N-(4-Chloro-3-methylphenyl)succinamic acid

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The title compound, $C_{11}H_{12}ClNO_3$, crystallizes with two independent molecules in the asymmetric unit in which the dihedral angles between the benzene ring and the amide group are 55.0 (2) and 28.2 (3)°. The two independent molecules are linked by an N—H···O hydrogen bond. In the crystal, molecules form inversion dimers via pairs of O—H···O hydrogen bonds. These dimers are linked into sheets parallel to (11$\bar{3}$) via N—H···O hydrogen bonds.

Related literature

For our studies on the effects of substituents on the structures and other aspects of N-(aryl)-amides, see: Gowda et al. (2000); Chaithanya et al. (2012), of N-chloroarylamides, see: Gowda & Rao (1989); Jyothi & Gowda (2004) and of N-bromoaryl-sulfonamides, see: Gowda & Mahadevappa (1983); Usha & Gowda (2006).
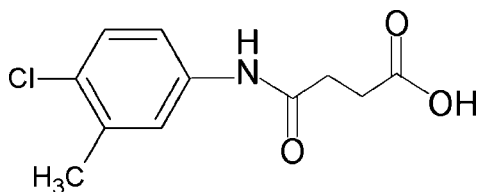
Experimental

Crystal data

$C_{11}H_{12}ClNO_3$	$a = 6.6253$ (8) Å
$M_r = 241.67$	$b = 7.9634$ (9) Å
Triclinic, $P\bar{1}$	$c = 21.545$ (3) Å
$\alpha = 88.57$ (1)°	Mo $K\alpha$ radiation
$\beta = 81.99$ (1)°	$\mu = 0.33$ mm^{-1}
$\gamma = 84.25$ (1)°	$T = 293$ K
$V = 1119.9$ (2) Å^3	$0.48 \times 0.16 \times 0.03$ mm
$Z = 4$	

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	Diffraction, 2009)
	$T_{min} = 0.857$, $T_{max} = 0.990$
Absorption correction: multi-scan (CrysAlis RED; Oxford	6984 measured reflections
	3864 independent reflections
	2640 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.090$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.155$	
$S = 1.33$	
3864 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
303 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
4 restraints	

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3O···O2[i]	0.82 (2)	1.85 (2)	2.668 (5)	176 (7)
N1—H1N···O4[ii]	0.86 (2)	2.09 (2)	2.934 (5)	169 (5)
O6—H6O···O5[iii]	0.82 (2)	1.86 (2)	2.685 (6)	177 (8)
N2—H2N···O1	0.85 (2)	2.12 (2)	2.944 (6)	163 (5)

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

Data collection: CrysAlis CCD (Oxford Diffraction, 2009); cell refinement: CrysAlis CCD; data reduction: CrysAlis RED (Oxford Diffraction, 2009); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: SHELXL97.

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supporting information

N-(4-Chloro-3-methylphenyl)succinamic acid

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S1. Comment

As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Gowda *et al.*, 2000; Chaithanya *et al.*, 2012), *N*-chloroarylsulfonamides (Gowda & Rao, 1989; Jyothi & Gowda, 2004) and *N*-bromo-aryl- sulfonamides (Gowda & Mahadevappa, 1983; Usha & Gowda, 2006), in the present work, the crystal structure of *N*-(4-Chloro-3-methylphenyl)succinamic acid has been determined (Fig. 1). The asymmetric unit of the structure contains two independent molecules. The conformations of the N—H bonds in the amide segments are *anti* to the *meta*–methyl groups in the benzene rings of both the molecules, similar to the *anti* conformation observed between the N—H bond and *meta*–chloro atoms in *N*-(3-Chloro-4-methylphenyl)succinamic acid (I) (Chaithanya *et al.*, 2012).

Further, the conformations of the amide oxygen and the carboxyl oxygen of the acid segments are *anti* to each other and both are *anti* to the H atoms on the adjacent –CH$_2$ groups.

The C=O and O—H bonds of the acid groups are in *syn* position to each other, similar to that observed in (I).

