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2-Amino-5-chlorobenzophenone

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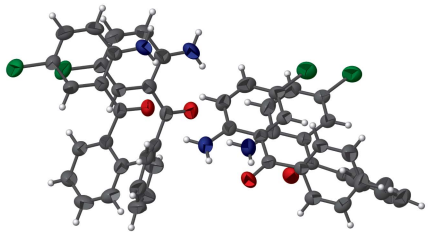
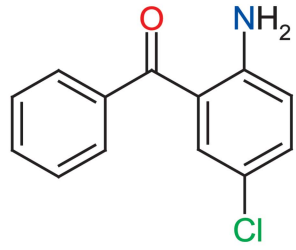
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound (systematic name: 2-benzoyl-4-chloroaniline), $C_{13}H_{10}ClNO$, contains four independent molecules with similar dihedral angles between the benzene rings [ranging from 53.7 (2) to 59.8 (2)°]. In the crystal, the molecules are linked by N—H···O hydrogen bonds into a three-dimensional supramolecular architecture.

3D view



Chemical scheme



Structure description

Benzophenone derivatives often exhibit biological activities and are used as antiallergic, anti-inflammatory, antiasthamatic, antimalarial, antimicrobial and antianaphylactic agents (Evans *et al.*, 1987; Wiesner *et al.*, 2002; Sieroń *et al.*, 2004). 2-Aminobenzophenone and its derivatives are important because of their applications in heterocyclic synthesis and medicines (Walsh, 1980). 2-Aminobenzophenone has been used as the starting material for the synthesis of 1,4-benzodiazepines (Sternbach *et al.*, 1962) and anti-inflammatory agents (Ottosen *et al.*, 2003) as well as peptidoaminobenzophenones, a class of open-ring derivatives of 1,4-benzodiazepines (Hirai *et al.*, 1980). 2-Aminobenzophenone derivatives have also been used as antimitotic agents (Liou *et al.*, 2002). Related molecules, *e.g.* benzophenones-1, -2, -3, -4, -6, -8 and -12 (Wang & Lee, 2003), are used as sunscreen agents. The crystal structures of chlorinated benzophenones, such as 4-chloro-2-(3,4,5-trimethoxybenzoyl)phenol (Hsieh *et al.*, 2003) and pestalone (Cueto *et al.*, 2001), have also been determined and their role as potential anticancer agents and antibiotics has been examined.

In the title compound (Fig. 1), an intramolecular hydrogen bond (Table 1) between the carbonyl O and an amine H atom from the 2-aminobenzoyl group stabilizes each of the four independent molecules and forms a six-membered ring. The dihedral angles between

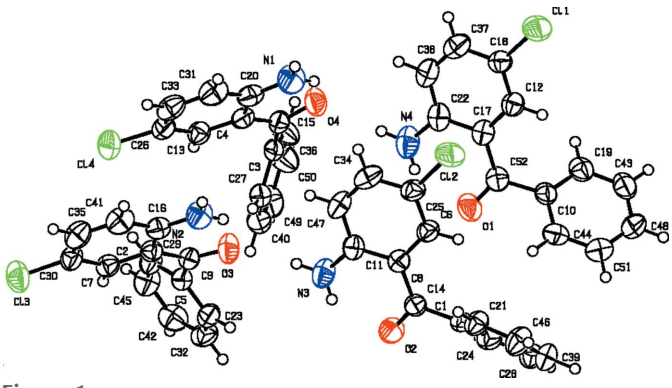


Figure 1
The four independent molecules in the asymmetric unit of the title compound showing the atom labelling scheme and 50% probability displacement ellipsoids.

the benzene rings range from 53.7 (2) to 59.8 (2)°. This compares with values of 56° for benzophenone (Fleischer *et al.*, 1968) and 54.39 (8)° for 2-methylamino-5-chlorobenzophenone (Cox *et al.*, 1997).

In the crystal, N—H···O hydrogen bonds (Table 1) link the molecules into chains along [1$\bar{2}$0].

Synthesis and crystallization

The title compound was purchased from Sigma Aldrich and recrystallized by slow evaporation of an MeOH solution, prismatic colourless single crystals being obtained after four days.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors are grateful to the Department of Chemistry, National Taras Shevchenko University, for financial support, and to the Department of Chemistry, Langat Singh College, B. R. A. Bihar University, Muzaffarpur, India, for providing a laboratory.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.86	2.21	2.971 (5)	147
N1—H1B···O4	0.86	2.01	2.642 (5)	130
N2—H2A···O1^{ii}	0.86	2.16	2.962 (4)	154
N2—H2B···O3	0.86	1.99	2.629 (4)	130
N3—H3A···O3	0.86	2.19	2.960 (4)	149
N3—H3B···O2	0.86	1.98	2.623 (4)	130
N4—H4A···O4	0.86	2.19	2.986 (5)	153
N4—H4B···O1	0.86	2.03	2.654 (5)	129

Symmetry codes: (i) $x-1, y+1, z$; (ii) $x, y+1, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{10}ClNO$
M_r	231.67
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	273
a, b, c (Å)	9.7335 (7), 10.6473 (7), 24.7554 (17)
α, β, γ (°)	87.443 (2), 84.272 (2), 62.978 (2)
V (Å^3)	2274.1 (3)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.31
Crystal size (mm)	0.25 × 0.15 × 0.10
Data collection	
Diffractometer	Bruker APEX CCD area detector
Absorption correction	Empirical (using intensity measurements) (*SADABS*; Bruker, 2001)
T_{min}, T_{max}	0.945, 0.969
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	34850, 11324, 4512
R_{int}	0.079
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.668
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.080, 0.225, 1.06
No. of reflections	11324
No. of parameters	577
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.27

Computer programs: *APEX2*, *SMART* and *SAINT-Plus* (Bruker, 2007), *SHELXS86*, *SHELXL97* and *SHELXTL* (Sheldrick, 2008) and *WinGX* (Farrugia, 2012).

