the title compound was synthesized and its structure determined. The structures of other amides determined by us are apparent in the literature [7–11].

The title structure shows the expected bonding pattern of an *ortho*-trifluoromethylanilide of hexanoic acid. The C=O and C–N bond lengths of 1.2222(19) Å and 1.351(2) Å, respectively, deviate from the expected value of a pure C=O double bond to longer and the expected value of a pure C–N single bond to shorter values in agreement with expected amide-type resonance along the C=O(NH) moiety. Both values are also in good agreement with values apparent for other carboxylic acid amides deposited with the Cambridge Structural Database [12]. The resonance appears to not extend to the aromatic system as well as the least-squares planes as defined by the carbon atoms of the aromatic system on the one hand and the non-hydrogen atoms of the hexanoic acid amide side chain on the other hand enclose an angle of 42.28(10)°. The pentyl chain adopts its ideal zig–zag conformation whose carbon atoms are almost co-planar.

In the crystal, classical hydrogen bonds of the N–H···O type as well as C–H···O contacts whose range falls by more

than 0.1 Å below the sum of van-der-Waals radii are observed. The latter are supported by one of the hydrogen atoms of the methylene group in α -position to the carbonyl group. In terms of graph-set analysis [13], the descriptor for these contacts is *C*₁¹(4)*C*₁¹(4). In total, the molecules are connected to infinite chains along the crystallographic *a* axis. π -Stacking is not prominent in the title crystal structure as the shortest distance between two centers of gravity was measured at 4.7889(12) Å.

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