1,8-Bis(diphenylmethylium)naphthalenediyl Dication as an Organic Oxidant: Synthesis of Benzidines via Self-Coupling of *N,N*-Dialkylanilines

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Supporting Information

General: NMR spectra were obtained on a JEOL AL-400 spectrometer or a Bruker Avance500 spectrometer. Chemical shift values were given in ppm relative to internal Me_4Si (for ^1H NMR: δ 0.00) and $CDCl_3$ (for ^{13}C NMR: δ 77.0). IR spectra were recorded on a Horiba FT-300S spectrometer. Mass spectra were taken with a JEOL JMS SX-102A spectrometer. Elemental analyses were performed with a YANAKO MT-6 CHN Corder apparatus. Column chromatography and preparative thin-layer chromatography (PTLC) were performed on silica gel.

1,1,3,3-Tetraphenyl-1*H*,3*H*-benzo[*de*]isochromene (**3**)
To a solution of 1,8-dibromonaphthalene (2.0 g, 7.0 mmol) in diethyl ether (35 ml) was added *n*-BuLi (6.6 ml, 2.60 M in hexane, 17.2 mmol) at 0 ^oC under argon. The reaction mixture was stirred for 2 h at room temperature and then benzophenone (3.1 g, 17.0 mmol) was added. After being refluxed for 5 h, the reaction was quenched with saturated aqueous NH_4Cl. Organic materials were extracted with EtOAc three times. The combined extracts were washed with brine and dried over Na_2SO_4. After removal of the solvent under reduced pressure, the resulting residue was dissolved in dichloromethane (10 ml), and a catalytic amount of trifluoroacetic acid (0.05 ml, 0.7 mmol) was added at room temperature. The reaction mixture was stirred for 10 h, and then MeOH (20 ml) was added. After stirring for 1 h at room temperature, the resulting precipitate was collected by filtration, washed with MeOH, and dried under reduced pressure to give **3** (2.7 g, 80%) as white crystals.
m.p. 244–245 ^oC (CH_2Cl_2–MeOH);
^1H NMR (400 MHz, $CDCl_3$) δ 6.95–7.15 (22H, m), 7.37 (2H, dd, J = 8.0, 7.2 Hz), 7.80 (2H, d, J = 8.0 Hz);
^{13}C NMR (100 MHz, $CDCl_3$) δ 84.3, 124.9, 126.4, 126.5, 126.6, 127.0, 129.5, 132.9, 136.1, 146.5;
IR (KBr) 1578, 1546, 1452, 1359, 1087, 704, 623 cm^{-1};
FAB HRMS calcd for $C_{36}H_{27}O$ 475.2062 (M+1); found 475.2067.

Trimethylsilyl perchlorate ($TMSClO_4$ toluene solution)
To a solution of $AgClO_4$ (4.6 g, 22.2 mmol) in toluene (30 ml) was added trimethylsilyl chloride (2.8 ml, 22.2 mmol) under argon. After this mixture was stirred for 0.5 h at room temperature and then left standing for 0.5 h without stirring, the supernatant was used as a reagent ($TMSClO_4$, 0.74 M in toluene) for the preparation of the dications.

1,8-Bis(diphenylmethlyium)naphthalenediyl dication (**4a**)
To a stirred solution of **3** (2.0 g, 4.2 mmol) in 1,1,1,3,3,3-hexafluoro-2-propanol (30 ml) was added

$TMSClO_4$ (17.1 ml, 0.74 M in toluene, 12.6 mmol) at room temperature under argon. This mixture was stirred for 2 days at the same temperature, and the solvent was removed under reduced pressure. Dichloromethane (3.0 ml) and diethyl ether (10 ml) were added to the resulting crude product. After stirring for 1 h at room temperature, the black precipitate was collected by filtration, washed with diethyl ether (10 ml) and then dichloromethane (3.0 ml) under argon, and dried under reduced pressure to give **4a** (2.6 g, 95%) as dark red crystals.
^{1}H NMR (400 MHz, CD_3CN) δ 6.54 (2H, brs), 6.73 (2H, brs), 7.37 (2H, brs), 7.41–7.65 (10H, m), 7.75 (2H, brs), 7.98 (2H, brs), 8.05 (2H, dd, *J* = 7.6, 7.6 Hz), 8.23 (2H, brs), 8.95 (2H, dd, *J* = 8.4, 1.6 Hz);
^{13}C NMR (100 MHz, CD_3CN) δ 127.7, 128.0, 129.7, 130.1, 131.9, 136.6, 137.5, 139.7, 144.8, 151.0, 207.6;
IR (KBr) 1489, 1444, 1217, 1184, 1018, 742, 694 cm^{-1}.

X-ray date: see below

Typical experimental procedure for the preparation of aniline derivatives is as follows:
To a suspension of NaH (3.9 g, 60% dispersion in mineral oil, 98 mmol) in THF (50 ml) at 0 ^oC was added an aniline (40 mmol) under argon, and the reaction mixture was stirred for 0.5 h at the same temperature. After alkyl iodide (96 mmol) was added, the mixture was stirred at room temperature for 3 h. The reaction was quenched with water, and organic materials were extracted with EtOAc three times. The combined extracts were washed with brine and dried over Na_2SO_4. After removal of the solvent under reduced pressure, the residue was recrystallized from ethanol.

N,N-Diethyl-3,5-dimethylaniline
white crystals, m.p. 44–45 ^oC (EtOH);
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.14 (6H, t, *J* = 6.8 Hz), 2.26 (6H, s), 3.31 (4H, q, *J* = 6.8 Hz), 6.31 (2H, s), 6.31 (1H, s);
^{13}C NMR (100 MHz, $CDCl_3$) δ 12.7, 21.9, 44.3, 109.8, 117.4, 138.7, 147.9;
IR (neat) 1595, 1487, 1356, 1219, 814 cm^{-1};
Anal. Calcd for $C_{12}H_{19}N$: C, 81.30; H, 10.80; N, 7.90%. Found: C, 81.14; H, 10.72; N, 7.70%.

