

Refinement

Refinement on <i>F</i>	(Δ/σ) _{max} = 0.01
<i>R</i> = 0.076	$\Delta\rho_{\text{max}} = 0.58 \text{ e } \text{Å}^{-3}$
<i>wR</i> = 0.087	$\Delta\rho_{\text{min}} = -0.49 \text{ e } \text{Å}^{-3}$
<i>S</i> = 2.96	Extinction correction: none
991 reflections	Atomic scattering factors
127 parameters	from <i>International Tables</i>
H-atom parameters not refined	for <i>X-ray Crystallography</i>
<i>w</i> = 1/ σ^2 (<i>F</i>)	(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{\text{eq}} = (1/3)\sum_i \sum_j B_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
Br	0.6383 (2)	0.4596 (3)	0.4548 (1)	5.20 (3)
O(1)	0.3021 (8)	-0.429 (3)	0.1175 (6)	5.7 (3)
O(2)	0.4326 (9)	-0.281 (3)	0.0399 (6)	6.0 (3)
O(3)	0.7089 (7)	0.256 (2)	0.2631 (7)	5.0 (2)
C(1)	0.347 (1)	-0.473 (3)	0.041 (1)	5.1 (4)
C(2)	0.452 (1)	-0.166 (3)	0.1193 (9)	4.1 (3)
C(3)	0.530 (1)	0.004 (3)	0.1488 (8)	3.8 (3)
C(4)	0.528 (1)	0.097 (3)	0.2315 (8)	3.4 (3)
C(5)	0.4474 (9)	0.002 (3)	0.2783 (8)	3.3 (3)
C(6)	0.369 (1)	-0.176 (3)	0.2467 (9)	4.2 (3)
C(7)	0.371 (1)	-0.263 (3)	0.1650 (9)	4.4 (3)
C(8)	0.602 (1)	0.286 (3)	0.2801 (8)	3.2 (3)
C(9)	0.586 (1)	0.188 (3)	0.3680 (8)	3.6 (3)
C(10)	0.470 (1)	0.124 (3)	0.3653 (8)	4.1 (3)

Table 2. Selected geometric parameters (Å, °)

C(1)—O(1)	1.43 (2)	C(5)—C(6)	1.36 (2)
C(1)—O(2)	1.42 (2)	C(5)—C(10)	1.52 (2)
C(2)—C(3)	1.33 (2)	C(6)—C(7)	1.39 (2)
C(2)—C(7)	1.41 (2)	C(7)—O(1)	1.35 (2)
C(2)—O(2)	1.39 (2)	C(8)—C(9)	1.53 (2)
C(3)—C(4)	1.41 (2)	C(8)—O(3)	1.41 (2)
C(4)—C(5)	1.41 (2)	C(9)—C(10)	1.49 (2)
C(4)—C(8)	1.47 (2)	C(9)—Br	1.98 (1)
C(1)—O(1)—C(7)	106 (1)	C(1)—O(2)—C(2)	107 (1)
C(2)—C(3)—C(4)	116 (1)	C(2)—C(7)—C(6)	119 (1)
C(2)—C(7)—O(1)	111 (1)	C(3)—C(2)—C(7)	125 (1)
C(3)—C(2)—O(2)	128 (1)	C(3)—C(4)—C(5)	120 (1)
C(3)—C(4)—C(8)	129 (1)	C(4)—C(5)—C(10)	108 (1)
C(4)—C(5)—C(6)	123 (1)	C(4)—C(8)—C(9)	99 (1)
C(4)—C(8)—O(3)	113 (1)	C(5)—C(4)—C(8)	112 (1)
C(5)—C(6)—C(7)	117 (1)	C(5)—C(10)—C(9)	101 (1)
C(6)—C(5)—C(10)	130 (1)	C(6)—C(7)—O(1)	130 (1)
C(7)—C(2)—O(2)	107 (1)	C(8)—C(9)—Br	113 (1)
C(8)—C(9)—C(10)	106 (1)	C(9)—C(8)—O(3)	112 (1)
C(10)—C(9)—Br	114 (1)	O(1)—C(1)—O(2)	107 (1)

