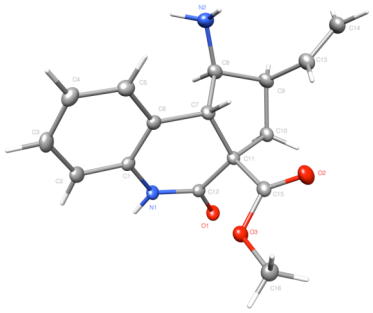
# *APPENDIX 7*

*X-ray Crystallography Reports*

*Relevant to Chapter 2*

## A7.1 CRYSTAL STRUCTURE ANALYSIS OF 240



**240**

**Note:** The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 820779.

*Table A7.1.1. Crystal data and structure refinement for **240** (CCDC 820779).*

| | |
|---|---|
| Empirical formula | $C_{16}H_{18}N_2O_3$ |
| Formula weight | 286.32 |
| Crystallization Solvent | Dichloromethane/hexane |
| Crystal Habit | Column |
| Crystal size | 0.36 x 0.19 x 0.19 $mm^3$ |
| Crystal color | Colorless |

## Data Collection

| | | |
|---|---|---|
| Type of diffractometer | Bruker SMART 1000 | |
| Wavelength | 0.71073 Å MoKα | |
| Data Collection Temperature | 100(2) K | |
| θ range for 8222 reflections used in lattice determination | 2.71 to 36.32° | |
| Unit cell dimensions | a = 7.6777(3) Å<br>b = 9.1864(3) Å<br>c = 10.8584(4) Å | α= 71.309(2)°<br>β= 69.829(2)°<br>γ = 75.7590(10)° |
| Volume | 673.01(4) $Å^3$ | |
| Z | 2 | |
| Crystal system | Triclinic | |
| Space group | P-1 | |
| Density (calculated) | 1.413 $Mg/m^3$ | |
| F(000) | 304 | |
| Data collection program | Bruker SMART v5.630 | |
| θ range for data collection | 2.07 to 36.35° | |
| Completeness to θ = 36.35° | 98.4 % | |
| Index ranges | $-12 \le h \le 12, -15 \le k \le 15, -18 \le l \le 18$ | |
| Data collection scan type | ω scans at 10 settings | |
| Data reduction program | Bruker SAINT-Plus v7.66A | |
| Reflections collected | 24459 | |
| Independent reflections | 6449 [$R_{int}$= 0.0431] | |
| Absorption coefficient | 0.099 $mm^{-1}$ | |
| Absorption correction | None | |
| Max. and min. transmission | 0.9815 and 0.9654 | |

*Table A7.1.1 (cont.)*

## Structure solution and Refinement

| | |
|---|---|
| Structure solution program | SHELXS-97 (Sheldrick, 2008) |
| Primary solution method | Direct methods |
| Secondary solution method | Difference Fourier map |
| Hydrogen placement | Difference Fourier map |
| Structure refinement program | SHELXL-97 (Sheldrick, 2008) |
| Refinement method | Full matrix least-squares on $F^2$ |
| Data / restraints / parameters | 6449 / 0 / 262 |
| Treatment of hydrogen atoms | Unrestrained |
| Goodness-of-fit on $F^2$ | 2.646 |
| Final R indices [I>2σ(I), 4636 reflections] | R1 = 0.0623, *w*R2 = 0.0933 |
| R indices (all data) | R1 = 0.0829, *w*R2 = 0.0954 |
| Type of weighting scheme used | Sigma |
| Weighting scheme used | $w=1/\sigma^2(Fo^2)$ |
| Max shift/error | 0.001 |
| Average shift/error | 0.000 |
| Largest diff. peak and hole | 0.666 and -0.395 e.Å$^{-3}$ |

## Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

Refinement of $F^2$ against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on $F^2$, conventional R-factors (R) are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

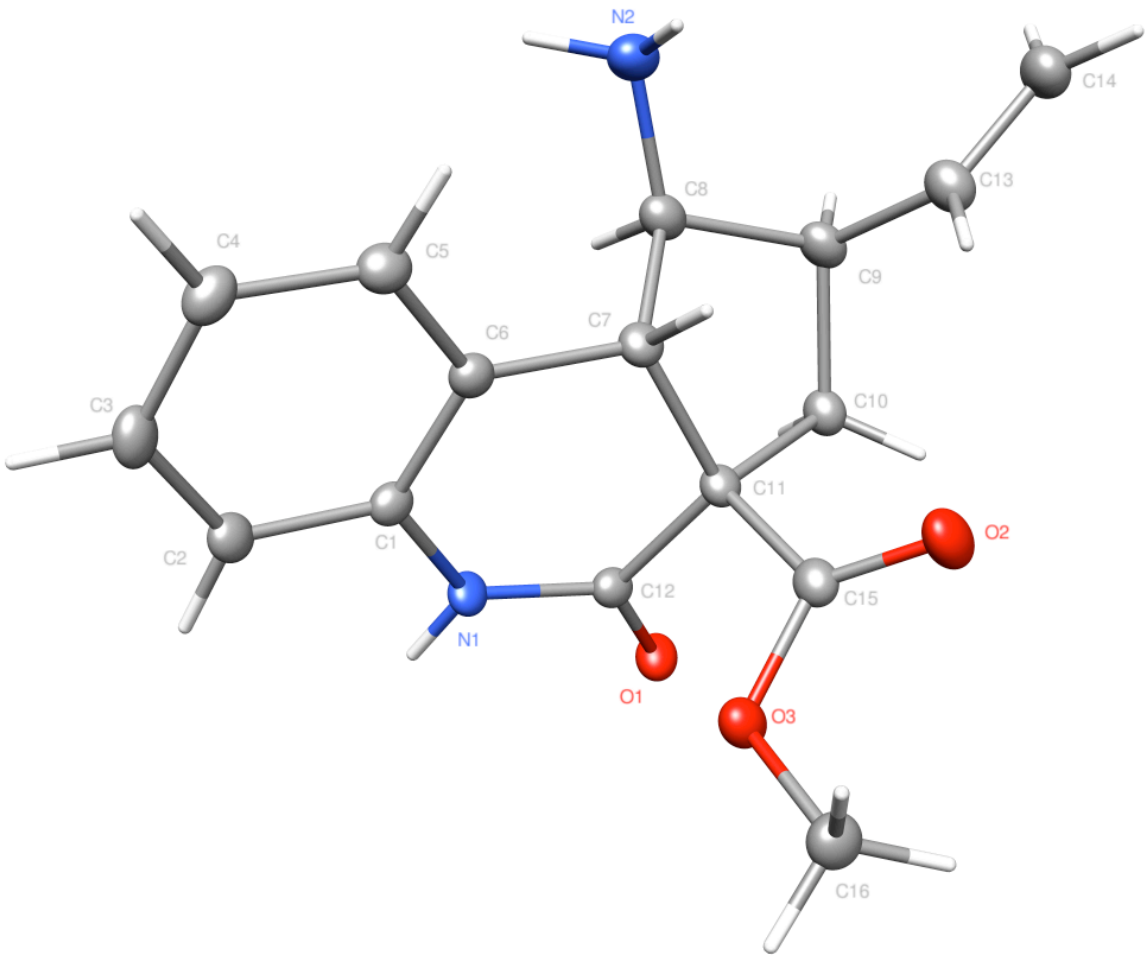

N2
C14
C13
C8
C4
C5
C9
C6
C7
C3
C10
C11
O2
C1
C2
C12
C15
N1
O1
O3
C16

*Table A7.1.2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters (Å$^2$x $10^3$) for XXa (CCDC 820779). U(eq) is defined as the trace of the orthogonalized $U^{ij}$ tensor.*

