Electronic Supplementary Information for

Oxidative Functionalization of Benzylic C-H Bonds by DDQ

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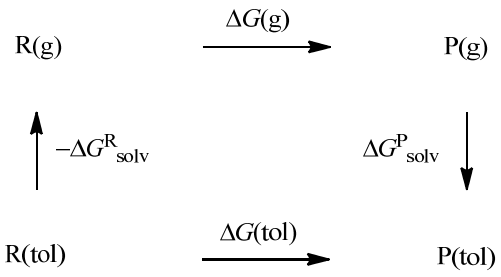
S1: Electronic Structure Calculations

As in previous studies,[1,2,3] we applied density functional theory to calculate free energies and characterize the structural and spin/electronic properties of reaction intermediates. We used the BH&H hybrid density functional (50% Hartree-Fock exchange and 50% LSDA exchange) implemented in Gaussian 09[4] because it is known to be capable of correctly reproducing π-stacking geometries and interactions.[1,5,6] All minimum energy structures and free-energy changes were calculated at the BH&H/6-311++G(d,p) level.

Changes in the free energy in toluene ΔG(tol) from reactants R to products P were found using the thermodynamic cycle:



where:

$$\Delta G(\text{tol}) = \Delta G(\text{g}) + \Delta G^{\text{P}}_{\text{solv}} - \Delta G^{\text{R}}_{\text{solv}},$$

and $\Delta G(\text{g}) = \Delta H(\text{g}) - T\Delta S(\text{g})$ is the free energy state transition in the gas phase. The solvation free energies ΔG_{solv} used to correct the gas phase free energies were calculated using the Polarizable Continuum Model of Gaussian 09 (dielectric constant of $\varepsilon = 2.37$ for toluene).[4]

Table S1. Atomic coordinates of the calculated minimum energy reaction intermediate structures referred to in Figure 3 of the main text.

1:1 Complex

C	0.298801	-1.095765	-1.091020
C	-0.722775	-0.039392	-1.250624
C	-0.469794	1.228635	-0.921922
C	0.853312	1.637258	-0.404368
C	1.862113	0.575456	-0.222487
C	1.604480	-0.690491	-0.537780
O	0.068540	-2.217092	-1.412783
O	1.088312	2.776690	-0.163823
Cl	3.326672	1.080332	0.439837
Cl	2.710284	-1.941302	-0.309631
C	-1.683477	-1.379143	1.320622
C	-2.662898	-0.398853	1.204376
C	-2.413355	0.896299	1.580750
C	-1.170901	1.246222	2.082221
C	-0.201810	0.278898	2.234913
C	-0.458185	-1.022092	1.855082
H	-3.625693	-0.664928	0.786149
H	-3.178432	1.651313	1.461244
H	-0.967045	2.271230	2.361547
H	0.765018	0.540438	2.646754
H	0.307605	-1.780539	1.971018
N	-2.198695	3.081352	-1.224118
C	-1.425915	2.257466	-1.084956
N	-2.950277	-0.806536	-2.232316
C	-1.960869	-0.457142	-1.791184
C	-1.955597	-2.768025	0.876719
H	-2.252028	-2.789341	-0.174251
H	-1.077381	-3.401175	0.986741
H	-2.771100	-3.206967	1.453110

TS - Hydride Mechanism Transition State

C	0.097714	-0.887367	-0.992469
C	-0.763601	0.224574	-1.116953
C	-0.395447	1.452518	-0.659774
C	0.963498	1.715014	-0.188291
C	1.870557	0.567093	-0.210238
C	1.448517	-0.658398	-0.557234
O	-0.276545	-2.044574	-1.376668
O	1.307960	2.799831	0.187661
Cl	3.446720	0.870210	0.317109
Cl	2.455599	-2.019872	-0.476848
C	-1.607026	-1.663278	1.140347
C	-2.656388	-0.708440	1.097214
C	-2.457735	0.560261	1.526855
C	-1.194511	0.956390	1.982956
C	-0.175049	0.034988	2.104177

C	-0.360764	-1.245580	1.655475
H	-3.612528	-1.008627	0.689353
H	-3.253609	1.289242	1.463986
H	-1.031820	1.983331	2.283797
H	0.785262	0.341179	2.496958
H	0.439744	-1.972341	1.724084
N	-2.015876	3.420746	-0.743260
C	-1.278600	2.552193	-0.719284
N	-3.054281	-0.218427	-2.160965
C	-2.038594	-0.008921	-1.690546
C	-1.737261	-2.913995	0.563707
H	-1.002181	-2.488635	-0.699997
H	-1.016244	-3.687538	0.798852
H	-2.700120	-3.232969	0.184501

