Supplementary Material

Unique cyclolignan’s architecture obtained via acid catalyzed cyclization/intramolecular Friedel-Crafts tandem reaction

Krzysztof K. Krawczyk, Kamil Lisiecki, and Zbigniew Czarnocki*

Faculty of Chemistry, University of Warsaw, Pasteura 1, 02-093 Warsaw, Poland
Email: czarnoz@chem.uw.edu.pl

Table of Contents

1H NMR spectrum of 6 (CDCl$_3$, 500 MHz) .........................................................S2
13C NMR spectrum of 6 (CDCl$_3$, 125 MHz) ..............................................................S2
1H NMR spectrum of 7 (CDCl$_3$, 500 MHz) ..............................................................S3
13C NMR spectrum of 7 (CDCl$_3$, 125 MHz) ..............................................................S3
1H NMR spectrum of mixture of 8 and 9 (CDCl$_3$, 500 MHz) ..................................S4
1H NMR spectrum of 10 (CDCl$_3$, 500 MHz) ............................................................S4
1H NMR spectrum of 11 (CDCl$_3$, 600 MHz) ............................................................S5
13C NMR spectrum of 11 (CDCl$_3$, 150 MHz) ..........................................................S5
HMBC spectrum of 11 (CDCl$_3$, 600 MHz) ..............................................................S6
HSQC spectrum of 11 (CDCl$_3$, 600 MHz) ..............................................................S6
COSY spectrum of 11 (CDCl$_3$, 600 MHz) ...............................................................S7
ROESY spectrum of 11 (CDCl$_3$, 600 MHz) ...............................................................S7
Details of X-Ray Crystal Structure analysis for 11.........................................................S8
$^1$H NMR spectrum of 6 (CDCl$_3$, 500 MHz)

$^{13}$C NMR spectrum of 6 (CDCl$_3$, 125 MHz)
The 1H NMR spectrum of 7 in CDCl₃, recorded at 500 MHz, is shown above. The spectrum reveals the chemical shifts and coupling constants of the protons in the molecule, providing insights into its structural features.

The 13C NMR spectrum of 7 in CDCl₃, recorded at 125 MHz, is shown below. This spectrum provides information on the chemical shifts of the carbon atoms, which can be correlated with the observed protons to further elucidate the molecular structure.
$^1$H NMR spectrum of mixture of 8 and 9 (CDCl$_3$, 500 MHz)

$^1$H NMR spectrum of 10 (CDCl$_3$, 500 MHz)
\(^1\)H NMR spectrum of 11 (CDCl\(_3\), 600 MHz)

\(^{13}\)C NMR spectrum of 11 (CDCl\(_3\), 150 MHz)
HMBC spectrum of 11 (CDCl₃, 600 MHz)

HSQC spectrum of 11 (CDCl₃, 600 MHz)
COSY spectrum of 11 (CDCl₃, 600 MHz)

ROESY spectrum of 11 (CDCl₃, 600 MHz)
Details of X-Ray Crystal Structure analysis for 11
The X-ray crystallographic experiment was performed using Oxford Diffraction Excalibur R single crystal diffractometer. The graphite-monochromatized Cu Kα radiation was applied. The preliminary experiment was used for calculation of unit cell dimensions and crystal symmetry assignment. Final experiment was then performed. Data were corrected for Lorenz-polarization effects and for absorption. Collected data were used for structure solution and refinement. Direct methods from SHELXS-97 and full matrix least-squares refinement from SHELXL-97 (G.M. Sheldrick, Acta Cryst. (2008). A64, 112–122) were applied for this purposes. To enable determination of an absolute structure all Friedel pairs were measured. During refinement correctness of the molecule configuration was verified using the Flack parameter (S. Parsons, P. Pattisonb, H.D. Flack, Acta Cryst. (2012). A68, 736–749) – the near-zero value correspond to a proper absolute structure and hence a proper enantiomer/diastereoisomer of the compound.

Crystal structure of 11. A good quality colorless block shaped crystal of dimensions 0.4×0.07×0.02 mm was mounted on the diffractometer. 24068 reflections were collected of which 4849 were classified as independent after merging. The crystals of 11 crystallize in the acentric orthorhombic P2_{1}2_{1}2_{1} space group. The experimental parameters for the structure are collected in Tables (vide infra). The conformation of the molecule is shown in Figure 1. The crystal packing is shown in Figure 2. The characteristic O-H····O hydrogen bonds are depicted as the dashed lines. The full experimental and structural parameters were deposited with Cambridge Structural Data Centre under the number CCDC 1977939.