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| O(1) | 4408(1) | 7089(1) | 4178(1) | 19(1) |
| O(2) | 7024(1) | 9577(1) | 237(1) | 26(1) |
| O(3) | 6615(1) | 7073(1) | 1117(1) | 19(1) |
| N(1) | 7276(1) | 5626(1) | 3956(1) | 16(1) |
| N(2) | 10859(2) | 9851(1) | 3092(1) | 24(1) |
| C(1) | 9253(1) | 5399(1) | 3465(1) | 15(1) |
| C(2) | 10235(2) | 3896(1) | 3734(1) | 18(1) |
| C(3) | 12183(2) | 3662(1) | 3300(1) | 20(1) |
| C(4) | 13148(2) | 4913(1) | 2592(1) | 20(1) |
| C(5) | 12157(2) | 6406(1) | 2289(1) | 19(1) |
| C(6) | 10199(2) | 6674(1) | 2726(1) | 15(1) |
| C(7) | 9129(2) | 8291(1) | 2420(1) | 16(1) |
| C(8) | 9097(2) | 9371(1) | 3279(1) | 20(1) |
| C(9) | 7562(2) | 10747(1) | 2922(1) | 19(1) |
| C(10) | 6011(2) | 9897(1) | 2999(1) | 18(1) |
| C(11) | 7013(1) | 8352(1) | 2576(1) | 14(1) |
| C(12) | 6129(2) | 6970(1) | 3640(1) | 15(1) |
| C(13) | 8229(2) | 11823(1) | 1535(1) | 22(1) |
| C(14) | 8438(2) | 13284(2) | 1290(1) | 26(1) |
| C(15) | 6863(2) | 8427(1) | 1186(1) | 16(1) |
| C(16) | 6494(2) | 7061(2) | -190(1) | 21(1) |

*Table A7.1.3. Bond lengths [Å] and angles [°] for **240** (CCDC 820779).*

| | |
|---|---|
| O(1)-C(12) | 1.2396(13) |
| O(2)-C(15) | 1.2129(13) |
| O(3)-C(15) | 1.3331(13) |
| O(3)-C(16) | 1.4571(14) |
| N(1)-C(12) | 1.3493(14) |
| N(1)-C(1) | 1.4101(14) |
| N(1)-H(1) | 0.894(14) |
| N(2)-C(8) | 1.4532(15) |
| N(2)-H(2A) | 0.874(16) |
| N(2)-H(2B) | 0.923(16) |
| C(1)-C(2) | 1.3935(15) |
| C(1)-C(6) | 1.3990(14) |
| C(2)-C(3) | 1.3881(16) |
| C(2)-H(2) | 0.958(12) |
| C(3)-C(4) | 1.3862(17) |
| C(3)-H(3) | 0.963(12) |
| C(4)-C(5) | 1.3918(16) |
| C(4)-H(4) | 0.941(14) |
| C(5)-C(6) | 1.3954(15) |
| C(5)-H(5) | 0.949(13) |
| C(6)-C(7) | 1.5046(15) |
| C(7)-C(8) | 1.5555(16) |
| C(7)-C(11) | 1.5630(14) |
| C(7)-H(7) | 1.000(13) |
| C(8)-C(9) | 1.5509(16) |
| C(8)-H(8) | 1.005(12) |
| C(9)-C(13) | 1.5060(16) |
| C(9)-C(10) | 1.5433(16) |
| C(9)-H(9) | 0.980(13) |
| C(10)-C(11) | 1.5665(15) |
| C(10)-H(10A) | 0.948(12) |
| C(10)-H(10B) | 0.995(12) |
| C(11)-C(15) | 1.5308(15) |
| C(11)-C(12) | 1.5333(14) |
| C(13)-C(14) | 1.3213(17) |
| C(13)-H(13) | 1.001(14) |
| C(14)-H(14A) | 0.955(14) |
| C(14)-H(14B) | 0.968(14) |
| C(16)-H(16A) | 0.990(13) |
| C(16)-H(16B) | 0.938(13) |
| C(16)-H(16C) | 0.944(14) |
| | |
| C(15)-O(3)-C(16) | 115.23(9) |
| C(12)-N(1)-C(1) | 125.72(9) |
| C(12)-N(1)-H(1) | 116.1(9) |
| C(1)-N(1)-H(1) | 118.1(9) |
| C(8)-N(2)-H(2A) | 111.6(10) |
| C(8)-N(2)-H(2B) | 111.6(9) |
| H(2A)-N(2)-H(2B) | 106.0(14) |
| C(2)-C(1)-C(6) | 121.00(10) |

| | |
|---|---|
| C(2)-C(1)-N(1) | 118.99(10) |
| C(6)-C(1)-N(1) | 120.01(10) |
| C(3)-C(2)-C(1) | 119.60(10) |
| C(3)-C(2)-H(2) | 121.6(8) |
| C(1)-C(2)-H(2) | 118.7(8) |
| C(2)-C(3)-C(4) | 120.27(11) |
| C(2)-C(3)-H(3) | 118.2(7) |
| C(4)-C(3)-H(3) | 121.5(7) |
| C(3)-C(4)-C(5) | 119.80(11) |
| C(3)-C(4)-H(4) | 119.3(8) |
| C(5)-C(4)-H(4) | 120.8(8) |
| C(4)-C(5)-C(6) | 121.02(11) |
| C(4)-C(5)-H(5) | 121.5(8) |
| C(6)-C(5)-H(5) | 117.5(8) |
| C(5)-C(6)-C(1) | 118.25(10) |
| C(5)-C(6)-C(7) | 121.02(10) |
| C(1)-C(6)-C(7) | 120.72(9) |
| C(6)-C(7)-C(8) | 116.70(9) |
| C(6)-C(7)-C(11) | 114.37(9) |
| C(8)-C(7)-C(11) | 104.20(9) |
| C(6)-C(7)-H(7) | 107.1(8) |
| C(8)-C(7)-H(7) | 105.8(7) |
| C(11)-C(7)-H(7) | 108.1(7) |
| N(2)-C(8)-C(9) | 113.55(10) |
| N(2)-C(8)-C(7) | 117.56(10) |
| C(9)-C(8)-C(7) | 102.10(9) |
| N(2)-C(8)-H(8) | 108.3(7) |
| C(9)-C(8)-H(8) | 107.3(7) |
| C(7)-C(8)-H(8) | 107.4(7) |
| C(13)-C(9)-C(10) | 111.69(10) |
| C(13)-C(9)-C(8) | 113.01(10) |
| C(10)-C(9)-C(8) | 101.94(9) |
| C(13)-C(9)-H(9) | 109.3(7) |
| C(10)-C(9)-H(9) | 112.2(7) |
| C(8)-C(9)-H(9) | 108.5(7) |
| C(9)-C(10)-C(11) | 107.07(9) |
| C(9)-C(10)-H(10A) | 113.9(7) |
| C(11)-C(10)-H(10A) | 109.5(7) |
| C(9)-C(10)-H(10B) | 109.0(7) |
| C(11)-C(10)-H(10B) | 108.6(7) |
| H(10A)-C(10)-H(10B) | 108.6(10) |
| C(15)-C(11)-C(12) | 109.43(9) |
| C(15)-C(11)-C(7) | 107.53(8) |
| C(12)-C(11)-C(7) | 114.40(9) |
| C(15)-C(11)-C(10) | 110.87(9) |
| C(12)-C(11)-C(10) | 110.03(9) |
| C(7)-C(11)-C(10) | 104.48(8) |
| O(1)-C(12)-N(1) | 121.40(10) |
| O(1)-C(12)-C(11) | 120.58(9) |
| N(1)-C(12)-C(11) | 118.01(9) |
| C(14)-C(13)-C(9) | 125.48(12) |
| C(14)-C(13)-H(13) | 120.0(8) |
| C(9)-C(13)-H(13) | 114.5(8) |
| C(13)-C(14)-H(14A) | 119.3(8) |

| | |
|---|---|
| C(13)-C(14)-H(14B) | 122.8(8) |
| H(14A)-C(14)-H(14B) | 117.8(11) |
| O(2)-C(15)-O(3) | 123.70(10) |
| O(2)-C(15)-C(11) | 123.68(10) |
| O(3)-C(15)-C(11) | 112.57(9) |
| O(3)-C(16)-H(16A) | 110.0(8) |
| O(3)-C(16)-H(16B) | 107.2(8) |
| H(16A)-C(16)-H(16B) | 108.9(11) |
| O(3)-C(16)-H(16C) | 109.2(8) |
| H(16A)-C(16)-H(16C) | 112.1(11) |
| H(16B)-C(16)-H(16C) | 109.3(1) |