Program used for data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Programs used for data reduction and structure solution: *SDP* (Frenz, 1978) via direct methods with *MULTAN80* (Main *et al.*, 1980) and difference Fourier syntheses. Program used for molecular graphics: *ORTEP* (Johnson, 1965). H atoms were introduced at idealized positions before the last cycle of refinement, which included anisotropic displacement parameters for all non-H atoms.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1083). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4-Amino-3-chlorobenzophenone

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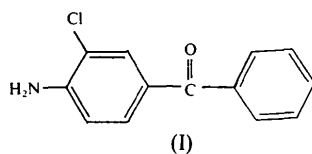
(Received 3 September 1993; accepted 25 January 1994)

Abstract

The title compound, C₁₃H₁₀ClNO, has two phenyl groups, one of which has three substituents. The dihedral angles between the plane of the carbonyl group and the planes of the substituted and unsubstituted phenyl rings are 151.4 (3) and 149.7 (3)°, respectively.

Comment

It is reported that 4-aminobenzophenone (ABP) has a large second-harmonic generation (SHG) efficiency (Frazier & Cockerham, 1987). We have synthesized ABP and its derivatives in order to investigate their non-linear optical properties. The title compound, (I), is one such compound, but has no SHG efficiency, which depends on the conjugation of donor and acceptor.



The dihedral angle between the C(1)–C(6) phenyl plane and the plane of the NH₂ donor group is 4.0 (8)°, whereas the dihedral angle between this phenyl plane and the plane of the C=O acceptor group is 151.4 (3)°. Such weak conjugation results in a small SHG effect. The centrosymmetric space group prohibits the title compound from showing any SHG properties.

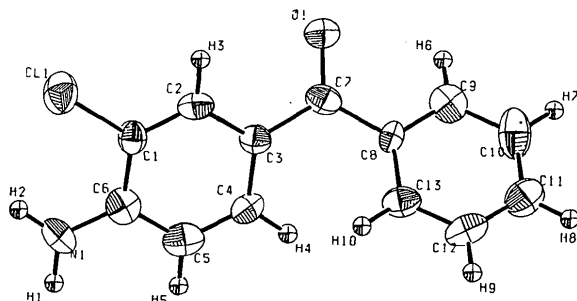


Fig. 1. The molecular configuration of 4-amino-3-chlorobenzophenone.

Experimental

Crystal data

C₁₃H₁₀ClNO

M_r = 231.68

Orthorhombic

Pbca

a = 24.07 (2) Å

b = 12.216 (7) Å

c = 7.406 (6) Å

V = 2178 (6) Å³

Z = 8

D_x = 1.414 Mg m⁻³

D_m = 1.41 Mg m⁻³

Mo *K*α radiation

λ = 0.71069 Å

Cell parameters from 20 reflections

θ = 6–9°

μ = 0.323 mm⁻¹

T = 293 K

Needle

1.2 × 0.5 × 0.15 mm

Colourless

Crystal source: from alcoholic solution

Data collection

Rigaku AFC-5R diffractometer

ω/2θ scans

Absorption correction:

empirical

T_{min} = 0.637, *T_{max}* = 1.000

4268 measured reflections

2243 independent reflections

673 observed reflections

[*I* > 3.0σ(*I*)]

θ_{max} = 20°

h = 0 → 29

k = -15 → 15

l = 0 → 9

3 standard reflections

monitored every 250 reflections

intensity variation: 0.1%

Refinement

Refinement on *F*

R = 0.062

wR = 0.072

S = 1.50

673 reflections

145 parameters

H-atom parameters not refined

w = 1/σ²(*F*)

