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9-Oxo-4,5-diazafluoren-4-ium tetrachloridoaurate(III)–4,5-diazafluoren-9-one (1/1)

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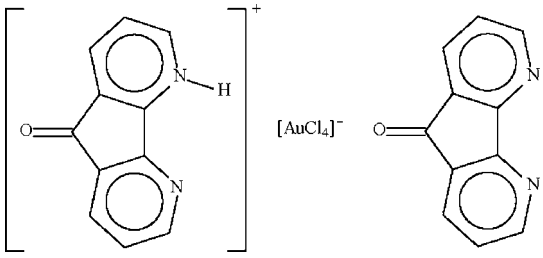
Key indicators: single-crystal X-ray study; T = 118 K; mean σ(C–C) = 0.005 Å; R factor = 0.025; wR factor = 0.067; data-to-parameter ratio = 16.7.

The Au^{III} atom in the title compound, $(C_{11}H_7N_2O)[AuCl_4]\cdot C_{11}H_6N_2O$, is in a nearly square-planar environment defined by four Cl atoms. The protonated 9-oxo-4,5-diazafluoren-4-ium cation forms an N—H···N hydrogen bond with the neutral 4,5-diazafluoren-9-one molecule.

Related literature

For other 9-oxo-4,5-diazafluoren-4-ium tetrachloridometallates, see: Kulkarni *et al.* (2003); Menon *et al.* (1994); Ravikumar *et al.* (1995); Ravikumar & Lakshmi (1994); Zhang *et al.* (2003). For the synthesis of 4,5-diazafluoren-9-one, see: Henderson *et al.* (1984).



Experimental

Crystal data

$(C_{11}H_7N_2O)[AuCl_4]\cdot C_{11}H_6N_2O$
M_r = 704.13
Triclinic, $P\overline{1}$
a = 7.1035 (1) Å
b = 12.6513 (2) Å
c = 13.2366 (2) Å
α = 73.285 (1)°
β = 78.410 (1)°
γ = 88.375 (1)°
V = 1115.51 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 7.10 mm^{-1}
T = 118 K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.331, T_{max} = 0.537
(expected range = 0.303–0.491)
9343 measured reflections
5044 independent reflections
4829 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.067
S = 1.04
5044 reflections
302 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 3.05 e Å^{-3}
$\Delta\rho_{min}$ = −1.27 e Å^{-3}

Table 1
Selected bond lengths (Å).

Au1—Cl1	2.2720 (8)	Au1—Cl3	2.2864 (8)
Au1—Cl2	2.2882 (8)	Au1—Cl4	2.2872 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N3	0.89 (1)	1.88 (1)	2.762 (4)	168 (4)

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2184).

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supplementary materials

Acta Cryst. (2009). E**65**, m344 [**doi:10.1107/S1600536809006734**]

9-Oxo-4,5-diazafluoren-4-ium tetrachloridoaurate(III)-4,5-diazafluoren-9-one (1/1)

N. Safari, V. Amani, B. Notash and S. W. Ng

Experimental

Chloroauric acid trihydrate (0.25 g, 0.73 mmol) dissolved in acetonitrile (5 ml) was mixed with 4,5-diazafluoren-9-one (Henderson *et al.*, 1984) (0.13 g, 0.73 mmol) dissolved in methanol (5 ml). The yellow solution was set aside for the growth of crystals, which appeared after two weeks (yield 70%; m.p. 483 K).

Refinement

C-bound H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.95 Å and with U_{iso}(H) = 1.2U_{eq}(C). The N-bound H atom was located in a difference Fourier map and refined isotropically, with a distance restraint of N—H = 0.88 (1) Å. The crystal diffracted strongly owing to the extremely heavy metal atom; however, its presence introduced severe absorption problems that could not be corrected analytically as the crystal did not have regular faces. The final difference Fourier map had a highest peak at 1.00 Å and a deepest hole at 1.00 Å from Au1 atom.

Figures

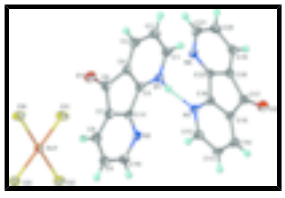


Fig. 1. Molecular structure of the title compound. Displacement ellipsoids are drawn at the 70% probability level. The dashed line denotes hydrogen bond.