*Table A7.1.4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for **240** (CCDC 820779). The anisotropic displacement factor exponent takes the form: $-2\pi^2[ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12} ]$*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| O(1) | 135(4) | 188(4) | 200(4) | -10(3) | -24(3) | -30(3) |
| O(2) | 364(5) | 196(4) | 198(4) | 13(3) | -101(4) | -86(4) |
| O(3) | 230(4) | 166(4) | 166(4) | -23(3) | -69(3) | -42(3) |
| N(1) | 132(4) | 148(4) | 154(4) | 6(3) | -27(3) | -38(3) |
| N(2) | 243(5) | 230(5) | 277(6) | -18(4) | -111(5) | -80(4) |
| C(1) | 131(5) | 176(5) | 124(5) | -31(4) | -36(4) | -24(4) |
| C(2) | 186(5) | 172(5) | 156(5) | -16(4) | -55(4) | -29(4) |
| C(3) | 204(6) | 191(6) | 178(5) | -36(4) | -66(4) | 26(4) |
| C(4) | 130(5) | 257(6) | 193(6) | -63(4) | -35(4) | 2(4) |
| C(5) | 155(5) | 209(6) | 189(5) | -37(4) | -28(4) | -49(4) |
| C(6) | 143(5) | 168(5) | 152(5) | -38(4) | -45(4) | -25(4) |
| C(7) | 135(5) | 160(5) | 165(5) | -13(4) | -22(4) | -42(4) |
| C(8) | 215(6) | 178(5) | 193(6) | -28(4) | -59(4) | -51(4) |
| C(9) | 219(6) | 163(5) | 193(5) | -50(4) | -38(4) | -41(4) |
| C(10) | 157(5) | 161(5) | 187(5) | -38(4) | -28(4) | -15(4) |
| C(11) | 120(5) | 142(5) | 146(5) | -18(4) | -19(4) | -31(4) |
| C(12) | 152(5) | 160(5) | 126(5) | -20(4) | -39(4) | -40(4) |
| C(13) | 202(6) | 185(6) | 227(6) | -39(4) | -35(4) | -34(4) |
| C(14) | 269(7) | 199(6) | 314(7) | -16(5) | -124(5) | -49(5) |
| C(15) | 128(5) | 158(5) | 183(5) | -35(4) | -29(4) | -15(4) |
| C(16) | 244(6) | 218(6) | 173(6) | -46(4) | -72(5) | -37(5) |

*Table A7.1.5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for **240** (CCDC 820779).*

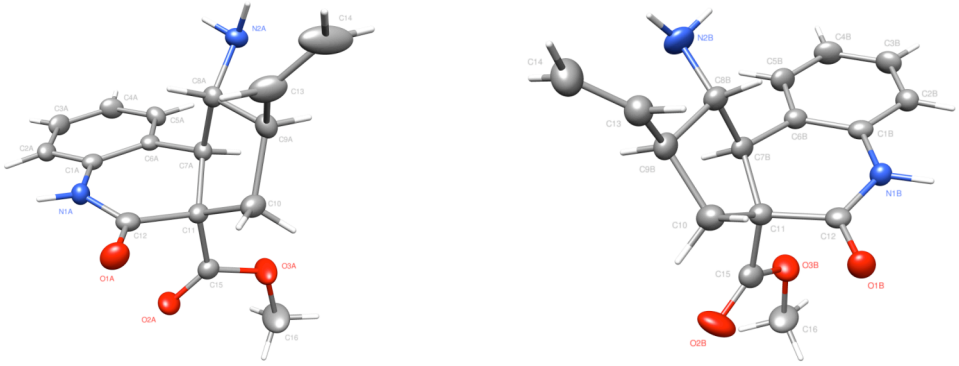
| | x | y | z | $U_{iso}$ |
|---|---|---|---|---|
| H(1) | 6719(19) | 4828(16) | 4556(14) | 29(4) |
| H(2) | 9539(17) | 3045(15) | 4204(13) | 21(3) |
| H(2A) | 11470(20) | 10154(18) | 2232(16) | 40(4) |
| H(2B) | 11660(20) | 9042(19) | 3473(16) | 43(4) |
| H(3) | 12833(17) | 2615(15) | 3504(12) | 18(3) |
| H(4) | 14470(20) | 4737(15) | 2302(14) | 29(4) |
| H(5) | 12784(18) | 7277(15) | 1776(13) | 26(4) |
| H(7) | 9747(18) | 8815(15) | 1456(13) | 24(3) |
| H(8) | 8599(16) | 8819(14) | 4261(12) | 14(3) |
| H(9) | 7155(17) | 11335(14) | 3609(13) | 19(3) |
| H(10A) | 5241(16) | 10483(14) | 2433(12) | 15(3) |
| H(10B) | 5202(16) | 9630(13) | 3954(12) | 14(3) |
| H(13) | 8473(18) | 11360(15) | 762(14) | 28(4) |
| H(14A) | 8124(19) | 13735(16) | 2033(14) | 32(4) |
| H(14B) | 8860(19) | 13939(16) | 381(14) | 28(4) |
| H(16A) | 5459(19) | 7866(15) | -436(13) | 24(3) |
| H(16B) | 6227(18) | 6081(16) | -92(13) | 22(3) |
| H(16C) | 7662(19) | 7209(15) | -849(14) | 25(4) |

*Table A7.1.6. Hydrogen bonds for **240** (CCDC 820779) [Å and °].*

| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| N(1)-H(1)...O(1)#1 | 0.894(14) | 2.029(14) | 2.9217(12) | 175.5(13) |
| N(2)-H(2A)...O(2)#2 | 0.874(16) | 2.483(16) | 3.3366(15) | 165.7(13) |
| N(2)-H(2B)...O(1)#3 | 0.923(16) | 2.576(16) | 3.4855(13) | 168.5(13) |

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+1
#2 -x+2,-y+2,-z
#3 x+1,y,z

## A7.2 CRYSTAL STRUCTURE ANALYSIS OF 241



**241**

**Note:** The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 820778."

*Table A7.2.1. Crystal data and structure refinement for **241** (CCDC 820778).*

| | |
|---|---|
| Empirical formula | $C_{16}H_{18}N_2O_3$ |
| Formula weight | 286.32 |
| Crystallization Solvent | Dichloromethane/hexane |
| Crystal Habit | Plate |
| Crystal size | 0.43 x 0.40 x 0.08 $mm^3$ |
| Crystal color | Colorless |

## Data Collection

| | | |
|---|---|---|
| Type of diffractometer | Bruker SMART 1000 | |
| Wavelength | 0.71073 Å MoKα | |
| Data Collection Temperature | 100(2) K | |
| θ range for 9885 reflections used in lattice determination | 2.42 to 28.07° | |
| Unit cell dimensions | a = 19.947(2) Å<br>b = 13.3646(15) Å<br>c = 11.2796(13) Å | α= 90°<br>β= 105.507(2)°<br>γ = 90° |
| Volume | 2897.5(6) $Å^3$ | |
| Z | 8 | |
| Crystal system | Monoclinic | |
| Space group | P2(1)/c | |
| Density (calculated) | 1.313 $Mg/m^3$ | |
| F(000) | 1216 | |
| Data collection program | Bruker SMART v5.630 | |
| θ range for data collection | 1.06 to 29.00° | |
| Completeness to θ = 29.00° | 92.1 % | |
| Index ranges | $-26 \le h \le 26, -17 \le k \le 17, -15 \le l \le 15$ | |
| Data collection scan type | ω scans at 5 settings | |
| Data reduction program | Bruker SAINT-Plus v7.66A | |
| Reflections collected | 44322 | |
| Independent reflections | 7100 [$R_{int}$= 0.0500] | |
| Absorption coefficient | 0.092 $mm^{-1}$ | |
| Absorption correction | None | |
| Max. and min. transmission | 0.9927 | and 0.9617 |