TS - Radical Mechanism Transition State

C	-0.558430	1.346837	-0.798783
C	0.520770	0.580263	-1.282713
C	0.493772	-0.800520	-1.245457
C	-0.633278	-1.507773	-0.721461
C	-1.785034	-0.703831	-0.365011
C	-1.748709	0.645439	-0.412169
O	-0.441029	2.578511	-0.600322
O	-0.607059	-2.716804	-0.547659
Cl	-3.132524	-1.546265	0.209739
Cl	-3.051089	1.599959	0.102918
C	1.530565	1.046406	1.498164
C	2.695302	0.366994	1.078993
C	2.805542	-0.980795	1.238525
C	1.752075	-1.703917	1.791823
C	0.586511	-1.059931	2.198604
C	0.472711	0.289214	2.054714
H	3.487883	0.938092	0.614097
H	3.692681	-1.498098	0.902608
H	1.828351	-2.778655	1.881595
H	-0.230420	-1.637173	2.608856
H	-0.429764	0.805286	2.358739
N	2.563023	-2.167607	-1.824610
C	1.624052	-1.563105	-1.589583
N	2.632344	1.837529	-1.969918
C	1.680285	1.276732	-1.689337
C	1.398624	2.455032	1.329980
H	0.683987	2.618766	0.373210
H	0.780942	2.948227	2.080059
H	2.316357	2.985377	1.093481

Ion Pair

C	0.198908	0.769222	-1.119828
C	1.506595	0.446401	-0.750260
C	1.847507	-0.803434	-0.291936
C	0.858013	-1.833963	-0.108304
C	-0.480843	-1.450445	-0.534983

C	-0.774664	-0.225804	-1.057919
O	-0.145182	1.971561	-1.556275
O	1.104357	-2.920991	0.362197
Cl	-1.664423	-2.655118	-0.410278
Cl	-2.334543	0.157066	-1.630573
C	-1.018922	2.038876	1.247195
C	0.264912	1.567572	1.625817
C	0.425358	0.305795	2.120105
C	-0.663789	-0.543781	2.168242
C	-1.958087	-0.082520	1.887603
C	-2.135361	1.175492	1.429338
H	1.111542	2.236835	1.530764
H	1.404844	-0.060956	2.393309
H	-0.517670	-1.576234	2.462097
H	-2.794774	-0.759893	1.978800
H	-3.118171	1.534528	1.154981
N	4.235118	-1.252000	0.475390
C	3.165686	-1.072219	0.120657
N	3.038184	2.474687	-0.940615
C	2.427942	1.515411	-0.841205
C	-1.149115	3.223731	0.606000
H	-2.109145	3.557869	0.236794
H	-0.298640	3.876912	0.465866
H	0.633077	2.512459	-1.722077

Radical Pair

C	-0.421887	-0.600179	-1.360708
C	-1.521493	0.135103	-0.914663
C	-1.350786	1.350893	-0.300699
C	-0.036332	1.906933	-0.110607
C	1.071605	1.117548	-0.615107
C	0.878650	-0.083229	-1.217837
O	-0.550127	-1.772465	-1.916517
O	0.130433	2.973298	0.443882
Cl	2.608861	1.765722	-0.404619
Cl	2.166015	-1.015695	-1.804491
C	-0.484374	-1.868732	1.525717
C	-0.695033	-0.648018	2.195024
C	0.359554	0.134305	2.580615
C	1.661940	-0.269361	2.325793
C	1.894137	-1.469656	1.676985
C	0.845670	-2.255598	1.278259
H	-1.711319	-0.332995	2.399457
H	0.176704	1.075176	3.082581
H	2.490736	0.355021	2.628552
H	2.908557	-1.783673	1.471956
H	1.028515	-3.187466	0.758417
N	-3.353949	2.630825	0.613087
C	-2.449230	2.072287	0.201545
N	-3.753131	-1.042213	-1.280517
C	-2.788225	-0.464675	-1.091285
C	-1.553941	-2.652569	1.103062

H	-1.381669	-3.599163	0.609411
H	-2.575252	-2.356170	1.298005
H	-1.463588	-2.078452	-1.897000

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S2: General Experimental Considerations

DDQ, triphenylphosphine and triethylphosphite were obtained from commercial sources and used as received. Liquid arylmethanes were stored over 4 Å molecular sieves prior to use. Toluene, diethyl ether and pentane were dried with a solvent purification system using a 1 m column containing activated alumina. Deuterated tetrachloroethane was distilled from CaH_2 and stored over 4 Å molecular sieves prior to use. All other deuterated solvents were used as received. ^{1}H and ^{13}C NMR spectra were taken on 400 and 500 MHz Bruker and 500 MHz Varian spectrometers.