### Table 1. Crystal data and structure refinement for ZC171-1abs

| Parameter                                  | Value                                      |
|--------------------------------------------|--------------------------------------------|
| Identification code                        | zc171-1abs                                 |
| Deposit number                             | CCDC 1977939                               |
| Empirical formula                          | C_{29}H_{32}NO_{9}                         |
| Formula weight                             | 538.56                                     |
| Temperature                                | 293(2) K                                   |
| Wavelength                                 | 1.54178 Å                                  |
| Crystal system                             | Orthorhombic                               |
| Space group                                | P 2_{1}2_{1}2_{1}                          |
| Unit cell dimensions                       | a = 5.02660(10) Å, b = 13.0853(2) Å, c = 39.8047(9) Å |
|                                           | b = 90°                                    |
|                                           | c = 90°                                    |
| Volume                                     | 2618.14(9) Å³                             |
| Z                                         | 4                                          |
| Density (calculated)                       | 1.366 Mg/m³                                |
| Absorption coefficient                     | 0.846 mm⁻¹                                 |
| F(000)                                     | 1140                                       |
| Crystal size                               | 0.3773 x 0.0721 x 0.0224 mm³               |
| Theta range for data collection            | 3.56 to 70.37°                             |
| Index ranges                               | -5≤h≤5, -15≤k≤15, -48≤l≤47                 |
| Reflections collected                      | 24068                                      |
| Independent reflections                    | 4849 [R(int) = 0.0590]                     |
Completeness to theta = 70.37° 97.9 %
Absorption correction Analytical
Max. and min. transmission 0.981 and 0.838
Refinement method Full-matrix least-squares on $F^2$
Data / restraints / parameters 4849 / 0 / 355
Goodness-of-fit on $F^2$ 0.840
Final R indices [$I>2\sigma(I)$] R1 = 0.0381, wR2 = 0.0790
R indices (all data) R1 = 0.0688, wR2 = 0.0862
Absolute structure parameter -0.1(2)
Largest diff. peak and hole 0.159 and -0.167 e.Å$^{-3}$
Table 2. Atomic coordinates ($x\times10^4$) and equivalent isotropic displacement parameters (Å$^2\times10^3$) for ZC171-1abs. U(eq) is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

|   | x     | y     | z     | U(eq) |
|---|-------|-------|-------|-------|
| O(1) | 6832(4) | 2111(1) | 9282(1) | 67(1) |
| O(2) | 6734(4) | 3760(1) | 8506(1) | 56(1) |
| O(3) | 9981(4) | 923(1)  | 9802(1) | 62(1) |
| O(4) | 13210(5) | -791(1) | 9863(1) | 71(1) |
| O(5) | 14682(5) | -1834(1) | 9324(1) | 71(1) |
| O(6) | 9261(4) | -1413(1) | 8254(1) | 70(1) |
| O(7) | 12312(4) | -1551(1) | 7655(1) | 60(1) |
| O(8) | 15188(5) | 57(1)  | 7457(1) | 67(1) |
| C(1) | 7909(5) | 1472(2) | 9107(1) | 48(1) |
| C(2) | 9610(5) | 618(2)  | 9208(1) | 44(1) |
| C(3) | 10615(6) | 352(2)  | 9525(1) | 51(1) |
| C(4) | 12242(6) | -492(2) | 9555(1) | 56(1) |
| C(5) | 13003(6) | -1041(2) | 9266(1) | 53(1) |
| C(6) | 11991(6) | -789(2) | 8952(1) | 48(1) |
| C(7) | 10248(5) | 27(2)   | 8928(1) | 44(1) |
| C(8) | 8757(6)  | 386(2)  | 8617(1) | 45(1) |
| C(9) | 10370(5) | 321(2)  | 8302(1) | 43(1) |
| C(10) | 10564(5) | -589(2) | 8118(1) | 47(1) |
| C(11) | 12096(6) | -642(2) | 7828(1) | 48(1) |
| C(12) | 13626(6) | 202(2)  | 7733(1) | 50(1) |
| C(13) | 13482(6) | 1103(2) | 7912(1) | 47(1) |
| C(14) | 11814(5) | 1170(2) | 8193(1) | 42(1) |
| C(15) | 11399(5) | 2152(2) | 8361(1) | 43(1) |
| C(16) | 9465(5)  | 2313(2) | 8583(1) | 43(1) |
| C(17) | 7770(5)  | 1465(2) | 8720(1) | 44(1) |
| C(18) | 8685(6)  | 3404(2) | 8656(1) | 45(1) |
| N(19) | 10254(5) | 3966(1) | 8853(1) | 50(1) |
| C(20) | 12140(6) | 3565(2) | 9102(1) | 63(1) |
| C(21) | 12172(11) | 4336(2) | 9364(1) | 120(2) |
| C(22) | 11177(8) | 5289(2) | 9235(1) | 76(1) |
| C(23) | 9778(6)  | 5074(2) | 8903(1) | 53(1) |
| C(24) | 12061(7) | 1562(2) | 9915(1) | 80(1) |
| C(25) | 11347(9) | -1390(3) | 10044(1) | 109(1) |
| C(26) | 15847(6) | -2333(2) | 9045(1) | 68(1) |
| C(27) | 7805(8)  | -2095(2) | 8056(1) | 81(1) |
| C(28) | 11236(8) | -1532(2) | 7319(1) | 77(1) |
| C(29) | 16838(8) | 878(2)  | 7350(1) | 76(1) |
| C(30) | 10785(6) | 5671(2) | 8605(1) | 64(1) |
Table 3. Bond lengths [Å] and angles [°] for ZC171-1abs