*Table A7.2.1. (cont.)*

## Structure solution and Refinement

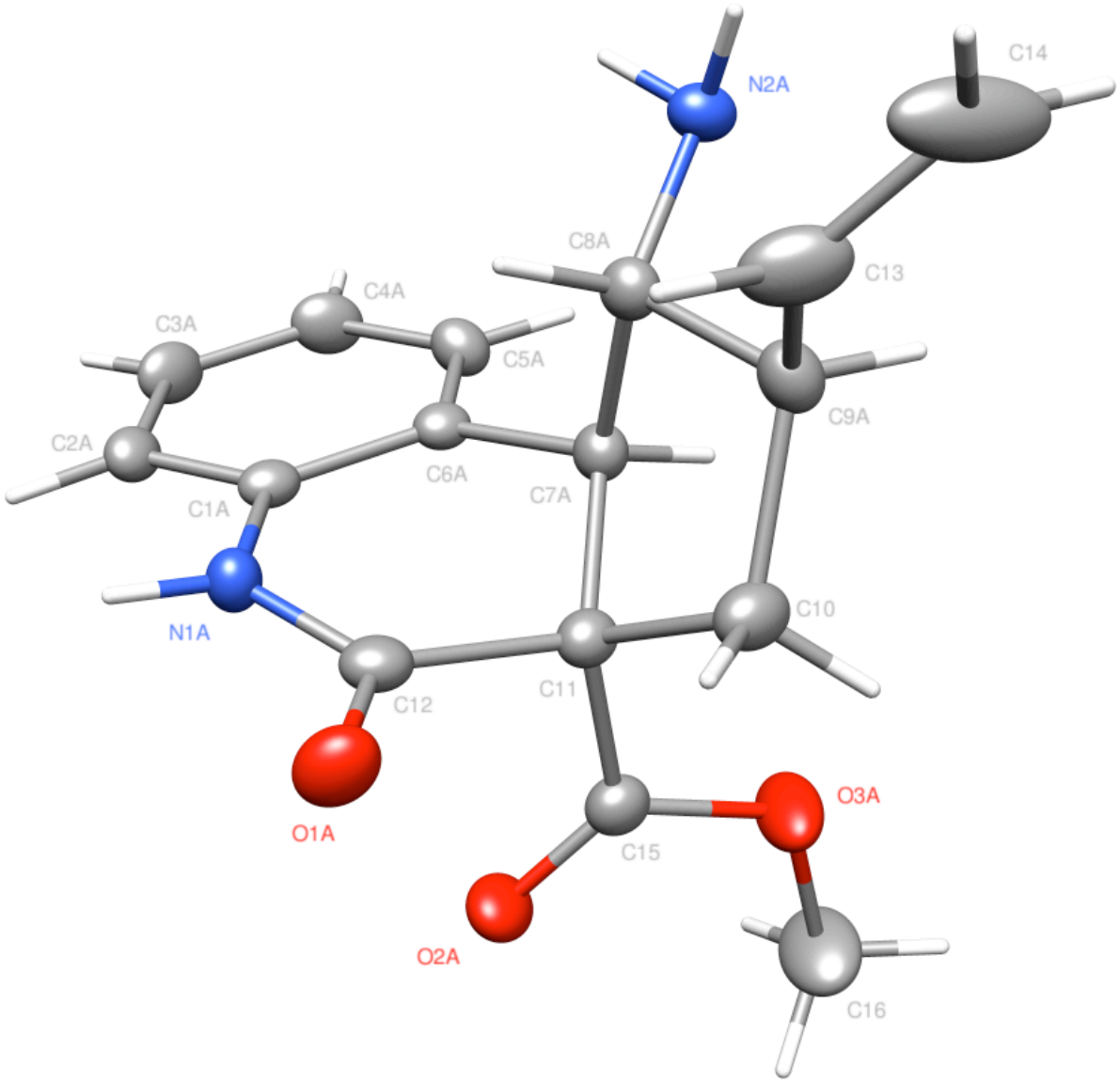
| | |
|---|---|
| Structure solution program | SHELXS-97 (Sheldrick, 2008) |
| Primary solution method | Direct methods |
| Secondary solution method | Difference Fourier map |
| Hydrogen placement | Difference Fourier map |
| Structure refinement program | SHELXL-97 (Sheldrick, 2008) |
| Refinement method | Full matrix least-squares on $F^2$ |
| Data / restraints / parameters | 7100 / 0 / 523 |
| Treatment of hydrogen atoms | Unrestrained |
| Goodness-of-fit on $F^2$ | 2.160 |
| Final R indices [I>2σ(I), 5019 reflections] | R1 = 0.0507, *w*R2 = 0.0645 |
| R indices (all data) | R1 = 0.0748, *w*R2 = 0.0664 |
| Type of weighting scheme used | Sigma |
| Weighting scheme used | $w=1/\sigma^2(Fo^2)$ |
| Max shift/error | 0.001 |
| Average shift/error | 0.000 |
| Largest diff. peak and hole | 0.362 and -0.287 e.Å$^{-3}$ |

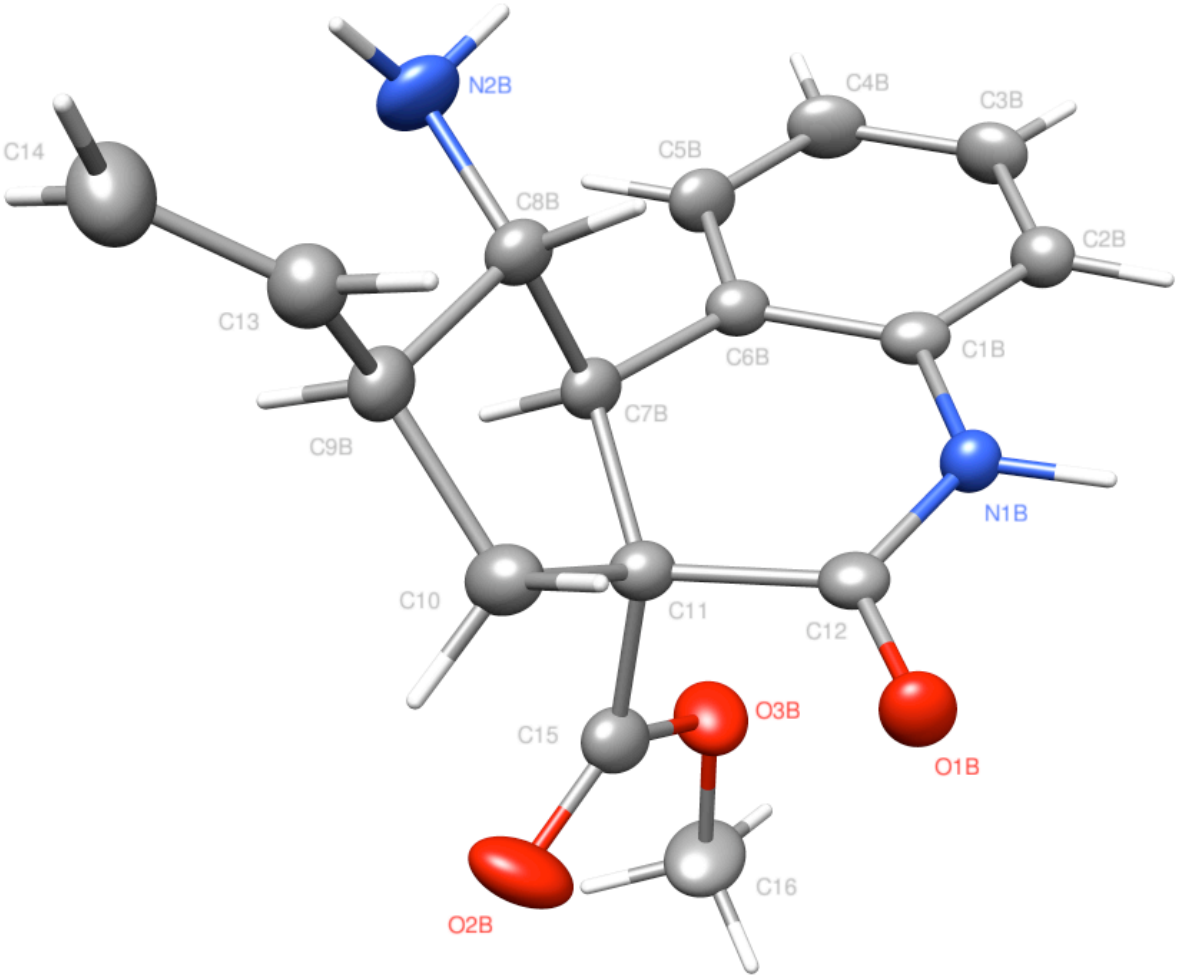
## Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

Refinement of $F^2$ against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on $F^2$, conventional R-factors (R) are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.


