Stereoselective Three-Component Synthesis from aldehydes, aniline and acetylcyclohexene: Efficient Entry to *trans-endo*-decahydroquinolin-4-one Derivatives

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Supporting information

Experimental section

(a) General All reactions were performed under 0.5ml ethyl ether condition at room temperature. Analytical thin layer chromatography was performed with E. Merck silica gel 60F glass plates and flash column chromatography used E. Merck silica gel 60 (230-400 mesh). MS or HRMS were measured using a JEOL JMS-D300 or a JEOL JMS-HX110 spectrometer. ¹H-NMR and ¹³C-NMR spectra were recorded with a Bruker Aavance EX 400 spectrometer. The single-crystal X-ray diffraction studies by Nonius Kappa CCD Axis.

(b) Chemical such as aldehyde (**1**), aniline (**2**), 1-acetylcyclohexene (**3**), 1-acetylcyclopentene (**17**), 2-cyclohex-1-one (**18**), and iodine and solvent were purchased from Aldrich and/or Acros Chemical Co and were not purified or dried before used.

(c) Typical experimental procedures for the synthesis of products **4, 8-16** (**4**: $R^1 = C_6H_5$, $R^2 = C_6H_5$; **8**: $R^1 = 4-ClC_6H_4$, $R^2 = C_6H_5$; **9**: $R^1 = 4-MeOC_6H_4$, $R^2 = C_6H_5$; **10**: $R^1 = 2-ClC_6H_4$, $R^2 = C_6H_5$; **11**: $R^1 = 2-MeOC_6H_4$, $R^2 = C_6H_5$; **12**: $R^1 = 3-MeOC_6H_4$, $R^2 = C_6H_5$; **13**: $R^1 = 2-thienyl$, $R^2 = C_6H_5$; **14**: $R^1 = 1-naphthyl$, $R^2 = C_6H_5$; **15**: $R^1 = C_6H_5$, $R^2 = 4-ClC_6H_4$, **16**: $R^1 = C_6H_5$, $R^2 = 4-MeOC_6H_4$): A mixture of aldehyde (**1**) (1.0 mmol), aniline (**2**) (1.5 mmol), 1-acetylcyclohexene (**3**) (2.0 mmol), and iodine (I_2) (1.0 mmol) were added to ethyl ether (0.5 mL) in one pot at the same time and the mixture was then stirred at room temperature for several hours. After completion of the reaction (monitored by TLC), the solution was washed with an ice cold saturated $Na_2S_2O_3(aq)$ solution (2×10 mL when iodine was used) and then extracted with CH_2Cl_2 (3×20 mL). The combined organic phases were washed sequentially with brine and ice water and dried over anhydrous Na_2SO_4 . Evaporation of the organic solvent afforded the crude products which were purified by short flash column chromatography followed by recrystallization from hexane and ethyl acetate if necessary.

(d) Typical experimental procedures for the synthesis of products **19**, *exo*-**20** and *endo*-**20** : The mixture of benzaldehyde (**1**) (1.0 mmol), aniline (**2**) (1.5 mmol), 1-acetylcyclopentene (**17**) or cyclohexenone (**18**) (2.0 mmol), and iodine (I₂) (1.0 mmol) were added to ethyl ether (0.5 mL) in one pot at the same time and the mixture was then stirred at room temperature over a period of several hours. After completion of the reaction (monitored by TLC), the solution was washed with an ice cold saturated Na₂S₂O_{3(aq)} solution (2×10 mL when iodine was used) and then extracted with CH₂Cl₂ (3×20 mL). The combined organic phases were washed sequentially with brine and ice water and dried over anhydrous Na₂SO₄. Evaporation of the organic solvent afforded the crude products which were purified by short flash column chromatography followed by recrystallization from hexane and ethyl acetate if necessary.

NMR Data

trans-endo-1,2-Diphenyldecahydroquinolin-4-one (**4**)

¹H NMR (400 MHz, CDCl₃) δ 7.23 (d, *J* = 7.32 Hz, 2H), 7.06–7.20 (m, 5H), 6.98 (d, *J* = 7.52 Hz, 2H), 6.94 (t, *J* = 7.32 Hz, 1H), 4.43 (dd, *J* = 9.96, 3.68 Hz, 1H), 2.93–3.01 (m, 2H), 2.66 (dd, *J* = 14.48, 3.68 Hz, 1H), 2.49 (td, *J* = 10.96, 2.92 Hz, 1H), 2.09 (d, *J* = 13.2 Hz, 1H), 1.61–1.77 (m, 3H), 1.06–1.38 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 209.31, 148.83, 142.30, 128.46, 128.19, 127.52, 127.01, 125.75, 124.28, 67.26, 65.29,

53.97, 49.41, 33.08, 24.77, 24.68, 24.24. MS m/z (relative intensity) 305 (M^+ , 82), 261 (100), 181 (16), 104 (15), 77 (14). HRMS calcd for $C_{21}H_{23}NO$ (M^+) 305.1780, found 305.1773.

trans-endo-2-(4-Chlorophenyl)-1-phenyldecahydroquinolin-4-one (8)

Melting point of this compound is 156-157 °C.

1H NMR (400 MHz, $CDCl_3$) δ 7.01–7.18 (m, 6H), 6.79 (d, J = 6.88 Hz, 3H), 4.37 (dd, J = 10.44, 3.56 Hz, 1H), 2.86–2.93 (m, 2H), 2.61 (dd, J = 3.56,

14.24 Hz, 1H), 2.48 (td, J = 10.96, 2.72 Hz, 1H), 2.07 (dt, J = 13.52, 2.76 Hz, 1H), 1.51–1.77 (m, 3H), 1.05–1.34 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.79, 148.50, 140.82, 132.59, 128.89, 128.60, 128.37, 126.23, 124.87, 66.94, 65.84, 54.16, 49.68, 33.10, 24.69, 24.65, 25.13. MS m/z (relative intensity) 339 (M^+ , 74), 295 (100), 215 (12), 158 (12), 104 (14), 57 (24). HRMS calcd for $C_{21}H_{22}ClNO$ (M^+) 339.1390, found 339.1386.

trans-endo-2-(4-Methoxyphenyl)-1-phenyldecahydroquinolin-4-one

(9)

Melting point of this compound is 145-146 °C.

1H NMR (400 MHz, $CDCl_3$) δ 7.12 (d, J = 8.64

Hz, 2H), 7.10 (m, 2H), 6.94 (m, 3H), 6.69 (d, $J = 11.6$ Hz, 2H), 4.36 (dd, $J = 10.12$, 3.60 Hz, 1H), 3.70 (s, 3H), 2.90–2.98 (m, 2H), 2.63 (dd, $J = 14.36$, 3.60 Hz, 1H), 2.48 (td, $J = 11.00$, 2.92 Hz, 1H), 2.07 (dt, $J = 13.36$, 2.44 Hz, 1H), 1.57–1.77 (m, 3H), 1.09–1.36 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 209.45, 158.36, 148.92, 134.45, 128.53, 128.45, 125.84, 124.28, 113.50, 66.73, 65.35, 55.06, 53.97, 49.64, 33.10, 24.75, 24.69, 24.22. MS m/z (relative intensity) 336 (M^+ , 23), 335 (100), 291 (25), 228 (36), 211 (21), 173 (35), 134 (28), 57 (15). HRMS calcd for $\text{C}_{22}\text{H}_{25}\text{NO}_2$ (M^+) 335.1885, found 335.1878.

trans-endo-2-(2-Chlorophenyl)-1-phenyldecahydroquinolin-4-one

(10)

^1H NMR (400 MHz, CDCl_3) δ 7.64 (d, $J = 7.64$ Hz, 1H), 7.05–7.13 (m, 6H), 6.96–6.98 (m, 2H), 4.98 (dd, $J = 11.24$, 3.48 Hz, 1H), 2.74–2.83 (m, 2H), 2.64 (dd, $J = 13.84$, 3.48 Hz, 1H), 2.53 (td, $J = 11.28$, 2.88 Hz, 1H), 2.08 (d, $J = 13.84$ Hz, 1H), 1.62–1.78 (m, 2H),

1.05–1.40 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 208.58, 148.32, 139.21, 132.28, 129.87, 129.11, 128.37, 127.92, 127.36, 126.69, 125.59, 67.11, 62.87, 54.67, 49.04, 33.29, 24.73, 24.03. MS m/z (relative intensity) 339(M^+ , 77), 296 (100), 215 (10),

158 (10), 104 (10), 77 (13), 57(6). HRMS calcd for $C_{21}H_{22}ClNO$ (M^+) 339.1390, found 339.1392.

trans-endo-2-(2-Methoxyphenyl)-1-phenyldecahydroquinolin-4-one

(11)

1H NMR (400 MHz, $CDCl_3$) δ 7.53 (dd, $J = 7.64$, 1.52 Hz, 1H), 6.99–7.12 (m, 5H), 6.93 (t, $J = 7.28$ Hz, 1H), 6.83 (t, $J = 7.28$ Hz, 1H), 6.65 (d, $J = 8.20$ Hz, 1H), 4.99 (dd, $J = 10.00$, 3.88 Hz, 1H), 3.70 (s, 3H), 2.90–2.97 (m, 2H), 2.64 (dd, $J = 14.40$, 3.88 Hz, 1H),

2.53 (td, $J = 10.72$, 2.6 Hz, 1H), 2.11 (dt, $J = 13.60$, 2.20 Hz, 1H), 1.78 (d, $J = 11.88$ Hz, 1H), 1.59–1.69 (m, 2H), 1.12–1.36 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.87, 155.99, 149.19, 130.50, 128.91, 128.13, 127.86, 126.09, 124.34, 120.42, 110.38, 65.61, 59.45, 55.30, 54.16, 48.44, 33.27, 24.92, 24.80, 24.36. MS m/z (relative intensity) 335 (M^+ , 52), 292 (37), 174 (100), 130 (17), 77 (11), 57 (8).

HRMS calcd for $C_{22}H_{25}NO_2$ (M^+) 335.1885, found 335.1884.

trans-endo-2-(3-Methoxyphenyl)-1-phenyldecahydroquinolin-4-one

(12)

1H NMR (400 MHz, $CDCl_3$) δ 7.13 (t, $J = 7.64$ Hz, 2H), 7.08 (t, $J = 7.88$ Hz, 1H), 6.98 (d, $J =$

7.88 Hz, 1H), 6.94 (t, $J = 7.64$ Hz, 1H), 6.84 (s, 1H), 6.81 (d, $J = 7.88$ Hz, 1H), 6.64 (d, $J = 8.16$ Hz, 1H), 4.44 (dd, $J = 9.52, 3.52$ Hz, 1H), 3.72 (s, 3H), 2.97 (m, 2H), 2.68 (dd, $J = 14.60, 3.52$ Hz, 1H), 2.48 (td, $J = 10.76, 2.20$ Hz, 1H), 2.08 (d, $J = 13.44$, 1H), 1.77 (d, $J = 11.36$ Hz, 1H), 1.64 (m, 2H), 1.10–1.32 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 209.23, 159.47, 148.84, 144.09, 129.20, 128.49, 125.32, 124.08, 119.79, 113.21, 112.38, 66.86, 64.90, 55.14, 53.76, 49.15, 33.04, 24.76, 24.66, 24.27. MS m/z (relative intensity) 335(M^+ , 89), 292(100), 228(11), 210(11), 104(8), 57(4). HRMS calcd for $\text{C}_{22}\text{H}_{25}\text{NO}_2$ (M^+) 335.1885, found 335.1892.

trans-endo-2-thiophene-1-phenyldecahydroquinolin-4-one (13)

Melting point of this compound is 108-109 °C.

^1H NMR (400 MHz, CDCl_3) δ 7.20 (t, $J = 7.24$ Hz, 1H), 7.12 (dd, $J = 5.04, 0.96$ Hz, 1H), 6.95–7.04 (m, 3H), 6.79 (t, $J = 3.68$ Hz, 1H), 6.70 (s, 1H), 4.81–4.84 (m, 1H), 3.15 (td, $J = 10.40, 3.56$ Hz, 1H), 3.04 (ddd, $J =$

14.40, 6.84, 0.76 Hz, 1H), 2.84 (ddd, $J = 14.40, 4.08, 1.44$ Hz, 1H), 2.51 (t, $J = 10.92$ Hz, 1H), 2.14 (d, $J = 13.6$ Hz, 1H), 1.77 (m, 3H), 1.14–1.29 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 208.45, 148.87, 148.15, 128.69, 126.63, 124.72, 124.32, 123.45, 123.31, 62.53, 61.86, 52.71, 48.37, 32.81, 24.80, 24.56, 25.53.

trans-endo-2-thiophene-1-phenyldecahydroquinolin-4-one (13 α)

Melting point of this compound is 141-142 °C.

^1H NMR(400 MHz, CDCl_3) δ 7.70(d, J =15.56 Hz, 1H),

7.38(d, J =5.00 Hz, 1H), 7.14(t, J =7.84, Hz, 2H),

7.05(t, J =4.56 Hz, 1H), 6.62–6.70(m, 4H), 3.71(td,

J =3.84, 10.44 Hz, 1H), 2.71(td, J =3.84, 11.64 Hz, 1H),

2.27(dd, J =4.44, 9.76 Hz, 1H), 1.96(d, J =12.68 Hz, 1H), 1.15–1.81(m, 6H). ^{13}C NMR

(100 MHz, CDCl_3) δ 201.75, 146.92, 140.04, 135.23, 131.75, 129.13, 128.62, 128.19,

123.78, 117.78, 114.08, 55.68, 54.10, 32.93, 29.41, 25.10, 24.79.

trans-endo-2-naphthalene-1-phenyldecahydroquinolin-4-one (14)

Melting point of this compound is 124-125 °C.