The dihedral angles between the phenyl ring and the amide group in the two independent molecules are 55.03 (22)° and 28.19 (31)°, compared to the values of 40.58 (22)° and 44.93 (27)° in (I).

In the crystal, the molecules form centrosymmetric dimers via O—H···O hydrogen bonds. These dimers are linked into sheets parallel to (1 1 -3) via N—H···O hydrogen bonds. (Table 1, Fig. 2).

S2. Experimental

The solution of succinic anhydride (0.01 mole) in toluene (25 ml) was treated dropwise with the solution of 4-chloro-3-methylaniline (0.01 mole) also in toluene (20 ml) with constant stirring. The resulting mixture was stirred for about one hour and set aside for an additional hour at room temperature for completion of the reaction. The mixture was then treated with dilute hydrochloric acid to remove the unreacted 4-chloro-3-methyl- aniline. The resultant title compound was filtered under suction and washed thoroughly with water to remove the unreacted succinic anhydride and succinic acid. It was recrystallized to constant melting point from ethanol. The purity of the compound was checked and characterized by its infrared and NMR spectra.

Plate like colorless single crystals used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation of the solvent at room temperature.

S3. Refinement

The H atoms of the NH groups and the OH groups were located in a difference map and later restrained to the distance N—H = 0.86 (2) Å and O—H = 0.82 (2) Å, respectively. The other H atoms were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93 Å and methylene C—H = 0.97 Å. All H atoms were refined with isotropic displacement parameters set at 1.2 U_{eq}(C-aromatic, N, O) and 1.5 U_{eq}(C-methyl).
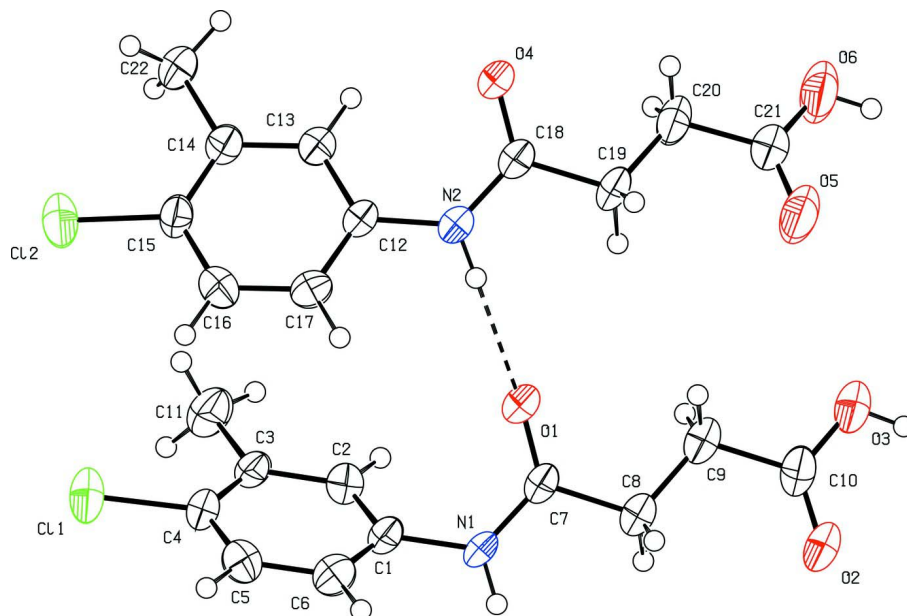
Figure 1