| Bond            | Length  | Angle  |
|-----------------|---------|--------|
| O(1)-C(1)       | 1.214(3)|        |
| O(2)-C(18)      | 1.240(3)|        |
| O(3)-C(3)       | 1.366(3)|        |
| O(3)-C(24)      | 1.412(4)|        |
| O(4)-C(4)       | 1.378(3)|        |
| O(4)-C(25)      | 1.418(4)|        |
| O(5)-C(5)       | 1.358(3)|        |
| O(5)-C(26)      | 1.416(3)|        |
| O(6)-C(10)      | 1.373(3)|        |
| O(6)-C(27)      | 1.397(3)|        |
| O(7)-C(11)      | 1.378(3)|        |
| O(7)-C(28)      | 1.444(3)|        |
| O(8)-C(12)      | 1.362(3)|        |
| O(8)-C(29)      | 1.423(4)|        |
| C(1)-C(2)       | 1.464(3)|        |
| C(1)-C(17)      | 1.543(3)|        |
| C(2)-C(7)       | 1.393(3)|        |
| C(2)-C(3)       | 1.404(3)|        |
| C(3)-C(4)       | 1.379(3)|        |
| C(4)-C(5)       | 1.408(3)|        |
| C(5)-C(6)       | 1.387(3)|        |
| C(6)-C(7)       | 1.384(3)|        |
| C(6)-H(6)       | 0.9300  |        |
| C(7)-C(8)       | 1.522(3)|        |
| C(8)-C(9)       | 1.497(3)|        |
| C(8)-C(17)      | 1.551(3)|        |
| C(8)-H(8)       | 0.9800  |        |
| C(9)-C(14)      | 1.396(3)|        |
| C(9)-C(10)      | 1.400(3)|        |
| C(10)-C(11)     | 1.390(3)|        |
| C(11)-C(12)     | 1.399(4)|        |
| C(12)-C(13)     | 1.379(3)|        |
| C(13)-C(14)     | 1.402(3)|        |
| C(13)-H(13)     | 0.9300  |        |
| C(14)-C(15)     | 1.462(3)|        |
| C(15)-C(16)     | 1.332(3)|        |
C(15)-H(15) 0.9300
C(16)-C(17) 1.501(3)
C(16)-C(18) 1.508(3)
C(17)-H(17) 0.9800
C(18)-N(19) 1.333(3)
N(19)-C(20) 1.470(3)
N(19)-C(23) 1.483(3)
C(20)-C(21) 1.449(4)
C(20)-H(20A) 0.9700
C(20)-H(20B) 0.9700
C(21)-C(22) 1.439(4)
C(21)-H(21) 0.9300
C(22)-C(23) 1.523(4)
C(22)-H(22A) 0.9700
C(22)-H(22B) 0.9700
C(23)-C(30) 1.507(4)
C(23)-H(23) 0.9800
C(24)-H(24A) 0.9600
C(24)-H(24B) 0.9600
C(24)-H(24C) 0.9600
C(25)-H(25A) 0.9600
C(25)-H(25B) 0.9600
C(25)-H(25C) 0.9600
C(26)-H(26A) 0.9600
C(26)-H(26B) 0.9600
C(26)-H(26C) 0.9600
C(27)-H(27A) 0.9600
C(27)-H(27B) 0.9600
C(27)-H(27C) 0.9600
C(28)-H(28A) 0.9600
C(28)-H(28B) 0.9600
C(28)-H(28C) 0.9600
C(29)-H(29A) 0.9600
C(29)-H(29B) 0.9600
C(29)-H(29C) 0.9600
C(30)-O(9) 1.434(4)
C(30)-H(30A) 0.9700
C(30)-H(30B) 0.9700
O(9)-H(9) 0.96(4)