9-Oxo-4,5-diazafluoren-4-ium tetrachloridoaurate(III)–4,5-diazafluoren-9-one (1/1)

Crystal data

$(C_{11}H_7N_2O)[AuCl_4]\cdot C_{11}H_6N_2O$	$Z = 2$
$M_r = 704.13$	$F_{000} = 672$
Triclinic, $P\overline{1}$	$D_x = 2.096$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.1035$ (1) Å	Cell parameters from 7741 reflections
$b = 12.6513$ (2) Å	$\theta = 2.7$–$28.4°$
$c = 13.2366$ (2) Å	$\mu = 7.10$ mm^{-1}
$\alpha = 73.285$ (1)°	$T = 118$ K
$\beta = 78.410$ (1)°	Block, yellow
$\gamma = 88.375$ (1)°	0.20 × 0.10 × 0.10 mm
$V = 1115.51$ (3) Å^3	

Data collection

Bruker APEXII CCD diffractometer	5044 independent reflections
Radiation source: fine-focus sealed tube	4829 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.016$
$T = 118$ K	$\theta_{max} = 27.5^{o}$
φ and ω scans	$\theta_{min} = 1.6^{o}$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9\rightarrow9$
$T_{min} = 0.331$, $T_{max} = 0.537$	$k = -16\rightarrow16$
9343 measured reflections	$l = -17\rightarrow17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.025$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.067$	$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.1124P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.001$
5044 reflections	$\Delta\rho_{max} = 3.05$ e Å^{-3}
302 parameters	$\Delta\rho_{min} = -1.27$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Au1	0.298203 (14)	0.304054 (8)	0.394619 (8)	0.01445 (6)
Cl1	0.28189 (13)	0.32712 (7)	0.56013 (7)	0.02448 (17)
Cl2	0.31382 (13)	0.49176 (6)	0.32181 (7)	0.02314 (17)
Cl3	0.31133 (12)	0.28082 (6)	0.22834 (6)	0.01975 (16)
Cl4	0.28622 (13)	0.11653 (6)	0.46788 (7)	0.02373 (17)
O1	0.1850 (4)	0.3950 (2)	0.7782 (2)	0.0258 (5)
O2	0.3590 (4)	1.22613 (19)	0.9995 (2)	0.0239 (5)
N1	0.2353 (4)	0.7025 (2)	0.9069 (2)	0.0163 (5)
H1	0.247 (6)	0.7760 (9)	0.885 (3)	0.029 (11)*
N2	0.2032 (4)	0.7897 (2)	0.6684 (2)	0.0187 (5)
N3	0.3136 (4)	0.9265 (2)	0.8548 (2)	0.0156 (5)
N4	0.1257 (4)	0.8534 (2)	1.0949 (2)	0.0168 (5)
C1	0.2546 (5)	0.6495 (3)	1.0088 (3)	0.0186 (6)
H1A	0.2662	0.6912	1.0567	0.022*
C2	0.2576 (5)	0.5354 (3)	1.0441 (3)	0.0203 (6)

