Table 1. Crystal data and structure refinement for **Chapingolide**.

| | | |
|---|---|---|
| Empirical formula | $C_9 H_{12} O_4$ | |
| Formula weight | 184.19 | |
| Temperature | 298(2) K | |
| Wavelength | 1.54178 Å | |
| Crystal system | Monoclinic | |
| Space group | $P 2_1$ | |
| Unit cell dimensions | $a$ = 5.1034(7) Å | α= 90° |
| | $b$ = 10.8665(15) Å | β= 92.595(6)° |
| | $c$ = 7.6481(10) Å | γ = 90° |
| Volume | 423.70(10) Å$^3$ | |
| $Z$ | 2 | |
| Density (calculated) | 1.444 Mg/m$^3$ | |
| Absorption coefficient | 0.960 mm$^{-1}$ | |
| $F$(000) | 196 | |
| Crystal size / colour / shape | 0.412 x 0.172 x 0.042 mm / colourless / lamina | |
| Theta range for data collection | 5.791 to 74.183° | |
| Index ranges | -6 $h$ 5, -13 $k$ 13, -9 $l$ 9 | |
| Reflections collected | 17267 | |
| Independent reflections | 1729 [$R$(int) = 0.0302] | |
| Completeness to theta = 67.679° | 100.0 % | |
| Measurement device | Bruker D8 Venture -geometry diffractometer 208039-01 | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.7538 and 0.6971 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 1729 / 1 / 124 | |
| Goodness-of-fit on $F^2$ | 1.045 | |
| Final $R$ indices [$I$>2($I$)] | $R$1 = 0.0285, $wR$2 = 0.0694 | |
| $R$ indices (all data) | $R$1 = 0.0301, $wR$2 = 0.0706 | |
| Absolute structure parameter | 0.09(5) | |
| Largest diff. peak and hole | 0.164 and -0.122 e.Å$^{-3}$ | |

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

| | x | y | z | U(eq) |
|---|---|---|---|---|
| O(1) | -1435(3) | 3649(2) | 11827(2) | 46(1) |
| C(1) | 3018(4) | 5572(2) | 9831(3) | 36(1) |
| O(2) | 583(3) | 5180(1) | 10591(2) | 37(1) |
| C(3) | 416(4) | 3991(2) | 11026(2) | 33(1) |
| C(4) | 2587(4) | 3178(2) | 10461(3) | 37(1) |
| C(5) | 3266(4) | 3444(2) | 8554(3) | 35(1) |
| C(6) | 1094(5) | 3082(2) | 7275(3) | 42(1) |
| C(7) | 297(4) | 3984(2) | 6242(3) | 40(1) |
| C(8) | 1835(4) | 5150(2) | 6556(2) | 33(1) |
| O(3) | 49(3) | 6145(1) | 6808(2) | 38(1) |
| C(9) | 3624(4) | 4846(2) | 8224(3) | 31(1) |
| C(10) | 3472(5) | 5464(3) | 4998(3) | 46(1) |
| O(4) | 4777(4) | 6601(2) | 5235(3) | 62(1) |

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

| | |
|---|---|
| O(1)-C(3) | 1.207(3) |
| C(1)-O(2) | 1.459(3) |
| C(1)-C(9) | 1.505(3) |
| C(1)-H(1A) | 0.9700 |
| C(1)-H(1B) | 0.9700 |
| O(2)-C(3) | 1.338(3) |
| C(3)-C(4) | 1.496(3) |
| C(4)-C(5) | 1.542(3) |
| C(4)-H(4B) | 0.9700 |
| C(4)-H(4C) | 0.9700 |
| C(5)-C(6) | 1.497(3) |
| C(5)-C(9) | 1.556(3) |
| C(5)-H(5) | 0.9800 |
| C(6)-C(7) | 1.312(4) |
| C(6)-H(6) | 0.9300 |
| C(7)-C(8) | 1.503(3) |
| C(7)-H(7) | 0.9300 |
| C(8)-O(3) | 1.433(3) |
| C(8)-C(10) | 1.525(3) |
| C(8)-C(9) | 1.569(2) |
| O(3)-H(3) | 0.84(4) |
| C(9)-H(9) | 0.9800 |
| C(10)-O(4) | 1.412(4) |
| C(10)-H(10A) | 0.9700 |
| C(10)-H(10B) | 0.9700 |
| O(4)-H(4) | 0.79(5) |
| | |
| O(2)-C(1)-C(9) | 112.47(16) |
| O(2)-C(1)-H(1A) | 109.1 |
| C(9)-C(1)-H(1A) | 109.1 |
| O(2)-C(1)-H(1B) | 109.1 |
| C(9)-C(1)-H(1B) | 109.1 |
| H(1A)-C(1)-H(1B) | 107.8 |
| C(3)-O(2)-C(1) | 116.51(16) |