C(3)-O(3)-C(24) 114.1(2)
C(4)-O(4)-C(25) 112.1(3)
C(5)-O(5)-C(26) 118.4(2)
C(10)-O(6)-C(27) 122.0(2)
| Bond          | Angle (°)  |
|--------------|-----------|
| C(11)-O(7)-C(28) | 114.71(18) |
| C(12)-O(8)-C(29) | 118.16(19) |
| O(1)-C(1)-C(2) | 129.0(2)   |
| O(1)-C(1)-C(17) | 123.8(2)   |
| C(2)-C(1)-C(17) | 107.2(2)   |
| C(7)-C(2)-C(3) | 119.9(2)   |
| C(7)-C(2)-C(1) | 109.8(2)   |
| C(3)-C(2)-C(1) | 130.3(2)   |
| O(3)-C(3)-C(4) | 120.6(2)   |
| O(3)-C(3)-C(2) | 120.3(2)   |
| C(4)-C(3)-C(2) | 119.1(2)   |
| O(4)-C(4)-C(3) | 120.8(2)   |
| O(4)-C(4)-C(5) | 119.1(2)   |
| C(3)-C(4)-C(5) | 120.1(2)   |
| O(5)-C(5)-C(6) | 124.3(2)   |
| O(5)-C(5)-C(4) | 114.8(2)   |
| C(6)-C(5)-C(4) | 120.9(2)   |
| C(7)-C(6)-C(5) | 118.5(2)   |
| C(7)-C(6)-H(6) | 120.8      |
| C(5)-C(6)-H(6) | 120.8      |
| C(6)-C(7)-C(2) | 112.2(2)   |
| C(6)-C(7)-C(8) | 127.3(2)   |
| C(2)-C(7)-C(8) | 111.4(2)   |
| C(9)-C(8)-C(7) | 113.5(2)   |
| C(9)-C(8)-C(17) | 116.44(17) |
| C(7)-C(8)-C(17) | 103.00(17) |
| C(9)-C(8)-H(8) | 107.8      |
| C(7)-C(8)-H(8) | 107.8      |
| C(17)-C(8)-H(8) | 107.8      |
| C(14)-C(9)-C(10) | 118.6(2)   |
| C(14)-C(9)-C(8) | 119.76(19) |
| C(10)-C(9)-C(8) | 121.6(2)   |
| O(6)-C(10)-C(11) | 123.5(2)   |
| O(6)-C(10)-C(9) | 115.4(2)   |
| C(11)-C(10)-C(9) | 121.0(2)   |
| O(7)-C(11)-C(10) | 120.1(2)   |
| O(7)-C(11)-C(12) | 120.2(2)   |
| C(10)-C(11)-C(12) | 119.4(2)   |
| O(8)-C(12)-C(13) | 124.5(2)   |
| O(8)-C(12)-C(11) | 115.2(2)   |
| C(13)-C(12)-C(11) | 120.3(2)   |
| C(12)-C(13)-C(14) | 119.9(2)   |
| C(12)-C(13)-H(13) | 120.0      |
| C(14)-C(13)-H(13) | 120.0      |
C(9)-C(14)-C(13)  120.5(2)
C(9)-C(14)-C(15)  118.9(2)
C(13)-C(14)-C(15)  120.3(2)
C(16)-C(15)-C(14)  123.1(2)
C(16)-C(15)-H(15)  118.4
C(14)-C(15)-H(15)  118.4
C(15)-C(16)-C(17)  122.5(2)
C(15)-C(16)-C(18)  117.9(2)
C(17)-C(16)-C(18)  118.9(2)
C(16)-C(17)-C(1)  109.37(19)
C(16)-C(17)-C(8)  113.3(2)
C(1)-C(17)-C(8)  104.70(17)
C(16)-C(17)-H(17)  109.8
C(1)-C(17)-H(17)  109.8
C(8)-C(17)-H(17)  109.8