N,N,3,5-Tetramethylaniline
yellow oil;
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.28 (6H, s), 2.91 (6H, s), 6.38 (2H, s), 6.39 (1H, s);
^{13}C NMR (100 MHz, $CDCl_3$) δ 21.8, 40.7, 110.7, 118.7, 138.5, 150.8;
IR (neat) 2914, 1597, 1489, 1354, 816 cm^{-1};
Anal. Calcd for $C_{10}H_{15}N$: C, 80.48; H, 10.13; N, 9.39%. Found: C, 80.50; H, 9.91; N, 9.21%.

3,5-Dichloro-*N,N*-diethylaniline
white crystals, m.p. 71–72 ^oC (EtOH);
^{1}H NMR (400 MHz, $CDCl_3$) δ 11.15 (6H, t, *J* = 7.2 Hz), 3.30 (4H, q, *J* = 7.2 Hz), 6.47 (2H, d, *J* = 2.0 Hz), 6.58 (1H, d, *J* = 2.0 Hz);
^{13}C NMR (100 MHz, $CDCl_3$) δ 12.4, 44.5, 109.6, 114.7, 135.5, 149.1;
IR (neat) 1585, 1549, 1468, 746 cm^{-1};
Anal. Calcd for $C_{10}H_{13}Cl_2N$: C, 55.06; H, 6.01; N, 6.42%. Found: C, 54.85; H, 5.98; N, 6.22%.

3-Bromo-*N,N*-diethylaniline
a pale yellow oil;

^{1}H NMR (400 MHz, $CDCl_3$) δ 1.15 (6H, t, *J* = 7.2 Hz), 3.32 (4H, q, *J* = 7.2 Hz), 6.56 (1H, dd, *J* = 8.0, 2.4 Hz), 6.72 (1H, dd, *J* = 7.6, 1.0 Hz), 6.76 (1H, dd, *J* = 2.4, 1.0 Hz), 7.05 (1H, dd, *J* = 8.0, 7.6 Hz);
^{13}C NMR (100 MHz, $CDCl_3$) δ 12.5, 44.3, 110.1, 114.2, 117.9, 123.5, 130.3, 148.9;
IR (neat) 1587, 1491, 1263, 978, 750 cm^{-1};
Anal. Calcd for $C_{10}H_{14}BrN$: C, 52.65; H, 6.19; N, 6.14%. Found: C, 52.47; H, 6.20; N, 5.92%.

N,N-Diallyl-3,5-dimethylaniline
pale yellow oil;
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.45 (6H, s), 3.87–3.90 (4H, m), 5.10–5.20 (4H, m), 5.78–5.91 (2H, m), 6.34 (2H, brs), 6.35 (1H, brs);
^{13}C NMR (100 MHz, $CDCl_3$) δ 21.8, 53.4, 110.3, 115.8, 118.3, 129.0, 138.5, 145.3;
IR (neat) 1597, 1192, 914, 816 cm^{-1};
Anal. Calcd for $C_{14}H_{19}N$: C, 83.53; H, 9.51; N, 6.96%. Found: C, 83.55; H, 9.51; N, 6.75%.

N,N-Dibenzyl-3,5-dimethylaniline
white crystals, m.p. 83–84 °C (EtOH);
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.20 (6H, s), 4.60 (4H, s), 6.37–6.41 (3H, m), 7.20–7.26 (6H, m), 7.28–7.34 (4H, m);
^{13}C NMR (100 MHz, $CDCl_3$) δ 21.8, 53.7, 110.2, 118.8, 126.6, 126.7, 128.5, 138.6, 138.7, 149,5;
IR (neat) 1595, 1493, 1450, 1360, 1192, 729, 694 cm^{-1};
Anal. Calcd for $C_{22}H_{23}N$: C, 87.66; H, 7.69; N, 4.65%. Found: C, 87.74; H, 7.82; N, 4.54%.

Typical experimental procedure is described for the oxidative coupling of *N,N*-diethyl-3,5-dimethylaniline: To a solution of *N,N*-diethyl-3,5-dimethylaniline (27.5 mg, 0.16 mmol) in dichloromethane (2.0 ml) was added **4a** (61.8 mg, 0.10 mmol) at –78 °C, and the reaction mixture was stirred for 1 h at the same temperature. After completion of the oxidative coupling (TLC monitoring), the reaction was quenched with saturated aqueous $NaHCO_3$. Organic materials were extracted with EtOAc three times, and the combined extracts were washed with brine and dried over Na_2SO_4. After removal of the solvent under reduced pressure, the resulting residue was purified by preparative TLC (silica gel) to give *N,N,N′,N′*-tetraethyl-2,2′,6,6′-tetramethylbenzidine (26.7 mg, 98%) along with acenaphthene **5**.

1,1,2,2-Tetraphenylacenaphthene (**5**)
white crystals, m.p. 257–258 °C (CH_2Cl_2–MeOH);
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.76–7.05 (20 H, m), 7.19 (2 H, d, *J* = 6.8 Hz), 7.55 (2 H, dd, *J* = 6.8, 8.4 Hz), 7.77 (2 H, d, *J* = 8.4 Hz);
^{13}C NMR (100 MHz, $CDCl_3$) δ 75.1, 123.2, 123.4, 125.3, 126.5, 128.1, 130.9, 131.4, 137.0, 144.9, 149.9;
IR (KBr) 1585, 1491, 1439, 906, 781, 727, 696 cm^{-1};
FAB HRMS calcd for $C_{36}H_{27}$ 459.2113 (M+1); found 459.2095.