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full crystallographic data

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2-Benzoyl-4-chloroaniline

Crystal data

$C_{13}H_{10}ClNO$
$M_r = 231.67$
Triclinic, $P\overline{1}$
$a = 9.7335$ (7) Å
$b = 10.6473$ (7) Å
$c = 24.7554$ (17) Å
$\alpha = 87.443$ (2)°
$\beta = 84.272$ (2)°
$\gamma = 62.978$ (2)°
$V = 2274.1$ (3) Å^3
$Z = 8$
$F(000) = 960$
$D_x = 1.353$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 11324 reflections
$\theta = 2.2$–$28.4°$
$\mu = 0.31$ mm^{-1}
$T = 273$ K
Prism, colorless
0.25 × 0.15 × 0.10 mm

Data collection

Bruker APEX CCD area detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: empirical (using intensity measurements) (SADABS; Bruker, 2001)
$T_{min} = 0.945$, $T_{max} = 0.969$
34850 measured reflections
11324 independent reflections
4512 reflections with $I > 2\sigma(I)$
$R_{int} = 0.079$
$\theta_{max} = 28.4°$, $\theta_{min} = 2.2°$
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -33 \rightarrow 33$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.080$
$wR(F^2) = 0.225$
$S = 1.06$
11324 reflections
577 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0827P)^2 + 0.4385P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

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. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.
All of the H atoms were placed in their calculated positions and then refined using the riding model with N—H = 0.88, C —H = 0.95 Å, and with $U_{iso}(H) = 1.18–1.21U_{eq}(C,N)$.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	−0.22973 (12)	0.40371 (11)	0.57327 (5)	0.0702 (4)
Cl4	0.32333 (13)	0.90689 (11)	0.92390 (5)	0.0699 (4)
Cl3	0.82354 (14)	0.92281 (11)	0.92846 (5)	0.0726 (4)
Cl2	0.25035 (13)	0.42264 (11)	0.57452 (5)	0.0742 (4)
O2	0.8276 (3)	0.0336 (3)	0.70037 (10)	0.0603 (8)
O1	0.3672 (3)	0.0469 (3)	0.69319 (11)	0.0660 (8)
O3	0.6319 (3)	0.5398 (3)	0.79903 (11)	0.0618 (8)
O4	0.0801 (3)	0.5417 (3)	0.80924 (11)	0.0638 (8)
N3	0.6669 (4)	0.2790 (3)	0.74839 (12)	0.0612 (9)
H3A	0.6444	0.3403	0.7735	0.073*
H3B	0.7476	0.1989	0.7494	0.073*
N2	0.5589 (4)	0.7840 (3)	0.75068 (12)	0.0608 (9)
H2A	0.5241	0.8453	0.7253	0.073*
H2B	0.5574	0.7041	0.7489	0.073*
C1	0.7822 (4)	−0.0228 (4)	0.61666 (14)	0.0399 (9)
C2	0.6802 (4)	0.7143 (4)	0.83548 (15)	0.0419 (9)
N4	0.1714 (4)	0.2712 (4)	0.75153 (13)	0.0708 (11)
H4A	0.1431	0.3318	0.7773	0.085*
H4B	0.2566	0.1947	0.7521	0.085*
C3	0.2427 (4)	0.4567 (4)	0.87849 (15)	0.0435 (9)
C4	0.1757 (4)	0.6974 (4)	0.83300 (15)	0.0446 (9)
C5	0.7346 (4)	0.4793 (4)	0.88307 (14)	0.0417 (9)
C6	0.5002 (4)	0.2495 (4)	0.62529 (15)	0.0448 (9)
H6	0.5168	0.1854	0.5981	0.054*
C7	0.7443 (4)	0.7518 (4)	0.87631 (15)	0.0446 (9)
H7	0.7884	0.6877	0.9038	0.054*
C8	0.6051 (4)	0.2112 (4)	0.66532 (14)	0.0414 (9)
C9	0.6786 (4)	0.5782 (4)	0.83669 (15)	0.0441 (9)
N1	0.0435 (4)	0.7667 (4)	0.75073 (13)	0.0697 (10)
H1A	0.0088	0.8263	0.7248	0.084*
H1B	0.0344	0.6900	0.7514	0.084*
C10	0.2946 (4)	−0.0435 (4)	0.62334 (15)	0.0460 (10)
C11	0.5753 (5)	0.3087 (4)	0.70726 (15)	0.0470 (10)
C12	0.0196 (4)	0.2353 (4)	0.62564 (15)	0.0466 (10)
H12	0.0446	0.1728	0.5970	0.056*
C13	0.2405 (4)	0.7356 (4)	0.87332 (15)	0.0447 (9)
H13	0.2788	0.6742	0.9021	0.054*
C14	0.7410 (4)	0.0742 (4)	0.66330 (15)	0.0422 (9)