^1H NMR (400 MHz, CDCl_3) δ 8.60 (d, J = 8.24 Hz,

1H), 7.69 (d, J = 8.12 Hz, 1H), 7.35–7.54 (m, 4H),

7.22 (t, J = 7.64 Hz, 1H), 7.04 (d, J = 7.72 Hz, 2H),

6.75 (t, J = 7.44 Hz, 2H), 6.81 (t, J = 7.28 Hz, 1H),

5.10 (dd, J = 11.08, 2.92 Hz, 1H), 3.23 (t, J = 13.36 Hz, 1H), 2.90 (td, J = 10.44, 3.00

Hz, 1H), 2.73 (dd, J = 14.24, 2.92 Hz, 1H), 2.67 (dd, J = 14.24, 2.32 Hz, 1H), 2.12 (d,

J = 13.16 Hz, 1H), 1.09–1.79 (m, 7H). ^{13}C NMR (100 MHz, CDCl_3) δ 209.07, 148.61,

137.40, 133.76, 130.44, 128.82, 128.13, 127.67, 126.82, 126.69, 125.53, 125.16,

125.12, 125.06, 123.60, 67.04, 65.74, 54.59, 49.12, 33.29, 24.70, 24.13. MS m/z (relative intensity). 355(M^+ , 100), 312(46), 228(28), 173(19), 132(11), 104(11), 57(13). HRMS calcd for $C_{25}H_{25}NO$ (M^+) 355.1936, found 355.1940.

trans-endo-2-phenyl-1-(4-Chlorophenyl)decahydroquinolin-4-one (15)

Melting point of this compound is 155-156 °C.

1H NMR (400 MHz, $CDCl_3$) δ 7.10–7.22 (m, 5H), 7.08 (d, J = 8.56 Hz, 2H), 6.91 (d, J = 8.56 Hz, 2H), 4.31 (dd, J = 10.24, 3.48 Hz, 1H), 2.99 (dd, J = 14.24, 10.44 Hz, 2H), 2.90 (td, J = 10.52, 3.12 Hz, 1H), 2.64 (dd, J = 14.24, 3.56 Hz, 1H), 2.48 (td, J = 11.04, 3.12

Hz, 1H), 2.08 (d, J = 13.4 Hz, 1H), 1.53–1.77 (m, 3H), 1.08–1.37 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.86, 147.45, 141.83, 129.72, 128.59, 128.35, 127.47, 127.36, 127.23, 67.49, 65.66, 54.02, 49.56, 33.01, 24.67, 24.64, 24.15. MS m/z (relative intensity) 339(M^+ , 94), 295(100), 215(18), 192(12), 138(14), 71(15), 57(19). HRMS calcd for $C_{21}H_{22}ClNO$ (M^+) 339.1380, found 339.1380.

trans-endo-2-Phenyl-1-(4-methoxyphenyl)decahydroquinolin-4-one (16)

1H NMR (400 MHz, $CDCl_3$) δ 7.19 (d, J = 7.32 Hz, 2H), 7.12 (t, J = 7.24 Hz, 2H), 7.04 (d, J = 7.16 Hz, 1H), 6.91 (d, J = 8.76 Hz, 2H), 6.62 (d, J = 8.76 Hz,

2H), 4.23 (dd, $J = 11.52, 3.08$ Hz, 1H), 2.91 (t, $J = 12.76$ Hz, 1H), 2.74 (td, $J = 10.52, 3.36$ Hz, 1H), 2.60 (dd, $J = 13.84, 3.2$ Hz, 1H), 2.05 (d, $J = 13.64$ Hz, 1H), 1.75 (d, $J = 12.52$ Hz, 1H), 1.61–1.65 (m, 2H), 1.07–1.43 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 209.46, 156.84, 142.21, 141.34, 128.33, 128.08, 127.82, 126.94, 113.50, 68.76, 67.43, 55.13, 54.73, 50.70, 33.20, 24.77, 24.71, 24.03. MS m/z (relative intensity) 335 (M^+ , 100), 291 (85), 188 (14), 134 (16), 85 (20), 77 (25), 57 (30). HRMS calcd for $\text{C}_{22}\text{H}_{25}\text{NO}_2$ (M^+) 335.1885, found 335.1885.

^1H of 4

^{13}C of 8

^1H of 9

^1H of 10

^{13}C of 11

^1H of 12

^{13}C of 13

^1H of 13α

^{13}C of 13α

^{13}C of 16

Single-Crystal X-ray Diffraction of 4

checkCIF/PLATON report

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 4155

Bond precision: C-C = 0.0092 Å Wavelength=0.71073

Cell: a=28.6990 (4) b=6.6330 (8) c=17.9410 (13)
 alpha=90 beta=92.198 (2) gamma=90

	Calculated	Reported
Volume	3412.7 (5)	3412.7 (5)
Space group	C 2/c	C 2/c
Hall group	-C 2yc	?
Moiety formula	C21 H23 N O	C21 H23 N1 O1
Sum formula	C21 H23 N O	C21 H23 N O
Mr	305.40	305.40
Dx, g cm-3	1.189	1.189
Z	8	8
Mu (mm-1)	0.072	0.072
F000	1312.0	1312.0
F000'	1312.50	
h,k,lmax	34,7,21	34,7,21
Nref	3016	2954
Tmin,Tmax	0.996,0.998	
Tmin'	0.995	

Correction method= Not given

Data completeness= Ratio = 0.98 Theta(max) = 25.03

R(reflections)= 0.1343 (1415) wR2(reflections)= 0.3632 (2954)

S = 1.096 Npar= 209

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

DIFMX01_ALERT_2_B The maximum difference density is > 0.1*ZMAX*1.00
 _refine_diff_density_max given = 0.835
 Test value = 0.800

RFACR01_ALERT_3_B The value of the weighted R factor is > 0.35
 Weighted R factor given 0.363

RINTA01_ALERT_3_B The value of Rint is greater than 0.15
 Rint given 0.169

PLAT020_ALERT_3_B	The value of Rint is greater than 0.10	0.17
PLAT084_ALERT_2_B	High R2 Value	0.36
PLAT097_ALERT_2_B	Maximum (Positive) Residual Density	0.83 e/A**3

🟡 **Alert level C**

DIFMX02_ALERT_1_C The maximum difference density is > 0.1*ZMAX*0.75
The relevant atom site should be identified.

HYDTR01_ALERT_1_C The hydrogen treatment should only be one of the following

keywords

- * refall
- * refxyz
- * refU
- * noref
- * undef
- * constr
- * none
- * mixed

Hydrogen treatment given as fixed

RFACG01_ALERT_3_C The value of the R factor is > 0.10

R factor given 0.134

PLAT026_ALERT_3_C	Ratio Observed / Unique Reflections too Low	48 Perc.
PLAT029_ALERT_3_C	diffn_measured_fraction_theta_full Low	0.98
PLAT082_ALERT_2_C	High R1 Value	0.13
PLAT147_ALERT_1_C	su on Symmetry Constrained Cell Angle(s)	?
PLAT331_ALERT_2_C	Small Average Phenyl C-C Dist. C8 -C13	1.37 Ang.
PLAT340_ALERT_3_C	Low Bond Precision on C-C bonds (x 1000) Ang ...	9

0 **ALERT level A** = In general: serious problem

6 **ALERT level B** = Potentially serious problem

9 **ALERT level C** = Check and explain

0 **ALERT level G** = General alerts; check

3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data

5 ALERT type 2 Indicator that the structure model may be wrong or deficient

7 ALERT type 3 Indicator that the structure quality may be low

0 ALERT type 4 Improvement, methodology, query or suggestion

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 12/04/2005; check.def file version of 22/03/2005

Datablock 4155 - ellipsoid plot

Table 1. Crystal data and structure refinement for 4155. resolved data

Identification code	4155	
Empirical formula	C ₂₁ H ₂₃ N O	
Formula weight	305.40	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 28.6990(4) Å	= 90.000(2)°.
	b = 6.6330(8) Å	= 92.198(2)°.
	c = 17.9410(13) Å	= 90.000(4)°.
Volume	3412.7(5) Å ³	
Z	8	
Density (calculated)	1.189 Mg/m ³	
Absorption coefficient	0.072 mm ⁻¹	
F(000)	1312	
Crystal size	0.07 x 0.05 x 0.03 mm ³	
Theta range for data collection	2.27 to 25.03°.	
Index ranges	-34<=h<=34, -7<=k<=7, -21<=l<=21	
Reflections collected	20573	

Independent reflections	2954 [R(int) = 0.1689]
Completeness to theta = 25.03°	97.9 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2954 / 0 / 209
Goodness-of-fit on F ²	1.096
Final R indices [I>2sigma(I)]	R1 = 0.1343, wR2 = 0.3042
R indices (all data)	R1 = 0.2396, wR2 = 0.3632
Extinction coefficient	0.012(2)
Largest diff. peak and hole	0.835 and -0.417 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 4155. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	7351(2)	-204(6)	-58(2)	57(1)
N(1)	8687(2)	1522(7)	358(3)	42(1)
C(1)	7766(2)	-158(8)	75(3)	37(2)
C(2)	7981(2)	-208(8)	851(3)	40(2)
C(3)	7629(2)	-179(10)	1460(3)	52(2)
C(4)	7866(2)	-267(11)	2227(4)	62(2)
C(5)	8225(2)	1321(12)	2342(4)	62(2)
C(6)	8578(2)	1292(11)	1723(3)	54(2)
C(7)	8343(2)	1497(9)	956(3)	46(2)
C(8)	9037(2)	3091(9)	510(3)	41(2)
C(9)	9478(2)	2602(10)	685(4)	53(2)
C(10)	9800(2)	4068(11)	865(4)	62(2)
C(11)	9694(3)	6032(11)	836(4)	59(2)
C(12)	9226(3)	6623(9)	645(3)	61(2)
C(13)	8908(2)	5057(9)	486(3)	46(2)
C(14)	8454(2)	1737(10)	-385(3)	50(2)
C(15)	8113(2)	-32(8)	-527(3)	45(2)
C(16)	8802(2)	1857(10)	-998(3)	49(2)
C(17)	9112(2)	278(11)	-1117(4)	56(2)
C(18)	9403(2)	347(12)	-1709(4)	64(2)
C(19)	9381(3)	1975(15)	-2214(4)	77(2)
C(20)	9075(3)	3531(15)	-2108(4)	83(3)
C(21)	8786(2)	3475(11)	-1491(4)	59(2)

Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for 4155.

O(1)-C(1)	1.206(6)
N(1)-C(8)	1.464(7)
N(1)-C(14)	1.475(7)
N(1)-C(7)	1.484(7)
C(1)-C(2)	1.501(8)
C(1)-C(15)	1.500(8)
C(2)-C(3)	1.514(8)
C(2)-C(7)	1.544(8)
C(2)-H(2)	0.9601
C(3)-C(4)	1.513(9)
C(3)-H(3A)	0.9609
C(3)-H(3B)	0.9614
C(4)-C(5)	1.482(9)
C(4)-H(4A)	0.9600
C(4)-H(4B)	0.9587
C(5)-C(6)	1.531(8)
C(5)-H(5A)	0.9594
C(5)-H(5B)	0.9603
C(6)-C(7)	1.514(8)
C(6)-H(6A)	0.9594
C(6)-H(6B)	0.9605
C(7)-H(7)	0.9607
C(8)-C(9)	1.333(8)
C(8)-C(13)	1.355(8)
C(9)-C(10)	1.371(9)
C(9)-H(9)	0.9594
C(10)-C(11)	1.338(10)
C(10)-H(10)	0.9598
C(11)-C(12)	1.428(9)
C(11)-H(11)	0.9616
C(12)-C(13)	1.405(8)
C(12)-H(12)	0.9603
C(13)-H(13)	0.9611
C(14)-C(16)	1.516(8)
C(14)-C(15)	1.544(8)
C(14)-H(14)	0.9609
C(15)-H(15A)	0.9611

C(15)-H(15B)	0.9579
C(16)-C(21)	1.390(9)
C(16)-C(17)	1.398(9)
C(17)-C(18)	1.376(9)
C(17)-H(17)	0.9601
C(18)-C(19)	1.410(10)
C(18)-H(18)	0.9605
C(19)-C(20)	1.374(11)
C(19)-H(19)	0.9582
C(20)-C(21)	1.410(9)
C(20)-H(20)	0.9598
C(21)-H(21)	0.9607

C(8)-N(1)-C(14)	112.5(4)
C(8)-N(1)-C(7)	110.1(4)
C(14)-N(1)-C(7)	111.3(4)
O(1)-C(1)-C(2)	123.5(5)
O(1)-C(1)-C(15)	122.4(5)
C(2)-C(1)-C(15)	114.1(5)
C(1)-C(2)-C(3)	114.0(5)
C(1)-C(2)-C(7)	110.4(5)
C(3)-C(2)-C(7)	111.5(5)
C(1)-C(2)-H(2)	105.5
C(3)-C(2)-H(2)	108.6
C(7)-C(2)-H(2)	106.3
C(4)-C(3)-C(2)	111.5(5)
C(4)-C(3)-H(3A)	108.1
C(2)-C(3)-H(3A)	108.7
C(4)-C(3)-H(3B)	109.5
C(2)-C(3)-H(3B)	109.8
H(3A)-C(3)-H(3B)	109.3
C(5)-C(4)-C(3)	112.7(5)
C(5)-C(4)-H(4A)	108.4
C(3)-C(4)-H(4A)	108.8
C(5)-C(4)-H(4B)	108.4
C(3)-C(4)-H(4B)	109.1
H(4A)-C(4)-H(4B)	109.4
C(4)-C(5)-C(6)	111.4(6)
C(4)-C(5)-H(5A)	109.1