S3: Experimental Procedures and Characterization

(**1a**)**:** To a flame dried 100 mL Schlenk flask with a magnetic stirrer under argon was added DDQ (2.27 mg, 10.0 mmol) and toluene (20 mL). The reaction mixture was heated to 110 °C with stirring. After 16 h of heating at 110 °C, the reaction mixture was allowed to cool. The reaction mixture was filtered and the obtained solid washed with cold diethyl ether to give a beige solid 2.49 g, 78 % yield). ^{13}C NMR (d_6-DMSO, 125 MHz): δ 154.8, 150.1, 135.1, 134.2, 129.0, 128.9, 128.6, 113.7, 113.1, 109.0, 101.9, 101.7, 77.0 ppm. ^{1}H NMR (CD_2Cl_2, 400 MHz): δ 7.52 (m, 2H, Ar-C*H*); 7.43 (m, 3H, Ar-*CH*); 5.19 (s, 2H, Ar-CH_2-O) ppm. HRMS: 319.00 [M+H].

Method A: To a flame-dried 20 mL Schlenk flask under an atmosphere of argon is added an arylmethane (2 mL), then DDQ was added (0.1 equivalent *versus* the arylmethane) and the reaction was heated to 110 °C. After completion the reaction it was cooled to room temperature and the solid collected by filtration. The solids were washed with petroleum ether to give pure product as demonstrated by ^{1}H and ^{13}C NMR.

Method B: To a flame-dried pressure tube under an argon atmosphere, the arylmethane (2 mmol) was dissolved in chlorobenzene. DDQ was then added (681 mg, 3 mmol), the reaction vessel sealed with a PTFE lined screw cap, and the mixture heated to 140 °C. After completion the reaction was cooled to room temperature, and the solid collected by filtration. The solid was washed with petroleum ether to give pure product as demonstrated by ^{1}H and ^{13}C NMR.

(**1b**). Method B: 397.2 mg, 67 % yield. ^{13}C NMR (d_6-DMSO, 125 MHz): δ 151.3, 150.3, 141.1, 140.0, 134.7, 130.0, 129.7, 129.4, 128.1, 127.3, 127.2, 114.2, 113.7, 109.3, 101.7, 76.7 ppm. ^{1}H NMR (d_6-DMSO, 500 MHz): δ 7.75 (m, 2H, Ar-C*H*); 7.70 (m, 2H, Ar-C*H*); 7.61 (m, 2H, Ar-C*H*); 7.48 (m, 2H, Ar-*CH*); 7.39 (m, 1H, Ar-C*H*); 5.16 (s, 2H, Ar-CH_2-O) ppm. HRMS unable to observe m/z due to hydrolysis.

(**1c**)**.** Method A: 388.4 mg, 65 % yield. Method B: 432.5 mg, 60% yield. ^{13}C NMR (d_6-DMSO, 126 MHz): δ 150.8, 150.0, 134.1, 133.6, 129.1, 128.6, 113.5, 113.1, 109.0, 101.9, 76.1 ppm. ^{1}H NMR (d_6-DMSO, 400 MHz): δ 7.51 (m, 4H, Ar-C*H*); 5.13 (s, 2H, Ar-CH_2-O) ppm. HRMS 352.96 $[M+H]^+$.

(**1d**). Method A: a beige solid, 614.1 mg, 81% yield. ^{13}C NMR (d_6-DMSO, 126 MHz): δ 154.8, 151.4, 150.8, 150.2, 134.1, 132.1, 129.1, 128.7, 125.3, 113.7, 108.9, 101.7, 76.9, 34.4, 31.1 ppm. ^{1}H NMR (d_6-DMSO, 400 MHz): δ 7.44 (m, 4H, Ar-C*H*); 5.08 (s, 2H, Ar-CH_2-O), 1.29 (s, 9H, -C(CH_3)$_3$) ppm. HRMS 375.06 $[M+H]^+$.