C14
N2A
C8A
C13
C4A
C3A
C5A
C9A
C2A
C6A
C7A
C1A
N1A
C10
C11
C12
O1A
O3A
C15
O2A
C16


N2B
C4B
C3B
C14
C5B
C8B
C2B
C13
C6B
C1B
C7B
C9B
N1B
C10
C11
C12
O3B
C15
O1B
C16
O2B

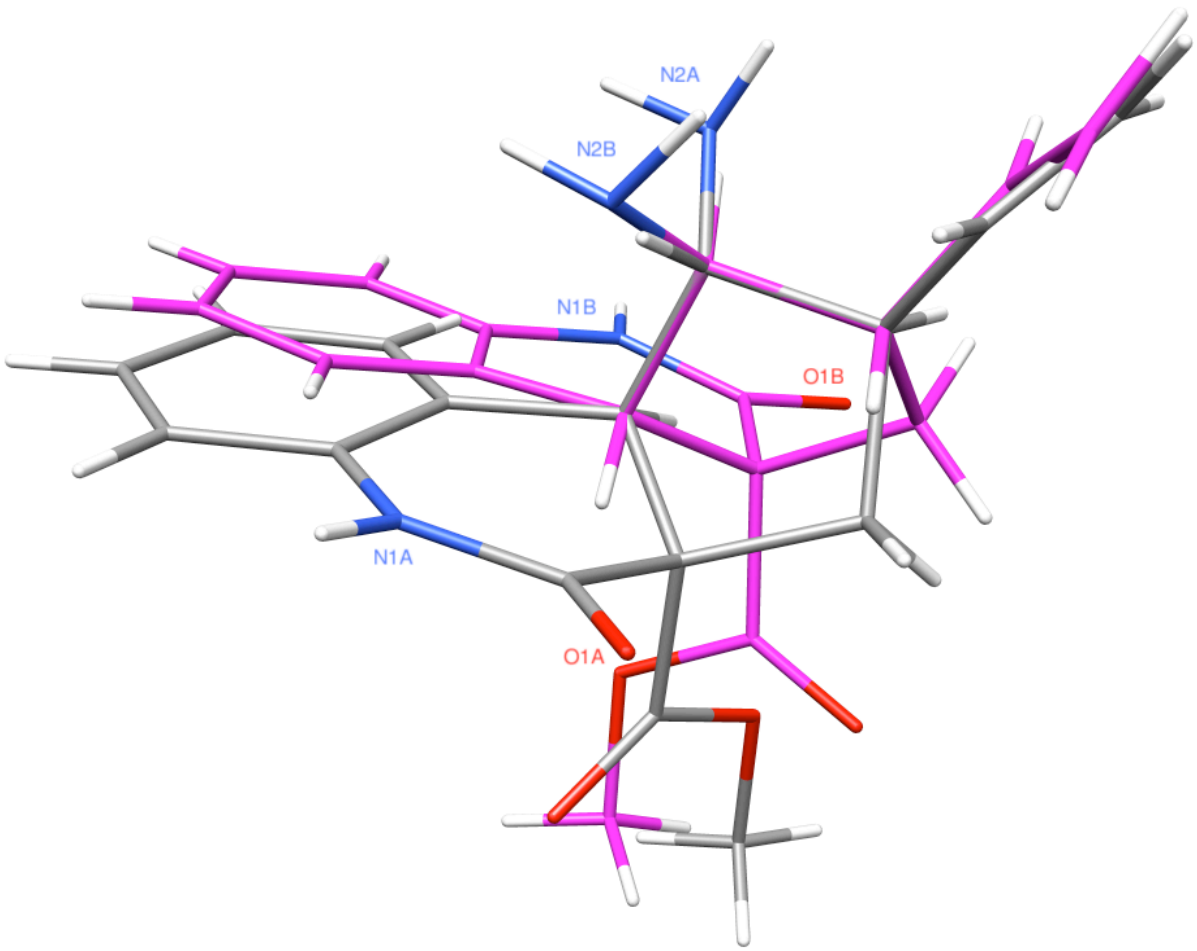

N2A
N2B
N1B
O1B
N1A
O1A

*Table A7.2.2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters (Å$^2$x $10^3$) for* ***241*** *(CCDC 820778). U(eq) is defined as the trace of the orthogonalized $U^{ij}$ tensor.*

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| O(1A) | 6702(1) | 965(1) | 10260(1) | 30(1) |
| O(2A) | 5913(1) | -380(1) | 7880(1) | 29(1) |
| O(3A) | 6207(1) | 438(1) | 6371(1) | 40(1) |
| N(1A) | 5544(1) | 1226(1) | 9712(1) | 21(1) |
| N(2A) | 5566(1) | 3969(1) | 7309(1) | 22(1) |
| C(1A) | 4910(1) | 1456(1) | 8853(1) | 18(1) |
| C(2A) | 4287(1) | 1269(1) | 9137(1) | 22(1) |
| C(3A) | 3667(1) | 1512(1) | 8294(1) | 27(1) |
| C(4A) | 3665(1) | 1922(1) | 7172(2) | 28(1) |
| C(5A) | 4288(1) | 2101(1) | 6890(1) | 23(1) |
| C(6A) | 4918(1) | 1880(1) | 7728(1) | 18(1) |
| C(7A) | 5609(1) | 2116(1) | 7503(1) | 19(1) |
| C(8A) | 5924(1) | 3121(1) | 8029(1) | 20(1) |
| C(9A) | 6683(1) | 3049(1) | 7979(1) | 22(1) |
| C(10A) | 6869(1) | 1925(1) | 8123(2) | 26(1) |
| C(11A) | 6188(1) | 1377(1) | 8134(1) | 20(1) |
| C(12A) | 6171(1) | 1172(1) | 9462(1) | 22(1) |
| C(13A) | 7153(1) | 3721(1) | 8882(2) | 32(1) |
| C(14A) | 7450(1) | 4528(1) | 8569(3) | 54(1) |
| C(15A) | 6091(1) | 375(1) | 7477(1) | 23(1) |
| C(16A) | 6124(1) | -490(2) | 5675(2) | 50(1) |
| | | | | |
| O(1B) | 9130(1) | 9861(1) | 5062(1) | 29(1) |
| O(2B) | 8139(1) | 9887(1) | 7206(1) | 45(1) |
| O(3B) | 9269(1) | 9904(1) | 8214(1) | 36(1) |
| N(1B) | 10085(1) | 9132(1) | 6282(1) | 22(1) |
| N(2B) | 9194(1) | 5996(1) | 7029(1) | 38(1) |
| C(1B) | 10439(1) | 8525(1) | 7274(1) | 22(1) |
| C(2B) | 11161(1) | 8550(1) | 7645(1) | 26(1) |
| C(3B) | 11516(1) | 7954(1) | 8606(1) | 29(1) |
| C(4B) | 11153(1) | 7340(1) | 9203(2) | 33(1) |
| C(5B) | 10430(1) | 7324(1) | 8825(1) | 30(1) |
| C(6B) | 10065(1) | 7911(1) | 7857(1) | 23(1) |
| C(7B) | 9289(1) | 7838(1) | 7351(1) | 24(1) |
| C(8B) | 9075(1) | 6972(1) | 6433(1) | 27(1) |
| C(9B) | 8305(1) | 7173(1) | 5848(2) | 28(1) |
| C(10B) | 8283(1) | 8303(1) | 5632(2) | 31(1) |
| C(11B) | 8929(1) | 8747(1) | 6584(1) | 24(1) |
| C(12B) | 9393(1) | 9284(1) | 5913(1) | 22(1) |
| C(13B) | 8014(1) | 6569(1) | 4712(2) | 33(1) |
| C(14B) | 7622(1) | 5776(1) | 4621(2) | 47(1) |
| C(15B) | 8714(1) | 9570(1) | 7353(1) | 29(1) |