N,N,N′,N′-Tetraethyl-2,2′,6,6′-tetramethylbenzidine
white crystals, m.p. 119–120 °C (EtOH);
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.18 (12H, t, *J* = 6.8 Hz), 1.87 (12H, s), 3.34 (8H, q, *J* = 6.8 Hz), 6.45 (4H, s);
^{13}C NMR (100 MHz, $CDCl_3$) δ 12.9, 20.8, 44.2, 110.0, 128.3, 137.0, 146.4;
IR (neat) 2964, 1601, 1473, 1373, 1286, 1198, 825 cm^{-1};
FAB HRMS calcd for $C_{24}H_{37}N_2$ 353.2957 (M+1); found 353.2935.

N,N,N′,N′,2,2′,6,6′-Octamethybenzidine
pale yellow crystals, m.p. 172–174 °C (EtOH);
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.88 (12H, s), 2.94 (12H, s), 6.51 (4H, s);
^{13}C NMR (100 MHz, $CDCl_3$) δ 20.6, 40.7, 111.6, 129.1, 136.9, 149.0;
IR (neat) 2913, 1602, 1489, 1440, 1352, 1228, 823 cm^{-1};
FAB HRMS calcd for $C_{20}H_{29}N_2$ 297.2331 (M+1); found 297.2326.

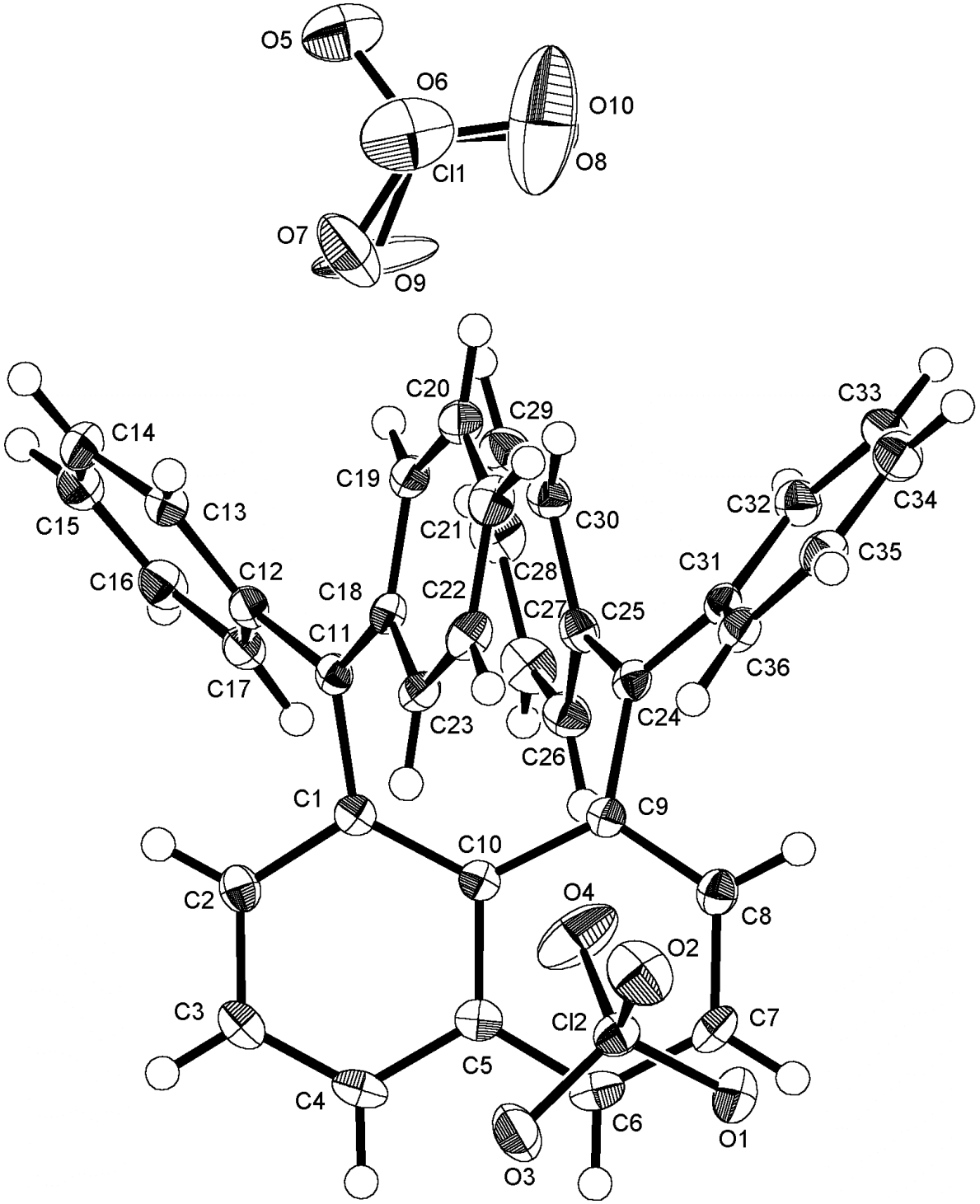
2,2′,6,6′-Tetrachloro-*N,N,N′,N′*-tetraethylbenzidine
white crystals, m.p. 215–217 °C (EtOH);
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.20 (12H, t, J = 7.2 Hz), 3.34 (8H, q, J = 7.2 Hz), 6.65 (4H, s);
^{13}C NMR (100 MHz, $CDCl_3$) δ 12.5, 44.3, 110.0, 121.6, 136.5, 148.2;
IR (neat) 2967, 1593, 1520, 1471, 1352, 1265, 1188 cm^{-1};
Anal. Calcd for $C_{20}H_{24}Cl_4N_2$: C, 55.32; H, 5.57; N, 6.45%. Found: C, 55.07; H, 5.72; N, 6.26%.