(**1e**). Method B: 266.1 mg, 40% yield. ^{13}C NMR (d_6-DMSO, 126 MHz): δ 171.9, 154.5, 150.2, 136.7, 133.5, 131.4 (d, J_{CF} = 8.7 Hz, C(3)), 117.8, 117.2, 115.4 (d, J_{CF} = 21.5 Hz, C(2)), 109.1, 102.0, 76.2, 70.7. ^{1}H NMR (d_6-DMSO, 400 MHz): δ 7.55 (m, 2H, Ar-C*H*); 7.26 (m, 2H, Ar-C*H*); 5.14 (s, 2H, Ar-CH_2-O) ppm. HRMS 336.99 $[M+H]^+$.

(**1f**)**.** Method A: 412.6 mg, 68 % yield. ^{13}C NMR (d_6-DMSO, 125 MHz): δ 162.1 (d, J_{CF} = 244 Hz, C(3)-F), 154.9, 149.9, 137.8 (d, J_{CF} = 7.6 Hz, C(1)$_{ipso}$), 134.1, 130.6 (d, J_{CF} = 8.2 Hz, C(5)), 129.2, 124.7 (d, J_{CF} = 2.8 Hz, C(6)), 115.6 (d, J_{CF} = 20.9 Hz), 115.3 (d, *J*CF = 21.7 Hz), 113.5, 113.0, 108.9, 101.8, 76.0 ppm. ^{1}H NMR (CD_2Cl_2, 400 MHz): δ 7.42 (m, 1H, Ar-C*H*); 7.28 (m, 2H, Ar-C*H*); 7.12 (m, 1H, Ar-C*H*); 5.18 (s, 2H, Ar-CH_2-O) ppm. HRMS 336.99 $[M+H]^+$.

Benzyl(triphenyl)phosphonium 2,3-dichloro-5,6-dicyanohydroquinolate (**2**). To a flame-dried 25 mL round-bottomed Schlenk flask under an atmosphere of argon was added **1** (319 mg, 1 mmol) and triphenylphoshine (262 mg, 1.41 mmol). The solids were dissolved in mesitylene (5

mL), a reflux condenser was attached to the flask and the mixture was heated to reflux for 3 hours. After cooling to room temperature, the reaction was filtered washing with pentane. The solid was dissolved in acetone and the pure product precipated with diethyl ether, and filtered to give **2** as a brown solid (540 mg, 93% yield). ^{13}C NMR (d_6-DMSO, 125 MHz): δ 135.1 (d, J_{CP} = 3.1 Hz), 134.0 (d, J_{CP} = 9.8 Hz), 130.8 (d, J_{CP} = 5.6 Hz), 130.1 (d, J_{CP} = 12.3 Hz), 129.5, 128.8 (d, J_{CP} = 3.3 Hz) 128.3 (d, J_{CP} = 3.9 Hz), 127.9 (d, J_{CP} = 8.6 Hz), 123.1, 117.8 (d, J_{CP} = 86 Hz), 114.1, 101.23, 28.1 (d, J_{CP} = 47 Hz) ppm. ^{1}H NMR (CD_2Cl_2, 400 MHz): δ 7.90 (m, 3H, Ar-C*H*); 7.75-7.65 (m, 12H, Ar-C*H*); 7.29 (m, 1H, Ar-C*H*); 7.22 (m, 2H, Ar-C*H*); 6.98 (m, 2H, Ar-*C*H), 5.20 (d, 2H, J_{PH} = 15.7 Hz, Ar-CH_2-P) ppm.

Synthesis of diethyl benzylphosphonic ester from **1a**. To a flame-dried 25 mL round-bottomed flask was added **1a** (323.1 mg, 1.01 mmol) and 2 mL of triethyl phosphite. A reflux condenser was attached to the flask and the mixture heated to reflux for 18 h. After cooling the reaction to room temperature, the excess triethyl phosphite was removed *via* vacuum distillation to give a brown oil. The crude material was then purified by column chromatography, eluting with 1:1 hexanes/ethyl acetate, to give a colourless oil (215 mg, 93% yield).