C15	0.1613 (4)	0.5656 (4)	0.83826 (15)	0.0467 (10)
C16	0.6155 (4)	0.8130 (4)	0.79311 (15)	0.0467 (10)
C17	0.1186 (4)	0.1997 (4)	0.66732 (15)	0.0456 (10)
C18	−0.1129 (4)	0.3602 (4)	0.62636 (15)	0.0463 (10)
C19	0.1906 (5)	−0.0987 (4)	0.61899 (15)	0.0518 (10)
H19	0.0956	−0.0609	0.6397	0.062*
C20	0.1119 (4)	0.7934 (4)	0.79014 (16)	0.0513 (10)
C21	0.7963 (4)	0.0193 (4)	0.56358 (15)	0.0505 (10)
H21	0.7745	0.1126	0.5561	0.061*
C22	0.0815 (5)	0.2965 (4)	0.70985 (15)	0.0499 (10)
C52	0.2646 (5)	0.0684 (4)	0.66352 (15)	0.0463 (10)
C23	0.8413 (4)	0.3404 (4)	0.87202 (16)	0.0501 (10)
H23	0.8788	0.3124	0.8363	0.060*
C24	0.8145 (4)	−0.1618 (4)	0.62641 (16)	0.0493 (10)
H24	0.8060	−0.1917	0.6619	0.059*
C25	0.3751 (4)	0.3784 (4)	0.62560 (16)	0.0479 (10)
C26	0.2493 (4)	0.8601 (4)	0.87180 (15)	0.0478 (10)
C27	0.4016 (4)	0.4008 (4)	0.88200 (15)	0.0491 (10)
H27	0.4591	0.4368	0.8603	0.059*
C28	0.8590 (4)	−0.2570 (4)	0.58436 (17)	0.0547 (11)
H28	0.8799	−0.3502	0.5916	0.066*
C29	0.6782 (4)	0.5194 (4)	0.93601 (16)	0.0510 (10)
H29	0.6060	0.6120	0.9441	0.061*
C30	0.7437 (4)	0.8804 (4)	0.87671 (16)	0.0502 (10)
C31	0.1272 (5)	0.9185 (4)	0.78939 (17)	0.0614 (12)
H31	0.0911	0.9811	0.7607	0.074*
C32	0.8920 (5)	0.2442 (4)	0.91293 (18)	0.0583 (11)
H32	0.9638	0.1515	0.9049	0.070*
C33	0.1927 (5)	0.9517 (4)	0.82900 (18)	0.0597 (12)
H33	0.1995	1.0362	0.8273	0.072*
C34	0.3474 (5)	0.4746 (4)	0.66614 (18)	0.0625 (12)
H34	0.2624	0.5631	0.6660	0.075*
C35	0.6778 (5)	0.9772 (4)	0.83648 (17)	0.0602 (12)
H35	0.6749	1.0657	0.8374	0.072*
C36	0.1628 (5)	0.3999 (4)	0.91087 (17)	0.0648 (12)
H36	0.0571	0.4343	0.9084	0.078*
C37	−0.1510 (5)	0.4535 (4)	0.66879 (18)	0.0589 (11)
H37	−0.2413	0.5382	0.6694	0.071*
C38	−0.0569 (5)	0.4218 (4)	0.70959 (17)	0.0598 (12)
H38	−0.0851	0.4851	0.7381	0.072*
C39	0.8723 (5)	−0.2140 (4)	0.53205 (19)	0.0630 (12)
H39	0.9014	−0.2776	0.5035	0.076*
C40	0.4736 (5)	0.2923 (4)	0.91764 (18)	0.0650 (12)
H40	0.5798	0.2551	0.9198	0.078*
C41	0.6172 (5)	0.9438 (4)	0.79555 (17)	0.0589 (11)
H41	0.5755	1.0095	0.7683	0.071*
C42	0.8373 (5)	0.2840 (5)	0.96577 (19)	0.0630 (12)
H42	0.8726	0.2191	0.9938	0.076*