2,2′-Dibromo-*N,N,N′,N′*-tetraethylbenzidine
pale yellow oil;
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.18 (12H, t, J = 7.2 Hz), 3.35 (8H, q, J = 7.2 Hz), 6.61 (2H, dd, J = 8.4, 2.4 Hz), 6.90 (2H, d, J = 2.4 Hz), 7.06 (2H, d, J = 8.4 Hz);
^{13}C NMR (100 MHz, $CDCl_3$) δ 12.6, 44.3, 110.0, 114.4, 125.5, 128.8, 132.0, 147.8;
IR (neat) 1597, 1489, 1352, 1196, 795 cm^{-1};
FAB HRMS calcd for $C_{20}H_{27}{}^{81}Br_2N_2$ 457.0500 (M+1); found 457.0505.

N,N,N′,N′-Tetraallyl-2,2′,6,6′-tetramethylbenzidine
pale yellow oil;
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.85 (12H, s), 3.90 (8H, d, J = 5.2 Hz), 5.18–5.30 (8H, m), 5.80–5.95 (4H, m), 6.47 (4H, s);
^{13}C NMR (100 MHz, $CDCl_3$) δ 20.7, 52.6, 111.4, 115.9, 128.9, 134.7, 136.9, 147.3;
IR (neat) 2910, 1601, 1473, 1190, 912, 771 cm^{-1};
FAB HRMS calcd for $C_{28}H_{37}N_2$ 401.2957 (M+1); found 401.2968.

N,N,N′,N′-Tetrabenzyl-2,2′,6,6′-tetramethylbenzidine
white crystals, m.p. 195–197 °C (EtOH);
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.82 (12H, s), 4.59 (8H, s), 6.52 (4H, s), 7.20–7.36 (20H, m);
^{13}C NMR (100 MHz, $CDCl_3$) δ 20.7, 53.7, 111.5, 126.6, 127.0, 128.4, 129.3, 137.0, 139.2, 148.0;
IR (neat) 1601, 1493, 1450, 1215, 1184, 748, 728, 694 cm^{-1};
Anal. Calcd for $C_{44}H_{44}N_2$: C, 87.96; H, 7.38; N, 4.66%. Found: C, 87.75; H, 7.57; N, 4.53%.

4,4′,5,5′-Tetrakis(dimethylamino)-1,1′-binaphthalene
pale yellow crystals, m.p. 192–193 °C (EtOH);
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.87 (24H, s), 6.88 (2H, d, J = 7.2 Hz), 6.93 (2H, d, J = 8.0 Hz), 6.99 (2H, d, J = 8.0 Hz), 7.08 (2H, dd, J = 7.2, 7.6 Hz), 7.25 (2H, d, J = 7.6 Hz);
^{13}C NMR (125 MHz, $CDCl_3$) δ 44.4 (br), 112.0, 112.2, 120.2, 120.6, 124.9, 127.7, 132.5, 137.3, 149.9, 150.5;
IR (neat) 2931, 2823, 2771, 1572, 1394, 1373, 1026, 725 cm^{-1};
Anal. Calcd for $C_{28}H_{34}N_4$: C, 78.83; H, 8.03; N, 13.13%. Found: C, 78.61; H, 8.12; N, 12.87%.


O5
O6
O10
Cl1
O8
O7
O9
C20
C29
C33
C14
C19
C32
C34
C13
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C35
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C16
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C27
C25
C11
C36
C17
C23
C24
C26
C1
C9
C10
C2
O4
C8
O2
C3
Cl2
C7
C5
C4
C6
O1
O3

Table 1. Crystal data and structure refinement for ichi0130.

Identification code	ichi0130	
Empirical formula	C36 H26 Cl2 O8	
Formula weight	657.47	
Temperature	120(2) K	
Wavelength	0.71070 Å	
Crystal system	monoclinic	
Space group	P21/a	
Unit cell dimensions	a = 16.090(7) Å	α= 90.0000(15)°.
	b = 10.544(4) Å	β= 104.6052(17)°.
	c = 17.881(7) Å	γ = 90.0000(15)°.
Volume	2936(2) $Å^3$	
Z	4	
Density (calculated)	1.488 Mg/m^3	
Absorption coefficient	0.279 mm^{-1}	
F(000)	1360	
Crystal size	0.50 x 0.30 x 0.20 mm^3	
Theta range for data collection	3.05 to 27.48°.	
Index ranges	-17<=h<=20, -13<=k<=13, -21<=l<=23	
Reflections collected	16652	
Independent reflections	5037 [R(int) = 0.0296]	
Completeness to theta = 27.48°	74.9 %	
Max. and min. transmission	0.9463 and 0.8732	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5037 / 0 / 434	
Goodness-of-fit on F^2	1.054	
Final R indices [I>2sigma(I)]	R1 = 0.0527, wR2 = 0.1420	
R indices (all data)	R1 = 0.0593, wR2 = 0.1467	
Largest diff. peak and hole	1.480 and -0.773 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ichi0130. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	5250(2)	1828(3)	8291(2)	17(1)
C(2)	6090(2)	2115(3)	8701(2)	21(1)
C(3)	6283(2)	3205(3)	9154(2)	24(1)
C(4)	5634(2)	4039(3)	9180(2)	24(1)
C(5)	4780(2)	3802(3)	8777(2)	20(1)
C(6)	4131(2)	4655(3)	8860(2)	24(1)
C(7)	3283(2)	4408(3)	8545(2)	24(1)
C(8)	3061(2)	3324(3)	8084(2)	21(1)
C(9)	3670(2)	2523(2)	7913(1)	16(1)
C(10)	4565(2)	2695(3)	8308(2)	16(1)
C(11)	5150(2)	544(3)	7956(1)	17(1)
C(12)	5680(2)	218(3)	7446(2)	19(1)
C(13)	6004(2)	-1023(3)	7419(2)	23(1)
C(14)	6540(2)	-1285(3)	6946(2)	27(1)
C(15)	6744(2)	-344(3)	6476(2)	28(1)
C(16)	6412(2)	878(3)	6478(2)	26(1)
C(17)	5906(2)	1171(3)	6976(2)	21(1)
C(18)	4600(2)	-352(2)	8200(2)	17(1)
C(19)	4253(2)	-1415(3)	7746(2)	19(1)
C(20)	3736(2)	-2257(3)	8003(2)	23(1)
C(21)	3549(2)	-2068(3)	8716(2)	25(1)
C(22)	3896(2)	-1034(3)	9180(2)	23(1)
C(23)	4407(2)	-180(3)	8926(2)	19(1)
C(24)	3340(2)	1684(2)	7247(2)	18(1)
C(25)	3722(2)	1736(3)	6596(2)	19(1)
C(26)	4038(2)	2897(3)	6396(2)	23(1)
C(27)	4327(2)	2986(3)	5733(2)	28(1)
C(28)	4340(2)	1920(3)	5284(2)	31(1)
C(29)	4058(2)	760(3)	5488(2)	27(1)
C(30)	3728(2)	663(3)	6125(2)	21(1)
C(31)	2574(2)	951(3)	7205(2)	18(1)
C(32)	2009(2)	639(3)	6477(2)	24(1)
C(33)	1278(2)	-63(3)	6453(2)	28(1)