C(6)-C(5)-H(5A)	107.7
C(4)-C(5)-H(5B)	110.5
C(6)-C(5)-H(5B)	108.6
H(5A)-C(5)-H(5B)	109.5
C(7)-C(6)-C(5)	112.0(5)
C(7)-C(6)-H(6A)	108.6
C(5)-C(6)-H(6A)	110.7
C(7)-C(6)-H(6B)	107.5
C(5)-C(6)-H(6B)	108.6
H(6A)-C(6)-H(6B)	109.5
N(1)-C(7)-C(6)	111.9(5)
N(1)-C(7)-C(2)	112.6(5)
C(6)-C(7)-C(2)	108.6(5)
N(1)-C(7)-H(7)	108.9
C(6)-C(7)-H(7)	107.4
C(2)-C(7)-H(7)	107.2
C(9)-C(8)-C(13)	119.8(6)
C(9)-C(8)-N(1)	120.6(5)
C(13)-C(8)-N(1)	119.6(5)
C(8)-C(9)-C(10)	120.6(6)
C(8)-C(9)-H(9)	119.2
C(10)-C(9)-H(9)	120.2
C(11)-C(10)-C(9)	122.0(7)
C(11)-C(10)-H(10)	118.7
C(9)-C(10)-H(10)	119.3
C(10)-C(11)-C(12)	119.2(6)
C(10)-C(11)-H(11)	120.7
C(12)-C(11)-H(11)	120.1
C(13)-C(12)-C(11)	116.3(6)
C(13)-C(12)-H(12)	120.9
C(11)-C(12)-H(12)	122.8
C(8)-C(13)-C(12)	122.0(6)
C(8)-C(13)-H(13)	119.5
C(12)-C(13)-H(13)	118.5
N(1)-C(14)-C(16)	111.9(5)
N(1)-C(14)-C(15)	109.8(5)
C(16)-C(14)-C(15)	110.5(5)
N(1)-C(14)-H(14)	109.3
C(16)-C(14)-H(14)	107.1

C(15)-C(14)-H(14)	108.0
C(1)-C(15)-C(14)	111.0(5)
C(1)-C(15)-H(15A)	110.4
C(14)-C(15)-H(15A)	109.0
C(1)-C(15)-H(15B)	107.8
C(14)-C(15)-H(15B)	109.1
H(15A)-C(15)-H(15B)	109.5
C(21)-C(16)-C(17)	119.2(6)
C(21)-C(16)-C(14)	119.7(6)
C(17)-C(16)-C(14)	120.9(6)
C(18)-C(17)-C(16)	120.1(7)
C(18)-C(17)-H(17)	120.5
C(16)-C(17)-H(17)	119.3
C(17)-C(18)-C(19)	120.6(7)
C(17)-C(18)-H(18)	119.7
C(19)-C(18)-H(18)	119.7
C(20)-C(19)-C(18)	119.9(7)
C(20)-C(19)-H(19)	119.9
C(18)-C(19)-H(19)	120.2
C(19)-C(20)-C(21)	119.3(7)
C(19)-C(20)-H(20)	120.0
C(21)-C(20)-H(20)	120.7
C(16)-C(21)-C(20)	120.9(7)
C(16)-C(21)-H(21)	119.7
C(20)-C(21)-H(21)	119.4

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 4155. The anisotropic displacement factor exponent takes the form: $-2 \pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	38(3)	77(3)	54(3)	0(2)	0(2)	4(2)
N(1)	45(3)	45(3)	38(3)	-1(2)	-2(2)	1(2)
C(1)	37(4)	30(3)	46(4)	2(3)	4(3)	5(3)
C(2)	35(3)	50(4)	36(4)	5(3)	2(3)	2(3)
C(3)	46(4)	72(4)	40(4)	5(3)	4(3)	-14(3)
C(4)	59(4)	94(5)	34(4)	6(4)	1(3)	-7(4)
C(5)	60(4)	87(5)	38(4)	8(4)	-1(3)	-6(4)
C(6)	47(4)	64(4)	51(4)	4(3)	0(3)	-9(3)
C(7)	46(4)	46(4)	45(4)	0(3)	1(3)	0(3)
C(8)	47(4)	39(4)	38(4)	0(3)	5(3)	0(3)
C(9)	54(4)	44(4)	62(5)	2(3)	5(3)	-8(3)
C(10)	63(5)	64(5)	59(5)	3(4)	-2(4)	-4(4)
C(11)	64(5)	65(5)	47(4)	-2(4)	1(3)	-27(4)
C(12)	108(6)	36(4)	39(4)	2(3)	19(4)	3(4)
C(13)	40(3)	56(4)	41(4)	-4(3)	0(3)	-2(3)
C(14)	56(4)	52(4)	43(4)	0(3)	8(3)	5(3)
C(15)	47(4)	48(4)	40(4)	-10(3)	1(3)	0(3)
C(16)	54(4)	61(4)	32(4)	3(3)	-3(3)	-9(3)
C(17)	53(4)	76(5)	38(4)	-5(3)	12(3)	3(4)
C(18)	46(4)	98(6)	47(5)	-11(4)	-3(3)	7(4)
C(19)	56(5)	127(8)	48(5)	-2(5)	2(4)	-15(5)
C(20)	79(6)	110(7)	60(6)	35(5)	6(5)	-16(5)
C(21)	56(4)	66(5)	55(5)	13(4)	2(4)	-3(4)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 4155.

	x	y	z	U(eq)
H(2)	8152	-1451	884	48
H(3A)	7455	1057	1423	63
H(3B)	7419	-1301	1398	63
H(4A)	7635	-98	2595	75
H(4B)	8013	-1556	2293	75
H(5A)	8076	2616	2332	74
H(5B)	8390	1139	2813	74
H(6A)	8800	2364	1791	65
H(6B)	8738	20	1738	65
H(7)	8175	2750	947	55
H(9)	9568	1209	687	64
H(10)	10111	3680	1020	75
H(11)	9930	7038	932	70
H(12)	9127	8006	636	73
H(13)	8590	5403	356	55
H(14)	8277	2967	-402	60
H(15A)	8288	-1264	-552	54
H(15B)	7944	183	-991	54
H(17)	9122	-847	-780	67
H(18)	9617	-740	-1786	77
H(19)	9579	1998	-2632	92
H(20)	9064	4653	-2446	99
H(21)	8572	4563	-1415	71

Single-Crystal X-ray Diffraction of 8

checkCIF/PLATON report

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 5503

Bond precision: C-C = 0.0045 Å Wavelength=0.71073

Cell: a=11.2065 (4) b=15.3110 (5) c=11.6237 (6)
 alpha=90 beta=114.464 (2) gamma=90

	Calculated	Reported
Volume	1815.37 (13)	1815.37 (13)
Space group	P 21/a	P 21/a
Hall group	-P 2yab	?
Moiety formula	C21 H22 Cl N O	C21 H22 Cl1 N1 O1
Sum formula	C21 H22 Cl N O	C21 H22 Cl N O
Mr	339.85	339.85
Dx, g cm-3	1.243	1.243
Z	4	4
Mu (mm-1)	0.217	0.217
F000	720.0	720.0
F000'	720.84	
h,k,lmax	13,18,14	13,18,13
Nref	3329	3301
Tmin,Tmax	0.959,0.974	0.922,1.000
Tmin'	0.953	

Correction method= 'MULTI-SCAN'

Data completeness= Ratio = 0.99 Theta(max) = 25.38

R(reflections)= 0.0527 (2024) wR2(reflections)= 0.1629 (3301)

S = 1.078 Npar= 218

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

ABSTY02_ALERT_1_C An _exptl_absorpt_correction_type has been given without
a literature citation. This should be contained in the
_exptl_absorpt_process_details field.