O(2)-C(18)-N(19)  123.0(2)
O(2)-C(18)-C(16)  118.0(2)
N(19)-C(18)-C(16)  118.7(2)
C(18)-N(19)-C(20)  125.60(19)
C(18)-N(19)-C(23)  121.5(2)
C(20)-N(19)-C(23)  111.28(19)
C(21)-C(20)-N(19)  104.1(2)
C(21)-C(20)-H(20A)  110.9
N(19)-C(20)-H(20A)  110.9
C(21)-C(20)-H(20B)  110.9
N(19)-C(20)-H(20B)  110.9
H(20A)-C(20)-H(20B)  109.0
C(22)-C(21)-C(20)  110.1(2)
C(22)-C(21)-H(21)  125.0
C(20)-C(21)-H(21)  125.0
C(21)-C(22)-C(23)  108.1(2)
C(21)-C(22)-H(22A)  110.1
C(23)-C(22)-H(22A)  110.1
C(21)-C(22)-H(22B)  110.1
C(23)-C(22)-H(22B)  110.1
H(22A)-C(22)-H(22B)  108.4
N(19)-C(23)-C(30)  110.4(2)
N(19)-C(23)-C(22)  102.8(2)
C(30)-C(23)-C(22)  115.5(2)
N(19)-C(23)-H(23)  109.3
C(30)-C(23)-H(23)  109.3
C(22)-C(23)-H(23)  109.3
O(3)-C(24)-H(24A)  109.5
O(3)-C(24)-H(24B)  109.5
H(24A)-C(24)-H(24B)  109.5
O(3)-C(24)-H(24C)  109.5
H(24A)-C(24)-H(24C)  109.5
H(24B)-C(24)-H(24C)  109.5
O(4)-C(25)-H(25A)  109.5
O(4)-C(25)-H(25B)  109.5
H(25A)-C(25)-H(25B)  109.5
O(4)-C(25)-H(25C)  109.5
H(25A)-C(25)-H(25C)  109.5
H(25B)-C(25)-H(25C)  109.5
O(5)-C(26)-H(26A)  109.5
O(5)-C(26)-H(26B)  109.5
H(26A)-C(26)-H(26B)  109.5
O(5)-C(26)-H(26C)  109.5
H(26A)-C(26)-H(26C)  109.5
H(26B)-C(26)-H(26C)  109.5
O(6)-C(27)-H(27A)  109.5
O(6)-C(27)-H(27B)  109.5
H(27A)-C(27)-H(27B)  109.5
O(6)-C(27)-H(27C)  109.5
H(27A)-C(27)-H(27C)  109.5
H(27B)-C(27)-H(27C)  109.5
O(7)-C(28)-H(28A)  109.5
O(7)-C(28)-H(28B)  109.5
H(28A)-C(28)-H(28B)  109.5
O(7)-C(28)-H(28C)  109.5
H(28A)-C(28)-H(28C)  109.5
H(28B)-C(28)-H(28C)  109.5
O(8)-C(29)-H(29A)  109.5
O(8)-C(29)-H(29B)  109.5
H(29A)-C(29)-H(29B)  109.5
O(8)-C(29)-H(29C)  109.5
H(29A)-C(29)-H(29C)  109.5
H(29B)-C(29)-H(29C)  109.5
O(9)-C(30)-C(23)  114.3(2)
O(9)-C(30)-H(30A)  108.7
C(23)-C(30)-H(30A)  108.7
O(9)-C(30)-H(30B)  108.7
C(23)-C(30)-H(30B)  108.7
H(30A)-C(30)-H(30B)  107.6
C(30)-O(9)-H(9)  106(2)
Table 4. Anisotropic displacement parameters (Å² x 10³) for ZC171-1abs. The anisotropic displacement factor exponent takes the form: -2π² [ h² a*² U₁₁ + ... + 2 h k a* b* U₁₂