(Δ/σ)_{max} = 0.0048

Δρ_{max} = 0.616 e Å⁻³

Δρ_{min} = -0.288 e Å⁻³

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV, Tables

2.2A, 2.3.1)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i>
Cl(1)	0.07249 (9)	0.2218 (2)	0.0264 (3)	4.4 (1)
O(1)	-0.1358 (3)	0.3307 (6)	0.125 (1)	4.5 (3)
N(1)	0.0673 (3)	0.0164 (6)	0.235 (1)	4.7 (4)
C(1)	0.0129 (3)	0.1618 (7)	0.114 (1)	2.8 (4)
C(2)	-0.0368 (4)	0.2111 (6)	0.089 (1)	3.0 (4)
C(3)	-0.0858 (3)	0.1672 (7)	0.157 (1)	2.5 (4)
C(4)	-0.0819 (3)	0.0662 (7)	0.246 (1)	3.1 (4)
C(5)	-0.0322 (4)	0.0176 (7)	0.274 (1)	3.5 (4)
C(6)	0.0175 (4)	0.0615 (8)	0.203 (1)	3.4 (4)
C(7)	-0.1383 (4)	0.2294 (7)	0.135 (1)	3.0 (4)
C(8)	-0.1927 (3)	0.1743 (7)	0.127 (1)	2.5 (4)
C(9)	-0.2391 (4)	0.2317 (8)	0.184 (1)	3.8 (4)
C(10)	-0.2912 (4)	0.187 (1)	0.162 (1)	4.6 (5)
C(11)	-0.2985 (4)	0.083 (1)	0.087 (1)	4.2 (5)
C(12)	-0.2515 (5)	0.0268 (7)	0.027 (1)	4.0 (4)
C(13)	-0.1993 (4)	0.0695 (7)	0.050 (1)	3.4 (4)

Table 2. Selected geometric parameters (Å, °)

Cl(1)–C(1)	1.734 (8)	C(5)–C(6)	1.41 (1)
O(1)–C(7)	1.241 (9)	C(7)–C(8)	1.47 (1)
N(1)–C(6)	1.34 (1)	C(8)–C(9)	1.38 (1)
C(1)–C(2)	1.35 (1)	C(8)–C(13)	1.41 (1)
C(1)–C(6)	1.40 (1)	C(9)–C(10)	1.38 (1)
C(2)–C(3)	1.39 (1)	C(10)–C(11)	1.39 (1)
C(3)–C(7)	1.48 (1)	C(11)–C(12)	1.40 (1)
C(4)–C(5)	1.35 (1)	C(12)–C(13)	1.37 (1)
C(2)–C(1)–C(6)	121.6 (8)	O(1)–C(7)–C(8)	119.8 (9)
C(2)–C(1)–Cl(1)	119.6 (7)	O(1)–C(7)–C(3)	118.4 (9)
C(6)–C(1)–Cl(1)	118.8 (7)	C(8)–C(7)–C(3)	121.9 (8)
C(1)–C(2)–C(3)	122.2 (7)	C(9)–C(8)–C(13)	119.5 (8)
C(2)–C(3)–C(4)	116.8 (8)	C(9)–C(8)–C(7)	118.3 (8)
C(2)–C(3)–C(7)	119.2 (7)	C(13)–C(8)–C(7)	121.9 (8)
C(4)–C(3)–C(7)	124.0 (8)	C(10)–C(9)–C(8)	119.8 (9)
C(5)–C(4)–C(3)	121.1 (8)	C(9)–C(10)–C(11)	121 (1)
C(4)–C(5)–C(6)	121.9 (8)	C(10)–C(11)–C(12)	118 (1)
N(1)–C(6)–C(1)	120.9 (9)	C(13)–C(12)–C(11)	121.1 (9)
N(1)–C(6)–C(5)	122.5 (8)	C(12)–C(13)–C(8)	119.7 (9)
C(1)–C(6)–C(5)	116.3 (9)		