Absorption correction given as multi-scan

HYDTR01_ALERT_1_C The hydrogen treatment should only be one of the following
keywords
* refall

```

* refxyz
* refU
* noref
* undef
* constr
* none
* mixed
Hydrogen treatment given as fixed
PLAT062_ALERT_4_C Rescale T(min) & T(max) by ..... 0.98
PLAT128_ALERT_4_C Non-standard setting of Space group P21/c .... P21/a

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0 ALERT level A = In general: serious problem
0 ALERT level B = Potentially serious problem
4 ALERT level C = Check and explain
0 ALERT level G = General alerts; check

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
0 ALERT type 2 Indicator that the structure model may be wrong or deficient
0 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion

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Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 12/04/2005; check.def file version of 22/03/2005

Datablock 5503 - ellipsoid plot

Table 1. Crystal data and structure refinement for 5503.

Identification code	5503	
Empirical formula	C ₂₁ H ₂₂ Cl N O	
Formula weight	339.85	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 2 ₁ /a	
Unit cell dimensions	a = 11.2065(4) Å	= 90°.
	b = 15.3110(5) Å	= 114.464(2)°.
	c = 11.6237(6) Å	= 90°.
Volume	1815.37(13) Å ³	
Z	4	
Density (calculated)	1.243 Mg/m ³	
Absorption coefficient	0.217 mm ⁻¹	
F(000)	720	
Crystal size	0.22 x 0.16 x 0.12 mm ³	
Theta range for data collection	2.34 to 25.38°.	
Index ranges	-13 ≤ h ≤ 13, -18 ≤ k ≤ 15, -12 ≤ l ≤ 13	
Reflections collected	12927	

Independent reflections	3301 [R(int) = 0.0829]
Completeness to theta = 25.38°	99.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9996 and 0.922
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3301 / 0 / 218
Goodness-of-fit on F ²	1.078
Final R indices [I>2sigma(I)]	R1 = 0.0527, wR2 = 0.1198
R indices (all data)	R1 = 0.1063, wR2 = 0.1629
Extinction coefficient	0.014(2)
Largest diff. peak and hole	0.321 and -0.278 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5503. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	1778(1)	-356(1)	413(1)	75(1)
O(1)	9897(2)	-275(1)	6594(2)	48(1)
N(1)	7484(2)	1495(1)	4212(2)	31(1)
C(1)	9042(3)	273(2)	6106(3)	37(1)
C(2)	9287(3)	1237(2)	6344(3)	35(1)
C(3)	10692(3)	1467(2)	7210(3)	46(1)
C(4)	10865(3)	2448(2)	7458(3)	52(1)
C(5)	10395(3)	2951(2)	6226(3)	44(1)
C(6)	8997(3)	2709(2)	5347(3)	39(1)
C(7)	8849(3)	1730(2)	5075(2)	32(1)
C(8)	7044(2)	1996(2)	3039(3)	30(1)
C(9)	6095(3)	2625(2)	2818(3)	38(1)
C(10)	5655(3)	3108(2)	1706(3)	43(1)
C(11)	6155(3)	2966(2)	822(3)	41(1)
C(12)	7111(3)	2343(2)	1050(3)	42(1)
C(13)	7554(3)	1856(2)	2154(3)	38(1)
C(14)	7321(3)	543(2)	3942(3)	34(1)
C(15)	7686(3)	46(2)	5181(3)	39(1)
C(16)	5939(3)	328(2)	3035(3)	34(1)
C(17)	4889(3)	639(2)	3253(3)	37(1)
C(18)	3612(3)	438(2)	2446(3)	42(1)
C(19)	3385(3)	-93(2)	1414(3)	44(1)

C(20)	4408(3)	-408(2)	1173(3)	48(1)
C(21)	5678(3)	-191(2)	1987(3)	43(1)

Table 3. Bond lengths [Å] and angles [°] for 5503.

Cl(1)-C(19)	1.739(3)
O(1)-C(1)	1.222(3)
N(1)-C(8)	1.461(3)
N(1)-C(7)	1.485(3)
N(1)-C(14)	1.486(3)
C(1)-C(15)	1.494(4)
C(1)-C(2)	1.505(4)
C(2)-C(3)	1.519(4)
C(2)-C(7)	1.546(4)
C(2)-H(2)	0.9600
C(3)-C(4)	1.525(4)
C(3)-H(3A)	0.9600
C(3)-H(3B)	0.9600
C(4)-C(5)	1.516(4)
C(4)-H(4A)	0.9600
C(4)-H(4B)	0.9599
C(5)-C(6)	1.519(4)
C(5)-H(5A)	0.9600
C(5)-H(5B)	0.9600
C(6)-C(7)	1.526(4)
C(6)-H(6A)	0.9599
C(6)-H(6B)	0.9600
C(7)-H(7)	0.9600
C(8)-C(9)	1.378(4)
C(8)-C(13)	1.385(4)
C(9)-C(10)	1.389(4)
C(9)-H(9)	0.9600
C(10)-C(11)	1.377(4)
C(10)-H(10)	0.9600
C(11)-C(12)	1.376(4)
C(11)-H(11)	0.9600
C(12)-C(13)	1.386(4)
C(12)-H(12)	0.9601
C(13)-H(13)	0.9600

C(14)-C(16)	1.505(4)
C(14)-C(15)	1.527(4)
C(14)-H(14)	0.9601
C(15)-H(15A)	0.9601
C(15)-H(15B)	0.9599
C(16)-C(21)	1.381(4)
C(16)-C(17)	1.386(4)
C(17)-C(18)	1.382(4)
C(17)-H(17)	0.9600
C(18)-C(19)	1.383(4)
C(18)-H(18)	0.9600
C(19)-C(20)	1.375(5)
C(20)-C(21)	1.385(4)
C(20)-H(20)	0.9600
C(21)-H(21)	0.9599

C(8)-N(1)-C(7)	110.95(19)
C(8)-N(1)-C(14)	110.7(2)
C(7)-N(1)-C(14)	112.50(19)
O(1)-C(1)-C(15)	122.9(3)
O(1)-C(1)-C(2)	122.6(3)
C(15)-C(1)-C(2)	114.5(2)
C(1)-C(2)-C(3)	114.2(2)
C(1)-C(2)-C(7)	110.0(2)
C(3)-C(2)-C(7)	110.5(2)
C(1)-C(2)-H(2)	106.2
C(3)-C(2)-H(2)	108.5
C(7)-C(2)-H(2)	107.0
C(2)-C(3)-C(4)	111.7(2)
C(2)-C(3)-H(3A)	109.7
C(4)-C(3)-H(3A)	109.5
C(2)-C(3)-H(3B)	108.5
C(4)-C(3)-H(3B)	107.9
H(3A)-C(3)-H(3B)	109.5
C(5)-C(4)-C(3)	110.8(2)
C(5)-C(4)-H(4A)	108.6
C(3)-C(4)-H(4A)	108.6
C(5)-C(4)-H(4B)	109.7
C(3)-C(4)-H(4B)	109.7

H(4A)-C(4)-H(4B)	109.5
C(4)-C(5)-C(6)	111.7(3)
C(4)-C(5)-H(5A)	109.8
C(6)-C(5)-H(5A)	108.5
C(4)-C(5)-H(5B)	108.7
C(6)-C(5)-H(5B)	108.7
H(5A)-C(5)-H(5B)	109.5
C(5)-C(6)-C(7)	111.9(2)
C(5)-C(6)-H(6A)	109.3
C(7)-C(6)-H(6A)	107.9
C(5)-C(6)-H(6B)	110.0
C(7)-C(6)-H(6B)	108.2
H(6A)-C(6)-H(6B)	109.5
N(1)-C(7)-C(6)	111.8(2)
N(1)-C(7)-C(2)	110.9(2)
C(6)-C(7)-C(2)	108.6(2)
N(1)-C(7)-H(7)	107.9
C(6)-C(7)-H(7)	109.1
C(2)-C(7)-H(7)	108.5
C(9)-C(8)-C(13)	119.6(3)
C(9)-C(8)-N(1)	118.5(2)
C(13)-C(8)-N(1)	121.9(2)
C(8)-C(9)-C(10)	119.7(3)
C(8)-C(9)-H(9)	119.2
C(10)-C(9)-H(9)	121.2
C(11)-C(10)-C(9)	120.9(3)
C(11)-C(10)-H(10)	118.9
C(9)-C(10)-H(10)	120.2
C(12)-C(11)-C(10)	119.4(3)
C(12)-C(11)-H(11)	120.7
C(10)-C(11)-H(11)	119.9
C(11)-C(12)-C(13)	120.2(3)
C(11)-C(12)-H(12)	120.1
C(13)-C(12)-H(12)	119.7
C(8)-C(13)-C(12)	120.3(3)
C(8)-C(13)-H(13)	119.0
C(12)-C(13)-H(13)	120.7
N(1)-C(14)-C(16)	111.2(2)
N(1)-C(14)-C(15)	109.1(2)

C(16)-C(14)-C(15)	110.3(2)
N(1)-C(14)-H(14)	108.8
C(16)-C(14)-H(14)	108.6
C(15)-C(14)-H(14)	108.7
C(1)-C(15)-C(14)	111.0(2)
C(1)-C(15)-H(15A)	108.9
C(14)-C(15)-H(15A)	109.4
C(1)-C(15)-H(15B)	109.7
C(14)-C(15)-H(15B)	108.4
H(15A)-C(15)-H(15B)	109.5
C(21)-C(16)-C(17)	118.2(3)
C(21)-C(16)-C(14)	121.6(3)
C(17)-C(16)-C(14)	120.2(3)
C(18)-C(17)-C(16)	121.3(3)
C(18)-C(17)-H(17)	119.1
C(16)-C(17)-H(17)	119.7
C(17)-C(18)-C(19)	119.1(3)
C(17)-C(18)-H(18)	119.8
C(19)-C(18)-H(18)	121.2
C(20)-C(19)-C(18)	120.9(3)
C(20)-C(19)-Cl(1)	120.1(2)
C(18)-C(19)-Cl(1)	118.9(3)
C(19)-C(20)-C(21)	118.9(3)
C(19)-C(20)-H(20)	119.6
C(21)-C(20)-H(20)	121.4
C(16)-C(21)-C(20)	121.6(3)
C(16)-C(21)-H(21)	119.8
C(20)-C(21)-H(21)	118.6

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5503. The anisotropic displacement factor exponent takes the form: $-2 \left[h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12} \right]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	47(1)	65(1)	83(1)	-21(1)	-2(1)	-8(1)
O(1)	46(1)	37(1)	55(1)	11(1)	15(1)	10(1)
N(1)	33(1)	28(1)	31(1)	3(1)	11(1)	1(1)

C(1)	43(2)	36(2)	35(2)	8(1)	19(1)	6(1)
C(2)	41(2)	33(2)	31(2)	4(1)	14(1)	6(1)
C(3)	51(2)	44(2)	34(2)	4(1)	7(2)	4(1)
C(4)	59(2)	47(2)	35(2)	-4(1)	5(2)	-3(2)
C(5)	52(2)	36(2)	38(2)	-5(1)	12(2)	-3(1)
C(6)	49(2)	32(2)	34(2)	2(1)	15(1)	1(1)
C(7)	33(2)	31(2)	30(2)	0(1)	13(1)	3(1)
C(8)	30(2)	27(1)	31(2)	0(1)	11(1)	-2(1)
C(9)	38(2)	38(2)	39(2)	5(1)	19(1)	7(1)
C(10)	38(2)	41(2)	45(2)	10(2)	13(2)	10(1)
C(11)	46(2)	42(2)	30(2)	6(1)	10(2)	1(1)
C(12)	50(2)	44(2)	37(2)	-2(1)	21(2)	1(1)
C(13)	39(2)	36(2)	37(2)	0(1)	13(1)	6(1)
C(14)	35(2)	29(2)	40(2)	2(1)	18(1)	0(1)
C(15)	42(2)	30(2)	45(2)	4(1)	19(2)	1(1)
C(16)	39(2)	27(2)	37(2)	2(1)	15(1)	-3(1)
C(17)	38(2)	34(2)	38(2)	-2(1)	15(1)	-2(1)
C(18)	39(2)	38(2)	48(2)	4(2)	18(2)	1(1)
C(19)	41(2)	38(2)	44(2)	0(2)	8(2)	-6(1)
C(20)	58(2)	41(2)	39(2)	-9(1)	14(2)	-6(2)
C(21)	47(2)	37(2)	46(2)	-3(1)	22(2)	1(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5503.

	x	y	z	U(eq)
H(2)	8727	1427	6735	42
H(3A)	10948	1163	7998	56
H(3B)	11249	1294	6807	56
H(4A)	10350	2622	7905	62
H(4B)	11771	2577	7962	62
H(5A)	10426	3566	6393	53
H(5B)	10958	2818	5813	53
H(6A)	8419	2867	5733	47
H(6B)	8742	3012	4557	47
H(7)	9412	1561	4676	38
H(9)	5735	2712	3428	45

H(10)	4996	3549	1546	51
H(11)	5837	3301	55	50
H(12)	7476	2243	445	51
H(13)	8217	1418	2318	46
H(14)	7905	368	3569	41
H(15A)	7643	-571	5018	47
H(15B)	7068	197	5525	47
H(17)	5046	1009	3968	44
H(18)	2900	668	2610	50
H(20)	4229	-766	442	58
H(21)	6388	-412	1814	51

Single-Crystal X-ray Diffraction of 9

checkCIF/PLATON report

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 5504a

Bond precision: C-C = 0.0046 Å

Wavelength=0.71073

Cell: a=17.6746(8)
 alpha=90

 b=6.6948(4)
 beta=119.352(2)

 c=17.8568(11)
 gamma=90

	Calculated	Reported
Volume	1841.71(18)	1841.71(18)
Space group	P 21/n	P 21/n
Hall group	-P 2yn	?
Moiety formula	C22 H25 N O2	C22 H25 N O2
Sum formula	C22 H25 N O2	C22 H25 N O2
Mr	335.43	335.43
Dx, g cm-3	1.210	1.210
Z	4	4
Mu (mm-1)	0.077	0.077
F000	720.0	720.0
F000'	720.30	
h,k,lmax	21,8,21	21,8,21
Nref	3365	3325
Tmin,Tmax	0.962,0.994	0.905,1.176
Tmin'	0.955	

Correction method= 'MULTI-SCAN'

Data completeness= Ratio = 0.99

Theta(max)= 25.36

R(reflections)= 0.0770(1900)

wR2(reflections)= 0.2244(3325)

S = 0.999

Npar= 227

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

DIFMX01_ALERT_2_B The maximum difference density is > 0.1*ZMAX*1.00

 _refine_diff_density_max given = 0.960

 Test value = 0.800

PLAT097_ALERT_2_B Maximum (Positive) Residual Density 0.96 e/Å**3

Alert level C

ABSTM02_ALERT_3_C The ratio of expected to reported Tmax/Tmin(RR') is < 0.90
 Tmin and Tmax reported: 0.905 1.176
 Tmin(prime) and Tmax expected: 0.954 0.994
 RR(prime) = 0.802
 Please check that your absorption correction is appropriate.

ABSTY02_ALERT_1_C An _exptl_absorpt_correction_type has been given without
 a literature citation. This should be contained in the
 _exptl_absorpt_process_details field.
 Absorption correction given as multi-scan

DIFMX02_ALERT_1_C The maximum difference density is > 0.1*ZMAX*0.75
 The relevant atom site should be identified.

HYDTR01_ALERT_1_C The hydrogen treatment should only be one of the following
 keywords
 * refall
 * refxyz
 * refU
 * noref
 * undef
 * constr
 * none
 * mixed
 Hydrogen treatment given as fixed

PLAT061_ALERT_3_C Tmax/Tmin Range Test RR' too Large 0.80
 PLAT062_ALERT_4_C Rescale T(min) & T(max) by 0.85
 PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density 2.24
 PLAT340_ALERT_3_C Low Bond Precision on C-C bonds (x 1000) Ang ... 5

Alert level G

ABSTM02_ALERT_3_G When printed, the submitted absorption T values will be
 replaced by the scaled T values. Since the ratio of scaled T's
 is identical to the ratio of reported T values, the scaling
 does not imply a change to the absorption corrections used in
 the study.
 Ratio of Tmax expected/reported 0.845