Representative example of a competition experiment used to determine the linear free energy relationship (see Figure 2). DDQ (4.7 mg, 0.021 mmol) was added to an oven-dried NMR tube and then purged with argon gas for 10 minutes. Toluene (22.0 µL, 0.21 mmol) and *p*-chlorotoluene (24.5 µL, 0.21 mmol) were then added *via* microsyringe, and all of the reagents dissolved in 0.7 mL of $C_2D_2Cl_4$. The reaction mixture was heated to 105 °C under an atmosphere of argon for 18 h, and then analyzed by 1H NMR. The ratio of products was 1:1.47 for *p*-H *versus* *p*-Cl.

Determination of the deuterium kinetic isotope effect. DDQ (54.3 mg, 0.239 mmol) was added to an oven dried 4-dram vial with a magnetic stirrer. The reaction vessel was purged with argon for 10 minutes. Toluene (0.25 mL, 2.35 mmol) and d_8-toluene (4.75 mL, 44.7 mmol) were then added and the reaction mixture heated to 110 °C for 4 days. The resulting mixture was transferred to a round-bottomed flask and concentrated. The resulting crude material was dissolved in a 1:1 mixture of DMSO and d_6-DMSO, and nitromethane and d_3-nitromethane added as internal standards. The amount of protiated and deuterated products was then determined and used to determine a DKIE of 5.2 ± 0.1 (average of two runs).

S4. X-ray Structure 1a

Data Collection

A colorless prism crystal of $O_2C_{15}H_8N_2Cl_2$ having approximate dimensions of 0.30 x 0.10 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku Mercury275R CCD (SCX mini) diffractometer using filtered Mo-Kα radiation. The crystal-to-detector distance was 49.90 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 12.036(5) Å
b = 23.769(9) Å β = 99.954(6)°
c = 10.114(4) Å
V = 2850.1(18) Å^3

For Z = 8 and F.W. = 319.15, the calculated density is 1.487 g/cm^3. The reflection conditions of:

h0l: l = 2n

0k0: k = 2n

uniquely determine the space group to be: $P2_1/c$ (#14).

The data were collected at a temperature of -50 °C to a maximum 2θ value of 50.1°. A total of 360 oscillation images were collected. A sweep of data was done using ω scans from -120.0 to 60.0° in 1.0° step, at χ = 54.0° and φ = 0.0°. The exposure rate was 60.0 [sec./°]. The detector swing angle was -28.40°. A second sweep was performed using ω scans from -120.0 to 60.0° in 1.0° step, at χ = 54.0° and φ = 120.0°. The exposure rate was 60.0 [sec./°]. The detector swing angle was -28.40°. The crystal-to-detector distance was 49.90 mm. Readout was performed in the 0.146 mm pixel mode.

Data Reduction

Of the 15658 reflections that were collected, 5016 were unique (R_{int} = 0.0419). Data were collected and processed using CrystalClear (Rigaku).

The linear absorption coefficient, μ, for Mo-Kα radiation is 4.590 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.651 to 0.955. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The positions of phenolic H atoms H1 and H3 were determined using a rotating group refinement. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 5007 of the 5016 observed reflections and 381 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0568$$

$$wR2 = [\Sigma (w (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1481$$

The standard deviation of an observation of unit weight[4] was 1.04. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.26 and -0.40 $e^-/Å^3$, respectively. Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL-97[10].

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(3) Least Squares function minimized: (SHELXL97)

$$\Sigma w(F_o^2-F_c^2)^2$$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$$

where N_o = number of observations
N_v = number of variables

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(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$O_2C_{15}H_8N_2Cl_2$
Formula Weight	319.15
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.30 X 0.10 X 0.10 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 12.036(5) Å b = 23.769(9) Å c = 10.114(4) Å β = 99.954(6) ° V = 2850.1(18) Å^3
Space Group	$P2_1/c$ (#14)
Z value	8
D_{calc}	1.487 g/cm^3
F_{000}	1296.00
μ(MoKα)	4.590 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku Mercury275R CCD (SCX mini)
Radiation	MoKα (λ = 0.71075 Å)
Voltage, Current	50kV, 40mA
Temperature	-50.0°C
Detector Aperture	75 mm (diameter)
Data Images	360 exposures
ω oscillation Range (χ=54.0, φ =0.0)	-120.0 - 60.0°
Exposure Rate	60.0 sec./°
Detector Swing Angle	-28.40°
ω oscillation Range (χ=54.0, φ=120.0)	-120.0 - 60.0°
Exposure Rate	60.0 sec./°
Detector Swing Angle	-28.40°
Detector Position	49.90 mm
Pixel Size	0.146 mm
$2\theta_{max}$	50.1°
No. of Reflections Measured	Total: 15658 Unique: 5016 (R_{int} = 0.0419)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.651 - 0.955)