| | | | | |
|---|---|---|---|---|
| C(16B) | 9139(1) | 10740(1) | 8951(2) | 42(1) |

*Table A7.2.3. Bond lengths [Å] and angles [°] for* ***241*** *(CCDC 820778).*

_____

| | |
|---|---|
| O(1A)-C(12A) | 1.2239(15) |
| O(2A)-C(15A) | 1.1996(15) |
| O(3A)-C(15A) | 1.3307(16) |
| O(3A)-C(16A) | 1.4539(19) |
| N(1A)-C(12A) | 1.3544(16) |
| N(1A)-C(1A) | 1.4057(16) |
| N(1A)-H(1A) | 0.877(14) |
| N(2A)-C(8A) | 1.4642(17) |
| N(2A)-H(2A2) | 0.878(14) |
| N(2A)-H(2A3) | 0.950(14) |
| C(1A)-C(2A) | 1.3868(18) |
| C(1A)-C(6A) | 1.3936(17) |
| C(2A)-C(3A) | 1.3823(19) |
| C(2A)-H(2A) | 0.968(12) |
| C(3A)-C(4A) | 1.378(2) |
| C(3A)-H(3A) | 0.953(11) |
| C(4A)-C(5A) | 1.3832(19) |
| C(4A)-H(4A) | 0.945(13) |
| C(5A)-C(6A) | 1.3878(18) |
| C(5A)-H(5A) | 0.941(11) |
| C(6A)-C(7A) | 1.4997(18) |
| C(7A)-C(8A) | 1.5328(18) |
| C(7A)-C(11A) | 1.5427(18) |
| C(7A)-H(7A) | 0.938(11) |
| C(8A)-C(9A) | 1.5334(18) |
| C(8A)-H(8A) | 0.975(11) |
| C(9A)-C(13A) | 1.487(2) |
| C(9A)-C(10A) | 1.5448(19) |
| C(9A)-H(9A) | 0.929(12) |
| C(10A)-C(11A) | 1.5453(19) |
| C(10A)-H(10A) | 0.949(13) |
| C(10A)-H(10B) | 0.966(13) |
| C(11A)-C(15A) | 1.5177(18) |
| C(11A)-C(12A) | 1.5333(18) |
| C(13A)-C(14A) | 1.323(2) |
| C(13A)-H(13A) | 0.972(13) |
| C(14A)-H(14A) | 0.99(2) |
| C(14A)-H(14B) | 0.958(17) |
| C(16A)-H(16A) | 0.984(16) |
| C(16A)-H(16B) | 0.945(18) |
| C(16A)-H(16C) | 0.977(16) |
| O(1B)-C(12B) | 1.2332(15) |
| O(2B)-C(15B) | 1.1911(16) |
| O(3B)-C(15B) | 1.3395(17) |
| O(3B)-C(16B) | 1.4563(18) |
| N(1B)-C(12B) | 1.3454(17) |
| N(1B)-C(1B) | 1.4098(17) |
| N(1B)-H(1B) | 0.984(15) |
| N(2B)-C(8B) | 1.4569(18) |
| N(2B)-H(2B2) | 0.944(17) |
| N(2B)-H(2B3) | 1.006(19) |
| C(1B)-C(2B) | 1.3877(18) |
| C(1B)-C(6B) | 1.3878(18) |
| C(2B)-C(3B) | 1.378(2) |

| | |
|---|---|
| C(2B)-H(2B) | 0.963(12) |
| C(3B)-C(4B) | 1.384(2) |
| C(3B)-H(3B) | 0.924(12) |
| C(4B)-C(5B) | 1.390(2) |
| C(4B)-H(4B) | 0.996(13) |
| C(5B)-C(6B) | 1.3835(19) |
| C(5B)-H(5B) | 0.958(13) |
| C(6B)-C(7B) | 1.5015(19) |
| C(7B)-C(8B) | 1.5351(19) |
| C(7B)-C(11B) | 1.5521(19) |
| C(7B)-H(7B) | 1.020(12) |
| C(8B)-C(9B) | 1.5252(19) |
| C(8B)-H(8B) | 1.039(12) |
| C(9B)-C(13B) | 1.494(2) |
| C(9B)-C(10B) | 1.529(2) |
| C(9B)-H(9B) | 1.001(13) |
| C(10B)-C(11B) | 1.558(2) |
| C(10B)-H(10C) | 0.992(14) |
| C(10B)-H(10D) | 1.061(15) |
| C(11B)-C(12B) | 1.5233(18) |
| C(11B)-C(15B) | 1.5307(19) |
| C(13B)-C(14B) | 1.304(2) |
| C(13B)-H(13B) | 1.017(15) |
| C(14B)-H(14C) | 1.009(16) |
| C(14B)-H(14D) | 1.028(18) |
| C(16B)-H(16D) | 1.006(15) |
| C(16B)-H(16E) | 1.000(16) |
| C(16B)-H(16F) | 1.011(15) |

| | |
|---|---|
| C(15A)-O(3A)-C(16A) | 115.42(13) |
| C(12A)-N(1A)-C(1A) | 125.33(13) |
| C(12A)-N(1A)-H(1A) | 116.4(9) |
| C(1A)-N(1A)-H(1A) | 118.3(9) |
| C(8A)-N(2A)-H(2A2) | 110.6(9) |
| C(8A)-N(2A)-H(2A3) | 112.2(8) |
| H(2A2)-N(2A)-H(2A3) | 102.5(12) |
| C(2A)-C(1A)-C(6A) | 120.81(13) |
| C(2A)-C(1A)-N(1A) | 119.86(13) |
| C(6A)-C(1A)-N(1A) | 119.33(12) |
| C(3A)-C(2A)-C(1A) | 119.39(14) |
| C(3A)-C(2A)-H(2A) | 121.8(7) |
| C(1A)-C(2A)-H(2A) | 118.8(7) |
| C(4A)-C(3A)-C(2A) | 120.60(15) |
| C(4A)-C(3A)-H(3A) | 119.6(7) |
| C(2A)-C(3A)-H(3A) | 119.7(7) |
| C(3A)-C(4A)-C(5A) | 119.74(15) |
| C(3A)-C(4A)-H(4A) | 118.2(8) |
| C(5A)-C(4A)-H(4A) | 122.1(8) |
| C(4A)-C(5A)-C(6A) | 120.88(14) |
| C(4A)-C(5A)-H(5A) | 121.6(7) |
| C(6A)-C(5A)-H(5A) | 117.5(7) |
| C(5A)-C(6A)-C(1A) | 118.56(13) |
| C(5A)-C(6A)-C(7A) | 123.09(13) |
| C(1A)-C(6A)-C(7A) | 118.29(12) |