 Tmax scaled 0.994 Tmin scaled 0.765

-
- 0 ALERT level A = In general: serious problem
 - 2 ALERT level B = Potentially serious problem
 - 8 ALERT level C = Check and explain
 - 1 ALERT level G = General alerts; check

 - 3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 - 3 ALERT type 2 Indicator that the structure model may be wrong or deficient
 - 4 ALERT type 3 Indicator that the structure quality may be low
 - 1 ALERT type 4 Improvement, methodology, query or suggestion
-
-

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 12/04/2005; check.def file version of 22/03/2005

Datablock 5504a - ellipsoid plot

Table 1. Crystal data and structure refinement for 5504a.

Identification code	5504a	
Empirical formula	C ₂₂ H ₂₅ N O ₂	
Formula weight	335.43	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 2 ₁ /n	
Unit cell dimensions	a = 17.6746(8) Å	= 90°.
	b = 6.6948(4) Å	= 119.352(2)°.

	$c = 17.8568(11) \text{ \AA}$	$= 90^\circ$.
Volume	$1841.71(18) \text{ \AA}^3$	
Z	4	
Density (calculated)	1.210 Mg/m^3	
Absorption coefficient	0.077 mm^{-1}	
F(000)	720	
Crystal size	$0.6 \times 0.42 \times 0.08 \text{ mm}^3$	
Theta range for data collection	$2.27 \text{ to } 25.36^\circ$.	
Index ranges	$-21 \leq h \leq 18, -8 \leq k \leq 7, -13 \leq l \leq 21$	
Reflections collected	10269	
Independent reflections	3325 [R(int) = 0.0789]	
Completeness to $\theta = 25.36^\circ$	98.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.1762 and 0.9049	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3325 / 0 / 227	
Goodness-of-fit on F^2	0.999	
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0770, wR2 = 0.1814$	
R indices (all data)	$R1 = 0.1361, wR2 = 0.2244$	
Extinction coefficient	0.042(6)	
Largest diff. peak and hole	$0.960 \text{ and } -0.429 \text{ e.\AA}^{-3}$	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5504a. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	7345(1)	1993(3)	7673(1)	47(1)
O(2)	12490(1)	1236(4)	8346(1)	56(1)
N(1)	8323(1)	873(3)	6131(1)	34(1)
C(1)	7651(2)	2274(4)	7197(2)	36(1)
C(2)	7107(2)	2473(4)	6241(2)	38(1)
C(3)	6134(2)	2449(5)	5911(2)	45(1)
C(4)	5626(2)	2628(5)	4942(2)	52(1)
C(5)	5886(2)	1048(5)	4515(2)	52(1)
C(6)	6862(2)	1055(5)	4846(2)	45(1)
C(7)	7371(2)	815(4)	5820(2)	35(1)
C(8)	8557(2)	-690(4)	5714(2)	35(1)
C(9)	8430(2)	-2685(4)	5809(2)	40(1)

C(10)	8619(2)	-4138(5)	5381(2)	49(1)
C(11)	8970(2)	-3591(5)	4856(2)	51(1)
C(12)	9104(2)	-1609(5)	4772(2)	49(1)
C(13)	8900(2)	-156(5)	5194(2)	44(1)
C(14)	8859(2)	699(4)	7084(2)	38(1)
C(15)	8606(2)	2328(4)	7515(2)	40(1)
C(16)	9818(2)	812(4)	7375(2)	37(1)
C(17)	10161(2)	2416(4)	7146(2)	40(1)
C(18)	11042(2)	2596(4)	7440(2)	42(1)
C(19)	11601(2)	1175(5)	7997(2)	41(1)
C(20)	11282(2)	-436(5)	8239(2)	47(1)
C(21)	10391(2)	-607(5)	7920(2)	43(1)
C(22)	12839(2)	2780(6)	8069(2)	62(1)

Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for 5504a.

O(1)-C(1)	1.223(3)
O(2)-C(19)	1.378(3)
O(2)-C(22)	1.413(4)
N(1)-C(8)	1.457(3)
N(1)-C(7)	1.491(3)
N(1)-C(14)	1.491(3)
C(1)-C(15)	1.495(4)
C(1)-C(2)	1.500(4)
C(2)-C(3)	1.519(4)
C(2)-C(7)	1.536(4)
C(2)-H(2)	0.9603
C(3)-C(4)	1.514(4)
C(3)-H(3A)	0.9604
C(3)-H(3B)	0.9597
C(4)-C(5)	1.503(4)
C(4)-H(4A)	0.9602
C(4)-H(4B)	0.9602
C(5)-C(6)	1.525(4)
C(5)-H(5A)	0.9593
C(5)-H(5B)	0.9601
C(6)-C(7)	1.524(4)
C(6)-H(6A)	0.9595
C(6)-H(6B)	0.9597

C(7)-H(7)	0.9601
C(8)-C(9)	1.379(4)
C(8)-C(13)	1.381(4)
C(9)-C(10)	1.374(4)
C(9)-H(9)	0.9603
C(10)-C(11)	1.404(4)
C(10)-H(10)	0.9600
C(11)-C(12)	1.369(5)
C(11)-H(11)	0.9599
C(12)-C(13)	1.382(4)
C(12)-H(12)	0.9600
C(13)-H(13)	0.9600
C(14)-C(16)	1.511(4)
C(14)-C(15)	1.523(4)
C(14)-H(14)	0.9595
C(15)-H(15A)	0.9605
C(15)-H(15B)	0.9600
C(16)-C(21)	1.382(4)
C(16)-C(17)	1.389(4)
C(17)-C(18)	1.383(4)
C(17)-H(17)	0.9597
C(18)-C(19)	1.381(4)
C(18)-H(18)	0.9598
C(19)-C(20)	1.380(4)
C(20)-C(21)	1.393(4)
C(20)-H(20)	0.9599
C(21)-H(21)	0.9593
C(22)-H(22A)	0.9601
C(22)-H(22B)	0.9597
C(22)-H(22C)	0.9605

C(19)-O(2)-C(22)	117.4(2)
C(8)-N(1)-C(7)	110.0(2)
C(8)-N(1)-C(14)	110.2(2)
C(7)-N(1)-C(14)	113.2(2)
O(1)-C(1)-C(15)	122.8(3)
O(1)-C(1)-C(2)	123.3(2)
C(15)-C(1)-C(2)	113.8(2)
C(1)-C(2)-C(3)	114.4(2)

C(1)-C(2)-C(7)	108.3(2)
C(3)-C(2)-C(7)	111.1(2)
C(1)-C(2)-H(2)	108.7
C(3)-C(2)-H(2)	106.3
C(7)-C(2)-H(2)	107.8
C(4)-C(3)-C(2)	111.5(2)
C(4)-C(3)-H(3A)	108.0
C(2)-C(3)-H(3A)	107.8
C(4)-C(3)-H(3B)	110.6
C(2)-C(3)-H(3B)	109.3
H(3A)-C(3)-H(3B)	109.5
C(5)-C(4)-C(3)	111.5(2)
C(5)-C(4)-H(4A)	109.9
C(3)-C(4)-H(4A)	109.6
C(5)-C(4)-H(4B)	108.1
C(3)-C(4)-H(4B)	108.1
H(4A)-C(4)-H(4B)	109.5
C(4)-C(5)-C(6)	111.8(3)
C(4)-C(5)-H(5A)	110.4
C(6)-C(5)-H(5A)	110.5
C(4)-C(5)-H(5B)	106.4
C(6)-C(5)-H(5B)	108.1
H(5A)-C(5)-H(5B)	109.5
C(5)-C(6)-C(7)	111.4(2)
C(5)-C(6)-H(6A)	108.1
C(7)-C(6)-H(6A)	108.6
C(5)-C(6)-H(6B)	109.8
C(7)-C(6)-H(6B)	109.5
H(6A)-C(6)-H(6B)	109.5
N(1)-C(7)-C(6)	110.4(2)
N(1)-C(7)-C(2)	110.9(2)
C(6)-C(7)-C(2)	109.4(2)
N(1)-C(7)-H(7)	109.0
C(6)-C(7)-H(7)	109.9
C(2)-C(7)-H(7)	107.2
C(9)-C(8)-C(13)	119.0(3)
C(9)-C(8)-N(1)	121.9(3)
C(13)-C(8)-N(1)	119.1(3)
C(10)-C(9)-C(8)	121.3(3)

C(10)-C(9)-H(9)	119.8
C(8)-C(9)-H(9)	119.0
C(9)-C(10)-C(11)	119.6(3)
C(9)-C(10)-H(10)	121.0
C(11)-C(10)-H(10)	119.4
C(12)-C(11)-C(10)	118.9(3)
C(12)-C(11)-H(11)	118.8
C(10)-C(11)-H(11)	122.3
C(11)-C(12)-C(13)	121.1(3)
C(11)-C(12)-H(12)	117.8
C(13)-C(12)-H(12)	121.1
C(12)-C(13)-C(8)	120.2(3)
C(12)-C(13)-H(13)	120.6
C(8)-C(13)-H(13)	119.2
N(1)-C(14)-C(16)	111.6(2)
N(1)-C(14)-C(15)	109.9(2)
C(16)-C(14)-C(15)	110.4(2)
N(1)-C(14)-H(14)	108.4
C(16)-C(14)-H(14)	108.3
C(15)-C(14)-H(14)	108.2
C(1)-C(15)-C(14)	110.4(2)
C(1)-C(15)-H(15A)	110.7
C(14)-C(15)-H(15A)	110.7
C(1)-C(15)-H(15B)	108.6
C(14)-C(15)-H(15B)	106.8
H(15A)-C(15)-H(15B)	109.5
C(21)-C(16)-C(17)	117.7(3)
C(21)-C(16)-C(14)	121.3(3)
C(17)-C(16)-C(14)	121.0(2)
C(18)-C(17)-C(16)	121.9(3)
C(18)-C(17)-H(17)	119.3
C(16)-C(17)-H(17)	118.8
C(19)-C(18)-C(17)	119.3(3)
C(19)-C(18)-H(18)	120.2
C(17)-C(18)-H(18)	120.5
O(2)-C(19)-C(20)	115.5(3)
O(2)-C(19)-C(18)	124.1(3)
C(20)-C(19)-C(18)	120.4(3)
C(19)-C(20)-C(21)	119.3(3)

C(19)-C(20)-H(20)	120.7
C(21)-C(20)-H(20)	119.9
C(16)-C(21)-C(20)	121.5(3)
C(16)-C(21)-H(21)	119.1
C(20)-C(21)-H(21)	119.4
O(2)-C(22)-H(22A)	109.3
O(2)-C(22)-H(22B)	109.3
H(22A)-C(22)-H(22B)	109.4
O(2)-C(22)-H(22C)	109.9
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.4

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5504a. The anisotropic displacement factor exponent takes the form: $-2 \left[h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12} \right]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	53(1)	59(2)	35(1)	1(1)	27(1)	8(1)
O(2)	41(1)	70(2)	54(2)	6(1)	22(1)	0(1)
N(1)	40(1)	37(1)	28(1)	-1(1)	18(1)	0(1)
C(1)	45(2)	33(2)	33(2)	-4(1)	22(2)	1(1)
C(2)	42(2)	44(2)	32(2)	2(1)	22(1)	4(1)
C(3)	41(2)	57(2)	38(2)	0(1)	20(2)	7(2)
C(4)	36(2)	78(3)	37(2)	5(2)	14(2)	8(2)
C(5)	43(2)	75(2)	30(2)	4(2)	12(2)	5(2)
C(6)	46(2)	58(2)	30(2)	1(2)	18(2)	5(2)
C(7)	38(2)	39(2)	29(2)	2(1)	16(1)	0(1)
C(8)	32(2)	40(2)	29(2)	-2(1)	11(1)	-2(1)
C(9)	47(2)	37(2)	36(2)	2(1)	20(2)	0(1)
C(10)	49(2)	43(2)	44(2)	-4(2)	14(2)	-2(2)
C(11)	42(2)	54(2)	51(2)	-17(2)	19(2)	7(2)
C(12)	54(2)	51(2)	53(2)	-13(2)	34(2)	-5(2)
C(13)	48(2)	46(2)	44(2)	-3(2)	26(2)	-5(2)
C(14)	43(2)	43(2)	26(2)	1(1)	16(1)	-1(1)
C(15)	41(2)	47(2)	27(2)	-5(1)	14(1)	0(1)
C(16)	41(2)	41(2)	28(2)	-1(1)	17(1)	0(1)
C(17)	39(2)	44(2)	32(2)	6(1)	13(1)	1(2)

C(18)	45(2)	45(2)	35(2)	0(1)	18(2)	-8(2)
C(19)	39(2)	52(2)	34(2)	-3(2)	18(1)	-1(2)
C(20)	42(2)	52(2)	45(2)	7(2)	21(2)	9(2)
C(21)	47(2)	45(2)	41(2)	2(2)	25(2)	-3(2)
C(22)	47(2)	75(3)	62(2)	-1(2)	25(2)	-18(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5504a.

	x	y	z	U(eq)
H(2)	7233	3734	6070	45
H(3A)	5991	1190	6068	54
H(3B)	5986	3513	6176	54
H(4A)	5016	2530	4750	62
H(4B)	5750	3910	4786	62
H(5A)	5575	1216	3903	62
H(5B)	5734	-212	4662	62
H(6A)	7013	2314	4698	54
H(6B)	7011	-7	4581	54
H(7)	7228	-437	5982	42
H(9)	8196	-3056	6174	48
H(10)	8523	-5525	5444	59
H(11)	9112	-4557	4547	61
H(12)	9349	-1262	4414	59
H(13)	8988	1232	5125	53
H(14)	8743	-572	7255	45
H(15A)	8926	2191	8129	47
H(15B)	8747	3581	7352	47
H(17)	9772	3400	6757	48
H(18)	11266	3722	7278	51
H(20)	11667	-1436	8621	56
H(21)	10167	-1754	8069	51
H(22A)	13459	2646	8351	74
H(22B)	12691	4048	8213	74
H(22C)	12606	2699	7458	74

Single-Crystal X-ray Diffraction of 13

checkCIF/PLATON report

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 5470

Bond precision: C-C = 0.0034 Å Wavelength=0.71073

Cell: a=11.1580(3) b=9.0370(3) c=16.5660(5)
 alpha=90 beta=103.986(2) gamma=90

	Calculated	Reported
Volume	1620.91(9)	1620.91(8)
Space group	P 21/a	P 21/a
Hall group	-P 2yab	?
Moiety formula	C19 H21 N O S	C19 H21 N1 O1 S1
Sum formula	C19 H21 N O S	C19 H21 N O S
Mr	311.44	311.43
Dx, g cm-3	1.276	1.276
Z	4	4
Mu (mm-1)	0.201	0.201
F000	664.0	664.0
F000'	664.73	
h,k,lmax	13,10,19	13,10,19
Nref	2967	2950
Tmin,Tmax	0.908,0.932	0.781,1.066
Tmin'	0.860	

Correction method= 'MULTI-SCAN'

Data completeness= Ratio = 0.99 Theta(max) = 25.33

R(reflections)= 0.0591(2214) wR2(reflections)= 0.1678(2950)

S = 1.152 Npar= 207

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level A

PLAT773_ALERT_2_A Suspect C-C Bond in CIF: C17A -C16 .. 1.70 Ång.

Alert level B

PLAT201_ALERT_2_B Isotropic non-H Atoms in Main Residue(s) 1
PLAT241_ALERT_2_B Check High Ueq as Compared to Neighbors for S1

● Alert level C

ABSTM02_ALERT_3_C The ratio of expected to reported Tmax/Tmin(RR') is < 0.90

Tmin and Tmax reported: 0.781 1.066
Tmin(prime) and Tmax expected: 0.858 0.932
RR(prime) = 0.796

Please check that your absorption correction is appropriate.

ABSTY02_ALERT_1_C An exptl absorpt correction type has been given without a literature citation. This should be contained in the exptl_absorpt_process_details field.

Absorption correction given as multi-scan

HYDTR01_ALERT_1_C The hydrogen treatment should only be one of the following

keywords

* refall
* refxyz
* refU
* noref
* undef
* constr
* none
* mixed

Hydrogen treatment given as fixed

PLAT061_ALERT_3_C Tmax/Tmin Range Test RR' too Large	0.79
PLAT062_ALERT_4_C Rescale T(min) & T(max) by	0.87
PLAT063_ALERT_3_C Crystal Probably too Large for Beam Size	0.75 mm
PLAT128_ALERT_4_C Non-standard setting of Space group P21/c	P21/a
PLAT152_ALERT_1_C Supplied and Calc Volume s.u. Inconsistent	?
PLAT161_ALERT_4_C Missing or Zero su (esd) on x-coordinate for ...	C17A
PLAT162_ALERT_4_C Missing or Zero su (esd) on y-coordinate for ...	C17A