| | |
|---|---|
| O(1)-C(3)-O(2) | 118.9(2) |
| O(1)-C(3)-C(4) | 124.9(2) |
| O(2)-C(3)-C(4) | 116.22(19) |
| C(3)-C(4)-C(5) | 111.31(17) |
| C(3)-C(4)-H(4B) | 109.4 |
| C(5)-C(4)-H(4B) | 109.4 |
| C(3)-C(4)-H(4C) | 109.4 |
| C(5)-C(4)-H(4C) | 109.4 |
| H(4B)-C(4)-H(4C) | 108.0 |
| C(6)-C(5)-C(4) | 112.18(19) |
| C(6)-C(5)-C(9) | 103.86(17) |
| C(4)-C(5)-C(9) | 111.74(17) |
| C(6)-C(5)-H(5) | 109.6 |
| C(4)-C(5)-H(5) | 109.6 |
| C(9)-C(5)-H(5) | 109.6 |
| C(7)-C(6)-C(5) | 113.4(2) |
| C(7)-C(6)-H(6) | 123.3 |
| C(5)-C(6)-H(6) | 123.3 |
| C(6)-C(7)-C(8) | 112.82(19) |
| C(6)-C(7)-H(7) | 123.6 |
| C(8)-C(7)-H(7) | 123.6 |
| O(3)-C(8)-C(7) | 109.05(16) |
| O(3)-C(8)-C(10) | 108.11(18) |
| C(7)-C(8)-C(10) | 111.39(19) |
| O(3)-C(8)-C(9) | 113.54(16) |
| C(7)-C(8)-C(9) | 103.45(17) |
| C(10)-C(8)-C(9) | 111.28(16) |
| C(8)-O(3)-H(3) | 106(2) |
| C(1)-C(9)-C(5) | 110.52(17) |
| C(1)-C(9)-C(8) | 114.86(17) |
| C(5)-C(9)-C(8) | 105.65(16) |
| C(1)-C(9)-H(9) | 108.5 |
| C(5)-C(9)-H(9) | 108.5 |
| C(8)-C(9)-H(9) | 108.5 |
| O(4)-C(10)-C(8) | 111.6(2) |
| O(4)-C(10)-H(10A) | 109.3 |

| | |
|---|---|
| C(8)-C(10)-H(10A) | 109.3 |
| O(4)-C(10)-H(10B) | 109.3 |
| C(8)-C(10)-H(10B) | 109.3 |
| H(10A)-C(10)-H(10B) | 108.0 |
| C(10)-O(4)-H(4) | 106(4) |

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

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| O(1) | 36(1) | 55(1) | 49(1) | 3(1) | 10(1) | -6(1) |
| C(1) | 40(1) | 32(1) | 36(1) | 1(1) | -4(1) | -8(1) |
| O(2) | 41(1) | 35(1) | 36(1) | -4(1) | 6(1) | 2(1) |
| C(3) | 31(1) | 38(1) | 29(1) | -1(1) | -3(1) | -3(1) |
| C(4) | 33(1) | 34(1) | 44(1) | 9(1) | 3(1) | 1(1) |
| C(5) | 33(1) | 32(1) | 41(1) | 2(1) | 5(1) | 6(1) |
| C(6) | 44(1) | 33(1) | 48(1) | -11(1) | 5(1) | -4(1) |
| C(7) | 33(1) | 45(1) | 41(1) | -12(1) | -3(1) | 0(1) |
| C(8) | 29(1) | 37(1) | 32(1) | 1(1) | -1(1) | 5(1) |
| O(3) | 32(1) | 34(1) | 47(1) | 0(1) | -1(1) | 6(1) |
| C(9) | 25(1) | 35(1) | 34(1) | 3(1) | -1(1) | -1(1) |
| C(10) | 39(1) | 64(2) | 35(1) | 6(1) | 2(1) | 8(1) |
| O(4) | 44(1) | 72(1) | 70(1) | 31(1) | 6(1) | -4(1) |

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

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1A) | 4452 | 5479 | 10698 | 44 |
| H(1B) | 2886 | 6437 | 9528 | 44 |
| H(4B) | 2065 | 2323 | 10565 | 45 |
| H(4C) | 4131 | 3308 | 11225 | 45 |
| H(5) | 4875 | 3005 | 8281 | 42 |
| H(6) | 380 | 2295 | 7218 | 50 |
| H(7) | -1074 | 3911 | 5406 | 48 |
| H(3) | 950(70) | 6790(30) | 6790(40) | 57 |
| H(9) | 5454 | 4997 | 7953 | 37 |
| H(10A) | 4756 | 4819 | 4844 | 55 |
| H(10B) | 2341 | 5500 | 3946 | 55 |
| H(4) | 6190(100) | 6440(40) | 5630(50) | 93 |

Table 6. Hydrogen bonds for **Chapingolide** [Å and °].

| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(4)-H(4B)...O(3)#1 | 0.97 | 2.65 | 3.362(3) | 130.3 |
| C(4)-H(4C)...O(1)#2 | 0.97 | 2.32 | 3.221(3) | 154.4 |
| O(3)-H(3)...O(1)#3 | 0.84(4) | 2.29(3) | 2.988(2) | 141(3) |
| O(3)-H(3)...O(4) | 0.84(4) | 2.34(3) | 2.789(3) | 114(3) |
| O(4)-H(4)...O(3)#2 | 0.79(5) | 2.15(5) | 2.939(3) | 175(5) |

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