H2	0.2704	0.4994	1.1159	0.024*
C3	0.2420 (5)	0.4737 (3)	0.9747 (3)	0.0205 (6)
H3	0.2447	0.3954	0.9977	0.025*
C4	0.2222 (4)	0.5296 (3)	0.8712 (3)	0.0164 (6)
C5	0.2212 (4)	0.6449 (2)	0.8388 (2)	0.0153 (6)
C6	0.2012 (4)	0.4900 (3)	0.7772 (3)	0.0181 (6)
C7	0.1980 (5)	0.5922 (3)	0.6874 (3)	0.0182 (6)
C8	0.1879 (5)	0.6097 (3)	0.5801 (3)	0.0212 (7)
H8	0.1825	0.5501	0.5504	0.025*
C9	0.1863 (5)	0.7187 (3)	0.5186 (3)	0.0212 (7)
H9	0.1809	0.7349	0.4445	0.025*
C10	0.1924 (5)	0.8047 (3)	0.5642 (3)	0.0223 (7)
H10	0.1888	0.8781	0.5195	0.027*
C11	0.2057 (4)	0.6848 (3)	0.7249 (3)	0.0155 (6)
C12	0.3999 (4)	0.9788 (3)	0.7520 (3)	0.0181 (6)
H12	0.4178	0.9380	0.7009	0.022*
C13	0.4636 (4)	1.0882 (3)	0.7171 (3)	0.0180 (6)
H13	0.5212	1.1206	0.6436	0.022*
C14	0.4435 (4)	1.1511 (2)	0.7894 (3)	0.0176 (6)
H14	0.4858	1.2264	0.7675	0.021*
C15	0.3585 (4)	1.0977 (2)	0.8947 (3)	0.0154 (6)
C16	0.2951 (4)	0.9874 (2)	0.9225 (2)	0.0139 (6)
C17	0.3142 (4)	1.1372 (2)	0.9930 (3)	0.0165 (6)
C18	0.2124 (4)	1.0415 (2)	1.0813 (3)	0.0162 (6)
C19	0.1345 (4)	1.0290 (3)	1.1887 (3)	0.0181 (6)
H19	0.1383	1.0876	1.2200	0.022*
C20	0.0503 (4)	0.9262 (3)	1.2485 (3)	0.0199 (6)
H20	−0.0062	0.9133	1.3226	0.024*
C21	0.0490 (4)	0.8422 (3)	1.1996 (3)	0.0195 (6)
H21	−0.0094	0.7729	1.2426	0.023*
C22	0.2039 (4)	0.9522 (2)	1.0394 (2)	0.0143 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Au1	0.01648 (8)	0.01391 (8)	0.01188 (8)	0.00013 (5)	−0.00036 (5)	−0.00370 (5)
Cl1	0.0377 (5)	0.0222 (4)	0.0142 (4)	0.0028 (3)	−0.0047 (3)	−0.0066 (3)
Cl2	0.0343 (4)	0.0151 (4)	0.0193 (4)	−0.0012 (3)	−0.0052 (3)	−0.0038 (3)
Cl3	0.0262 (4)	0.0187 (4)	0.0144 (4)	−0.0011 (3)	−0.0020 (3)	−0.0061 (3)
Cl4	0.0333 (4)	0.0151 (4)	0.0194 (4)	0.0019 (3)	−0.0005 (3)	−0.0030 (3)
O1	0.0391 (14)	0.0147 (11)	0.0241 (13)	−0.0037 (10)	−0.0044 (11)	−0.0072 (10)
O2	0.0348 (13)	0.0135 (11)	0.0251 (13)	0.0004 (9)	−0.0077 (11)	−0.0074 (10)
N1	0.0197 (12)	0.0112 (12)	0.0171 (13)	0.0000 (9)	−0.0020 (10)	−0.0038 (10)
N2	0.0205 (13)	0.0146 (12)	0.0195 (14)	−0.0029 (10)	−0.0032 (11)	−0.0030 (10)
N3	0.0171 (12)	0.0149 (12)	0.0136 (12)	−0.0002 (9)	−0.0016 (10)	−0.0033 (10)
N4	0.0180 (12)	0.0155 (12)	0.0162 (13)	−0.0018 (9)	−0.0016 (10)	−0.0048 (10)
C1	0.0229 (15)	0.0171 (14)	0.0161 (15)	−0.0012 (11)	−0.0014 (12)	−0.0068 (12)
C2	0.0255 (16)	0.0161 (15)	0.0177 (16)	0.0011 (12)	−0.0038 (13)	−0.0031 (12)

C3	0.0228 (16)	0.0151 (14)	0.0211 (16)	0.0004 (12)	−0.0026 (13)	−0.0024 (12)
C4	0.0178 (14)	0.0136 (14)	0.0189 (15)	−0.0004 (11)	−0.0024 (12)	−0.0071 (12)
C5	0.0146 (13)	0.0143 (14)	0.0164 (15)	−0.0007 (10)	0.0001 (11)	−0.0054 (12)
C6	0.0189 (14)	0.0171 (15)	0.0191 (15)	0.0002 (11)	−0.0012 (12)	−0.0081 (12)
C7	0.0190 (14)	0.0161 (14)	0.0199 (16)	0.0005 (11)	−0.0021 (12)	−0.0071 (12)
C8	0.0212 (15)	0.0241 (16)	0.0198 (16)	−0.0026 (12)	−0.0037 (13)	−0.0089 (13)
C9	0.0194 (15)	0.0265 (17)	0.0169 (16)	−0.0016 (12)	−0.0025 (12)	−0.0054 (13)
C10	0.0224 (16)	0.0218 (16)	0.0197 (17)	−0.0023 (12)	−0.0048 (13)	−0.0007 (13)
C11	0.0137 (13)	0.0137 (14)	0.0181 (15)	−0.0001 (10)	−0.0023 (11)	−0.0035 (12)
C12	0.0190 (14)	0.0188 (15)	0.0162 (15)	0.0008 (11)	−0.0023 (12)	−0.0056 (12)
C13	0.0184 (14)	0.0176 (14)	0.0141 (14)	−0.0006 (11)	−0.0007 (12)	−0.0002 (12)
C14	0.0169 (14)	0.0130 (13)	0.0214 (16)	−0.0009 (11)	−0.0040 (12)	−0.0024 (12)
C15	0.0156 (13)	0.0128 (13)	0.0177 (15)	0.0032 (10)	−0.0046 (12)	−0.0035 (12)
C16	0.0122 (13)	0.0133 (13)	0.0155 (14)	0.0014 (10)	−0.0027 (11)	−0.0032 (11)
C17	0.0178 (14)	0.0131 (13)	0.0190 (15)	0.0030 (11)	−0.0057 (12)	−0.0040 (12)
C18	0.0149 (13)	0.0157 (14)	0.0183 (15)	0.0011 (11)	−0.0043 (11)	−0.0049 (12)
C19	0.0193 (14)	0.0184 (14)	0.0193 (15)	0.0028 (11)	−0.0060 (12)	−0.0084 (12)
C20	0.0170 (14)	0.0270 (16)	0.0148 (15)	0.0024 (12)	−0.0009 (12)	−0.0064 (13)
C21	0.0181 (14)	0.0172 (14)	0.0206 (16)	−0.0011 (11)	−0.0021 (12)	−0.0025 (12)
C22	0.0120 (12)	0.0149 (13)	0.0165 (14)	0.0020 (10)	−0.0036 (11)	−0.0047 (11)

Geometric parameters (Å, °)

Au1—Cl1	2.2720 (8)	C7—C8	1.389 (5)
Au1—Cl2	2.2882 (8)	C7—C11	1.405 (4)
Au1—Cl3	2.2864 (8)	C8—C9	1.386 (5)
Au1—Cl4	2.2872 (8)	C8—H8	0.9500
O1—C6	1.208 (4)	C9—C10	1.393 (5)
O2—C17	1.209 (4)	C9—H9	0.9500
N1—C5	1.331 (4)	C10—H10	0.9500
N1—C1	1.355 (4)	C12—C13	1.385 (4)
N1—H1	0.89 (1)	C12—H12	0.9500
N2—C11	1.325 (4)	C13—C14	1.396 (4)
N2—C10	1.355 (4)	C13—H13	0.9500
N3—C16	1.326 (4)	C14—C15	1.381 (4)
N3—C12	1.354 (4)	C14—H14	0.9500
N4—C22	1.326 (4)	C15—C16	1.400 (4)
N4—C21	1.352 (4)	C15—C17	1.498 (4)
C1—C2	1.384 (4)	C16—C22	1.493 (4)
C1—H1A	0.9500	C17—C18	1.500 (4)
C2—C3	1.387 (5)	C18—C19	1.382 (4)
C2—H2	0.9500	C18—C22	1.402 (4)
C3—C4	1.383 (5)	C19—C20	1.392 (4)
C3—H3	0.9500	C19—H19	0.9500
C4—C5	1.397 (4)	C20—C21	1.396 (4)
C4—C6	1.503 (4)	C20—H20	0.9500
C5—C11	1.471 (4)	C21—H21	0.9500
C6—C7	1.486 (5)		
Cl1—Au1—Cl3	179.43 (3)	N2—C10—C9	123.9 (3)

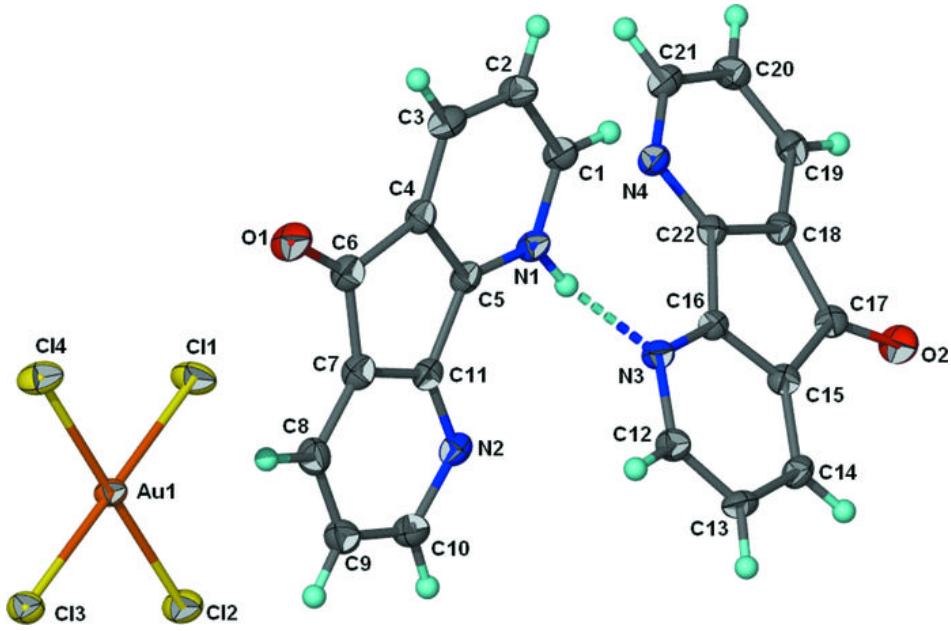
Cl1—Au1—Cl4	90.27 (3)	N2—C10—H10	118.1
Cl3—Au1—Cl4	89.73 (3)	C9—C10—H10	118.1
Cl1—Au1—Cl2	89.47 (3)	N2—C11—C7	126.7 (3)
Cl3—Au1—Cl2	90.55 (3)	N2—C11—C5	125.6 (3)
Cl4—Au1—Cl2	179.30 (3)	C7—C11—C5	107.7 (3)
C5—N1—C1	120.1 (3)	N3—C12—C13	123.8 (3)
C5—N1—H1	122 (3)	N3—C12—H12	118.1
C1—N1—H1	118 (3)	C13—C12—H12	118.1
C11—N2—C10	114.0 (3)	C12—C13—C14	120.3 (3)
C16—N3—C12	115.2 (3)	C12—C13—H13	119.8
C22—N4—C21	115.0 (3)	C14—C13—H13	119.8
N1—C1—C2	120.9 (3)	C15—C14—C13	116.1 (3)
N1—C1—H1A	119.6	C15—C14—H14	121.9
C2—C1—H1A	119.6	C13—C14—H14	121.9
C1—C2—C3	120.1 (3)	C14—C15—C16	119.8 (3)
C1—C2—H2	120.0	C14—C15—C17	131.3 (3)
C3—C2—H2	120.0	C16—C15—C17	108.9 (3)
C4—C3—C2	118.0 (3)	N3—C16—C15	124.8 (3)
C4—C3—H3	121.0	N3—C16—C22	126.9 (3)
C2—C3—H3	121.0	C15—C16—C22	108.3 (3)
C3—C4—C5	120.0 (3)	O2—C17—C15	126.5 (3)
C3—C4—C6	132.0 (3)	O2—C17—C18	128.0 (3)
C5—C4—C6	107.9 (3)	C15—C17—C18	105.4 (2)
N1—C5—C4	121.0 (3)	C19—C18—C22	119.4 (3)
N1—C5—C11	129.2 (3)	C19—C18—C17	132.1 (3)
C4—C5—C11	109.8 (3)	C22—C18—C17	108.5 (3)
O1—C6—C7	128.9 (3)	C18—C19—C20	116.5 (3)
O1—C6—C4	126.0 (3)	C18—C19—H19	121.8
C7—C6—C4	105.1 (3)	C20—C19—H19	121.8
C8—C7—C11	118.2 (3)	C19—C20—C21	120.0 (3)
C8—C7—C6	132.5 (3)	C19—C20—H20	120.0
C11—C7—C6	109.4 (3)	C21—C20—H20	120.0
C9—C8—C7	116.6 (3)	N4—C21—C20	124.0 (3)
C9—C8—H8	121.7	N4—C21—H21	118.0
C7—C8—H8	121.7	C20—C21—H21	118.0
C8—C9—C10	120.7 (3)	N4—C22—C18	125.2 (3)
C8—C9—H9	119.7	N4—C22—C16	126.0 (3)
C10—C9—H9	119.7	C18—C22—C16	108.8 (3)
C5—N1—C1—C2	−0.8 (5)	C16—N3—C12—C13	−0.8 (5)
N1—C1—C2—C3	0.4 (5)	N3—C12—C13—C14	1.0 (5)
C1—C2—C3—C4	−0.4 (5)	C12—C13—C14—C15	0.1 (5)
C2—C3—C4—C5	0.9 (5)	C13—C14—C15—C16	−1.3 (4)
C2—C3—C4—C6	−179.7 (3)	C13—C14—C15—C17	179.7 (3)
C1—N1—C5—C4	1.2 (5)	C12—N3—C16—C15	−0.5 (4)
C1—N1—C5—C11	−177.8 (3)	C12—N3—C16—C22	179.1 (3)
C3—C4—C5—N1	−1.3 (5)	C14—C15—C16—N3	1.5 (5)
C6—C4—C5—N1	179.2 (3)	C17—C15—C16—N3	−179.3 (3)
C3—C4—C5—C11	177.9 (3)	C14—C15—C16—C22	−178.0 (3)
C6—C4—C5—C11	−1.6 (3)	C17—C15—C16—C22	1.2 (3)

C3—C4—C6—O1	5.2 (6)	C14—C15—C17—O2	−5.5 (6)
C5—C4—C6—O1	−175.3 (3)	C16—C15—C17—O2	175.4 (3)
C3—C4—C6—C7	−176.8 (3)	C14—C15—C17—C18	177.2 (3)
C5—C4—C6—C7	2.8 (3)	C16—C15—C17—C18	−1.9 (3)
O1—C6—C7—C8	−4.5 (6)	O2—C17—C18—C19	3.9 (6)
C4—C6—C7—C8	177.6 (4)	C15—C17—C18—C19	−178.8 (3)
O1—C6—C7—C11	175.0 (3)	O2—C17—C18—C22	−175.3 (3)
C4—C6—C7—C11	−2.9 (3)	C15—C17—C18—C22	2.0 (3)
C11—C7—C8—C9	0.0 (5)	C22—C18—C19—C20	−0.3 (4)
C6—C7—C8—C9	179.4 (3)	C17—C18—C19—C20	−179.5 (3)
C7—C8—C9—C10	−0.7 (5)	C18—C19—C20—C21	0.4 (5)
C11—N2—C10—C9	−0.6 (5)	C22—N4—C21—C20	−0.4 (5)
C8—C9—C10—N2	1.0 (5)	C19—C20—C21—N4	−0.1 (5)
C10—N2—C11—C7	−0.2 (5)	C21—N4—C22—C18	0.5 (4)
C10—N2—C11—C5	178.5 (3)	C21—N4—C22—C16	−178.9 (3)
C8—C7—C11—N2	0.5 (5)	C19—C18—C22—N4	−0.1 (5)
C6—C7—C11—N2	−179.1 (3)	C17—C18—C22—N4	179.2 (3)
C8—C7—C11—C5	−178.4 (3)	C19—C18—C22—C16	179.3 (3)
C6—C7—C11—C5	2.0 (4)	C17—C18—C22—C16	−1.3 (3)
N1—C5—C11—N2	0.0 (5)	N3—C16—C22—N4	0.0 (5)
C4—C5—C11—N2	−179.1 (3)	C15—C16—C22—N4	179.5 (3)
N1—C5—C11—C7	178.9 (3)	N3—C16—C22—C18	−179.5 (3)
C4—C5—C11—C7	−0.2 (4)	C15—C16—C22—C18	0.1 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3	0.89 (1)	1.88 (1)	2.762 (4)	168 (4)

Fig. 1


C21
C20
C2
C3
C1
C19
N4
C4
C22
C18
O1
C6
N1
C5
C16
C17
Cl4
Cl1
C7
C11
N3
O2
C15
C8
N2
C12
C14
Au1
C13
C10
C9
Cl3
Cl2