C(34)	1098(2)	-476(3)	7133(2)	29(1)
C(35)	1649(2)	-187(3)	7849(2)	25(1)
C(36)	2370(2)	548(3)	7888(2)	22(1)
Cl(1)	3862(1)	-3192(1)	5744(1)	36(1)
Cl(2)	3528(1)	2372(1)	10443(1)	22(1)
O(1)	2919(1)	3394(2)	10254(1)	34(1)
O(2)	3163(2)	1339(2)	10781(1)	41(1)
O(3)	4289(1)	2808(2)	10993(2)	41(1)
O(4)	3722(2)	1931(3)	9752(2)	51(1)
O(5)	4258(2)	-3967(3)	5254(2)	55(1)
O(6)	3803(2)	-3932(3)	6400(2)	65(1)
O(7)	4524(8)	-2309(16)	6058(8)	59(4)
O(8)	3159(12)	-2503(17)	5193(10)	83(6)
O(9)	4380(11)	-2129(10)	5990(8)	76(4)
O(10)	3088(9)	-2920(20)	5417(9)	120(5)

Table 3. Bond lengths [Å] and angles [°] for ichi0130.

C(1)-C(2)	1.398(4)	C(20)-C(21)	1.396(4)
C(1)-C(10)	1.438(4)	C(20)-H(13)	0.9500
C(1)-C(11)	1.474(4)	C(21)-C(22)	1.399(4)
C(2)-C(3)	1.396(4)	C(21)-H(14)	0.9500
C(2)-H(1)	0.9500	C(22)-C(23)	1.371(4)
C(3)-C(4)	1.375(4)	C(22)-H(15)	0.9500
C(3)-H(2)	0.9500	C(23)-H(16)	0.9500
C(4)-C(5)	1.404(4)	C(24)-C(31)	1.442(4)
C(4)-H(3)	0.9500	C(24)-C(25)	1.447(4)
C(5)-C(6)	1.413(4)	C(25)-C(26)	1.406(4)
C(5)-C(10)	1.428(4)	C(25)-C(30)	1.413(4)
C(6)-C(7)	1.364(4)	C(26)-C(27)	1.382(4)
C(6)-H(4)	0.9500	C(26)-H(17)	0.9500
C(7)-C(8)	1.402(4)	C(27)-C(28)	1.384(5)
C(7)-H(5)	0.9500	C(27)-H(18)	0.9500
C(8)-C(9)	1.386(4)	C(28)-C(29)	1.385(5)
C(8)-H(6)	0.9500	C(28)-H(19)	0.9500
C(9)-C(10)	1.447(4)	C(29)-C(30)	1.376(4)
C(9)-C(24)	1.471(4)	C(29)-H(20)	0.9500
C(11)-C(18)	1.436(4)	C(30)-H(21)	0.9500
C(11)-C(12)	1.437(4)	C(31)-C(36)	1.407(4)
C(12)-C(13)	1.414(4)	C(31)-C(32)	1.425(4)
C(12)-C(17)	1.415(4)	C(32)-C(33)	1.382(4)
C(13)-C(14)	1.381(4)	C(32)-H(22)	0.9500
C(13)-H(7)	0.9500	C(33)-C(34)	1.389(4)
C(14)-C(15)	1.392(4)	C(33)-H(23)	0.9500
C(14)-H(8)	0.9500	C(34)-C(35)	1.394(4)
C(15)-C(16)	1.395(5)	C(34)-H(24)	0.9500
C(15)-H(9)	0.9500	C(35)-C(36)	1.382(4)
C(16)-C(17)	1.384(4)	C(35)-H(25)	0.9500
C(16)-H(10)	0.9500	C(36)-H(26)	0.9500
C(17)-H(11)	0.9500	Cl(1)-O(10)	1.269(16)
C(18)-C(19)	1.414(4)	Cl(1)-O(9)	1.399(10)
C(18)-C(23)	1.421(4)	Cl(1)-O(7)	1.420(12)
C(19)-C(20)	1.373(4)	Cl(1)-O(6)	1.432(3)
C(19)-H(12)	0.9500	Cl(1)-O(5)	1.456(3)