PLAT163_ALERT_4_C Missing or Zero su (esd) on z-coordinate for ...	C17A
PLAT220_ALERT_2_C Large Non-Solvent C Ueq(max)/Ueq(min) ...	3.21 Ratio
PLAT222_ALERT_3_C Large Non-Solvent H Ueq(max)/Ueq(min) ...	3.20 Ratio
PLAT241_ALERT_2_C Check High Ueq as Compared to Neighbors for	C18
PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for	C17A
PLAT242_ALERT_2_C Check Low Ueq as Compared to Neighbors for	C17
PLAT301_ALERT_3_C Main Residue Disorder	8.00 Perc.
PLAT366_ALERT_2_C Short? C(sp?)-C(sp?) Bond C18 - C19	1.32 Ang.
PLAT779_ALERT_2_C Suspect or Irrelevant (Bond) Angle in CIF	6.80 Deg.
C17 -C16 -S1A 1.555 1.555 1.555	
PLAT779_ALERT_2_C Suspect or Irrelevant (Bond) Angle in CIF	3.92 Deg.
S1 -C16 -C17A 1.555 1.555 1.555	
PLAT779_ALERT_2_C Suspect or Irrelevant (Bond) Angle in CIF	18.20 Deg.
S1A -C18 -C17 1.555 1.555 1.555	
PLAT779_ALERT_2_C Suspect or Irrelevant (Bond) Angle in CIF	5.33 Deg.
S1 -C19 -C17A 1.555 1.555 1.555	

● Alert level G

ABSTM02_ALERT_3_G When printed, the submitted absorption T values will be replaced by the scaled T values. Since the ratio of scaled T's is identical to the ratio of reported T values, the scaling does not imply a change to the absorption corrections used in the study.

Ratio of Tmax-expected/reported 0.874
Tmax scaled 0.932 Tmin scaled 0.683

-
- 1 ALERT level A = In general; serious problem
 - 2 ALERT level B = Potentially serious problem
 - 22 ALERT level C = Check and explain
 - 1 ALERT level G = General alerts; check

- 3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 12 ALERT type 2 Indicator that the structure model may be wrong or deficient
 6 ALERT type 3 Indicator that the structure quality may be low
 5 ALERT type 4 Improvement, methodology, query or suggestion

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 12/04/2005; check.def file version of 22/03/2005

Datablock 5470 - ellipsoid plot

Table 1. Crystal data and structure refinement for 5470.

Identification code	5470
Empirical formula	C ₁₉ H ₂₁ N O S
Formula weight	311.43
Temperature	200(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P 21/a
Unit cell dimensions	a = 11.1580(3) Å = 90°.
	b = 9.0370(3) Å = 103.986(2)°.
	c = 16.5660(5) Å = 90°.
Volume	1620.91(8) Å ³

Z	4
Density (calculated)	1.276 Mg/m ³
Absorption coefficient	0.201 mm ⁻¹
F(000)	664
Crystal size	0.75 x 0.4 x 0.35 mm ³
Theta range for data collection	2.53 to 25.33°.
Index ranges	-13<=h<=13, -10<=k<=10, -19<=l<=18
Reflections collected	10012
Independent reflections	2950 [R(int) = 0.0584]
Completeness to theta = 25.33°	99.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0659 and 0.781
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2950 / 0 / 207
Goodness-of-fit on F ²	1.152
Final R indices [I>2sigma(I)]	R1 = 0.0591, wR2 = 0.1551
R indices (all data)	R1 = 0.0783, wR2 = 0.1678
Extinction coefficient	0.023(4)
Largest diff. peak and hole	0.371 and -0.577 e.Å ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å²x 10³) for 5470. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	4780(2)	2598(2)	3517(1)	90(1)
C(17A)	4780	2680	3428	25(1)
O(1)	1175(2)	-1472(2)	3312(1)	73(1)
N(1)	4154(2)	-94(2)	2426(1)	36(1)
C(1)	2050(2)	-1008(3)	3075(2)	49(1)
C(2)	1909(2)	121(3)	2384(1)	41(1)
C(3)	628(2)	137(3)	1795(2)	52(1)
C(4)	487(2)	1367(3)	1166(2)	54(1)
C(5)	1481(2)	1303(3)	688(2)	52(1)
C(6)	2756(2)	1274(2)	1278(1)	39(1)
C(7)	2896(2)	-29(2)	1888(1)	34(1)
C(8)	5153(2)	-359(2)	2075(1)	33(1)
C(9)	4995(2)	-887(2)	1262(1)	38(1)
C(10)	5991(2)	-1079(3)	915(2)	46(1)

C(11)	7172(2)	-778(3)	1357(2)	51(1)
C(12)	7346(2)	-276(3)	2165(2)	48(1)
C(13)	6364(2)	-60(3)	2519(1)	41(1)
C(14)	4298(2)	-346(3)	3320(1)	40(1)
C(15)	3348(2)	-1478(3)	3461(2)	51(1)
C(16)	4238(2)	1099(3)	3773(1)	41(1)
C(17)	3920(14)	1255(19)	4409(10)	38(5)
S(1A)	3620(2)	1333(2)	4593(1)	56(1)
C(18)	3909(3)	2980(4)	4698(2)	80(1)
C(19)	4435(3)	3655(3)	4168(2)	68(1)

Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for 5470.

S(1)-C(19)	1.557(3)
S(1)-C(16)	1.582(3)
C(17A)-C(19)	1.629(3)
C(17A)-C(16)	1.702(2)
C(17A)-H(17A)	0.9300
O(1)-C(1)	1.212(3)
N(1)-C(8)	1.398(3)
N(1)-C(14)	1.468(3)
N(1)-C(7)	1.471(3)
C(1)-C(15)	1.496(4)
C(1)-C(2)	1.513(3)
C(2)-C(3)	1.523(3)
C(2)-C(7)	1.531(3)
C(2)-H(2)	0.9800
C(3)-C(4)	1.505(4)
C(3)-H(3A)	0.9700
C(3)-H(3B)	0.9700
C(4)-C(5)	1.512(4)
C(4)-H(4A)	0.9700
C(4)-H(4B)	0.9700
C(5)-C(6)	1.519(3)
C(5)-H(5A)	0.9700
C(5)-H(5B)	0.9700
C(6)-C(7)	1.534(3)
C(6)-H(6A)	0.9700
C(6)-H(6B)	0.9700

C(7)-H(7)	0.9800
C(8)-C(13)	1.400(3)
C(8)-C(9)	1.398(3)
C(9)-C(10)	1.380(3)
C(9)-H(9)	0.9300
C(10)-C(11)	1.371(3)
C(10)-H(10)	0.9300
C(11)-C(12)	1.382(4)
C(11)-H(11)	0.9300
C(12)-C(13)	1.376(3)
C(12)-H(12)	0.9300
C(13)-H(13)	0.9300
C(14)-C(16)	1.515(3)
C(14)-C(15)	1.530(3)
C(14)-H(14)	0.9800
C(15)-H(15A)	0.9700
C(15)-H(15B)	0.9700
C(16)-C(17)	1.199(15)
C(16)-S(1A)	1.680(3)
C(17)-C(18)	1.632(17)
C(17)-H(17)	0.9300
S(1A)-C(18)	1.524(4)
C(18)-C(19)	1.317(4)
C(18)-H(18)	0.9300
C(19)-H(19)	0.9300
C(19)-S(1)-C(16)	99.55(19)
C(19)-C(17A)-C(16)	91.96(13)
C(19)-C(17A)-H(17A)	134.0
C(16)-C(17A)-H(17A)	134.0
C(8)-N(1)-C(14)	119.36(17)
C(8)-N(1)-C(7)	119.60(16)
C(14)-N(1)-C(7)	118.27(16)
O(1)-C(1)-C(15)	122.4(2)
O(1)-C(1)-C(2)	122.2(3)
C(15)-C(1)-C(2)	115.32(19)
C(1)-C(2)-C(3)	113.65(19)
C(1)-C(2)-C(7)	112.92(19)
C(3)-C(2)-C(7)	110.04(18)

C(1)-C(2)-H(2)	106.6
C(3)-C(2)-H(2)	106.6
C(7)-C(2)-H(2)	106.6
C(4)-C(3)-C(2)	112.01(19)
C(4)-C(3)-H(3A)	109.2
C(2)-C(3)-H(3A)	109.2
C(4)-C(3)-H(3B)	109.2
C(2)-C(3)-H(3B)	109.2
H(3A)-C(3)-H(3B)	107.9
C(3)-C(4)-C(5)	111.6(2)
C(3)-C(4)-H(4A)	109.3
C(5)-C(4)-H(4A)	109.3
C(3)-C(4)-H(4B)	109.3
C(5)-C(4)-H(4B)	109.3
H(4A)-C(4)-H(4B)	108.0
C(4)-C(5)-C(6)	110.8(2)
C(4)-C(5)-H(5A)	109.5
C(6)-C(5)-H(5A)	109.5
C(4)-C(5)-H(5B)	109.5
C(6)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	108.1
C(5)-C(6)-C(7)	111.55(18)
C(5)-C(6)-H(6A)	109.3
C(7)-C(6)-H(6A)	109.3
C(5)-C(6)-H(6B)	109.3
C(7)-C(6)-H(6B)	109.3
H(6A)-C(6)-H(6B)	108.0
N(1)-C(7)-C(2)	112.49(17)
N(1)-C(7)-C(6)	111.11(17)
C(2)-C(7)-C(6)	108.24(18)
N(1)-C(7)-H(7)	108.3
C(2)-C(7)-H(7)	108.3
C(6)-C(7)-H(7)	108.3
N(1)-C(8)-C(13)	120.89(19)
N(1)-C(8)-C(9)	122.15(19)
C(13)-C(8)-C(9)	116.93(19)
C(10)-C(9)-C(8)	121.1(2)
C(10)-C(9)-H(9)	119.4
C(8)-C(9)-H(9)	119.4

C(11)-C(10)-C(9)	121.3(2)
C(11)-C(10)-H(10)	119.3
C(9)-C(10)-H(10)	119.3
C(10)-C(11)-C(12)	118.3(2)
C(10)-C(11)-H(11)	120.8
C(12)-C(11)-H(11)	120.8
C(13)-C(12)-C(11)	121.3(2)
C(13)-C(12)-H(12)	119.4
C(11)-C(12)-H(12)	119.4
C(12)-C(13)-C(8)	121.0(2)
C(12)-C(13)-H(13)	119.5
C(8)-C(13)-H(13)	119.5
N(1)-C(14)-C(16)	110.94(18)
N(1)-C(14)-C(15)	110.27(18)
C(16)-C(14)-C(15)	112.74(18)
N(1)-C(14)-H(14)	107.6
C(16)-C(14)-H(14)	107.6
C(15)-C(14)-H(14)	107.6
C(1)-C(15)-C(14)	112.3(2)
C(1)-C(15)-H(15A)	109.1
C(14)-C(15)-H(15A)	109.1
C(1)-C(15)-H(15B)	109.1
C(14)-C(15)-H(15B)	109.1
H(15A)-C(15)-H(15B)	107.9
C(17)-C(16)-C(14)	126.4(8)
C(17)-C(16)-S(1)	110.4(8)
C(14)-C(16)-S(1)	122.75(17)
C(17)-C(16)-S(1A)	6.8(8)
C(14)-C(16)-S(1A)	125.73(17)
S(1)-C(16)-S(1A)	111.52(16)
C(17)-C(16)-C(17A)	113.6(8)
C(14)-C(16)-C(17A)	119.76(15)
S(1)-C(16)-C(17A)	3.92(7)
S(1A)-C(16)-C(17A)	114.46(14)
C(16)-C(17)-C(18)	113.3(11)
C(16)-C(17)-H(17)	123.4
C(18)-C(17)-H(17)	123.4
C(18)-S(1A)-C(16)	95.61(17)
C(19)-C(18)-S(1A)	119.5(2)

C(19)-C(18)-C(17)	102.2(6)
S(1A)-C(18)-C(17)	18.2(5)
C(19)-C(18)-H(18)	128.9
S(1A)-C(18)-H(18)	111.4
C(17)-C(18)-H(18)	128.9
C(18)-C(19)-S(1)	113.7(3)
C(18)-C(19)-C(17A)	118.4(2)
S(1)-C(19)-C(17A)	5.33(8)
C(18)-C(19)-H(19)	123.2
S(1)-C(19)-H(19)	123.2
C(17A)-C(19)-H(19)	118.4

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5470. The anisotropic displacement factor exponent takes the form: $-2 \text{ }^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	152(3)	59(1)	58(1)	-10(1)	23(1)	-16(1)
O(1)	73(1)	86(2)	75(1)	-8(1)	48(1)	-28(1)
N(1)	33(1)	46(1)	31(1)	-2(1)	11(1)	2(1)
C(1)	58(2)	49(2)	50(1)	-16(1)	30(1)	-19(1)
C(2)	37(1)	41(1)	48(1)	-15(1)	18(1)	-7(1)
C(3)	33(1)	60(2)	66(2)	-26(1)	18(1)	-10(1)
C(4)	35(1)	56(2)	65(2)	-19(1)	1(1)	6(1)
C(5)	45(2)	52(2)	54(1)	-3(1)	1(1)	6(1)
C(6)	37(1)	39(1)	42(1)	-2(1)	10(1)	0(1)
C(7)	33(1)	33(1)	38(1)	-8(1)	13(1)	-2(1)
C(8)	33(1)	30(1)	37(1)	2(1)	11(1)	2(1)
C(9)	38(1)	36(1)	42(1)	-3(1)	14(1)	1(1)
C(10)	49(2)	45(1)	51(1)	0(1)	24(1)	6(1)
C(11)	44(2)	49(2)	69(2)	7(1)	31(1)	6(1)
C(12)	33(1)	48(2)	63(2)	10(1)	11(1)	1(1)
C(13)	38(1)	42(1)	40(1)	3(1)	7(1)	-1(1)
C(14)	45(1)	42(1)	33(1)	3(1)	12(1)	-1(1)
C(15)	66(2)	43(1)	48(1)	2(1)	21(1)	-10(1)
C(16)	42(1)	50(1)	31(1)	-3(1)	7(1)	-3(1)
S(1A)	64(1)	65(1)	41(1)	-9(1)	19(1)	-5(1)

C(18)	73(2)	115(3)	51(2)	-37(2)	15(2)	-4(2)
C(19)	81(2)	49(2)	69(2)	-18(1)	8(2)	-7(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5470.

	x	y	z	U(eq)
H(17A)	5135	2901	2989	30
H(2)	2028	1095	2651	49
H(3A)	480	-803	1506	62
H(3B)	13	255	2116	62
H(4A)	530	2312	1450	64
H(4B)	-317	1294	780	64
H(5A)	1412	2159	327	63
H(5B)	1368	423	343	63
H(6A)	2894	2194	1589	47
H(6B)	3376	1197	959	47
H(7)	2741	-947	1564	40
H(9)	4205	-1114	951	45
H(10)	5859	-1419	371	55
H(11)	7839	-909	1119	61
H(12)	8143	-80	2476	57
H(13)	6507	290	3062	49
H(14)	5120	-771	3541	47
H(15A)	3505	-2421	3226	61
H(15B)	3445	-1615	4054	61
H(17)	3702	471	4707	46
H(18)	3609	3383	5127	96
H(19)	4586	4668	4182	81

Single-Crystal X-ray Diffraction of 13 α

checkCIF/PLATON report

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 5518

Bond precision: C-C = 0.0061 Å Wavelength=0.71073

Cell: a=9.4810(4) b=11.5940(5) c=15.4390(7)
 alpha=90 beta=91.988(2) gamma=90

	Calculated	Reported
Volume	1696.08(13)	1696.08(13)
Space group	P 21/a	P 21/a
Hall group	-P 2yab	?
Moiety formula	C19 H21 N O S	C19 H21 N O S
Sum formula	C19 H21 N O S	C19 H21 N O S
Mr	311.44	311.43
Dx, g cm-3	1.220	1.220
Z	4	4
Mu (mm-1)	0.192	0.192
F000	664.0	664.0
F000'	664.73	
h,k,lmax	11,13,18	11,13,18
Nref	3094	3089
Tmin,Tmax	0.966,0.990	0.953,1.014
Tmin'	0.966	

Correction method= 'MULTI-SCAN'

Data completeness= Ratio = 1.00 Theta(max)= 25.34

R(reflections)= 0.0648(1830) wR2(reflections)= 0.2283(3089)

S = 1.040 Npar= 200

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level C

ABSTY02_ALERT_1_C An _exptl_absorpt_correction_type has been given without a literature citation. This should be contained in the _exptl_absorpt_process_details field.

Absorption correction given as multi-scan

HYDTR01_ALERT_1_C The hydrogen treatment should only be one of the following

keywords

* reall