| | |
|---|---|
| C(6A)-C(7A)-C(8A) | 114.80(11) |
| C(6A)-C(7A)-C(11A) | 113.34(11) |
| C(8A)-C(7A)-C(11A) | 101.70(11) |
| C(6A)-C(7A)-H(7A) | 109.6(7) |
| C(8A)-C(7A)-H(7A) | 107.1(7) |
| C(11A)-C(7A)-H(7A) | 109.9(7) |
| N(2A)-C(8A)-C(7A) | 112.03(11) |
| N(2A)-C(8A)-C(9A) | 111.57(12) |
| C(7A)-C(8A)-C(9A) | 103.79(11) |
| N(2A)-C(8A)-H(8A) | 111.9(7) |
| C(7A)-C(8A)-H(8A) | 107.7(7) |
| C(9A)-C(8A)-H(8A) | 109.5(7) |
| C(13A)-C(9A)-C(8A) | 112.64(12) |
| C(13A)-C(9A)-C(10A) | 115.17(13) |
| C(8A)-C(9A)-C(10A) | 105.56(11) |
| C(13A)-C(9A)-H(9A) | 108.2(8) |
| C(8A)-C(9A)-H(9A) | 106.8(7) |
| C(10A)-C(9A)-H(9A) | 108.1(7) |
| C(9A)-C(10A)-C(11A) | 105.98(11) |
| C(9A)-C(10A)-H(10A) | 112.4(8) |
| C(11A)-C(10A)-H(10A) | 109.9(8) |
| C(9A)-C(10A)-H(10B) | 111.1(8) |
| C(11A)-C(10A)-H(10B) | 111.2(8) |
| H(10A)-C(10A)-H(10B) | 106.4(11) |
| C(15A)-C(11A)-C(12A) | 106.60(11) |
| C(15A)-C(11A)-C(7A) | 110.96(11) |
| C(12A)-C(11A)-C(7A) | 110.81(11) |
| C(15A)-C(11A)-C(10A) | 114.37(11) |
| C(12A)-C(11A)-C(10A) | 110.02(12) |
| C(7A)-C(11A)-C(10A) | 104.15(11) |
| O(1A)-C(12A)-N(1A) | 121.61(13) |
| O(1A)-C(12A)-C(11A) | 121.19(13) |
| N(1A)-C(12A)-C(11A) | 117.20(12) |
| C(14A)-C(13A)-C(9A) | 123.67(19) |
| C(14A)-C(13A)-H(13A) | 119.8(8) |
| C(9A)-C(13A)-H(13A) | 116.6(8) |
| C(13A)-C(14A)-H(14A) | 124.0(13) |
| C(13A)-C(14A)-H(14B) | 116.5(11) |
| H(14A)-C(14A)-H(14B) | 119.5(16) |
| O(2A)-C(15A)-O(3A) | 123.49(13) |
| O(2A)-C(15A)-C(11A) | 124.74(13) |
| O(3A)-C(15A)-C(11A) | 111.73(12) |
| O(3A)-C(16A)-H(16A) | 109.5(10) |
| O(3A)-C(16A)-H(16B) | 104.6(11) |
| H(16A)-C(16A)-H(16B) | 112.8(15) |
| O(3A)-C(16A)-H(16C) | 109.5(10) |
| H(16A)-C(16A)-H(16C) | 106.8(14) |
| H(16B)-C(16A)-H(16C) | 113.6(15) |
| C(15B)-O(3B)-C(16B) | 115.53(13) |
| C(12B)-N(1B)-C(1B) | 126.00(13) |
| C(12B)-N(1B)-H(1B) | 115.0(9) |
| C(1B)-N(1B)-H(1B) | 118.7(9) |
| C(8B)-N(2B)-H(2B2) | 111.7(11) |
| C(8B)-N(2B)-H(2B3) | 104.7(10) |

| | |
|---|---|
| H(2B2)-N(2B)-H(2B3) | 112.9(14) |
| C(2B)-C(1B)-C(6B) | 121.21(13) |
| C(2B)-C(1B)-N(1B) | 119.00(13) |
| C(6B)-C(1B)-N(1B) | 119.78(13) |
| C(3B)-C(2B)-C(1B) | 119.80(15) |
| C(3B)-C(2B)-H(2B) | 121.2(7) |
| C(1B)-C(2B)-H(2B) | 119.0(7) |
| C(2B)-C(3B)-C(4B) | 119.97(15) |
| C(2B)-C(3B)-H(3B) | 120.0(8) |
| C(4B)-C(3B)-H(3B) | 120.0(8) |
| C(3B)-C(4B)-C(5B) | 119.63(15) |
| C(3B)-C(4B)-H(4B) | 120.6(8) |
| C(5B)-C(4B)-H(4B) | 119.7(8) |
| C(6B)-C(5B)-C(4B) | 121.26(15) |
| C(6B)-C(5B)-H(5B) | 118.1(8) |
| C(4B)-C(5B)-H(5B) | 120.6(8) |
| C(5B)-C(6B)-C(1B) | 118.13(14) |
| C(5B)-C(6B)-C(7B) | 122.38(13) |
| C(1B)-C(6B)-C(7B) | 119.26(13) |
| C(6B)-C(7B)-C(8B) | 112.21(12) |
| C(6B)-C(7B)-C(11B) | 115.94(12) |
| C(8B)-C(7B)-C(11B) | 102.14(11) |
| C(6B)-C(7B)-H(7B) | 109.4(7) |
| C(8B)-C(7B)-H(7B) | 107.4(7) |
| C(11B)-C(7B)-H(7B) | 109.2(7) |
| N(2B)-C(8B)-C(9B) | 112.24(13) |
| N(2B)-C(8B)-C(7B) | 112.46(13) |
| C(9B)-C(8B)-C(7B) | 103.05(12) |
| N(2B)-C(8B)-H(8B) | 112.5(6) |
| C(9B)-C(8B)-H(8B) | 107.8(7) |
| C(7B)-C(8B)-H(8B) | 108.2(7) |
| C(13B)-C(9B)-C(8B) | 113.00(13) |
| C(13B)-C(9B)-C(10B) | 114.15(13) |
| C(8B)-C(9B)-C(10B) | 103.03(12) |
| C(13B)-C(9B)-H(9B) | 109.4(7) |
| C(8B)-C(9B)-H(9B) | 104.5(7) |
| C(10B)-C(9B)-H(9B) | 112.3(7) |
| C(9B)-C(10B)-C(11B) | 106.44(12) |
| C(9B)-C(10B)-H(10C) | 111.1(8) |
| C(11B)-C(10B)-H(10C) | 109.8(8) |
| C(9B)-C(10B)-H(10D) | 109.6(8) |
| C(11B)-C(10B)-H(10D) | 107.0(8) |
| H(10C)-C(10B)-H(10D) | 112.8(12) |
| C(12B)-C(11B)-C(15B) | 104.11(11) |
| C(12B)-C(11B)-C(7B) | 113.14(11) |
| C(15B)-C(11B)-C(7B) | 113.85(12) |
| C(12B)-C(11B)-C(10B) | 109.70(12) |
| C(15B)-C(11B)-C(10B) | 111.03(12) |
| C(7B)-C(11B)-C(10B) | 105.12(11) |
| O(1B)-C(12B)-N(1B) | 121.49(13) |
| O(1B)-C(12B)-C(11B) | 119.52(13) |
| N(1B)-C(12B)-C(11B) | 118.97(13) |
| C(14B)-C(13B)-C(9B) | 126.38(18) |
| C(14B)-C(13B)-H(13B) | 120.7(9) |

| | |
|---|---|
| C(9B)-C(13B)-H(13B) | 112.9(8) |
| C(13B)-C(14B)-H(14C) | 117.8(10) |
| C(13B)-C(14B)-H(14D) | 119.9(10) |
| H(14C)-C(14B)-H(14D) | 122.0(14) |
| O(2B)-C(15B)-O(3B) | 124.10(14) |
| O(2B)-C(15B)-C(11B) | 125.52(15) |
| O(3B)-C(15B)-C(11B) | 110.37(13) |
| O(3B)-C(16B)-H(16D) | 108.7(9) |
| O(3B)-C(16B)-H(16E) | 102.4(9) |
| H(16D)-C(16B)-H(16E) | 117.4(13) |
| O(3B)-C(16B)-H(16F) | 111.0(9) |
| H(16D)-C(16B)-H(16F) | 104.4(12) |
| H(16E)-C(16B)-H(16F) | 113.0(13) |