|     | U₁₁     | U₂₂     | U₃₃     | U₂₃     | U₁₃     | U₁₂     |
|-----|---------|---------|---------|---------|---------|---------|
| O(1)| 80(2)   | 65(1)   | 56(1)   | -5(1)   | 6(1)    | 28(1)   |
| O(2)| 57(1)   | 47(1)   | 63(1)   | -1(1)   | -14(1)  | 10(1)   |
| O(3)| 72(1)   | 67(1)   | 47(1)   | -11(1)  | 4(1)    | 6(1)    |
| O(4)| 89(2)   | 74(1)   | 51(1)   | 9(1)    | -9(1)   | 17(1)   |
| O(5)| 95(2)   | 60(1)   | 57(1)   | 4(1)    | -6(1)   | 29(1)   |
| O(6)| 106(2)  | 49(1)   | 54(1)   | -5(1)   | -3(1)   | -26(1)  |
| O(7)| 87(2)   | 42(1)   | 51(1)   | -13(1)  | -8(1)   | 8(1)    |
| O(8)| 86(2)   | 55(1)   | 61(1)   | -10(1)  | 16(1)   | 1(1)    |
| C(1)| 50(2)   | 47(1)   | 48(1)   | -1(1)   | 5(1)    | 0(1)    |
| C(2)| 52(2)   | 38(1)   | 42(1)   | 0(1)    | 3(1)    | 0(1)    |
| C(3)| 62(2)   | 51(1)   | 42(1)   | -2(1)   | 5(1)    | 3(1)    |
| C(4)| 75(2)   | 49(1)   | 43(1)   | 4(1)    | -3(1)   | 7(1)    |
| C(5)| 67(2)   | 42(1)   | 51(2)   | 5(1)    | -5(2)   | 10(1)   |
| C(6)| 64(2)   | 37(1)   | 44(1)   | -4(1)   | 2(1)    | 2(1)    |
| C(7)| 54(2)   | 36(1)   | 43(1)   | 4(1)    | -1(1)   | -4(1)   |
| C(8)| 51(2)   | 39(1)   | 44(1)   | -1(1)   | -4(1)   | -8(1)   |
| C(9)| 48(2)   | 41(1)   | 40(1)   | -1(1)   | -8(1)   | 1(1)    |
| C(10)| 59(2)  | 38(1)   | 43(1)   | 0(1)    | -5(1)   | -4(1)   |
| C(11)| 64(2)  | 35(1)   | 46(1)   | -8(1)   | -8(1)   | 8(1)    |
| C(12)| 59(2)  | 49(1)   | 41(1)   | -4(1)   | 4(1)    | 7(1)    |
| C(13)| 52(2)  | 39(1)   | 49(1)   | 0(1)    | 1(1)    | -2(1)   |
| C(14)| 46(2)  | 38(1)   | 41(1)   | 1(1)    | -6(1)   | 0(1)    |
| C(15)| 49(2)  | 39(1)   | 43(1)   | -2(1)   | -5(1)   | -1(1)   |
| C(16)| 46(2)  | 39(1)   | 43(1)   | -2(1)   | -6(1)   | 8(1)    |
| C(17)| 46(2)  | 45(1)   | 43(1)   | 0(1)    | -3(1)   | 2(1)    |
| C(18)| 53(2)  | 43(1)   | 40(1)   | 2(1)    | 3(1)    | 6(1)    |
| N(19)| 59(2)  | 40(1)   | 50(1)   | -7(1)   | -11(1)  | 9(1)    |
| C(20)| 66(2)  | 57(2)   | 64(2)   | -2(1)   | -15(2)  | 10(2)   |
| C(21)| 218(5) | 69(2)   | 73(2)   | -22(2)  | -72(3)  | 38(3)   |
| C(22)| 105(3) | 61(2)   | 61(2)   | -22(1)  | -12(2)  | 5(2)    |
| C(23)| 57(2)  | 41(1)   | 59(2)   | -10(1)  | 1(2)    | 6(1)    |
| C(24)| 96(3)  | 73(2)   | 69(2)   | -17(2)  | 6(2)    | -9(2)   |
| C(25)| 149(4) | 107(2)  | 71(2)   | 34(2)   | 4(3)    | -5(3)   |
| C(26)| 80(2)  | 47(1)   | 76(2)   | -1(1)   | 0(2)    | 15(2)   |
| C(27)| 92(3)  | 67(2)   | 84(2)   | -17(2)  | -10(2)  | -23(2)  |
| C(28)| 116(3) | 65(2)   | 52(2)   | -17(1)  | -14(2)  | -1(2)   |
| C(29)| 96(3)  | 62(2)   | 70(2)   | 5(1)    | 26(2)   | 5(2)    |
| C(30)| 74(3)  | 45(1)   | 74(2)   | -1(1)   | -10(2)  | 7(1)    |
| O(9)| 70(2)  | 52(1)   | 103(2)  | -1(1)   | 12(1)   | 3(1)    |
Table 5. Hydrogen coordinates ($x \times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^3$) for ZC171-1abs