Cl(1)-O(8)	1.489(11)	C(12)-C(11)-C(1)	117.2(2)
Cl(2)-O(4)	1.428(2)	C(13)-C(12)-C(17)	119.0(3)
Cl(2)-O(1)	1.438(2)	C(13)-C(12)-C(11)	121.6(3)
Cl(2)-O(3)	1.438(2)	C(17)-C(12)-C(11)	119.3(3)
Cl(2)-O(2)	1.440(2)	C(14)-C(13)-C(12)	119.9(3)
		C(14)-C(13)-H(7)	120.1
C(2)-C(1)-C(10)	119.8(2)	C(12)-C(13)-H(7)	120.1
C(2)-C(1)-C(11)	113.8(2)	C(13)-C(14)-C(15)	120.5(3)
C(10)-C(1)-C(11)	126.0(2)	C(13)-C(14)-H(8)	119.8
C(3)-C(2)-C(1)	121.8(3)	C(15)-C(14)-H(8)	119.8
C(3)-C(2)-H(1)	119.1	C(14)-C(15)-C(16)	120.5(3)
C(1)-C(2)-H(1)	119.1	C(14)-C(15)-H(9)	119.8
C(4)-C(3)-C(2)	119.1(3)	C(16)-C(15)-H(9)	119.8
C(4)-C(3)-H(2)	120.5	C(17)-C(16)-C(15)	119.8(3)
C(2)-C(3)-H(2)	120.5	C(17)-C(16)-H(10)	120.1
C(3)-C(4)-C(5)	121.5(3)	C(15)-C(16)-H(10)	120.1
C(3)-C(4)-H(3)	119.2	C(16)-C(17)-C(12)	120.3(3)
C(5)-C(4)-H(3)	119.2	C(16)-C(17)-H(11)	119.9
C(4)-C(5)-C(6)	119.1(3)	C(12)-C(17)-H(11)	119.9
C(4)-C(5)-C(10)	120.4(3)	C(19)-C(18)-C(23)	118.7(2)
C(6)-C(5)-C(10)	120.5(3)	C(19)-C(18)-C(11)	122.0(2)
C(7)-C(6)-C(5)	121.5(3)	C(23)-C(18)-C(11)	119.2(2)
C(7)-C(6)-H(4)	119.2	C(20)-C(19)-C(18)	120.3(3)
C(5)-C(6)-H(4)	119.2	C(20)-C(19)-H(12)	119.8
C(6)-C(7)-C(8)	118.7(3)	C(18)-C(19)-H(12)	119.8
C(6)-C(7)-H(5)	120.7	C(19)-C(20)-C(21)	120.1(3)
C(8)-C(7)-H(5)	120.7	C(19)-C(20)-H(13)	119.9
C(9)-C(8)-C(7)	122.4(3)	C(21)-C(20)-H(13)	119.9
C(9)-C(8)-H(6)	118.8	C(20)-C(21)-C(22)	120.5(3)
C(7)-C(8)-H(6)	118.8	C(20)-C(21)-H(14)	119.7
C(8)-C(9)-C(10)	119.2(2)	C(22)-C(21)-H(14)	119.7
C(8)-C(9)-C(24)	114.5(2)	C(23)-C(22)-C(21)	119.9(3)
C(10)-C(9)-C(24)	125.6(2)	C(23)-C(22)-H(15)	120.1
C(5)-C(10)-C(1)	117.3(2)	C(21)-C(22)-H(15)	120.1
C(5)-C(10)-C(9)	116.9(2)	C(22)-C(23)-C(18)	120.4(3)
C(1)-C(10)-C(9)	125.7(2)	C(22)-C(23)-H(16)	119.8
C(18)-C(11)-C(12)	123.1(2)	C(18)-C(23)-H(16)	119.8
C(18)-C(11)-C(1)	119.5(2)	C(31)-C(24)-C(25)	120.6(2)