*Table A7.2.4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for **241** (CCDC 820778). The anisotropic displacement factor exponent takes the form: $-2\pi^2[ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}$*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| O(1A) | 253(6) | 371(7) | 237(6) | -16(5) | 14(5) | 87(5) |
| O(2A) | 349(6) | 203(6) | 340(6) | -11(5) | 151(5) | -42(5) |
| O(3A) | 730(8) | 230(6) | 323(7) | -81(5) | 306(6) | -52(6) |
| N(1A) | 236(7) | 231(7) | 176(7) | 22(6) | 75(6) | 28(6) |
| N(2A) | 235(8) | 169(7) | 260(7) | -7(6) | 66(6) | -6(6) |
| C(1A) | 191(8) | 137(7) | 206(8) | -36(6) | 40(6) | 1(6) |
| C(2A) | 278(9) | 173(8) | 245(9) | -15(7) | 112(7) | -24(7) |
| C(3A) | 211(9) | 257(9) | 359(10) | -34(8) | 112(8) | -40(7) |
| C(4A) | 198(9) | 274(9) | 334(10) | -11(8) | 10(8) | -2(7) |
| C(5A) | 279(9) | 203(8) | 207(9) | 7(7) | 44(7) | -25(7) |
| C(6A) | 205(8) | 129(7) | 202(8) | -32(6) | 59(6) | -10(6) |
| C(7A) | 228(8) | 188(8) | 161(8) | -3(6) | 63(7) | 1(6) |
| C(8A) | 233(8) | 194(8) | 163(8) | 2(7) | 55(7) | 4(7) |
| C(9A) | 226(8) | 222(9) | 206(8) | 27(7) | 55(7) | -15(7) |
| C(10A) | 232(9) | 221(9) | 338(10) | -20(8) | 112(8) | 8(7) |
| C(11A) | 205(8) | 176(8) | 213(8) | -15(6) | 73(6) | 0(6) |
| C(12A) | 253(9) | 158(8) | 237(9) | -19(7) | 51(7) | 22(7) |
| C(13A) | 235(9) | 288(10) | 381(11) | -83(8) | -17(8) | 55(8) |
| C(14A) | 224(10) | 263(11) | 1040(20) | -139(13) | 28(12) | -15(8) |
| C(15A) | 214(8) | 235(9) | 241(9) | -3(7) | 69(7) | 31(7) |
| C(16A) | 862(19) | 309(12) | 416(13) | -147(10) | 319(13) | -61(12) |
| O(1B) | 262(6) | 314(6) | 272(6) | 94(5) | 53(5) | 48(5) |
| O(2B) | 376(7) | 523(8) | 461(7) | -28(6) | 106(6) | 209(6) |
| O(3B) | 371(7) | 321(7) | 408(7) | -122(5) | 149(6) | -33(5) |
| N(1B) | 212(7) | 210(7) | 226(7) | 33(6) | 54(6) | 5(6) |
| N(2B) | 411(10) | 219(8) | 492(10) | 91(7) | 91(8) | 14(7) |
| C(1B) | 263(9) | 184(8) | 203(8) | -19(6) | 46(7) | 39(7) |
| C(2B) | 248(9) | 242(9) | 273(9) | -7(7) | 66(7) | 4(7) |
| C(3B) | 228(9) | 308(10) | 290(9) | -1(8) | 8(8) | 46(8) |
| C(4B) | 355(10) | 328(10) | 257(9) | 54(8) | 18(8) | 98(8) |
| C(5B) | 362(10) | 266(9) | 280(9) | 74(7) | 124(8) | 38(8) |
| C(6B) | 258(9) | 206(8) | 231(8) | 4(7) | 72(7) | 26(7) |
| C(7B) | 268(9) | 203(8) | 275(9) | 48(7) | 110(7) | 10(7) |
| C(8B) | 278(9) | 202(9) | 337(10) | 32(7) | 101(8) | 2(7) |
| C(9B) | 271(9) | 234(9) | 365(10) | 11(8) | 120(8) | -32(7) |
| C(10B) | 248(9) | 237(10) | 431(11) | 0(8) | 47(8) | 5(7) |
| C(11B) | 242(8) | 201(8) | 277(9) | 15(7) | 79(7) | 22(7) |
| C(12B) | 244(9) | 174(8) | 238(9) | -29(7) | 55(7) | 21(7) |
| C(13B) | 266(10) | 279(10) | 449(11) | -18(8) | 99(8) | -21(8) |
| C(14B) | 375(11) | 426(12) | 656(15) | -141(11) | 227(11) | -88(9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(15B) | 316(10) | 266(9) | 298(9) | 61(7) | 118(8) | 29(8) |
| C(16B) | 580(14) | 312(11) | 432(12) | -127(9) | 232(12) | -18(10) |

*Table A7.2.5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters (Å$^2$x $10^3$) for **241** (CCDC 820778).*

| | x | y | z | $U_{iso}$ |
|---|---|---|---|---|
| H(1A) | 5539(7) | 1098(10) | 10472(13) | 34(5) |
| H(2A2) | 5783(7) | 4531(10) | 7573(13) | 34(5) |
| H(2A3) | 5119(7) | 4079(10) | 7431(12) | 38(5) |
| H(2A) | 4299(6) | 966(9) | 9922(11) | 16(4) |
| H(3A) | 3237(6) | 1368(9) | 8474(11) | 15(4) |
| H(4A) | 3231(7) | 2080(9) | 6620(12) | 31(4) |
| H(5A) | 4301(6) | 2378(9) | 6130(11) | 16(4) |
| H(7A) | 5564(5) | 2131(8) | 6654(11) | 9(3) |
| H(8A) | 5908(6) | 3154(8) | 8884(11) | 11(3) |
| H(9A) | 6693(6) | 3246(9) | 7194(11) | 15(4) |
| H(10A) | 7040(6) | 1680(9) | 7471(12) | 29(4) |
| H(10B) | 7229(7) | 1801(9) | 8872(12) | 27(4) |
| H(13A) | 7228(7) | 3553(10) | 9747(13) | 36(5) |
| H(14A) | 7393(10) | 4747(16) | 7705(19) | 113(10) |
| H(14B) | 7747(9) | 4902(13) | 9228(15) | 66(6) |
| H(16A) | 6398(8) | -1022(13) | 6186(15) | 61(6) |
| H(16B) | 6281(9) | -339(12) | 4975(17) | 70(6) |
| H(16C) | 5637(9) | -701(12) | 5475(15) | 59(6) |
| | | | | |
| H(1B) | 10359(8) | 9538(11) | 5853(14) | 56(5) |
| H(2B2) | 9672(9) | 5832(13) | 7271(16) | 73(7) |
| H(2B3) | 8911(9) | 5518(14) | 6404(17) | 88(7) |
| H(2B) | 11404(6) | 8983(9) | 7218(11) | 18(4) |
| H(3B) | 11996(7) | 7978(9) | 8865(12) | 25(4) |
| H(4B) | 11405(7) | 6906(9) | 9899(12) | 31(4) |
| H(5B) | 10172(7) | 6897(10) | 9223(12) | 33(4) |
| H(7B) | 9067(6) | 7713(9) | 8053(11) | 19(4) |
| H(8B) | 9335(6) | 7055(8) | 5755(11) | 18(4) |
| H(9B) | 8070(6) | 6974(9) | 6494(12) | 29(4) |
| H(10C) | 7851(7) | 8599(10) | 5759(13) | 43(5) |
| H(10D) | 8346(7) | 8456(11) | 4745(14) | 52(5) |
| H(13B) | 8177(7) | 6812(11) | 3980(14) | 52(5) |
| H(14C) | 7503(8) | 5535(12) | 5387(16) | 67(6) |
| H(14D) | 7481(9) | 5385(13) | 3807(17) | 86(7) |
| H(16D) | 8958(8) | 11320(12) | 8388(14) | 52(5) |
| H(16E) | 9600(8) | 10826(12) | 9569(15) | 60(6) |
| H(16F) | 8750(8) | 10579(11) | 9338(14) | 51(5) |

*Table A7.2.6. Hydrogen bonds for **241** (CCDC 820778) [Å and °].*

| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| N(1A)-H(1A)...N(2A)#1 | 0.877(14) | 2.060(14) | 2.9300(18) | 171.0(13) |
| N(2A)-H(2A3)...O(2A)#2 | 0.950(14) | 2.120(14) | 3.0283(17) | 159.6(12) |
| N(1B)-H(1B)...O(1B)#3 | 0.984(15) | 1.820(16) | 2.7986(15) | 172.3(13) |
| N(2B)-H(2B2)...O(3B)#4 | 0.944(17) | 2.630(17) | 3.4707(18) | 148.6(14) |

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+1/2,z+1/2
#2 -x+1,y+1/2,-z+3/2
#3 -x+2,-y+2,-z+1
#4 -x+2,y-1/2,-z+3/2