```

* refxyz
* refU
* noref
* undef
* constr
* none
* mixed
Hydrogen treatment given as fixed
RINTA01_ALERT_3_C The value of Rint is greater than 0.10
      Rint given    0.106
PLAT020_ALERT_3_C The value of Rint is greater than 0.10 .....      0.11
PLAT062_ALERT_4_C Rescale T(min) & T(max) by .....      0.98
PLAT128_ALERT_4_C Non-standard setting of Space group P21/c ....      P21/a
PLAT340_ALERT_3_C Low Bond Precision on C-C bonds (x 1000) Ang ...      6

```

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0 ALERT level A = In general: serious problem
0 ALERT level B = Potentially serious problem
7 ALERT level C = Check and explain
0 ALERT level G = General alerts; check

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
0 ALERT type 2 Indicator that the structure model may be wrong or deficient
3 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion

```

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 12/04/2005; check.def file version of 22/03/2005

Datablock 5518 - ellipsoid plot

Table 1. Crystal data and structure refinement for 5518.

Identification code	5518	
Empirical formula	C ₁₉ H ₂₁ N O S	
Formula weight	311.43	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 2 ₁ /a	
Unit cell dimensions	a = 9.4810(4) Å	= 90°.
	b = 11.5940(5) Å	= 91.988(2)°.
	c = 15.4390(7) Å	= 90°.
Volume	1696.08(13) Å ³	
Z	4	
Density (calculated)	1.220 Mg/m ³	
Absorption coefficient	0.192 mm ⁻¹	
F(000)	664	
Crystal size	0.18 x 0.17 x 0.05 mm ³	
Theta range for data collection	2.20 to 25.34°.	
Index ranges	-11 ≤ h ≤ 11, -13 ≤ k ≤ 13, -16 ≤ l ≤ 18	
Reflections collected	14184	

Independent reflections	3089 [R(int) = 0.1060]
Completeness to theta = 25.34°	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0139 and 0.9527
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3089 / 0 / 200
Goodness-of-fit on F ²	1.040
Final R indices [I>2sigma(I)]	R1 = 0.0648, wR2 = 0.1680
R indices (all data)	R1 = 0.1268, wR2 = 0.2283
Extinction coefficient	0.025(5)
Largest diff. peak and hole	0.707 and -0.386 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5518. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	2232(1)	4469(1)	-204(1)	58(1)
O(1)	-296(3)	6873(3)	2136(2)	45(1)
N(1')	2024(3)	8528(3)	3005(2)	39(1)
C(1)	3476(5)	3594(5)	-627(3)	65(1)
C(2)	4512(5)	3333(4)	-54(3)	63(1)
C(3)	4323(5)	3851(4)	755(3)	56(1)
C(4)	3113(4)	4502(4)	790(3)	44(1)
C(5)	2622(4)	5159(4)	1509(3)	42(1)
C(6)	1416(4)	5754(4)	1512(2)	39(1)
C(7)	866(4)	6402(3)	2226(2)	36(1)
C(8)	1654(4)	6466(3)	3106(2)	33(1)
C(9)	1121(4)	5465(3)	3664(2)	37(1)
C(10)	1751(4)	5499(4)	4590(3)	40(1)
C(11)	1485(4)	6652(4)	5015(2)	39(1)
C(12)	2058(4)	7625(4)	4470(3)	39(1)
C(13)	1437(4)	7631(3)	3544(2)	33(1)
C(14)	1639(4)	9671(3)	3044(2)	34(1)
C(15)	2404(4)	10494(4)	2576(3)	40(1)
C(16)	1996(5)	11646(4)	2562(3)	51(1)
C(17)	820(5)	12005(4)	3008(3)	55(1)
C(18)	91(5)	11209(4)	3473(3)	50(1)
C(19)	489(4)	10064(4)	3502(3)	43(1)

Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for 5518.

S(1)-C(1)	1.703(5)
S(1)-C(4)	1.722(4)
O(1)-C(7)	1.233(4)
N(1')-C(14)	1.376(5)
N(1')-C(13)	1.455(5)
N(1')-H(1')	0.9764
C(1)-C(2)	1.334(7)
C(1)-H(1)	0.9589
C(2)-C(3)	1.402(6)
C(2)-H(2)	0.9600
C(3)-C(4)	1.375(6)
C(3)-H(3)	0.9600
C(4)-C(5)	1.437(6)
C(5)-C(6)	1.335(6)
C(5)-H(5)	0.9593
C(6)-C(7)	1.447(5)
C(6)-H(6)	0.9600
C(7)-C(8)	1.529(5)
C(8)-C(13)	1.527(5)
C(8)-C(9)	1.541(5)
C(8)-H(8)	0.9596
C(9)-C(10)	1.530(5)
C(9)-H(9A)	0.9595
C(9)-H(9B)	0.9597
C(10)-C(11)	1.514(6)
C(10)-H(10A)	0.9595
C(10)-H(10B)	0.9600
C(11)-C(12)	1.520(6)
C(11)-H(11A)	0.9599
C(11)-H(11B)	0.9605
C(12)-C(13)	1.527(5)
C(12)-H(12A)	0.9610
C(12)-H(12B)	0.9598
C(13)-H(13)	0.9595
C(14)-C(19)	1.397(6)
C(14)-C(15)	1.412(5)
C(15)-C(16)	1.390(6)

C(15)-H(15)	0.9610
C(16)-C(17)	1.394(7)
C(16)-H(16)	0.9604
C(17)-C(18)	1.370(7)
C(17)-H(17)	0.9609
C(18)-C(19)	1.380(6)
C(18)-H(18)	0.9596
C(19)-H(19)	0.9595

C(1)-S(1)-C(4)	91.9(2)
C(14)-N(1')-C(13)	123.8(3)
C(14)-N(1')-H(1')	114.8
C(13)-N(1')-H(1')	116.9
C(2)-C(1)-S(1)	112.6(4)
C(2)-C(1)-H(1)	128.0
S(1)-C(1)-H(1)	119.4
C(1)-C(2)-C(3)	112.4(4)
C(1)-C(2)-H(2)	127.5
C(3)-C(2)-H(2)	120.1
C(4)-C(3)-C(2)	113.7(4)
C(4)-C(3)-H(3)	118.6
C(2)-C(3)-H(3)	127.7
C(3)-C(4)-C(5)	127.9(4)
C(3)-C(4)-S(1)	109.4(3)
C(5)-C(4)-S(1)	122.6(3)
C(6)-C(5)-C(4)	125.3(4)
C(6)-C(5)-H(5)	118.6
C(4)-C(5)-H(5)	116.1
C(5)-C(6)-C(7)	127.1(4)
C(5)-C(6)-H(6)	113.3
C(7)-C(6)-H(6)	119.6
O(1)-C(7)-C(6)	119.2(4)
O(1)-C(7)-C(8)	119.1(3)
C(6)-C(7)-C(8)	121.6(3)
C(13)-C(8)-C(7)	111.6(3)
C(13)-C(8)-C(9)	111.5(3)
C(7)-C(8)-C(9)	107.5(3)
C(13)-C(8)-H(8)	108.5
C(7)-C(8)-H(8)	109.3

C(9)-C(8)-H(8)	108.5
C(10)-C(9)-C(8)	112.2(3)
C(10)-C(9)-H(9A)	108.1
C(8)-C(9)-H(9A)	108.7
C(10)-C(9)-H(9B)	108.6
C(8)-C(9)-H(9B)	109.6
H(9A)-C(9)-H(9B)	109.5
C(11)-C(10)-C(9)	111.3(3)
C(11)-C(10)-H(10A)	109.6
C(9)-C(10)-H(10A)	108.7
C(11)-C(10)-H(10B)	109.0
C(9)-C(10)-H(10B)	108.6
H(10A)-C(10)-H(10B)	109.5
C(10)-C(11)-C(12)	110.3(3)
C(10)-C(11)-H(11A)	109.7
C(12)-C(11)-H(11A)	108.8
C(10)-C(11)-H(11B)	109.7
C(12)-C(11)-H(11B)	109.0
H(11A)-C(11)-H(11B)	109.4
C(11)-C(12)-C(13)	112.8(3)
C(11)-C(12)-H(12A)	109.3
C(13)-C(12)-H(12A)	107.9
C(11)-C(12)-H(12B)	109.4
C(13)-C(12)-H(12B)	107.9
H(12A)-C(12)-H(12B)	109.5
N(1')-C(13)-C(12)	113.3(3)
N(1')-C(13)-C(8)	108.6(3)
C(12)-C(13)-C(8)	110.9(3)
N(1')-C(13)-H(13)	107.9
C(12)-C(13)-H(13)	107.6
C(8)-C(13)-H(13)	108.5
N(1')-C(14)-C(19)	123.3(3)
N(1')-C(14)-C(15)	119.2(3)
C(19)-C(14)-C(15)	117.5(4)
C(16)-C(15)-C(14)	120.7(4)
C(16)-C(15)-H(15)	120.1
C(14)-C(15)-H(15)	119.2
C(15)-C(16)-C(17)	120.4(4)
C(15)-C(16)-H(16)	120.0

C(17)-C(16)-H(16)	119.7
C(18)-C(17)-C(16)	119.0(4)
C(18)-C(17)-H(17)	121.4
C(16)-C(17)-H(17)	119.7
C(17)-C(18)-C(19)	121.5(4)
C(17)-C(18)-H(18)	118.3
C(19)-C(18)-H(18)	120.2
C(18)-C(19)-C(14)	121.0(4)
C(18)-C(19)-H(19)	119.9
C(14)-C(19)-H(19)	119.1

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5518. The anisotropic displacement factor exponent takes the form: $-2 \pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	59(1)	75(1)	40(1)	-18(1)	-7(1)	11(1)
O(1)	31(1)	65(2)	41(2)	4(1)	5(1)	6(1)
N(1')	35(2)	31(2)	52(2)	5(2)	15(2)	-3(2)
C(1)	80(3)	72(4)	44(3)	-19(3)	4(2)	9(3)
C(2)	67(3)	59(3)	62(3)	-15(3)	13(3)	19(3)
C(3)	55(3)	70(3)	44(3)	-7(2)	-2(2)	17(2)
C(4)	46(2)	49(3)	36(2)	-7(2)	2(2)	2(2)
C(5)	40(2)	52(3)	34(2)	-4(2)	0(2)	-3(2)
C(6)	36(2)	50(3)	32(2)	-3(2)	-1(2)	-2(2)
C(7)	33(2)	37(2)	37(2)	1(2)	7(2)	-3(2)
C(8)	28(2)	38(2)	35(2)	-1(2)	4(2)	0(2)
C(9)	42(2)	34(2)	36(2)	-1(2)	8(2)	-2(2)
C(10)	39(2)	42(2)	39(2)	8(2)	1(2)	4(2)
C(11)	39(2)	48(3)	31(2)	0(2)	0(2)	0(2)
C(12)	37(2)	41(2)	40(2)	-10(2)	4(2)	-3(2)
C(13)	32(2)	32(2)	34(2)	3(2)	7(2)	-3(2)
C(14)	35(2)	33(2)	33(2)	2(2)	-1(2)	-1(2)
C(15)	38(2)	42(3)	40(2)	4(2)	-6(2)	-4(2)
C(16)	63(3)	34(3)	54(3)	9(2)	-17(2)	-11(2)
C(17)	65(3)	34(3)	64(3)	-6(2)	-16(2)	4(2)
C(18)	52(2)	39(3)	60(3)	-11(2)	-3(2)	8(2)

C(19)	43(2)	39(3)	46(2)	-3(2)	2(2)	-3(2)
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Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5518.

	x	y	z	U(eq)
H(1')	2963	8390	2786	47
H(1)	3374	3353	-1220	78
H(2)	5321	2855	-143	75
H(3)	4928	3797	1265	68
H(5)	3213	5148	2026	50
H(6)	903	5740	965	47
H(8)	2645	6361	3026	40
H(9A)	114	5521	3693	44
H(9B)	1366	4743	3405	44
H(10A)	2748	5364	4572	48
H(10B)	1322	4901	4921	48
H(11A)	489	6763	5071	47
H(11B)	1945	6671	5579	47
H(12A)	1838	8352	4732	47
H(12B)	3063	7547	4440	47
H(13)	442	7768	3575	39
H(15)	3212	10249	2265	48
H(16)	2525	12199	2243	61
H(17)	535	12800	2983	66
H(18)	-706	11467	3787	61
H(19)	-33	9525	3837	51

Single-Crystal X-ray Diffraction of 15

checkCIF/PLATON report

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 5505

Bond precision: C-C = 0.0061 Å Wavelength=0.71073

Cell: a=34.5090(10) b=6.8140(3) c=18.0570(7)
 alpha=90 beta=120.888(2) gamma=90

	Calculated	Reported
Volume	3643.8(3)	3643.8(2)
Space group	C 2/c	C 2/c
Hall group	-C 2yc	?
Moiety formula	C21 H22 Cl N O	C21 H22 Cl1 N1 O1
Sum formula	C21 H22 Cl N O	C21 H22 Cl N O
Mr	339.85	339.85
Dx, g cm-3	1.239	1.239
Z	8	8
Mu (mm-1)	0.216	0.216
F000	1440.0	1440.0
F000'	1441.69	
h,k,lmax	41,8,21	41,8,21
Nref	3331	3319
Tmin,Tmax	0.962,0.979	0.887,1.046
Tmin'	0.952	

Correction method= 'MULTI-SCAN'

Data completeness= Ratio = 1.00 Theta(max) = 25.32

R(reflections)= 0.0595(1886) wR2(reflections)= 0.2216(3319)

S = 1.017 Npar= 217

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

ABSTM02_ALERT_3_C The ratio of expected to reported Tmax/Tmin(RR') is < 0.90
Tmin and Tmax reported: 0.887 1.046
Tmin(prime) and Tmax expected: 0.951 0.979
RR(prime) = 0.873
Please check that your absorption correction is appropriate.
ABSTY02_ALERT_1_C An _exptl_absorpt_correction_type has been given without
a literature citation. This should be contained in the

```

      _exptl absorpt_process_details field.
      Absorption correction given as multi-scan
HYDTR01_ALERT_1_C The hydrogen treatment should only be one of the following
      keywords
      * refall
      * refxyz
      * refU
      * noref
      * undef
      * constr
      * none
      * mixed
      Hydrogen treatment given as fixed
PLAT061_ALERT_3_C Tmax/Tmin Range Test RR' too Large ..... 0.87
PLAT062_ALERT_4_C Rescale T(min) & T(max) by ..... 0.94
PLAT152_ALERT_1_C Supplied and Calc Volume s.u. Inconsistent ..... ?
PLAT230_ALERT_2_C Hirshfeld Test Diff for C19 - C20 .. 6.20 su
PLAT340_ALERT_3_C Low Bond Precision on C-C bonds (x 1000) Ang ... 6

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Alert level G

ABSTM02_ALERT_3_G When printed, the submitted absorption T values will be replaced by the scaled T values. Since the ratio of scaled T's is identical to the ratio of reported T values, the scaling does not imply a change to the absorption corrections used in the study.