|   | x   | y   | z   | $U_{eq}$ |
|---|-----|-----|-----|----------|
| H(6) | 12472 | -1160 | 8763 | 58       |
| H(8)  | 7193  | -53  | 8589 | 54       |
| H(13) | 14490 | 1664 | 7846 | 56       |
| H(15) | 12539 | 2689 | 8309 | 52       |
| H(17) | 5924  | 1558 | 8647 | 53       |
| H(20A) | 11542 | 2913 | 9190 | 75       |
| H(20B) | 13895 | 3480 | 9005 | 75       |
| H(21) | 12751 | 4230 | 9583 | 144      |
| H(22A) | 12633 | 5764 | 9200 | 91       |
| H(22B) | 9939  | 5591 | 9393 | 91       |
| H(23) | 7865  | 5195 | 8929 | 63       |
| H(24A) | 11480 | 1936 | 10109 | 119    |
| H(24B) | 12540 | 2032 | 9740 | 119      |
| H(24C) | 13576 | 1152 | 9973 | 119      |
| H(25A) | 12099 | -1583 | 10257 | 163    |
| H(25B) | 10926 | -1993 | 9918 | 163      |
| H(25C) | 9756  | -1000 | 10081 | 163    |
| H(26A) | 16974 | -2876 | 9122 | 101      |
| H(26B) | 16884 | -1852 | 8918 | 101      |
| H(26C) | 14471 | -2609 | 8904 | 101      |
| H(27A) | 7017  | -2608 | 8197 | 121      |
| H(27B) | 8968  | -2415 | 7896 | 121      |
| H(27C) | 6431  | -1731 | 7939 | 121      |
| H(28A) | 11453 | -2192 | 7217 | 116      |
| H(28B) | 12160 | -1029 | 7188 | 116      |
| H(28C) | 9378  | -1364 | 7328 | 116      |
| H(29A) | 17828 | 673  | 7155 | 114      |
| H(29B) | 18046 | 1058 | 7527 | 114      |
| H(29C) | 15750 | 1458 | 7295 | 114      |
| H(30A) | 9949  | 5407 | 8403 | 77       |
| H(30B) | 10241 | 6378 | 8630 | 77       |
| H(9)   | 14060(70) | 4940(30) | 8520(8) | 112    |
Table 6. Torsion angles [°] for ZC171-1abs