C. Structure Solution and Refinement

Structure Solution	Direct Methods

Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\sigma^2(Fo^2) + (0.0712 \cdot P)^2 + 0.8456 \cdot P]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	50.1^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	5007
No. Variables	381
Reflection/Parameter Ratio	13.14
Residuals: R1 (I>2.00σ(I))	0.0568
Residuals: R (All reflections)	0.0906
Residuals: wR2 (All reflections)	0.1481
Goodness of Fit Indicator	1.045
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.26 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.40 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Cl(1)	0.60146(9)	-0.02921(4)	0.66364(12)	6.56(3)
Cl(2)	0.72036(10)	0.05807(4)	0.50650(12)	6.48(3)
Cl(3)	0.87585(8)	0.22498(4)	-0.19684(9)	5.04(2)
Cl(4)	0.75687(9)	0.31275(3)	-0.04265(10)	5.51(2)
O(1)	0.68703(19)	-0.14570(8)	0.6474(2)	4.19(5)
O(2)	0.90921(18)	0.01071(9)	0.3903(2)	3.93(4)
O(3)	0.8020(2)	0.10694(9)	-0.1616(2)	4.76(5)
O(4)	0.60379(18)	0.26255(9)	0.1256(2)	4.04(4)
N(1)	0.8717(3)	-0.22799(13)	0.5159(4)	6.44(8)
N(2)	1.0313(3)	-0.11203(14)	0.3183(4)	6.35(8)
N(3)	0.6422(3)	0.02378(12)	0.0055(3)	5.24(7)
N(4)	0.4856(3)	0.14063(13)	0.2036(3)	5.94(8)
C(1)	0.7367(3)	-0.10655(12)	0.5848(3)	3.34(6)
C(2)	0.7070(3)	-0.04962(13)	0.5815(3)	3.73(6)
C(3)	0.7605(3)	-0.01125(12)	0.5126(3)	3.71(6)
C(4)	0.8485(3)	-0.02722(12)	0.4479(3)	3.35(6)
C(5)	0.8768(3)	-0.08352(13)	0.4494(3)	3.42(6)
C(6)	0.8220(3)	-0.12323(12)	0.5175(3)	3.29(6)
C(7)	0.8506(3)	-0.18189(15)	0.5177(3)	4.20(7)
C(8)	0.9640(3)	-0.10024(14)	0.3782(3)	4.28(7)
C(9)	0.8651(4)	0.02351(19)	0.2524(4)	6.32(10)
C(10)	0.9491(3)	0.05998(15)	0.1993(3)	4.45(7)
C(11)	1.0387(4)	0.03597(16)	0.1522(4)	5.37(8)
C(12)	1.1147(4)	0.06911(18)	0.1003(4)	5.98(9)
C(13)	1.1003(4)	0.12686(17)	0.0954(4)	5.55(9)
C(14)	1.0139(4)	0.15035(16)	0.1439(4)	5.40(8)
C(15)	0.9390(3)	0.11749(16)	0.1966(4)	5.19(8)
C(16)	0.7546(3)	0.14655(13)	-0.0972(3)	3.39(6)
C(17)	0.7780(3)	0.20399(13)	-0.1038(3)	3.46(6)
C(18)	0.7259(3)	0.24262(12)	-0.0337(3)	3.55(6)
C(19)	0.6496(3)	0.22569(12)	0.0473(3)	3.46(6)
C(20)	0.6262(3)	0.16892(13)	0.0541(3)	3.39(6)
C(21)	0.6785(3)	0.12945(12)	-0.0173(3)	3.24(6)
C(22)	0.6578(3)	0.06981(15)	-0.0063(3)	3.94(6)
C(23)	0.5481(3)	0.15163(14)	0.1382(3)	4.14(6)
C(24)	0.5013(3)	0.28957(17)	0.0581(3)	5.65(9)
C(25)	0.4501(3)	0.32101(14)	0.1601(3)	3.98(6)
C(26)	0.3765(3)	0.29492(15)	0.2296(4)	4.94(8)
C(27)	0.3255(3)	0.32407(18)	0.3202(4)	5.80(9)
C(28)	0.3487(4)	0.38044(18)	0.3411(4)	5.57(9)
C(29)	0.4212(3)	0.40657(15)	0.2735(4)	5.23(8)
C(30)	0.4717(3)	0.37766(15)	0.1832(4)	4.72(7)