C43	0.2284 (5)	−0.2083 (4)	0.58428 (18)	0.0632 (12)
H43	0.1597	−0.2459	0.5818	0.076*
C44	0.4348 (5)	−0.1013 (4)	0.59149 (18)	0.0649 (12)
H44	0.5056	−0.0659	0.5939	0.078*
C45	0.7294 (5)	0.4214 (4)	0.97693 (16)	0.0623 (12)
H45	0.6906	0.4484	1.0127	0.075*
C46	0.8425 (5)	−0.0766 (4)	0.52181 (16)	0.0657 (12)
H46	0.8536	−0.0480	0.4863	0.079*
C47	0.4442 (5)	0.4398 (4)	0.70622 (16)	0.0581 (11)
H47	0.4230	0.5047	0.7336	0.070*
C48	0.3678 (5)	−0.2631 (4)	0.5530 (2)	0.0725 (14)
H48	0.3928	−0.3369	0.5291	0.087*
C49	0.3894 (6)	0.2389 (4)	0.94993 (19)	0.0742 (14)
H49	0.4380	0.1656	0.9739	0.089*
C50	0.2337 (6)	0.2945 (5)	0.94652 (19)	0.0765 (14)
H50	0.1758	0.2600	0.9688	0.092*
C51	0.4694 (5)	−0.2091 (5)	0.5569 (2)	0.0806 (15)
H51	0.5634	−0.2464	0.5356	0.097*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0616 (7)	0.0552 (7)	0.0837 (8)	−0.0151 (6)	−0.0241 (6)	0.0102 (6)
Cl4	0.0772 (8)	0.0564 (7)	0.0854 (9)	−0.0362 (6)	−0.0124 (6)	−0.0117 (6)
Cl3	0.0900 (9)	0.0613 (7)	0.0841 (9)	−0.0463 (7)	−0.0240 (7)	−0.0006 (6)
Cl2	0.0572 (7)	0.0612 (7)	0.0912 (9)	−0.0117 (6)	−0.0290 (6)	0.0080 (6)
O2	0.0649 (19)	0.0551 (17)	0.0468 (17)	−0.0116 (15)	−0.0217 (15)	0.0016 (13)
O1	0.072 (2)	0.0572 (18)	0.070 (2)	−0.0249 (16)	−0.0351 (17)	0.0057 (15)
O3	0.087 (2)	0.0549 (17)	0.0497 (17)	−0.0348 (16)	−0.0201 (16)	−0.0029 (13)
O4	0.075 (2)	0.0559 (18)	0.0651 (19)	−0.0282 (16)	−0.0307 (16)	−0.0019 (14)
N3	0.081 (3)	0.053 (2)	0.048 (2)	−0.0266 (19)	−0.009 (2)	−0.0068 (17)
N2	0.075 (2)	0.058 (2)	0.044 (2)	−0.0249 (19)	−0.0133 (18)	0.0072 (17)
C1	0.036 (2)	0.036 (2)	0.043 (2)	−0.0107 (17)	−0.0071 (17)	0.0017 (17)
C2	0.040 (2)	0.043 (2)	0.042 (2)	−0.0187 (19)	−0.0002 (18)	−0.0018 (18)
N4	0.100 (3)	0.063 (2)	0.055 (2)	−0.038 (2)	−0.019 (2)	−0.0048 (18)
C3	0.044 (2)	0.035 (2)	0.052 (2)	−0.0168 (19)	−0.010 (2)	−0.0053 (18)
C4	0.041 (2)	0.041 (2)	0.041 (2)	−0.0099 (19)	−0.0013 (18)	−0.0035 (18)
C5	0.044 (2)	0.041 (2)	0.042 (2)	−0.0208 (19)	−0.0040 (19)	−0.0014 (18)
C6	0.044 (2)	0.036 (2)	0.051 (2)	−0.0144 (19)	−0.0028 (19)	−0.0020 (18)
C7	0.046 (2)	0.036 (2)	0.049 (2)	−0.0172 (19)	−0.0029 (19)	0.0027 (17)
C8	0.045 (2)	0.039 (2)	0.042 (2)	−0.0213 (19)	−0.0009 (19)	0.0024 (18)
C9	0.043 (2)	0.045 (2)	0.041 (2)	−0.0176 (19)	−0.0026 (18)	−0.0065 (19)
N1	0.084 (3)	0.064 (2)	0.051 (2)	−0.020 (2)	−0.028 (2)	0.0092 (18)
C10	0.045 (3)	0.037 (2)	0.054 (3)	−0.015 (2)	−0.018 (2)	0.0035 (18)
C11	0.056 (3)	0.045 (2)	0.044 (2)	−0.027 (2)	−0.003 (2)	−0.0013 (19)
C12	0.052 (3)	0.040 (2)	0.048 (2)	−0.021 (2)	−0.005 (2)	0.0022 (18)
C13	0.049 (2)	0.039 (2)	0.043 (2)	−0.0172 (19)	−0.0048 (19)	0.0006 (17)
C14	0.043 (2)	0.038 (2)	0.045 (2)	−0.0177 (19)	−0.007 (2)	0.0018 (18)