C(31)-C(24)-C(9)	119.6(2)	C(33)-C(34)-H(24)	119.7
C(25)-C(24)-C(9)	119.3(2)	C(35)-C(34)-H(24)	119.7
C(26)-C(25)-C(30)	119.2(3)	C(36)-C(35)-C(34)	120.0(3)
C(26)-C(25)-C(24)	119.4(2)	C(36)-C(35)-H(25)	120.0
C(30)-C(25)-C(24)	121.2(2)	C(34)-C(35)-H(25)	120.0
C(27)-C(26)-C(25)	119.9(3)	C(35)-C(36)-C(31)	120.1(3)
C(27)-C(26)-H(17)	120.0	C(35)-C(36)-H(26)	120.0
C(25)-C(26)-H(17)	120.0	C(31)-C(36)-H(26)	120.0
C(26)-C(27)-C(28)	120.0(3)	O(10)-Cl(1)-O(9)	113.4(9)
C(26)-C(27)-H(18)	120.0	O(10)-Cl(1)-O(7)	125.6(9)
C(28)-C(27)-H(18)	120.0	O(9)-Cl(1)-O(7)	12.2(10)
C(27)-C(28)-C(29)	120.7(3)	O(10)-Cl(1)-O(6)	104.0(7)
C(27)-C(28)-H(19)	119.6	O(9)-Cl(1)-O(6)	109.6(6)
C(29)-C(28)-H(19)	119.6	O(7)-Cl(1)-O(6)	103.4(7)
C(30)-C(29)-C(28)	120.2(3)	O(10)-Cl(1)-O(5)	112.3(7)
C(30)-C(29)-H(20)	119.9	O(9)-Cl(1)-O(5)	108.8(6)
C(28)-C(29)-H(20)	119.9	O(7)-Cl(1)-O(5)	102.0(7)
C(29)-C(30)-C(25)	119.8(3)	O(6)-Cl(1)-O(5)	108.48(19)
C(29)-C(30)-H(21)	120.1	O(10)-Cl(1)-O(8)	24.4(9)
C(25)-C(30)-H(21)	120.1	O(9)-Cl(1)-O(8)	96.3(7)
C(36)-C(31)-C(32)	119.3(3)	O(7)-Cl(1)-O(8)	108.1(8)
C(36)-C(31)-C(24)	120.0(2)	O(6)-Cl(1)-O(8)	127.7(12)
C(32)-C(31)-C(24)	120.7(3)	O(5)-Cl(1)-O(8)	104.5(7)
C(33)-C(32)-C(31)	119.5(3)	O(4)-Cl(2)-O(1)	108.96(15)
C(33)-C(32)-H(22)	120.2	O(4)-Cl(2)-O(3)	111.18(18)
C(31)-C(32)-H(22)	120.2	O(1)-Cl(2)-O(3)	109.43(14)
C(32)-C(33)-C(34)	120.3(3)	O(4)-Cl(2)-O(2)	108.59(17)
C(32)-C(33)-H(23)	119.8	O(1)-Cl(2)-O(2)	109.88(15)
C(34)-C(33)-H(23)	119.8	O(3)-Cl(2)-O(2)	108.78(15)
C(33)-C(34)-C(35)	120.7(3)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for ichi0130. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	19(1)	19(1)	14(1)	1(1)	6(1)	-2(1)
C(2)	18(1)	23(1)	22(1)	3(1)	5(1)	-2(1)
C(3)	21(1)	28(2)	21(1)	1(1)	2(1)	-6(1)
C(4)	30(2)	22(1)	19(1)	-4(1)	5(1)	-10(1)
C(5)	24(2)	19(1)	17(1)	1(1)	6(1)	-2(1)
C(6)	35(2)	17(1)	20(1)	-2(1)	9(1)	1(1)
C(7)	28(2)	20(1)	26(2)	0(1)	10(1)	9(1)
C(8)	20(1)	23(1)	20(1)	1(1)	7(1)	2(1)
C(9)	17(1)	17(1)	15(1)	2(1)	4(1)	1(1)
C(10)	18(1)	17(1)	15(1)	2(1)	6(1)	-2(1)
C(11)	17(1)	20(1)	12(1)	2(1)	1(1)	3(1)
C(12)	16(1)	25(1)	16(1)	-2(1)	5(1)	-1(1)
C(13)	21(1)	23(2)	24(1)	-3(1)	5(1)	0(1)
C(14)	23(2)	30(2)	29(2)	-5(1)	7(1)	6(1)
C(15)	22(2)	42(2)	23(2)	-9(1)	9(1)	-3(1)
C(16)	23(2)	34(2)	22(1)	-3(1)	9(1)	-6(1)
C(17)	19(1)	25(2)	20(1)	-2(1)	4(1)	-3(1)
C(18)	16(1)	16(1)	18(1)	2(1)	5(1)	3(1)
C(19)	19(1)	20(1)	19(1)	0(1)	6(1)	3(1)
C(20)	22(1)	19(1)	29(2)	-1(1)	6(1)	0(1)
C(21)	24(2)	21(1)	31(2)	5(1)	11(1)	1(1)
C(22)	26(2)	23(1)	21(1)	3(1)	12(1)	4(1)
C(23)	21(1)	18(1)	19(1)	0(1)	6(1)	3(1)
C(24)	20(1)	16(1)	16(1)	3(1)	2(1)	5(1)
C(25)	17(1)	23(1)	15(1)	2(1)	2(1)	0(1)
C(26)	25(2)	24(2)	20(1)	1(1)	3(1)	-1(1)
C(27)	32(2)	31(2)	22(2)	9(1)	8(1)	-4(1)
C(28)	32(2)	44(2)	18(1)	3(1)	10(1)	-3(1)
C(29)	29(2)	31(2)	19(1)	-3(1)	5(1)	-2(1)
C(30)	22(1)	22(1)	18(1)	1(1)	3(1)	0(1)
C(31)	17(1)	17(1)	21(1)	1(1)	4(1)	3(1)
C(32)	25(2)	24(2)	21(1)	1(1)	4(1)	1(1)
C(33)	23(2)	26(2)	31(2)	-4(1)	1(1)	-2(1)

C(34)	24(2)	23(2)	42(2)	0(1)	11(1)	-5(1)
C(35)	27(2)	21(1)	29(2)	1(1)	11(1)	1(1)
C(36)	22(1)	20(1)	23(1)	1(1)	6(1)	4(1)
Cl(1)	42(1)	35(1)	30(1)	-4(1)	9(1)	-3(1)
Cl(2)	24(1)	22(1)	22(1)	-1(1)	8(1)	1(1)
O(1)	26(1)	33(1)	39(1)	-1(1)	5(1)	9(1)
O(2)	44(2)	37(1)	42(1)	11(1)	12(1)	-10(1)
O(3)	25(1)	33(1)	57(2)	-1(1)	-7(1)	-2(1)
O(4)	84(2)	45(2)	35(1)	0(1)	35(1)	19(2)
O(5)	74(2)	46(2)	50(2)	-13(1)	25(2)	-1(1)
O(6)	91(2)	55(2)	63(2)	0(2)	44(2)	-14(2)
O(7)	28(5)	111(11)	38(5)	-28(5)	8(4)	-17(5)
O(8)	81(9)	80(8)	57(7)	-16(6)	-39(6)	52(6)
O(9)	165(12)	19(4)	77(7)	-23(3)	91(8)	-40(5)
O(10)	75(6)	194(13)	107(8)	78(10)	52(7)	60(8)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for ichi0130.

	x	y	z	U(eq)
H(1)	6541	1552	8670	25
H(2)	6855	3369	9441	29
H(3)	5766	4792	9477	28
H(4)	4290	5418	9143	29
H(5)	2852	4960	8635	29
H(6)	2470	3132	7881	25
H(7)	5853	-1675	7727	27
H(8)	6771	-2113	6940	33
H(9)	7112	-535	6150	34
H(10)	6533	1506	6140	31
H(11)	5710	2016	7002	26
H(12)	4379	-1548	7260	23
H(13)	3505	-2970	7695	28
H(14)	3183	-2646	8886	30
H(15)	3779	-924	9671	27
H(16)	4633	531	9238	23
H(17)	4053	3620	6717	28
H(18)	4518	3778	5586	34
H(19)	4543	1986	4831	37
H(20)	4092	29	5186	32
H(21)	3506	-123	6247	25
H(22)	2135	913	6011	28
H(23)	897	-266	5969	33
H(24)	594	-959	7111	35
H(25)	1530	-497	8309	30
H(26)	2729	782	8376	26

Table 6. Torsion angles [°] for ichi0130.