$$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H(1)	0.6415	-0.1306	0.6894	5.03
H(3)	0.8555	0.1207	-0.1926	5.72
H(9A)	0.7929	0.0432	0.2456	7.59
H(9B)	0.8527	-0.0113	0.1999	7.59
H(11)	1.0481	-0.0033	0.1555	6.44
H(12)	1.1759	0.0526	0.0683	7.18
H(13)	1.1509	0.1497	0.0584	6.66
H(14)	1.0050	0.1896	0.1415	6.48
H(15)	0.8797	0.1346	0.2314	6.22
H(24A)	0.4483	0.2612	0.0144	6.78
H(24B)	0.5189	0.3155	-0.0107	6.78
H(26)	0.3605	0.2565	0.2151	5.93
H(27)	0.2753	0.3057	0.3674	6.96
H(28)	0.3141	0.4007	0.4026	6.68
H(29)	0.4370	0.4450	0.2886	6.28
H(30)	0.5215	0.3965	0.1363	5.67

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	0.0912(8)	0.0616(6)	0.1167(9)	0.0060(6)	0.0744(7)	-0.0046(5)
Cl(2)	0.0935(8)	0.0387(5)	0.1262(9)	0.0042(5)	0.0526(7)	0.0034(5)
Cl(3)	0.0666(6)	0.0665(6)	0.0677(6)	-0.0125(5)	0.0383(5)	0.0018(4)
Cl(4)	0.0972(8)	0.0408(5)	0.0781(6)	-0.0083(5)	0.0342(5)	0.0005(4)
O(1)	0.0593(14)	0.0468(13)	0.0610(14)	-0.0086(12)	0.0326(11)	0.0030(10)
O(2)	0.0506(12)	0.0559(13)	0.0436(12)	-0.0145(11)	0.0099(10)	0.0103(10)
O(3)	0.0680(16)	0.0502(13)	0.0746(16)	-0.0034(12)	0.0452(13)	-0.0108(11)
O(4)	0.0591(14)	0.0519(13)	0.0444(12)	0.0140(12)	0.0147(10)	-0.0055(10)
N(1)	0.088(3)	0.0510(20)	0.107(3)	0.0154(19)	0.020(2)	-0.0013(18)
N(2)	0.075(2)	0.082(2)	0.100(3)	0.0046(19)	0.058(2)	-0.0055(19)
N(3)	0.074(2)	0.0458(18)	0.080(2)	-0.0073(17)	0.0160(17)	0.0065(15)
N(4)	0.086(2)	0.077(2)	0.076(2)	-0.0083(19)	0.0529(19)	0.0059(17)
C(1)	0.0417(17)	0.0438(18)	0.0446(17)	-0.0059(15)	0.0169(14)	-0.0008(13)
C(2)	0.0482(18)	0.0458(18)	0.0534(18)	-0.0005(16)	0.0244(15)	-0.0061(14)
C(3)	0.0507(19)	0.0357(17)	0.0571(19)	-0.0041(16)	0.0163(16)	-0.0011(14)
C(4)	0.0426(17)	0.0418(18)	0.0442(17)	-0.0070(15)	0.0109(14)	0.0002(13)
C(5)	0.0398(17)	0.0516(19)	0.0413(16)	-0.0030(16)	0.0148(13)	-0.0027(14)
C(6)	0.0419(17)	0.0396(17)	0.0448(17)	-0.0011(15)	0.0112(14)	-0.0024(13)
C(7)	0.050(2)	0.048(2)	0.065(2)	0.0023(18)	0.0192(16)	-0.0024(16)
C(8)	0.0514(20)	0.052(2)	0.064(2)	0.0002(17)	0.0251(18)	0.0010(16)
C(9)	0.076(3)	0.103(3)	0.055(2)	-0.031(2)	-0.0028(19)	0.024(2)
C(10)	0.065(2)	0.066(2)	0.0378(17)	-0.016(2)	0.0080(16)	0.0113(15)
C(11)	0.097(3)	0.052(2)	0.057(2)	-0.010(2)	0.019(2)	0.0023(17)