C15	0.048 (2)	0.043 (2)	0.045 (2)	−0.015 (2)	−0.008 (2)	−0.0077 (18)
C16	0.044 (2)	0.046 (2)	0.043 (2)	−0.014 (2)	−0.0020 (19)	0.0022 (19)
C17	0.057 (3)	0.043 (2)	0.040 (2)	−0.026 (2)	−0.003 (2)	0.0036 (18)
C18	0.043 (2)	0.041 (2)	0.054 (3)	−0.019 (2)	−0.0020 (19)	0.0025 (19)
C19	0.052 (3)	0.048 (2)	0.055 (3)	−0.021 (2)	−0.016 (2)	0.009 (2)
C20	0.045 (2)	0.047 (2)	0.046 (3)	−0.007 (2)	−0.002 (2)	0.001 (2)
C21	0.058 (3)	0.038 (2)	0.047 (3)	−0.014 (2)	−0.007 (2)	0.0028 (19)
C22	0.069 (3)	0.052 (3)	0.040 (2)	−0.038 (2)	−0.001 (2)	0.003 (2)
C52	0.056 (3)	0.040 (2)	0.045 (2)	−0.022 (2)	−0.013 (2)	0.0093 (18)
C23	0.053 (3)	0.043 (2)	0.053 (3)	−0.021 (2)	0.002 (2)	−0.008 (2)
C24	0.046 (2)	0.041 (2)	0.056 (3)	−0.015 (2)	−0.006 (2)	0.004 (2)
C25	0.043 (2)	0.037 (2)	0.057 (3)	−0.012 (2)	−0.0074 (19)	0.0043 (19)
C26	0.047 (2)	0.040 (2)	0.054 (3)	−0.0183 (19)	0.003 (2)	−0.0048 (19)
C27	0.048 (3)	0.046 (2)	0.055 (3)	−0.022 (2)	−0.008 (2)	−0.005 (2)
C28	0.053 (3)	0.041 (2)	0.071 (3)	−0.022 (2)	−0.002 (2)	−0.004 (2)
C29	0.059 (3)	0.040 (2)	0.052 (3)	−0.021 (2)	−0.004 (2)	−0.003 (2)
C30	0.057 (3)	0.039 (2)	0.054 (3)	−0.020 (2)	−0.005 (2)	−0.0039 (19)
C31	0.068 (3)	0.049 (3)	0.054 (3)	−0.016 (2)	−0.007 (2)	0.013 (2)
C32	0.053 (3)	0.039 (2)	0.077 (3)	−0.016 (2)	−0.002 (2)	0.002 (2)
C33	0.062 (3)	0.040 (2)	0.069 (3)	−0.019 (2)	0.011 (2)	0.001 (2)
C34	0.056 (3)	0.040 (2)	0.073 (3)	−0.007 (2)	0.003 (2)	−0.003 (2)
C35	0.073 (3)	0.041 (2)	0.064 (3)	−0.025 (2)	0.001 (2)	0.003 (2)
C36	0.059 (3)	0.069 (3)	0.083 (3)	−0.041 (3)	−0.023 (3)	0.019 (3)
C37	0.057 (3)	0.044 (2)	0.070 (3)	−0.021 (2)	0.012 (2)	−0.005 (2)
C38	0.073 (3)	0.052 (3)	0.057 (3)	−0.032 (3)	0.005 (3)	−0.012 (2)
C39	0.067 (3)	0.055 (3)	0.065 (3)	−0.025 (2)	−0.001 (2)	−0.021 (2)
C40	0.061 (3)	0.045 (3)	0.084 (3)	−0.016 (2)	−0.027 (3)	−0.003 (2)
C41	0.069 (3)	0.046 (3)	0.054 (3)	−0.020 (2)	−0.008 (2)	0.015 (2)
C42	0.064 (3)	0.058 (3)	0.069 (3)	−0.027 (3)	−0.018 (3)	0.021 (2)
C43	0.064 (3)	0.047 (3)	0.084 (3)	−0.026 (2)	−0.032 (3)	0.005 (2)
C44	0.039 (3)	0.062 (3)	0.091 (3)	−0.019 (2)	−0.007 (2)	−0.012 (3)
C45	0.083 (3)	0.060 (3)	0.046 (3)	−0.034 (3)	−0.004 (2)	0.004 (2)
C46	0.080 (3)	0.059 (3)	0.047 (3)	−0.023 (3)	0.000 (2)	−0.005 (2)
C47	0.072 (3)	0.043 (2)	0.051 (3)	−0.019 (2)	0.004 (2)	−0.012 (2)
C48	0.062 (3)	0.044 (3)	0.097 (4)	−0.007 (2)	−0.019 (3)	−0.018 (2)
C49	0.098 (4)	0.047 (3)	0.084 (4)	−0.034 (3)	−0.044 (3)	0.021 (2)
C50	0.080 (4)	0.084 (4)	0.087 (4)	−0.054 (3)	−0.030 (3)	0.033 (3)
C51	0.049 (3)	0.074 (3)	0.107 (4)	−0.016 (3)	−0.002 (3)	−0.028 (3)

Geometric parameters (Å, º)

Cl1—C18	1.732 (4)	C18—C37	1.381 (5)
Cl4—C26	1.732 (4)	C19—C43	1.368 (5)
Cl3—C30	1.737 (4)	C19—H19	0.9300
Cl2—C25	1.740 (4)	C20—C31	1.405 (5)
O2—C14	1.236 (4)	C21—C46	1.378 (5)
O1—C52	1.230 (4)	C21—H21	0.9300
O3—C9	1.237 (4)	C22—C38	1.401 (5)