```

Ratio of Tmax expected/reported      0.936
Tmax scaled      0.979 Tmin scaled    0.830

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- 0 ALERT level A = In general: serious problem
 - 0 ALERT level B = Potentially serious problem
 - 8 ALERT level C = Check and explain
 - 1 ALERT level G = General alerts; check

 - 3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 - 1 ALERT type 2 Indicator that the structure model may be wrong or deficient
 - 4 ALERT type 3 Indicator that the structure quality may be low
 - 1 ALERT type 4 Improvement, methodology, query or suggestion
-

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 12/04/2005; check.def file version of 22/03/2005

Datablock 5505 - ellipsoid plot

Table 1. Crystal data and structure refinement for 5505.

Identification code	5505	
Empirical formula	C ₂₁ H ₂₂ Cl N O	
Formula weight	339.85	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 34.5090(10) Å	= 90°.
	b = 6.8140(3) Å	= 120.888(2)°.
	c = 18.0570(7) Å	= 90°.
Volume	3643.8(2) Å ³	
Z	8	
Density (calculated)	1.239 Mg/m ³	
Absorption coefficient	0.216 mm ⁻¹	
F(000)	1440	
Crystal size	0.23 x 0.15 x 0.1 mm ³	
Theta range for data collection	2.26 to 25.32°.	
Index ranges	-41<=h<=41, -7<=k<=8, -20<=l<=21	
Reflections collected	12249	

Independent reflections	3319 [R(int) = 0.0982]
Completeness to theta = 25.32°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0458 and 0.8869
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3319 / 0 / 217
Goodness-of-fit on F ²	1.017
Final R indices [I>2sigma(I)]	R1 = 0.0595, wR2 = 0.1590
R indices (all data)	R1 = 0.1280, wR2 = 0.2216
Largest diff. peak and hole	0.369 and -0.390 e.Å ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å²x 10³) for 5505. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	7757(1)	5238(5)	224(2)	36(1)
C(2)	7970(1)	5371(5)	-324(2)	36(1)
C(3)	7631(1)	5480(6)	-1285(3)	51(1)
C(4)	7862(1)	5665(7)	-1810(3)	61(1)
C(5)	8206(1)	4047(7)	-1585(3)	54(1)
C(6)	8543(1)	3947(6)	-623(2)	42(1)
C(7)	8307(1)	3682(5)	-107(2)	32(1)
C(8)	8396(1)	3214(5)	1319(2)	35(1)
C(9)	8087(1)	4959(5)	1173(3)	40(1)
C(10)	8723(1)	2866(5)	2269(2)	37(1)
C(11)	8696(1)	1160(6)	2650(3)	53(1)
C(12)	8980(2)	833(8)	3518(3)	74(2)
C(13)	9294(2)	2222(8)	4027(3)	67(1)
C(14)	9320(1)	3920(8)	3659(3)	60(1)
C(15)	9041(1)	4254(6)	2790(3)	46(1)
C(16)	8970(1)	2048(5)	1021(2)	32(1)
C(17)	8861(1)	57(5)	927(3)	40(1)
C(18)	9184(1)	-1345(6)	1082(2)	43(1)
C(19)	9617(1)	-757(5)	1334(2)	40(1)
C(20)	9733(1)	1202(6)	1443(3)	47(1)
C(21)	9406(1)	2596(5)	1289(3)	41(1)
N(1)	8635(1)	3559(4)	838(2)	32(1)
O(1)	7351(1)	5285(4)	-75(2)	44(1)

Cl(1)	10024(1)	-2498(2)	1524(1)	60(1)
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Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for 5505.

C(1)-O(1)	1.214(4)
C(1)-C(9)	1.506(5)
C(1)-C(2)	1.510(5)
C(2)-C(3)	1.517(5)
C(2)-C(7)	1.536(5)
C(2)-H(2)	0.9600
C(3)-C(4)	1.527(6)
C(3)-H(3A)	0.9601
C(3)-H(3B)	0.9600
C(4)-C(5)	1.515(6)
C(4)-H(4A)	0.9600
C(4)-H(4B)	0.9600
C(5)-C(6)	1.517(5)
C(5)-H(5A)	0.9601
C(5)-H(5B)	0.9600
C(6)-C(7)	1.532(5)
C(6)-H(6A)	0.9601
C(6)-H(6B)	0.9599
C(7)-N(1)	1.489(4)
C(7)-H(7)	0.9600
C(8)-N(1)	1.492(4)
C(8)-C(10)	1.512(5)
C(8)-C(9)	1.527(5)
C(8)-H(8)	0.9600
C(9)-H(9A)	0.9600
C(9)-H(9B)	0.9600
C(10)-C(11)	1.378(5)
C(10)-C(15)	1.388(5)
C(11)-C(12)	1.374(6)
C(11)-H(11)	0.9601
C(12)-C(13)	1.375(7)
C(12)-H(12)	0.9600
C(13)-C(14)	1.360(6)
C(13)-H(13)	0.9600
C(14)-C(15)	1.376(6)

C(14)-H(14)	0.9600
C(15)-H(15)	0.9600
C(16)-C(21)	1.377(5)
C(16)-C(17)	1.394(5)
C(16)-N(1)	1.453(4)
C(17)-C(18)	1.381(5)
C(17)-H(17)	0.9599
C(18)-C(19)	1.380(5)
C(18)-H(18)	0.9600
C(19)-C(20)	1.378(5)
C(19)-Cl(1)	1.734(3)
C(20)-C(21)	1.389(5)
C(20)-H(20)	0.9600
C(21)-H(21)	0.9600

O(1)-C(1)-C(9)	122.4(3)
O(1)-C(1)-C(2)	123.1(3)
C(9)-C(1)-C(2)	114.5(3)
C(1)-C(2)-C(3)	113.7(3)
C(1)-C(2)-C(7)	110.3(3)
C(3)-C(2)-C(7)	111.6(3)
C(1)-C(2)-H(2)	106.7
C(3)-C(2)-H(2)	105.4
C(7)-C(2)-H(2)	108.9
C(2)-C(3)-C(4)	111.8(3)
C(2)-C(3)-H(3A)	108.8
C(4)-C(3)-H(3A)	108.5
C(2)-C(3)-H(3B)	108.5
C(4)-C(3)-H(3B)	109.6
H(3A)-C(3)-H(3B)	109.5
C(5)-C(4)-C(3)	111.1(3)
C(5)-C(4)-H(4A)	109.5
C(3)-C(4)-H(4A)	109.8
C(5)-C(4)-H(4B)	108.2
C(3)-C(4)-H(4B)	108.8
H(4A)-C(4)-H(4B)	109.5
C(4)-C(5)-C(6)	112.0(3)
C(4)-C(5)-H(5A)	109.2
C(6)-C(5)-H(5A)	109.9

C(4)-C(5)-H(5B)	107.5
C(6)-C(5)-H(5B)	108.7
H(5A)-C(5)-H(5B)	109.5
C(5)-C(6)-C(7)	111.5(3)
C(5)-C(6)-H(6A)	108.3
C(7)-C(6)-H(6A)	108.6
C(5)-C(6)-H(6B)	109.4
C(7)-C(6)-H(6B)	109.5
H(6A)-C(6)-H(6B)	109.5
N(1)-C(7)-C(6)	112.0(3)
N(1)-C(7)-C(2)	111.4(3)
C(6)-C(7)-C(2)	108.9(3)
N(1)-C(7)-H(7)	107.5
C(6)-C(7)-H(7)	108.0
C(2)-C(7)-H(7)	108.9
N(1)-C(8)-C(10)	111.8(3)
N(1)-C(8)-C(9)	108.9(3)
C(10)-C(8)-C(9)	111.4(3)
N(1)-C(8)-H(8)	107.7
C(10)-C(8)-H(8)	108.9
C(9)-C(8)-H(8)	108.0
C(1)-C(9)-C(8)	110.3(3)
C(1)-C(9)-H(9A)	109.4
C(8)-C(9)-H(9A)	109.0
C(1)-C(9)-H(9B)	108.9
C(8)-C(9)-H(9B)	109.7
H(9A)-C(9)-H(9B)	109.5
C(11)-C(10)-C(15)	118.0(4)
C(11)-C(10)-C(8)	119.8(3)
C(15)-C(10)-C(8)	122.1(3)
C(12)-C(11)-C(10)	121.0(4)
C(12)-C(11)-H(11)	120.2
C(10)-C(11)-H(11)	118.8
C(11)-C(12)-C(13)	120.5(4)
C(11)-C(12)-H(12)	121.1
C(13)-C(12)-H(12)	118.4
C(14)-C(13)-C(12)	119.1(5)
C(14)-C(13)-H(13)	120.4
C(12)-C(13)-H(13)	120.5

C(13)-C(14)-C(15)	121.0(4)
C(13)-C(14)-H(14)	119.5
C(15)-C(14)-H(14)	119.6
C(14)-C(15)-C(10)	120.5(4)
C(14)-C(15)-H(15)	120.0
C(10)-C(15)-H(15)	119.4
C(21)-C(16)-C(17)	118.9(3)
C(21)-C(16)-N(1)	119.1(3)
C(17)-C(16)-N(1)	122.0(3)
C(18)-C(17)-C(16)	120.7(3)
C(18)-C(17)-H(17)	119.4
C(16)-C(17)-H(17)	119.9
C(19)-C(18)-C(17)	119.3(3)
C(19)-C(18)-H(18)	120.2
C(17)-C(18)-H(18)	120.5
C(20)-C(19)-C(18)	120.9(3)
C(20)-C(19)-Cl(1)	119.2(3)
C(18)-C(19)-Cl(1)	119.9(3)
C(19)-C(20)-C(21)	119.2(3)
C(19)-C(20)-H(20)	121.0
C(21)-C(20)-H(20)	119.8
C(16)-C(21)-C(20)	120.9(3)
C(16)-C(21)-H(21)	119.3
C(20)-C(21)-H(21)	119.8
C(16)-N(1)-C(7)	110.4(2)
C(16)-N(1)-C(8)	111.6(3)
C(7)-N(1)-C(8)	110.7(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5505. The anisotropic displacement factor exponent takes the form: $-2 \sum [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	34(2)	30(2)	45(3)	-6(2)	21(2)	0(2)
C(2)	34(2)	34(2)	40(2)	-2(2)	19(2)	0(2)
C(3)	43(2)	61(3)	47(3)	8(2)	21(2)	14(2)
C(4)	52(3)	84(3)	45(3)	17(2)	24(2)	16(2)

C(5)	41(2)	84(3)	35(3)	8(2)	19(2)	5(2)
C(6)	39(2)	51(2)	40(3)	4(2)	23(2)	4(2)
C(7)	31(2)	35(2)	31(2)	-3(2)	16(2)	-3(2)
C(8)	33(2)	35(2)	41(2)	-4(2)	23(2)	-3(2)
C(9)	34(2)	45(2)	45(3)	-3(2)	23(2)	3(2)
C(10)	36(2)	47(2)	33(2)	0(2)	22(2)	1(2)
C(11)	55(3)	58(3)	48(3)	11(2)	28(2)	-2(2)
C(12)	74(3)	88(4)	62(4)	34(3)	37(3)	2(3)
C(13)	50(3)	113(4)	37(3)	15(3)	21(2)	0(3)
C(14)	46(3)	92(4)	43(3)	-8(3)	23(2)	-14(2)
C(15)	45(2)	58(3)	39(3)	-1(2)	23(2)	-8(2)
C(16)	33(2)	33(2)	31(2)	0(2)	16(2)	3(2)
C(17)	33(2)	34(2)	50(3)	-7(2)	20(2)	-2(2)
C(18)	45(2)	32(2)	45(3)	-2(2)	18(2)	0(2)
C(19)	36(2)	38(2)	37(2)	0(2)	13(2)	11(2)
C(20)	31(2)	63(3)	45(3)	1(2)	18(2)	5(2)
C(21)	32(2)	37(2)	54(3)	1(2)	22(2)	0(2)
N(1)	31(2)	34(2)	33(2)	1(1)	18(1)	2(1)
O(1)	33(1)	42(2)	58(2)	-2(1)	24(1)	2(1)
Cl(1)	47(1)	60(1)	64(1)	-1(1)	21(1)	21(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5505.

	x	y	z	U(eq)
H(2)	8131	6593	-182	43
H(3A)	7452	4304	-1459	61
H(3B)	7438	6594	-1392	61
H(4A)	7640	5609	-2415	73
H(4B)	8017	6902	-1679	73
H(5A)	8361	4258	-1892	64
H(5B)	8044	2827	-1765	64
H(6A)	8709	5157	-450	50
H(6B)	8747	2875	-505	50
H(7)	8143	2466	-282	39
H(8)	8210	2071	1077	41
H(9A)	8268	6120	1404	48

H(9B)	7922	4731	1461	48
H(11)	8475	193	2298	64
H(12)	8962	-354	3785	89
H(13)	9494	1989	4632	81
H(14)	9538	4897	4012	72
H(15)	9066	5454	2538	55
H(17)	8560	-350	753	48
H(18)	9108	-2715	1016	51
H(20)	10035	1611	1627	57
H(21)	9485	3963	1371	49

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DEPT-135 of 4

HMQC of 4

NOESY of 10

COSY of 10

DEPT-135 of 10

HMQC of 10

NOESY of 11

NOESY of 12