C(12)	0.088(3)	0.083(3)	0.064(2)	0.000(3)	0.033(2)	-0.005(2)
C(13)	0.081(3)	0.075(3)	0.058(2)	-0.024(2)	0.022(2)	0.0107(19)
C(14)	0.088(3)	0.052(2)	0.065(2)	-0.005(2)	0.015(2)	0.0098(18)
C(15)	0.070(2)	0.073(3)	0.056(2)	-0.000(2)	0.0151(19)	0.0092(18)
C(16)	0.0421(17)	0.0477(18)	0.0425(16)	0.0018(16)	0.0175(14)	-0.0047(13)
C(17)	0.0441(17)	0.0461(18)	0.0447(17)	-0.0071(15)	0.0166(14)	0.0031(14)
C(18)	0.0543(19)	0.0409(17)	0.0418(17)	0.0014(16)	0.0143(15)	0.0015(13)
C(19)	0.0472(18)	0.0436(18)	0.0431(17)	0.0062(16)	0.0149(14)	-0.0016(13)
C(20)	0.0422(17)	0.0494(19)	0.0406(16)	0.0006(15)	0.0166(13)	0.0010(13)
C(21)	0.0457(17)	0.0360(16)	0.0444(17)	0.0005(15)	0.0159(14)	0.0023(13)
C(22)	0.0497(20)	0.055(2)	0.0481(18)	-0.0010(18)	0.0168(15)	0.0007(15)
C(23)	0.059(2)	0.0533(20)	0.0512(19)	0.0008(18)	0.0260(17)	-0.0029(15)
C(24)	0.076(3)	0.086(3)	0.052(2)	0.035(2)	0.0099(19)	-0.0054(19)
C(25)	0.0525(20)	0.057(2)	0.0426(17)	0.0142(18)	0.0110(15)	0.0007(15)
C(26)	0.070(2)	0.047(2)	0.072(2)	0.0079(19)	0.014(2)	0.0048(17)
C(27)	0.069(3)	0.089(3)	0.072(3)	0.013(2)	0.038(2)	0.020(2)
C(28)	0.073(3)	0.080(3)	0.061(2)	0.030(2)	0.019(2)	-0.009(2)
C(29)	0.071(2)	0.050(2)	0.074(3)	0.010(2)	0.004(2)	-0.0134(18)
C(30)	0.055(2)	0.062(2)	0.064(2)	0.0008(19)	0.0134(17)	0.0038(18)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Cl(1)	C(2)	1.704(4)	Cl(2)	C(3)	1.715(3)
Cl(3)	C(17)	1.704(3)	Cl(4)	C(18)	1.714(3)
O(1)	C(1)	1.325(4)	O(2)	C(4)	1.354(4)
O(2)	C(9)	1.437(4)	O(3)	C(16)	1.328(4)
O(4)	C(19)	1.361(4)	O(4)	C(24)	1.452(4)
N(1)	C(7)	1.126(5)	N(2)	C(8)	1.127(5)
N(3)	C(22)	1.120(5)	N(4)	C(23)	1.116(5)
C(1)	C(2)	1.399(4)	C(1)	C(6)	1.385(4)
C(2)	C(3)	1.374(5)	C(3)	C(4)	1.390(5)
C(4)	C(5)	1.380(4)	C(5)	C(6)	1.398(4)
C(5)	C(8)	1.428(5)	C(6)	C(7)	1.436(5)
C(9)	C(10)	1.500(6)	C(10)	C(11)	1.375(6)
C(10)	C(15)	1.372(5)	C(11)	C(12)	1.378(6)
C(12)	C(13)	1.383(6)	C(13)	C(14)	1.345(6)
C(14)	C(15)	1.369(6)	C(16)	C(17)	1.398(4)
C(16)	C(21)	1.383(4)	C(17)	C(18)	1.376(4)
C(18)	C(19)	1.392(5)	C(19)	C(20)	1.383(4)
C(20)	C(21)	1.398(4)	C(20)	C(23)	1.432(5)
C(21)	C(22)	1.447(5)	C(24)	C(25)	1.491(5)
C(25)	C(26)	1.371(5)	C(25)	C(30)	1.383(5)
C(26)	C(27)	1.375(6)	C(27)	C(28)	1.378(6)
C(28)	C(29)	1.349(6)	C(29)	C(30)	1.366(6)