O4—C15	1.232 (4)	C23—C32	1.366 (5)
N3—C11	1.352 (5)	C23—H23	0.9300
N3—H3A	0.8600	C24—C28	1.380 (5)
N3—H3B	0.8600	C24—H24	0.9300
N2—C16	1.342 (5)	C25—C34	1.384 (5)
N2—H2A	0.8600	C26—C33	1.383 (5)
N2—H2B	0.8600	C27—C40	1.379 (5)
C1—C24	1.381 (5)	C27—H27	0.9300
C1—C21	1.383 (5)	C28—C39	1.368 (5)
C1—C14	1.483 (5)	C28—H28	0.9300
C2—C7	1.399 (5)	C29—C45	1.378 (5)
C2—C16	1.426 (5)	C29—H29	0.9300
C2—C9	1.455 (5)	C30—C35	1.379 (5)
N4—C22	1.358 (5)	C31—C33	1.361 (6)
N4—H4A	0.8600	C31—H31	0.9300
N4—H4B	0.8600	C32—C42	1.371 (5)
C3—C36	1.368 (5)	C32—H32	0.9300
C3—C27	1.393 (5)	C33—H33	0.9300
C3—C15	1.478 (5)	C34—C47	1.359 (6)
C4—C13	1.398 (5)	C34—H34	0.9300
C4—C20	1.419 (5)	C35—C41	1.358 (5)
C4—C15	1.471 (5)	C35—H35	0.9300
C5—C29	1.377 (5)	C36—C50	1.358 (5)
C5—C23	1.387 (5)	C36—H36	0.9300
C5—C9	1.490 (5)	C37—C38	1.359 (6)
C6—C25	1.359 (5)	C37—H37	0.9300
C6—C8	1.406 (5)	C38—H38	0.9300
C6—H6	0.9300	C39—C46	1.372 (5)
C7—C30	1.367 (5)	C39—H39	0.9300
C7—H7	0.9300	C40—C49	1.373 (6)
C8—C11	1.415 (5)	C40—H40	0.9300
C8—C14	1.456 (5)	C41—H41	0.9300
N1—C20	1.343 (5)	C42—C45	1.379 (5)
N1—H1A	0.8600	C42—H42	0.9300
N1—H1B	0.8600	C43—C48	1.377 (6)
C10—C44	1.389 (5)	C43—H43	0.9300
C10—C19	1.397 (5)	C44—C51	1.358 (6)
C10—C52	1.491 (5)	C44—H44	0.9300
C11—C47	1.401 (5)	C45—H45	0.9300
C12—C18	1.368 (5)	C46—H46	0.9300
C12—C17	1.403 (5)	C47—H47	0.9300
C12—H12	0.9300	C48—C51	1.363 (6)
C13—C26	1.366 (5)	C48—H48	0.9300
C13—H13	0.9300	C49—C50	1.363 (6)
C16—C41	1.404 (5)	C49—H49	0.9300
C17—C22	1.408 (5)	C50—H50	0.9300
C17—C52	1.471 (5)	C51—H51	0.9300

C11—N3—H3A	120.0	C28—C24—C1	121.1 (4)
C11—N3—H3B	120.0	C28—C24—H24	119.4
H3A—N3—H3B	120.0	C1—C24—H24	119.4
C16—N2—H2A	120.0	C6—C25—C34	120.2 (4)
C16—N2—H2B	120.0	C6—C25—Cl2	119.7 (3)
H2A—N2—H2B	120.0	C34—C25—Cl2	120.0 (3)
C24—C1—C21	118.5 (3)	C13—C26—C33	119.1 (4)
C24—C1—C14	119.1 (3)	C13—C26—Cl4	120.8 (3)
C21—C1—C14	122.3 (3)	C33—C26—Cl4	120.0 (3)
C7—C2—C16	118.4 (3)	C40—C27—C3	120.0 (4)
C7—C2—C9	120.6 (3)	C40—C27—H27	120.0
C16—C2—C9	121.0 (3)	C3—C27—H27	120.0
C22—N4—H4A	120.0	C39—C28—C24	119.8 (4)
C22—N4—H4B	120.0	C39—C28—H28	120.1
H4A—N4—H4B	120.0	C24—C28—H28	120.1
C36—C3—C27	118.0 (4)	C5—C29—C45	119.5 (4)
C36—C3—C15	119.4 (3)	C5—C29—H29	120.3
C27—C3—C15	122.4 (4)	C45—C29—H29	120.3
C13—C4—C20	118.9 (4)	C7—C30—C35	120.0 (4)
C13—C4—C15	119.8 (3)	C7—C30—Cl3	120.0 (3)
C20—C4—C15	121.0 (4)	C35—C30—Cl3	120.0 (3)
C29—C5—C23	119.2 (3)	C33—C31—C20	122.5 (4)
C29—C5—C9	122.2 (3)	C33—C31—H31	118.7
C23—C5—C9	118.5 (3)	C20—C31—H31	118.7
C25—C6—C8	121.3 (4)	C23—C32—C42	120.1 (4)
C25—C6—H6	119.4	C23—C32—H32	119.9
C8—C6—H6	119.4	C42—C32—H32	119.9
C30—C7—C2	121.6 (4)	C31—C33—C26	120.2 (4)
C30—C7—H7	119.2	C31—C33—H33	119.9
C2—C7—H7	119.2	C26—C33—H33	119.9
C6—C8—C11	118.5 (3)	C47—C34—C25	120.1 (4)
C6—C8—C14	120.4 (3)	C47—C34—H34	120.0
C11—C8—C14	121.1 (3)	C25—C34—H34	120.0
O3—C9—C2	121.3 (3)	C41—C35—C30	120.1 (4)
O3—C9—C5	117.5 (3)	C41—C35—H35	119.9
C2—C9—C5	121.3 (3)	C30—C35—H35	119.9
C20—N1—H1A	120.0	C50—C36—C3	121.8 (4)
C20—N1—H1B	120.0	C50—C36—H36	119.1
H1A—N1—H1B	120.0	C3—C36—H36	119.1
C44—C10—C19	118.6 (4)	C38—C37—C18	120.2 (4)
C44—C10—C52	119.1 (4)	C38—C37—H37	119.9
C19—C10—C52	122.2 (4)	C18—C37—H37	119.9
N3—C11—C47	119.2 (4)	C37—C38—C22	121.8 (4)
N3—C11—C8	122.5 (3)	C37—C38—H38	119.1
C47—C11—C8	118.3 (4)	C22—C38—H38	119.1
C18—C12—C17	121.2 (4)	C28—C39—C46	119.7 (4)
C18—C12—H12	119.4	C28—C39—H39	120.1
C17—C12—H12	119.4	C46—C39—H39	120.1

C26—C13—C4	122.2 (4)	C49—C40—C27	120.3 (4)
C26—C13—H13	118.9	C49—C40—H40	119.8
C4—C13—H13	118.9	C27—C40—H40	119.8
O2—C14—C8	121.4 (3)	C35—C41—C16	122.1 (4)
O2—C14—C1	117.0 (3)	C35—C41—H41	119.0
C8—C14—C1	121.6 (3)	C16—C41—H41	119.0
O4—C15—C4	121.2 (4)	C32—C42—C45	119.2 (4)
O4—C15—C3	117.1 (3)	C32—C42—H42	120.4
C4—C15—C3	121.7 (3)	C45—C42—H42	120.4
N2—C16—C41	119.4 (4)	C19—C43—C48	120.2 (4)
N2—C16—C2	122.9 (4)	C19—C43—H43	119.9
C41—C16—C2	117.7 (4)	C48—C43—H43	119.9
C12—C17—C22	118.9 (4)	C51—C44—C10	120.5 (4)
C12—C17—C52	119.6 (3)	C51—C44—H44	119.8
C22—C17—C52	121.3 (4)	C10—C44—H44	119.8
C12—C18—C37	119.8 (4)	C29—C45—C42	121.1 (4)
C12—C18—Cl1	120.1 (3)	C29—C45—H45	119.5
C37—C18—Cl1	120.0 (3)	C42—C45—H45	119.5
C43—C19—C10	120.0 (4)	C39—C46—C21	120.7 (4)
C43—C19—H19	120.0	C39—C46—H46	119.6
C10—C19—H19	120.0	C21—C46—H46	119.6
N1—C20—C31	120.3 (4)	C34—C47—C11	121.7 (4)
N1—C20—C4	122.7 (4)	C34—C47—H47	119.2
C31—C20—C4	117.0 (4)	C11—C47—H47	119.2
C46—C21—C1	120.1 (4)	C51—C48—C43	120.0 (4)
C46—C21—H21	120.0	C51—C48—H48	120.0
C1—C21—H21	120.0	C43—C48—H48	120.0
N4—C22—C38	119.1 (4)	C50—C49—C40	119.4 (4)
N4—C22—C17	122.8 (4)	C50—C49—H49	120.3
C38—C22—C17	118.0 (4)	C40—C49—H49	120.3
O1—C52—C17	121.2 (4)	C36—C50—C49	120.4 (4)
O1—C52—C10	117.4 (3)	C36—C50—H50	119.8
C17—C52—C10	121.4 (3)	C49—C50—H50	119.8
C32—C23—C5	120.9 (4)	C44—C51—C48	120.8 (4)
C32—C23—H23	119.6	C44—C51—H51	119.6
C5—C23—H23	119.6	C48—C51—H51	119.6

C16—C2—C7—C30	1.0 (5)	C44—C10—C52—O1	−47.5 (5)
C9—C2—C7—C30	−178.4 (3)	C19—C10—C52—O1	128.3 (4)
C25—C6—C8—C11	1.8 (5)	C44—C10—C52—C17	132.0 (4)
C25—C6—C8—C14	−178.1 (3)	C19—C10—C52—C17	−52.2 (5)
C7—C2—C9—O3	−175.1 (3)	C29—C5—C23—C32	−0.8 (6)
C16—C2—C9—O3	5.5 (6)	C9—C5—C23—C32	−177.6 (4)
C7—C2—C9—C5	4.3 (5)	C21—C1—C24—C28	−0.4 (5)
C16—C2—C9—C5	−175.1 (3)	C14—C1—C24—C28	−177.5 (3)
C29—C5—C9—O3	−126.8 (4)	C8—C6—C25—C34	−0.8 (6)
C23—C5—C9—O3	49.9 (5)	C8—C6—C25—Cl2	178.8 (3)
C29—C5—C9—C2	53.8 (5)	C4—C13—C26—C33	−0.1 (6)

