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N-Benzoyl-*N*′-(1,1-dimethyl-3-oxobutyl)-hydrazine hydrochloride

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.075
Data-to-parameter ratio = 15.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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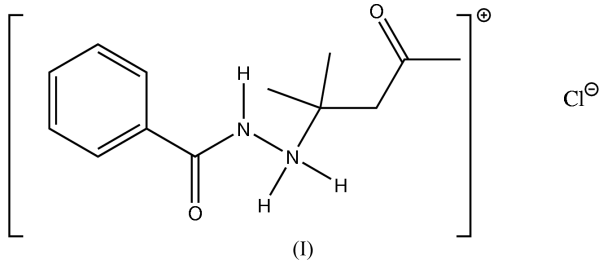
The crystal structure of the title compound, $C_{13}H_{19}N_2O_2^+\cdot Cl^-$, is stabilized by intra- and intermolecular hydrogen bonds, forming centrosymmetric dimers.

Comment

The aim of our investigations (Ton, 2004) was to synthesize 1-benzoyl-2-(2-propylidene)hydrazone by reacting acetone and benzoic acid hydrazide in the presence of HCl, in order to examine the intra- and intermolecular hydrogen-bond interactions in the solid state. Unfortunately, we obtained crystals of (I) instead.



(I)

The bond lengths and angles in (I) do not show unusual values. One of the H atoms bonded to N2 forms two intramolecular hydrogen bonds to both carbonyl O atoms. The other one and the H atom bonded to N1 form a hydrogen bond to the Cl^- ion. This results in the formation of centrosymmetric dimers (Fig. 2).

Experimental

In a 100 ml flask, ten drops of concentrated HCl were added dropwise to 20 ml acetone. 2 g of anhydrous sodium sulfate and 0.97 g (0.0071 mol) benzhydrazide were then added to the solution, which was stirred for 20 h and subsequently filtered. The excess acetone was removed. Afterwards, the residue was crystallized from acetone.

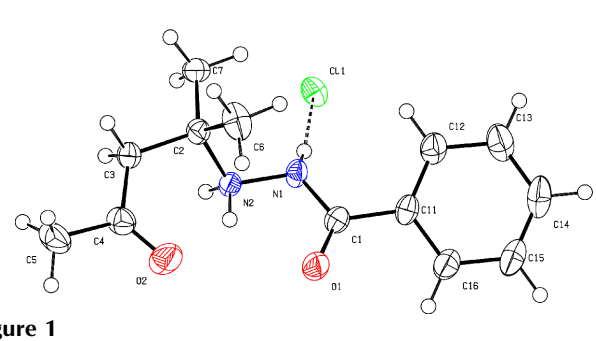


Figure 1
Perspective view of the title compound, with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates a hydrogen bond.

Crystal data

$C_{13}H_{19}N_2O_2^+\cdot Cl^-$
$M_r = 270.75$
Monoclinic, $P2_1/c$
$a = 7.087\ (1)$ Å
$b = 21.023\ (2)$ Å
$c = 10.019\ (1)$ Å
$\beta = 99.042\ (9)^\circ$
$V = 1474.2\ (3)$ Å^3
$Z = 4$
$D_x = 1.220$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11582 reflections
$\theta = 3.5$–25.8°
$\mu = 0.26$ mm^{-1}
$T = 173\ (2)$ K
Block, colourless
0.30 × 0.23 × 0.21 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 1990; Blessing, 1995)
$T_{min} = 0.927$, $T_{max} = 0.957$
16453 measured reflections
2832 independent reflections
2190 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 25.9^\circ$
$h = -8 \rightarrow 8$
$k = -25 \rightarrow 25$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.075$
$S = 0.95$
2832 reflections
178 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0461P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.3564 (19)	N2—C2	1.5192 (18)
N1—N2	1.4185 (16)		
C1—N1—N2	114.56 (12)	N2—N1—H1	115.3 (13)
C1—N1—H1	123.8 (13)	N1—N2—C2	114.32 (11)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···Cl1^{i}	0.97 (2)	2.05 (2)	3.0184 (13)	171.7 (16)
N1—H1···Cl1	0.839 (19)	2.29 (2)	3.1291 (13)	174.4 (17)
N2—H2A···O1	0.855 (18)	2.056 (18)	2.5818 (16)	119.1 (14)
N2—H2A···O2	0.855 (18)	2.071 (18)	2.7323 (16)	133.6 (15)

Symmetry code: (i) $-x, -y, 1-z$.

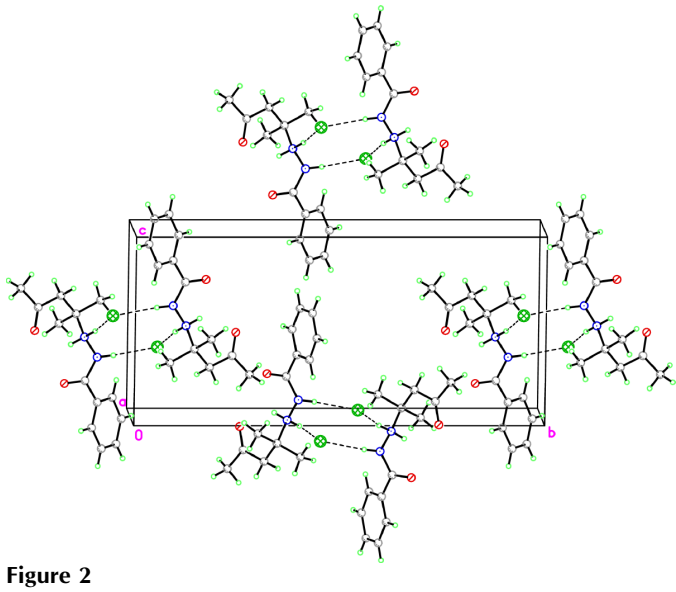


Figure 2
Packing diagram of the title compound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

H atoms bonded to C atoms were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl})] using a riding model, with aromatic C—H = 0.95 Å, methyl C—H = 0.98 Å and methylene C—H = 0.99 Å. The methyl groups were allowed to rotate but not to tip. H atoms bonded to N atoms were refined isotropically.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELX*97 and *PLATON*.

References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
Ton, Q. C. (2004). Diploma thesis, University of Frankfurt, Germany.