A colourless transparent crystal was mounted in a random orientation on a glass fibre. Data were collected using *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). The data were corrected for Lorentz and polarization factors, and the structure was solved by direct methods using *MITHRIL* (Gilmore, 1983). H atoms were placed in geometrically calculated positions with C–H = 0.95 Å, but were not included in the refinement. The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for C, N, O and Cl atoms. All calculations were performed on a VAX 11/785 computer using the *TEXSAN* program package (Molecular Structure Corporation, 1987). Molecular graphics were produced using *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, bond distances and angles involving H atoms and selected torsion angles have been deposited with the IUCr (Reference: AB1116). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-Phenyl-2-phenyliminotetrahydro-1,3-thiazole-2-carbothioamide, C₁₆H₁₅N₃S₂

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Abstract

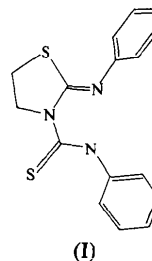
The thiazolidine ring in the title compound has an envelope conformation, *E*₄, with a C atom out of the plane of the ring. The C—S and C—N bond lengths clearly show the typical resonance of the thiourea group. The molecules are linked by van der Waals forces and an intramolecular N⋯N hydrogen bond is present.

Comment

A large number of thiazole or thiazine derivatives may be obtained by the reaction of haloalkylisothiocyanates with amines. Compounds containing the thiazoline or thiazine structural fragment have biological properties as potential herbicides and anti-fungal agents (Behner & Stendel, 1971; Sumitomo

Chemical Co. Ltd, 1982). Some of them also display medical properties (Johnson, Boukma & Kim, 1970).

The two phenyl groups in the title compound, (I), are planar (the maximum deviation from the least-squares plane is 0.007 Å). Bond lengths and angles are normal. The thiourea group is also planar (the maximum deviation from the least-squares plane is 0.006 Å). The S—C distance of 1.661 (2) Å and the C—N distances of 1.390 (3) and 1.344 (3) Å clearly show the resonance of the thiourea system (Kunchur & Truter, 1958; Valle, Cojazzi, Busetti & Mammi, 1970).



The thiazolidine ring is not planar, and in terms of the ring-puckering coordinates (Cremer & Pople, 1975) the amplitude and phase magnitudes are $Q = 0.353$ (3) Å and $\varphi = -33.6$ (4)° for the sequence of atoms S1, C2, N3, C4 and C5. The conformation of the ring is an envelope with C5 lying 0.316 (3) Å out of the least-squares plane of the ring. However, Nardelli (1983*a*) asymmetry parameters (Table 3) show a departure from ideal *E* symmetry. The substituent atoms C31 and N21 are -0.143 (2) and 0.296 (2) Å from the least-squares plane. Bond distances and angles agree well with those found for a

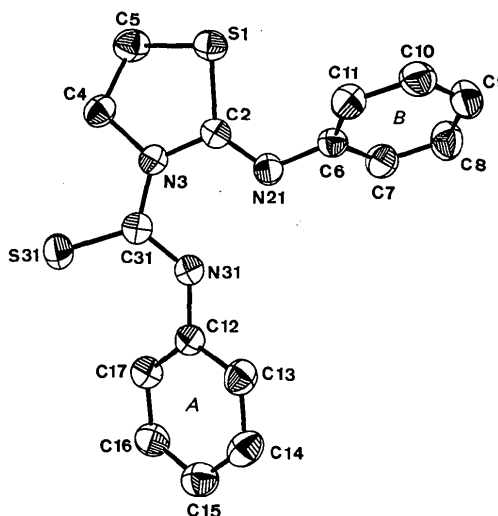


Fig. 1. An ORTEPII (Johnson, 1976) view of the molecule viewed down the *c* axis, showing the atomic numbering scheme. Displacement ellipsoids are shown at the 50% probability level.