C(10)-C(1)-C(2)-C(3)	2.5(4)	C(14)-C(15)-C(16)-C(17)	2.8(4)
C(11)-C(1)-C(2)-C(3)	-170.2(2)	C(15)-C(16)-C(17)-C(12)	-4.0(4)
C(1)-C(2)-C(3)-C(4)	-2.0(4)	C(13)-C(12)-C(17)-C(16)	2.3(4)
C(2)-C(3)-C(4)-C(5)	1.6(4)	C(11)-C(12)-C(17)-C(16)	-179.7(2)
C(3)-C(4)-C(5)-C(6)	176.3(3)	C(12)-C(11)-C(18)-C(19)	-27.1(4)
C(3)-C(4)-C(5)-C(10)	-1.7(4)	C(1)-C(11)-C(18)-C(19)	158.9(2)
C(4)-C(5)-C(6)-C(7)	-173.4(3)	C(12)-C(11)-C(18)-C(23)	151.8(2)
C(10)-C(5)-C(6)-C(7)	4.6(4)	C(1)-C(11)-C(18)-C(23)	-22.2(4)
C(5)-C(6)-C(7)-C(8)	-4.4(4)	C(23)-C(18)-C(19)-C(20)	0.4(4)
C(6)-C(7)-C(8)-C(9)	-2.6(4)	C(11)-C(18)-C(19)-C(20)	179.4(3)
C(7)-C(8)-C(9)-C(10)	9.1(4)	C(18)-C(19)-C(20)-C(21)	0.0(4)
C(7)-C(8)-C(9)-C(24)	-162.0(3)	C(19)-C(20)-C(21)-C(22)	-1.1(4)
C(4)-C(5)-C(10)-C(1)	2.1(4)	C(20)-C(21)-C(22)-C(23)	1.7(4)
C(6)-C(5)-C(10)-C(1)	-175.9(2)	C(21)-C(22)-C(23)-C(18)	-1.3(4)
C(4)-C(5)-C(10)-C(9)	179.9(2)	C(19)-C(18)-C(23)-C(22)	0.2(4)
C(6)-C(5)-C(10)-C(9)	2.0(4)	C(11)-C(18)-C(23)-C(22)	-178.8(2)
C(2)-C(1)-C(10)-C(5)	-2.4(4)	C(8)-C(9)-C(24)-C(31)	-49.9(3)
C(11)-C(1)-C(10)-C(5)	169.2(2)	C(10)-C(9)-C(24)-C(31)	139.6(3)
C(2)-C(1)-C(10)-C(9)	179.9(2)	C(8)-C(9)-C(24)-C(25)	121.9(3)
C(11)-C(1)-C(10)-C(9)	-8.4(4)	C(10)-C(9)-C(24)-C(25)	-48.7(4)
C(8)-C(9)-C(10)-C(5)	-8.5(4)	C(31)-C(24)-C(25)-C(26)	138.7(3)
C(24)-C(9)-C(10)-C(5)	161.6(2)	C(9)-C(24)-C(25)-C(26)	-32.9(4)
C(8)-C(9)-C(10)-C(1)	169.1(3)	C(31)-C(24)-C(25)-C(30)	-36.8(4)
C(24)-C(9)-C(10)-C(1)	-20.8(4)	C(9)-C(24)-C(25)-C(30)	151.6(2)
C(2)-C(1)-C(11)-C(18)	117.9(3)	C(30)-C(25)-C(26)-C(27)	1.8(4)
C(10)-C(1)-C(11)-C(18)	-54.2(4)	C(24)-C(25)-C(26)-C(27)	-173.8(3)
C(2)-C(1)-C(11)-C(12)	-56.5(3)	C(25)-C(26)-C(27)-C(28)	-2.7(4)
C(10)-C(1)-C(11)-C(12)	131.4(3)	C(26)-C(27)-C(28)-C(29)	0.5(5)
C(18)-C(11)-C(12)-C(13)	-29.0(4)	C(27)-C(28)-C(29)-C(30)	2.7(5)
C(1)-C(11)-C(12)-C(13)	145.1(3)	C(28)-C(29)-C(30)-C(25)	-3.5(4)
C(18)-C(11)-C(12)-C(17)	153.0(3)	C(26)-C(25)-C(30)-C(29)	1.3(4)
C(1)-C(11)-C(12)-C(17)	-32.8(3)	C(24)-C(25)-C(30)-C(29)	176.8(3)
C(17)-C(12)-C(13)-C(14)	0.7(4)	C(25)-C(24)-C(31)-C(36)	158.5(2)
C(11)-C(12)-C(13)-C(14)	-177.3(3)	C(9)-C(24)-C(31)-C(36)	-29.9(4)
C(12)-C(13)-C(14)-C(15)	-1.9(4)	C(25)-C(24)-C(31)-C(32)	-22.4(4)
C(13)-C(14)-C(15)-C(16)	0.2(4)	C(9)-C(24)-C(31)-C(32)	149.3(3)

C(36)-C(31)-C(32)-C(33)	-0.7(4)
C(24)-C(31)-C(32)-C(33)	-179.9(3)
C(31)-C(32)-C(33)-C(34)	-0.7(4)
C(32)-C(33)-C(34)-C(35)	0.0(5)
C(33)-C(34)-C(35)-C(36)	2.0(5)
C(34)-C(35)-C(36)-C(31)	-3.4(4)
C(32)-C(31)-C(36)-C(35)	2.8(4)
C(24)-C(31)-C(36)-C(35)	-178.1(3)

Symmetry transformations used to generate equivalent atoms: