Redetermination of the structure of *N*-(2-benzoyl-4-chlorophenyl)-2-chloroacetamide, $C_{15}H_{11}Cl_2NO_2$

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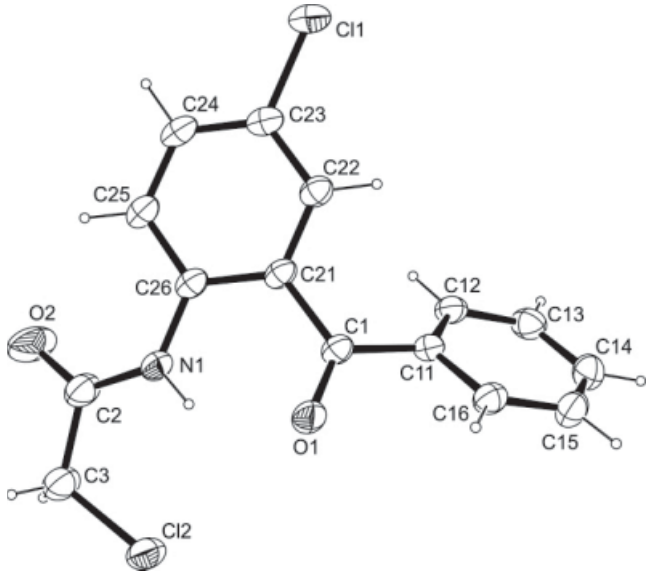
Received October 18, 2012, accepted December 03, 2012, available online May 31, 2012, CCDC no. 1267/3934



Abstract

$C_{15}H_{11}Cl_2NO_2$, triclinic, $P\bar{1}$ (no. 2), a = 5.9681(3) Å, b = 9.9888(5) Å, c = 11.9936(6) Å, α = 81.800(2)°, β = 77.113(2)°, γ = 81.364(2)°, V = 684.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.0314, $wR_{ref}(F^2)$ = 0.0818, T = 200 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.29×0.43×0.56 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.73 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	56.76°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11299, 3287
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3048
$N(param)_{refined}$:	185
Programs:	SHELX [11], ORTEP-3 [12], MERCURY [13], PLATON [14]

Source of material

The title compound was obtained as a gift sample from R. L. Fine Chem., Bengaluru, India. The compound was recrystallized from dichloromethane & acetone (*v*:*v* = 1:1) by slow evaporation.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å for aromatic carbon atoms and C–H 0.99 Å for methylene groups) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). The nitrogen-bound H atom was located from a Difference Fourier map and refined freely.

Discussion

Phenylmethanones are a class of compounds having various pharmacological properties. Benzophenone and related compounds have been reported to show antiallergic, anti-inflammatory, antiasthmatic, antimalarial, anti-microbial and antianaphylactic activity [1]. 4-Aminobenzophenones have high anti-inflammatory activity [2], a benzophenyl cyano derivative acts as a vasorelaxant [3] and the piperidinyl derivative produces analgesia [4]. Certain aminothiophenes of phenylmethanone are positive allosteric regulators of the human A1 adenosine receptor [5] and biphenyl derivatives show moderate to high activity against Mycobacterium tuberculosis in in vitro studies [6]. Recently a related compound has been reported by our research group [7].The structure of the title compound - that has been reported at room temperature only so far [8] - was re-determined at 200 K to allow for the comparison of metrical parameters with other members of the series to be investigated. While one of the two aromatic moieties of the benzophenone is an unsubstituted phenyl group, the other one bears a chloro atom and a 2-chloroacteamide substituent in *ortho* and *meta* position, respectively. The amide moiety features a terminal chloromethyl group with the chlorine atom nearly in-plane with the nitrogen atom. The respective N–C–C–Cl angle is found at -12.1(2)°. The least-squares planes defined by the respective carbon atoms of both aromatic substituents intersect at an angle of 61.72(4)° (Fig.). In the crystal, the nitrogen-bound hydrogen atom forms an intramolecular hydrogen bond with the oxygen atom of the benzophenone core. In addition, a C–H···O contact whose range falls by more than 0.1 Å below the sum of van-der-Waals radii is observed between the hydrogen atom in *ortho* position to the amide-derived substituent and the oxygen atom of the amide group in a neighbouring molecule. The latter contacts connect the molecules to centrosymmetric dimers. In terms of graph-set analysis [5, 6], the unary level descriptors for these contacts are $S(6)$ and R^2_2. In addition, a C–H···π interaction is observed that is supported by one of the hydrogen atoms of the methylene group and the aromatic system of the unsubstituted phenyl group. A dispersive interaction between two chlorine atoms (d(Cl···Cl: 3.3043(6) Å) is apparent in the crystal structure as well.

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	2*i*	0.614(3)	0.348(2)	0.365(2)	0.040(5)
H(3A)	2*i*	0.3787	0.2248	0.6250	0.047
H(3B)	2*i*	0.2969	0.1571	0.5305	0.047
H(12)	2*i*	1.1754	0.6268	0.2648	0.032
H(13)	2*i*	1.2348	0.8564	0.2394	0.039
H(14)	2*i*	1.0020	1.0189	0.1416	0.042
H(15)	2*i*	0.7081	0.9533	0.0695	0.041
H(16)	2*i*	0.6319	0.7272	0.1042	0.033
H(22)	2*i*	1.2433	0.4678	0.1091	0.030
H(24)	2*i*	1.4249	0.0873	0.2531	0.036
H(25)	2*i*	1.0606	0.0884	0.3749	0.034

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	2*i*	1.63233(6)	0.27130(4)	0.07500(3)	0.0248(2)	0.0384(2)	0.0394(2)	0.0048(1)	–0.0028(1)	–0.0120(1)
Cl(2)	2*i*	0.23669(6)	0.38977(4)	0.49668(3)	0.0303(2)	0.0358(2)	0.0364(2)	0.0066(1)	–0.0058(1)	–0.0031(1)
O(1)	2*i*	0.6069(2)	0.5104(1)	0.2474(1)	0.0204(4)	0.0277(5)	0.0518(6)	–0.0007(4)	–0.0117(4)	0.0028(4)
O(2)	2*i*	0.7466(3)	0.1016(2)	0.5206(1)	0.0586(8)	0.0636(9)	0.0581(8)	0.0325(7)	0.0103(7)	0.0288(7)
N(1)	2*i*	0.7076(2)	0.2830(1)	0.3848(1)	0.0229(5)	0.0228(5)	0.0326(6)	0.0026(4)	–0.0076(4)	–0.0009(4)
C(1)	2*i*	0.8153(2)	0.5181(1)	0.2249(1)	0.0217(6)	0.0225(6)	0.0275(6)	0.0009(4)	–0.0086(5)	–0.0033(5)
C(2)	2*i*	0.6328(3)	0.1997(2)	0.4804(1)	0.0373(7)	0.0304(7)	0.0309(7)	0.0063(6)	–0.0038(6)	0.0006(5)
C(3)	2*i*	0.3833(3)	0.2297(2)	0.5415(1)	0.0386(8)	0.0351(7)	0.0345(7)	0.0078(6)	0.0004(6)	0.0040(6)
C(11)	2*i*	0.8938(2)	0.6554(1)	0.1897(1)	0.0202(5)	0.0223(5)	0.0245(5)	0.0009(4)	–0.0028(4)	–0.0038(4)
C(12)	2*i*	1.0768(2)	0.6935(1)	0.2277(1)	0.0204(6)	0.0271(6)	0.0306(6)	0.0001(5)	–0.0043(5)	–0.0049(5)
C(13)	2*i*	1.1140(2)	0.8294(2)	0.2109(1)	0.0263(6)	0.0318(7)	0.0386(7)	–0.0062(5)	–0.0026(5)	–0.0091(6)
C(14)	2*i*	0.9757(3)	0.9260(1)	0.1529(1)	0.0394(8)	0.0239(6)	0.0391(7)	–0.0055(6)	0.0006(6)	–0.0033(5)
C(15)	2*i*	0.7994(3)	0.8874(1)	0.1114(1)	0.0377(7)	0.0268(6)	0.0338(7)	0.0035(5)	–0.0065(6)	0.0017(5)
C(16)	2*i*	0.7558(2)	0.7530(1)	0.1308(1)	0.0260(6)	0.0280(6)	0.0279(6)	0.0016(5)	–0.0072(5)	–0.0028(5)
C(21)	2*i*	0.9863(2)	0.3944(1)	0.2354(1)	0.0212(5)	0.0206(5)	0.0301(6)	0.0011(4)	–0.0105(5)	–0.0062(5)
C(22)	2*i*	1.2043(2)	0.3910(1)	0.1627(1)	0.0235(6)	0.0233(6)	0.0313(6)	–0.0006(5)	–0.0091(5)	–0.0065(5)
C(23)	2*i*	1.3635(2)	0.2759(1)	0.1686(1)	0.0214(6)	0.0283(6)	0.0320(6)	0.0012(5)	–0.0078(5)	–0.0119(5)
C(24)	2*i*	1.3119(2)	0.1646(1)	0.2481(1)	0.0277(6)	0.0238(6)	0.0394(7)	0.0065(5)	–0.0120(5)	–0.0095(5)
C(25)	2*i*	1.0957(2)	0.1653(1)	0.3207(1)	0.0293(6)	0.0213(6)	0.0353(7)	0.0024(5)	–0.0108(5)	–0.0049(5)
C(26)	2*i*	0.9295(2)	0.2789(1)	0.3142(1)	0.0234(6)	0.0212(5)	0.0294(6)	0.0012(4)	–0.0104(5)	–0.0060(5)

Acknowledgments. Thammarse S. Yamuna thanks the University of Mysore for research facilities.

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