Molecular structure of the title compound, showing the atom labelling scheme and with displacement ellipsoids drawn at the 50% probability level.
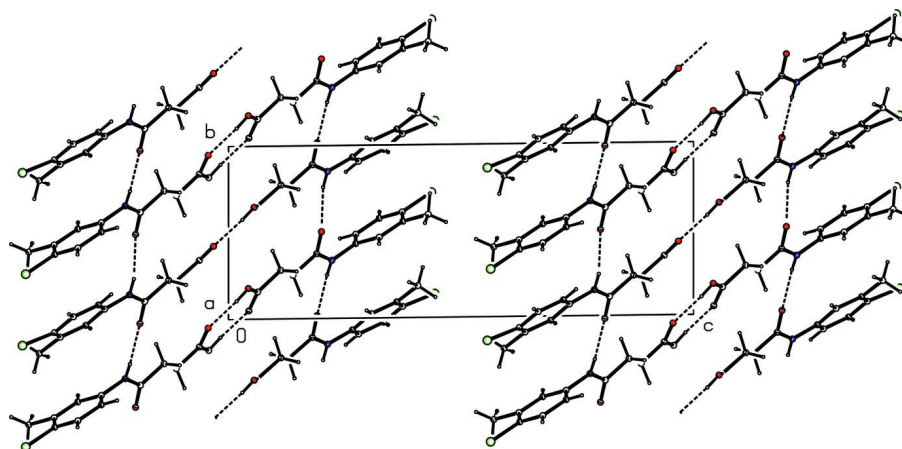
Figure 2

Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

N-(4-Chloro-3-methylphenyl)succinamic acid

Crystal data

$C_{11}H_{12}ClNO_3$	$\gamma = 84.25\ (1)°$
$M_r = 241.67$	$V = 1119.9\ (2)$ Å^3
Triclinic, $P\bar{1}$	$Z = 4$
Hall symbol: -P 1	$F(000) = 504$
$a = 6.6253\ (8)$ Å	$D_x = 1.433$ Mg m^{-3}
$b = 7.9634\ (9)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$c = 21.545\ (3)$ Å	Cell parameters from 1701 reflections
$\alpha = 88.57\ (1)°$	$\theta = 2.6–27.4°$
$\beta = 81.99\ (1)°$	$\mu = 0.33$ mm^{-1}

$T = 293$ K	$0.48 \times 0.16 \times 0.03$ mm
Plate, colourless	

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	6984 measured reflections
	3864 independent reflections
Radiation source: fine-focus sealed tube	2640 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.027$
Rotation method data acquisition using ω and phi scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.6°$
	$h = -7 \rightarrow 7$
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	$k = -9 \rightarrow 8$
	$l = -23 \rightarrow 25$
$T_{min} = 0.857$, $T_{max} = 0.990$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.090$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.155$	
$S = 1.33$	H atoms treated by a mixture of independent and constrained refinement
3864 reflections	
303 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0064P)^2 + 2.5267P]$ where $P = (F_o^2 + 2F_c^2)/3$
4 restraints	
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} < 0.001$
	$\Delta\rho_{max} = 0.35$ e Å^{-3}
	$\Delta\rho_{min} = -0.29$ e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlis RED (Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	1.1028 (3)	0.7244 (2)	0.43623 (7)	0.0669 (5)
O1	0.6519 (6)	0.5020 (5)	0.19988 (18)	0.0599 (12)
O2	0.6714 (7)	0.0487 (6)	0.0438 (2)	0.0788 (15)
O3	0.3608 (7)	0.1779 (6)	0.0426 (2)	0.0824 (15)
H3O	0.352 (11)	0.105 (6)	0.017 (3)	0.099*
N1	0.9291 (7)	0.3357 (5)	0.2222 (2)	0.0454 (12)
H1N	1.017 (6)	0.251 (5)	0.212 (2)	0.055*
C1	0.9694 (8)	0.4313 (6)	0.2738 (2)	0.0394 (13)
C2	0.8186 (8)	0.4669 (6)	0.3242 (2)	0.0404 (13)
H2	0.6905	0.4290	0.3238	0.049*
C3	0.8529 (8)	0.5574 (6)	0.3753 (2)	0.0380 (12)

C4	1.0473 (8)	0.6116 (6)	0.3735 (2)	0.0402 (13)
C5	1.1991 (8)	0.5761 (7)	0.3242 (2)	0.0473 (14)
H5	1.3276	0.6130	0.3247	0.057*
C6	1.1619 (8)	0.4854 (7)	0.2735 (2)	0.0464 (14)
H6	1.2643	0.4614	0.2399	0.056*
C7	0.7719 (8)	0.3752 (7)	0.1897 (2)	0.0409 (13)
C8	0.7563 (9)	0.2520 (7)	0.1387 (3)	0.0535 (16)
H8A	0.8685	0.2620	0.1051	0.064*
H8B	0.7703	0.1380	0.1556	0.064*
C9	0.5581 (9)	0.2808 (7)	0.1124 (3)	0.0561 (16)
H9A	0.5458	0.3940	0.0948	0.067*
H9B	0.4461	0.2737	0.1462	0.067*
C10	0.5379 (10)	0.1570 (8)	0.0627 (3)	0.0557 (16)
C11	0.6861 (9)	0.5963 (7)	0.4295 (2)	0.0574 (16)
H11A	0.5651	0.5470	0.4223	0.069*
H11B	0.6561	0.7163	0.4333	0.069*
H11C	0.7308	0.5502	0.4675	0.069*
Cl2	0.6698 (2)	1.1376 (2)	0.44331 (7)	0.0594 (4)
O4	0.1817 (6)	1.0173 (5)	0.19059 (18)	0.0632 (12)
O5	0.1879 (7)	0.5752 (6)	0.0324 (2)	0.0846 (16)
O6	−0.1414 (8)	0.6470 (7)	0.0550 (2)	0.0920 (17)
H6O	−0.157 (11)	0.576 (7)	0.029 (3)	0.110*
N2	0.4570 (7)	0.8506 (5)	0.2130 (2)	0.0428 (11)
H2N	0.531 (7)	0.759 (4)	0.204 (2)	0.051*
C12	0.5057 (8)	0.9318 (6)	0.2666 (2)	0.0354 (12)
C13	0.3572 (8)	1.0248 (6)	0.3070 (2)	0.0414 (13)
H13	0.2243	1.0431	0.2973	0.050*
C14	0.4031 (8)	1.0913 (6)	0.3619 (2)	0.0372 (12)
C15	0.6025 (8)	1.0644 (6)	0.3742 (2)	0.0397 (13)
C16	0.7513 (8)	0.9745 (7)	0.3338 (2)	0.0477 (14)
H16	0.8849	0.9587	0.3430	0.057*
C17	0.7044 (8)	0.9074 (7)	0.2796 (2)	0.0446 (14)
H17	0.8054	0.8468	0.2524	0.054*
C18	0.3015 (8)	0.8908 (7)	0.1804 (2)	0.0433 (13)
C19	0.2833 (9)	0.7696 (7)	0.1289 (2)	0.0512 (15)
H19A	0.3750	0.7969	0.0917	0.061*
H19B	0.3266	0.6558	0.1420	0.061*
C20	0.0720 (9)	0.7745 (8)	0.1128 (3)	0.0642 (18)
H20A	0.0298	0.8884	0.0995	0.077*
H20B	−0.0193	0.7489	0.1503	0.077*
C21	0.0466 (10)	0.6560 (8)	0.0627 (3)	0.0535 (15)
C22	0.2383 (8)	1.1841 (7)	0.4074 (2)	0.0525 (15)
H22A	0.1150	1.2044	0.3885	0.063*
H22B	0.2824	1.2899	0.4177	0.063*
H22C	0.2127	1.1171	0.4448	0.063*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0740 (11)	0.0742 (11)	0.0584 (9)	−0.0042 (9)	−0.0273 (8)	−0.0302 (8)
O1	0.072 (3)	0.048 (2)	0.061 (3)	0.024 (2)	−0.030 (2)	−0.029 (2)
O2	0.085 (3)	0.071 (3)	0.084 (3)	0.020 (3)	−0.038 (3)	−0.052 (3)
O3	0.082 (3)	0.083 (4)	0.087 (3)	0.016 (3)	−0.041 (3)	−0.054 (3)
N1	0.050 (3)	0.042 (3)	0.043 (3)	0.016 (2)	−0.014 (2)	−0.025 (2)
C1	0.048 (3)	0.035 (3)	0.034 (3)	0.007 (3)	−0.008 (3)	−0.012 (2)
C2	0.043 (3)	0.039 (3)	0.041 (3)	−0.002 (2)	−0.011 (3)	−0.007 (2)
C3	0.044 (3)	0.038 (3)	0.030 (3)	0.008 (2)	−0.005 (2)	−0.010 (2)
C4	0.047 (3)	0.039 (3)	0.036 (3)	0.001 (3)	−0.011 (3)	−0.014 (2)
C5	0.040 (3)	0.051 (4)	0.053 (3)	−0.003 (3)	−0.016 (3)	−0.009 (3)
C6	0.044 (3)	0.051 (4)	0.043 (3)	0.004 (3)	−0.003 (3)	−0.010 (3)
C7	0.050 (3)	0.037 (3)	0.036 (3)	0.004 (3)	−0.009 (3)	−0.014 (2)
C8	0.065 (4)	0.046 (4)	0.051 (3)	0.011 (3)	−0.021 (3)	−0.024 (3)
C9	0.065 (4)	0.049 (4)	0.057 (4)	0.004 (3)	−0.019 (3)	−0.026 (3)
C10	0.072 (4)	0.051 (4)	0.048 (3)	−0.003 (3)	−0.020 (3)	−0.018 (3)
C11	0.064 (4)	0.060 (4)	0.046 (3)	0.002 (3)	0.001 (3)	−0.015 (3)
Cl2	0.0647 (10)	0.0710 (11)	0.0479 (8)	−0.0085 (8)	−0.0223 (7)	−0.0176 (7)
O4	0.077 (3)	0.053 (3)	0.062 (3)	0.029 (2)	−0.036 (2)	−0.033 (2)
O5	0.072 (3)	0.096 (4)	0.089 (3)	0.003 (3)	−0.020 (3)	−0.062 (3)
O6	0.071 (3)	0.114 (4)	0.097 (4)	0.000 (3)	−0.027 (3)	−0.070 (3)
N2	0.046 (3)	0.040 (3)	0.042 (2)	0.010 (2)	−0.012 (2)	−0.018 (2)
C12	0.040 (3)	0.030 (3)	0.036 (3)	0.001 (2)	−0.007 (2)	−0.009 (2)
C13	0.039 (3)	0.042 (3)	0.044 (3)	0.004 (2)	−0.010 (3)	−0.013 (3)
C14	0.042 (3)	0.034 (3)	0.036 (3)	−0.002 (2)	−0.007 (2)	−0.006 (2)
C15	0.051 (3)	0.039 (3)	0.031 (3)	−0.002 (3)	−0.011 (2)	−0.009 (2)
C16	0.040 (3)	0.056 (4)	0.050 (3)	0.001 (3)	−0.018 (3)	−0.008 (3)
C17	0.037 (3)	0.052 (4)	0.042 (3)	0.009 (3)	−0.002 (3)	−0.014 (3)
C18	0.050 (3)	0.040 (3)	0.040 (3)	0.007 (3)	−0.010 (3)	−0.016 (3)
C19	0.068 (4)	0.042 (3)	0.044 (3)	0.009 (3)	−0.016 (3)	−0.023 (3)
C20	0.060 (4)	0.071 (4)	0.065 (4)	0.006 (3)	−0.019 (3)	−0.043 (3)
C21	0.058 (4)	0.051 (4)	0.053 (4)	0.002 (3)	−0.017 (3)	−0.019 (3)
C22	0.055 (4)	0.058 (4)	0.042 (3)	0.011 (3)	−0.009 (3)	−0.022 (3)

Geometric parameters (Å, º)

Cl1—C4	1.743 (5)	Cl2—C15	1.742 (5)
O1—C7	1.226 (6)	O4—C18	1.223 (6)
O2—C10	1.207 (7)	O5—C21	1.203 (6)
O3—C10	1.300 (7)	O6—C21	1.288 (7)
O3—H3O	0.82 (2)	O6—H6O	0.82 (2)
N1—C7	1.341 (6)	N2—C18	1.336 (6)
N1—C1	1.433 (6)	N2—C12	1.427 (6)
N1—H1N	0.855 (19)	N2—H2N	0.849 (19)
C1—C2	1.383 (7)	C12—C17	1.379 (7)
C1—C6	1.385 (7)	C12—C13	1.387 (6)

C2—C3	1.388 (6)	C13—C14	1.392 (6)
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.393 (7)	C14—C15	1.378 (7)
C3—C11	1.508 (7)	C14—C22	1.507 (7)
C4—C5	1.371 (7)	C15—C16	1.376 (7)
C5—C6	1.388 (7)	C16—C17	1.382 (7)
C5—H5	0.9300	C16—H16	0.9300
C6—H6	0.9300	C17—H17	0.9300
C7—C8	1.512 (6)	C18—C19	1.514 (6)
C8—C9	1.496 (7)	C19—C20	1.485 (7)
C8—H8A	0.9700	C19—H19A	0.9700
C8—H8B	0.9700	C19—H19B	0.9700
C9—C10	1.502 (7)	C20—C21	1.488 (7)
C9—H9A	0.9700	C20—H20A	0.9700
C9—H9B	0.9700	C20—H20B	0.9700
C11—H11A	0.9600	C22—H22A	0.9600
C11—H11B	0.9600	C22—H22B	0.9600
C11—H11C	0.9600	C22—H22C	0.9600
C10—O3—H3O	111 (5)	C21—O6—H6O	114 (5)
C7—N1—C1	124.2 (4)	C18—N2—C12	128.7 (4)
C7—N1—H1N	121 (4)	C18—N2—H2N	117 (4)
C1—N1—H1N	115 (4)	C12—N2—H2N	114 (4)
C2—C1—C6	120.1 (5)	C17—C12—C13	120.0 (4)
C2—C1—N1	120.4 (5)	C17—C12—N2	118.1 (4)
C6—C1—N1	119.5 (5)	C13—C12—N2	121.8 (4)
C1—C2—C3	121.9 (5)	C12—C13—C14	121.3 (5)
C1—C2—H2	119.0	C12—C13—H13	119.3
C3—C2—H2	119.0	C14—C13—H13	119.3
C2—C3—C4	116.8 (5)	C15—C14—C13	117.7 (5)
C2—C3—C11	121.1 (5)	C15—C14—C22	121.3 (4)
C4—C3—C11	122.1 (4)	C13—C14—C22	120.9 (5)
C5—C4—C3	122.1 (4)	C16—C15—C14	121.3 (4)
C5—C4—Cl1	118.4 (4)	C16—C15—Cl2	118.3 (4)
C3—C4—Cl1	119.5 (4)	C14—C15—Cl2	120.3 (4)
C4—C5—C6	120.3 (5)	C15—C16—C17	120.8 (5)
C4—C5—H5	119.8	C15—C16—H16	119.6
C6—C5—H5	119.8	C17—C16—H16	119.6
C1—C6—C5	118.8 (5)	C12—C17—C16	119.0 (5)
C1—C6—H6	120.6	C12—C17—H17	120.5
C5—C6—H6	120.6	C16—C17—H17	120.5
O1—C7—N1	122.9 (4)	O4—C18—N2	122.9 (5)
O1—C7—C8	122.5 (5)	O4—C18—C19	121.6 (5)
N1—C7—C8	114.6 (4)	N2—C18—C19	115.5 (5)
C9—C8—C7	112.8 (4)	C20—C19—C18	113.1 (4)
C9—C8—H8A	109.0	C20—C19—H19A	109.0
C7—C8—H8A	109.0	C18—C19—H19A	109.0
C9—C8—H8B	109.0	C20—C19—H19B	109.0

C7—C8—H8B	109.0	C18—C19—H19B	109.0
H8A—C8—H8B	107.8	H19A—C19—H19B	107.8
C8—C9—C10	113.5 (5)	C19—C20—C21	114.9 (5)
C8—C9—H9A	108.9	C19—C20—H20A	108.6
C10—C9—H9A	108.9	C21—C20—H20A	108.6
C8—C9—H9B	108.9	C19—C20—H20B	108.6
C10—C9—H9B	108.9	C21—C20—H20B	108.6
H9A—C9—H9B	107.7	H20A—C20—H20B	107.5
O2—C10—O3	123.6 (5)	O5—C21—O6	122.9 (5)
O2—C10—C9	123.5 (5)	O5—C21—C20	123.4 (6)
O3—C10—C9	112.9 (5)	O6—C21—C20	113.7 (5)
C3—C11—H11A	109.5	C14—C22—H22A	109.5
C3—C11—H11B	109.5	C14—C22—H22B	109.5
H11A—C11—H11B	109.5	H22A—C22—H22B	109.5
C3—C11—H11C	109.5	C14—C22—H22C	109.5
H11A—C11—H11C	109.5	H22A—C22—H22C	109.5
H11B—C11—H11C	109.5	H22B—C22—H22C	109.5
C7—N1—C1—C2	54.0 (8)	C18—N2—C12—C17	−158.1 (6)
C7—N1—C1—C6	−127.2 (6)	C18—N2—C12—C13	25.7 (9)
C6—C1—C2—C3	0.4 (8)	C17—C12—C13—C14	−1.7 (8)
N1—C1—C2—C3	179.2 (5)	N2—C12—C13—C14	174.4 (5)
C1—C2—C3—C4	0.2 (7)	C12—C13—C14—C15	1.2 (8)
C1—C2—C3—C11	179.5 (5)	C12—C13—C14—C22	−176.6 (5)
C2—C3—C4—C5	−0.8 (8)	C13—C14—C15—C16	−0.2 (8)
C11—C3—C4—C5	179.9 (5)	C22—C14—C15—C16	177.7 (5)
C2—C3—C4—Cl1	−179.4 (4)	C13—C14—C15—Cl2	−177.8 (4)
C11—C3—C4—Cl1	1.3 (7)	C22—C14—C15—Cl2	0.0 (7)
C3—C4—C5—C6	0.8 (8)	C14—C15—C16—C17	−0.4 (8)
Cl1—C4—C5—C6	179.5 (4)	Cl2—C15—C16—C17	177.3 (4)
C2—C1—C6—C5	−0.4 (8)	C13—C12—C17—C16	1.0 (8)
N1—C1—C6—C5	−179.2 (5)	N2—C12—C17—C16	−175.2 (5)
C4—C5—C6—C1	−0.2 (8)	C15—C16—C17—C12	0.0 (9)
C1—N1—C7—O1	3.0 (9)	C12—N2—C18—O4	5.3 (10)
C1—N1—C7—C8	−177.9 (5)	C12—N2—C18—C19	−175.2 (5)
O1—C7—C8—C9	−12.4 (8)	O4—C18—C19—C20	−25.0 (8)
N1—C7—C8—C9	168.6 (5)	N2—C18—C19—C20	155.5 (5)
C7—C8—C9—C10	−178.7 (5)	C18—C19—C20—C21	−179.4 (5)
C8—C9—C10—O2	−2.8 (10)	C19—C20—C21—O5	−7.9 (10)
C8—C9—C10—O3	176.6 (6)	C19—C20—C21—O6	171.8 (6)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3O···O2ⁱ	0.82 (2)	1.85 (2)	2.668 (5)	176 (7)
N1—H1N···O4ⁱⁱ	0.86 (2)	2.09 (2)	2.934 (5)	169 (5)

O6—H6*O*···O5ⁱ	0.82 (2)	1.86 (2)	2.685 (6)	177 (8)
N2—H2*N*···O1	0.85 (2)	2.12 (2)	2.944 (6)	163 (5)

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