C23—C5—C9—C2	−129.5 (4)	C4—C13—C26—Cl4	177.4 (3)
C6—C8—C11—N3	177.7 (3)	C36—C3—C27—C40	−0.5 (6)
C14—C8—C11—N3	−2.3 (6)	C15—C3—C27—C40	−175.4 (3)
C6—C8—C11—C47	−1.2 (5)	C1—C24—C28—C39	0.3 (6)
C14—C8—C11—C47	178.7 (3)	C23—C5—C29—C45	0.4 (6)
C20—C4—C13—C26	−2.4 (6)	C9—C5—C29—C45	177.1 (4)
C15—C4—C13—C26	−176.6 (3)	C2—C7—C30—C35	0.5 (6)
C6—C8—C14—O2	−173.9 (3)	C2—C7—C30—Cl3	179.6 (3)
C11—C8—C14—O2	6.2 (5)	N1—C20—C31—C33	178.6 (4)
C6—C8—C14—C1	6.3 (5)	C4—C20—C31—C33	−3.2 (6)
C11—C8—C14—C1	−173.6 (3)	C5—C23—C32—C42	0.2 (6)
C24—C1—C14—O2	49.8 (5)	C20—C31—C33—C26	0.8 (6)
C21—C1—C14—O2	−127.2 (4)	C13—C26—C33—C31	0.9 (6)
C24—C1—C14—C8	−130.4 (4)	Cl4—C26—C33—C31	−176.6 (3)
C21—C1—C14—C8	52.6 (5)	C6—C25—C34—C47	−0.8 (6)
C13—C4—C15—O4	166.5 (3)	Cl2—C25—C34—C47	179.6 (3)
C20—C4—C15—O4	−7.7 (6)	C7—C30—C35—C41	−1.8 (6)
C13—C4—C15—C3	−13.3 (5)	Cl3—C30—C35—C41	179.1 (3)
C20—C4—C15—C3	172.5 (3)	C27—C3—C36—C50	1.6 (6)
C36—C3—C15—O4	−47.7 (5)	C15—C3—C36—C50	176.6 (4)
C27—C3—C15—O4	127.2 (4)	C12—C18—C37—C38	0.3 (6)
C36—C3—C15—C4	132.2 (4)	Cl1—C18—C37—C38	−177.8 (3)
C27—C3—C15—C4	−53.0 (5)	C18—C37—C38—C22	1.1 (6)
C7—C2—C16—N2	176.4 (3)	N4—C22—C38—C37	179.4 (4)
C9—C2—C16—N2	−4.2 (6)	C17—C22—C38—C37	−2.6 (6)
C7—C2—C16—C41	−1.3 (5)	C24—C28—C39—C46	0.5 (6)
C9—C2—C16—C41	178.1 (3)	C3—C27—C40—C49	−0.2 (6)
C18—C12—C17—C22	−1.5 (5)	C30—C35—C41—C16	1.6 (6)
C18—C12—C17—C52	−176.4 (3)	N2—C16—C41—C35	−177.7 (4)
C17—C12—C18—C37	−0.1 (6)	C2—C16—C41—C35	0.0 (6)
C17—C12—C18—Cl1	178.0 (3)	C23—C32—C42—C45	0.8 (6)
C44—C10—C19—C43	0.6 (6)	C10—C19—C43—C48	−1.0 (6)
C52—C10—C19—C43	−175.2 (3)	C19—C10—C44—C51	0.1 (6)
C13—C4—C20—N1	−178.0 (3)	C52—C10—C44—C51	176.1 (4)
C15—C4—C20—N1	−3.8 (6)	C5—C29—C45—C42	0.6 (6)
C13—C4—C20—C31	3.9 (5)	C32—C42—C45—C29	−1.2 (7)
C15—C4—C20—C31	178.1 (3)	C28—C39—C46—C21	−1.3 (7)
C24—C1—C21—C46	−0.4 (6)	C1—C21—C46—C39	1.2 (6)
C14—C1—C21—C46	176.7 (4)	C25—C34—C47—C11	1.4 (6)
C12—C17—C22—N4	−179.4 (3)	N3—C11—C47—C34	−179.3 (4)
C52—C17—C22—N4	−4.5 (6)	C8—C11—C47—C34	−0.4 (6)
C12—C17—C22—C38	2.7 (5)	C19—C43—C48—C51	0.7 (7)
C52—C17—C22—C38	177.5 (3)	C27—C40—C49—C50	−0.2 (7)
C12—C17—C52—O1	165.7 (3)	C3—C36—C50—C49	−1.9 (7)
C22—C17—C52—O1	−9.1 (6)	C40—C49—C50—C36	1.2 (7)
C12—C17—C52—C10	−13.9 (5)	C10—C44—C51—C48	−0.4 (7)
C22—C17—C52—C10	171.3 (3)	C43—C48—C51—C44	0.0 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.86	2.21	2.971 (5)	147
N1—H1*B*···O4	0.86	2.01	2.642 (5)	130
N2—H2*A*···O1^{ii}	0.86	2.16	2.962 (4)	154
N2—H2*B*···O3	0.86	1.99	2.629 (4)	130
N3—H3*A*···O3	0.86	2.19	2.960 (4)	149
N3—H3*B*···O2	0.86	1.98	2.623 (4)	130
N4—H4*A*···O4	0.86	2.19	2.986 (5)	153
N4—H4*B*···O1	0.86	2.03	2.654 (5)	129

Symmetry codes: (i) *x*−1, *y*+1, *z*; (ii) *x*, *y*+1, *z*.