| Bond | Torsion Angle [°] |
|------|-------------------|
| O(1)-C(1)-C(2)-C(7) | 175.3(3) |
| C(17)-C(1)-C(2)-C(7) | -7.1(3) |
| O(1)-C(1)-C(2)-C(3) | -5.5(5) |
| C(17)-C(1)-C(2)-C(3) | 172.2(3) |
| C(24)-O(3)-C(3)-C(4) | 75.4(3) |
| C(24)-O(3)-C(3)-C(2) | -104.8(3) |
| C(7)-C(2)-C(3)-O(3) | -179.5(2) |
| C(1)-C(2)-C(3)-O(3) | 1.4(4) |
| C(7)-C(2)-C(3)-C(4) | 0.4(4) |
| C(1)-C(2)-C(3)-C(4) | -178.7(3) |
| C(25)-O(4)-C(4)-C(3) | 82.9(3) |
| C(25)-O(4)-C(4)-C(5) | -99.1(3) |
| O(3)-C(3)-C(4)-O(4) | 1.5(4) |
| C(2)-C(3)-C(4)-O(4) | -178.3(3) |
| O(3)-C(3)-C(4)-C(5) | -176.4(3) |
| C(2)-C(3)-C(4)-C(5) | 3.7(4) |
| C(26)-O(5)-C(5)-C(6) | 11.3(4) |
| C(26)-O(5)-C(5)-C(4) | -171.0(3) |
| O(4)-C(4)-C(5)-O(5) | -0.3(4) |
| C(3)-C(4)-C(5)-O(5) | 177.7(3) |
| O(4)-C(4)-C(5)-C(6) | 177.5(3) |
| C(3)-C(4)-C(5)-C(6) | -4.5(4) |
| O(5)-C(5)-C(6)-C(7) | 178.7(2) |
| C(4)-C(5)-C(6)-C(7) | 1.1(4) |
| C(5)-C(6)-C(7)-C(2) | 3.0(4) |
| C(5)-C(6)-C(7)-C(8) | -175.3(2) |
| C(3)-C(2)-C(7)-C(6) | -3.8(4) |
| C(1)-C(2)-C(7)-C(6) | 175.5(2) |
| C(3)-C(2)-C(7)-C(8) | 174.7(2) |
| C(1)-C(2)-C(7)-C(8) | -6.0(3) |
| C(6)-C(7)-C(8)-C(9) | -38.6(3) |
| C(2)-C(7)-C(8)-C(9) | 142.9(2) |
| C(6)-C(7)-C(8)-C(17) | -165.4(2) |
| C(2)-C(7)-C(8)-C(17) | 16.1(3) |
| C(7)-C(8)-C(9)-C(14) | -93.2(3) |
| C(17)-C(8)-C(9)-C(14) | 26.2(3) |
| C(7)-C(8)-C(9)-C(10) | 84.0(3) |
| C(17)-C(8)-C(9)-C(10) | -156.6(2) |
| C(27)-O(6)-C(10)-C(11) | -44.7(4) |
| C(27)-O(6)-C(10)-C(9) | 138.5(3) |
| C(14)-C(9)-C(10)-O(6) | 175.0(2) |
| C(8)-C(9)-C(10)-O(6) | -2.2(4) |
C(14)-C(9)-C(10)-C(11) -1.9(4)
C(8)-C(9)-C(10)-C(11) -179.1(2)
C(28)-O(7)-C(11)-C(10) 116.6(3)
C(28)-O(7)-C(11)-C(12) -69.9(3)
O(6)-C(10)-C(11)-O(7) 1.8(4)
C(9)-C(10)-C(11)-O(7) 178.5(2)
O(6)-C(10)-C(11)-C(12) -171.7(2)
C(9)-C(10)-C(11)-C(12) 171.1(2)
C(29)-O(8)-C(12)-C(13) 1.6(4)
C(29)-O(8)-C(12)-C(11) -178.8(3)
O(7)-C(11)-C(12)-O(8) 2.5(4)
C(10)-C(11)-C(12)-O(8) 176.1(2)
O(7)-C(11)-C(12)-C(13) -177.8(2)
C(10)-C(11)-C(12)-C(13) -4.3(4)
O(8)-C(12)-C(13)-C(14) -179.7(2)
C(11)-C(12)-C(13)-C(14) 0.6(4)
C(10)-C(9)-C(14)-C(13) -1.8(4)
C(8)-C(9)-C(14)-C(13) 175.5(2)
C(10)-C(9)-C(14)-C(15) 173.3(2)
C(8)-C(9)-C(14)-C(15) -9.4(3)
C(12)-C(13)-C(14)-C(15) 2.5(4)
C(12)-C(13)-C(14)-C(15) -172.6(2)
C(9)-C(14)-C(15)-C(16) -9.0(4)
C(13)-C(14)-C(15)-C(16) 166.2(2)
C(14)-C(15)-C(16)-C(17) 8.9(4)
C(14)-C(15)-C(16)-C(18) -161.1(2)
C(15)-C(16)-C(17)-C(1) 124.8(2)
C(18)-C(16)-C(17)-C(1) -65.3(3)
C(15)-C(16)-C(17)-C(8) 8.4(3)
C(18)-C(16)-C(17)-C(8) 178.3(2)
O(1)-C(1)-C(17)-C(16) 72.7(3)
C(2)-C(1)-C(17)-C(16) -105.1(2)
O(1)-C(1)-C(17)-C(16) -165.5(3)
C(2)-C(1)-C(17)-C(8) 16.7(3)
C(9)-C(8)-C(17)-C(16) -24.9(3)
C(7)-C(8)-C(17)-C(16) 100.0(2)
C(9)-C(8)-C(17)-C(1) -144.0(2)
C(7)-C(8)-C(17)-C(1) -19.2(3)
C(15)-C(16)-C(18)-O(2) 97.1(3)
C(17)-C(16)-C(18)-O(2) -73.3(3)
C(15)-C(16)-C(18)-N(19) -76.7(3)
C(17)-C(16)-C(18)-N(19) 112.9(3)
O(2)-C(18)-N(19)-C(20) 163.4(2)
C(16)-C(18)-N(19)-C(20) -23.1(4)
Table 7. Hydrogen bonds for ZC171-1abs [Å and °]

| D-H····A          | d(D-H)   | d(H····A) | d(D····A) | <(DHA) |
|-------------------|----------|-----------|-----------|--------|
| O(9)-H(9)····O(2)#1 | 0.96(4)  | 2.05(4)   | 2.923(3)  | 151(3) |

Symmetry transformations used to generate equivalent atoms:

#1 x+1,y,z