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# Diosgenone: a second $P2_1$ polymorph

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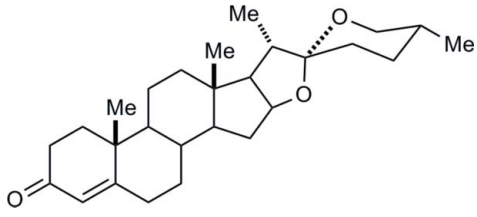
Key indicators: single-crystal X-ray study; $T$ = 136 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.052; $wR$ factor = 0.099; data-to-parameter ratio = 9.7.

Diosgenone [(20*S*,22*R*,25*R*)-spirost-4-en-3-one, $C_{27}H_{40}O_3$] has been proposed as a new therapeutic alternative for the treatment of malaria. The first X-ray structure report for diosgenone was by Piro *et al.* [(2002). *Z. Naturforsch. Teil C*, **57**, 947–950] in the space group $P2_1$ ($Z'$ = 2). We now report a new polymorph in the same space group, with two molecules in the asymmetric unit. Both molecules have similar conformations, characterized by a skewed envelope *A* ring, which contains the C=C bond conjugated with the ketone functionality at C3. The dimorphism results from a modification of the relative orientation of the molecules in the asymmetric unit: two independent molecules were arranged antiparallel in the Piro report, while they are parallel in the present determination.

## Related literature

For the potential application of diosgenone as an antimalarial drug, see: Saez *et al.* (1998); Echeverri *et al.* (2001). For a biotransformation of diosgenone, see: Wang *et al.* (2007, 2009). For the synthesis of diosgenone, see: Hunter & Priest (2006). For the structure of a monoclinic polymorph of diosgenone, see: Piro *et al.* (2002).




## Experimental

### Crystal data

$C_{27}H_{40}O_3$
$M_r$ = 412.59
Monoclinic, $P2_1$
$a$ = 10.3396 (6) Å
$b$ = 7.6466 (4) Å
$c$ = 29.9511 (17) Å
$\beta$ = 97.207 (5)°
$V$ = 2349.3 (2) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.07 mm$^{-1}$
$T$ = 136 K
0.44 × 0.19 × 0.05 mm

### Data collection

Oxford Diffraction Xcalibur (Atlas, Gemini) diffractometer
Absorption correction: analytical [*CrysAlis PRO* (Oxford Diffraction, 2009); based on expressions derived by Clark & Reid (1995)]
$T_{min}$ = 0.984, $T_{max}$ = 0.996
11305 measured reflections
5346 independent reflections
3908 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.042

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.099
$S$ = 1.03
5346 reflections
549 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2086).

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# supporting information

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### S1. Comment

A natural source of diosgenone is *Solanun nudum* (Saez *et al.*, 1998), present in different regions of South America. This steroid is also synthesized by oxidation of diosgenin, for instance, through the Swern oxidation. We obtained it using a modified Jones oxidation described in the literature (Hunter & Priest, 2006). The interest in diosgenone is currently growing, since it has been proposed by a group working in Colombia as a new therapeutic alternative for the treatment of malaria (Echeverri *et al.*, 2001; Saez *et al.*, 1998). This claim is in line with the fact that *Solanun nudum* is used by the community of Tumaco (Narino, Colombia) as a cure for malaria. A more academic interest is related to the biotransformation of diosgenone to isonuatigenone (C25-hydroxylation), which rearranges in acid media into nuatigenone, a rare nuatigenin-type steroid (Wang *et al.*, 2007, 2009). These studies allow a postulate for a new pathway of diosgenin metabolism.

The X-ray structure for diosgenone was described in space group $P2_1$ (Piro *et al.*, 2002; CSD refcode: LUKXAQ). We have now discovered that a second polymorph in the same space group may be obtained if the crystallization is carried out by slow evaporation of an AcOEt/acetone (4:1) solution, while Piro *et al.* crystallized diosgenone from an ethanolic solution. The asymmetric unit, as in the previous report, contains two independent molecules (Fig. 1), with very similar conformations. The r.m.s. deviation for the fitted molecules is less than 0.2 Å. No significant conformational modification is observed by comparing molecules in both polymorphs: calculated r.m.s. deviations for pairs of molecules taken in different crystal forms are in the range 0.17 to 0.32 Å, the largest deviations being for the methyl group bonded to C25 in the *F* ring. However, simulated X-ray powder patterns for each form (Fig. 2) show clearly that these crystal forms are dimorphic. The crystal modification should thus be due to a reorientation of independent molecules in the asymmetric unit. In the case of the previously described structure (Piro *et al.*, 2002), the asymmetric unit may be described with two molecules arranged in such a way that the *A*→*F* rings sequence of one molecule is oriented antiparallel to the *A*→*F* sequence of the other one. In contrast, the asymmetric unit of the title polymorph described herein includes two parallel molecules (see insets in Fig. 2).

### S2. Experimental

The title steroid was synthesized from diosgenin using a Jones oxidation described previously (Hunter & Priest, 2006). Diosgenine (2 g, 4.8 mmol) was dissolved in a $CH_2Cl_2$/acetone mixture (40 and 132 ml) and this solution was cooled to 263 K. Under stirring, the Jones reagent was added slowly, over 10 min., maintaining the temperature below 283 K. After addition, the mixture was further stirred at room temperature, until the color turned from orange to green. 2-Propanol was then added in order to eliminate the unreacted Jones reagent, and the product was extracted with AcOEt, washed with water, and dried over $Na_2SO_4$. The crude product was purified by chromatography on silica gel (AcOEt/hexane, 1:9 *v*/*v*), affording the title steroid (yield: 20%) and the $\Delta^4$-3,6 dione derivative. $^{13}$C-NMR for diosgenone: $\delta$ = 36.6 (C-1), 33.9

(C-2), 199.5 (C-3), 123.8 (C-4), 171.1 (C-5), 32.8 (C-6), 32.1 (C-7), 35.1 (C-8), 53.7 (C-9), 38.6 (C-10), 20.8 (C-11), 39.6 (C-12), 40.3 (C-13), 55.6 (C-14), 31.6 (C-15), 80.6 (C-16), 61.9 (C-17), 17.3 (C-18), 16.3 (C-19), 41.6 (C-20), 14.5 (C-21), 109.2 (C-22), 31.3 (C-23), 28.7 (C-24), 30.2 (C-25), 66.8 (C-26), 17.1 (C-27). Suitable single crystals were obtained by slow evaporation of an AcOEt/acetone (8:2) solution.

## S3. Refinement

All H atoms were placed in idealized positions and refined as riding on their carrier atoms. Isotropic displacement parameters were calculated as $U_{iso}$(H) = $xU_{eq}$(carrier atom) where $x$ = 1.5 for methyl H atoms and $x$ = 1.2 otherwise. Absolute configuration was assigned from chiral centers with known configuration in the steroidal nucleus, and measured Friedel pairs (3382) were merged.

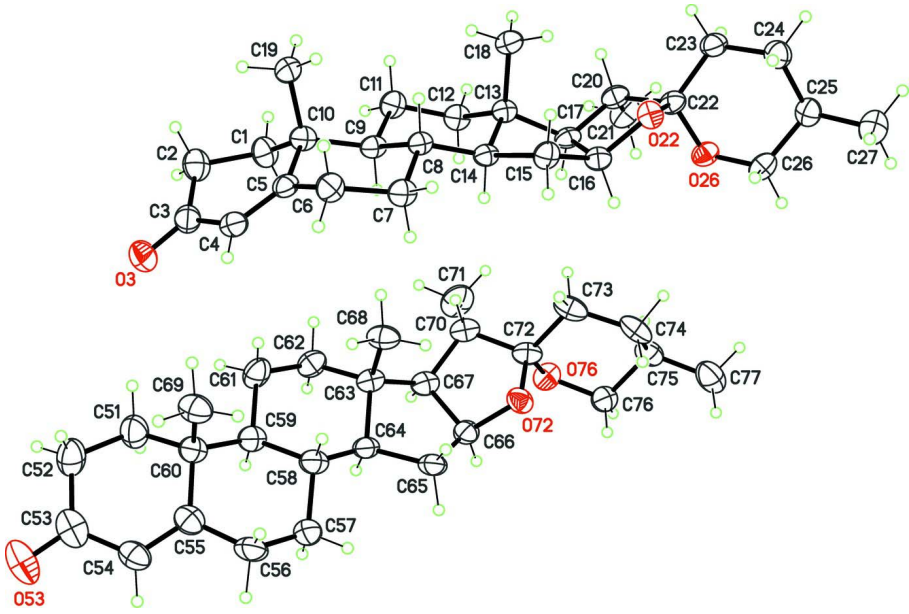


**Figure 1**

The asymmetric unit of the title compound. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

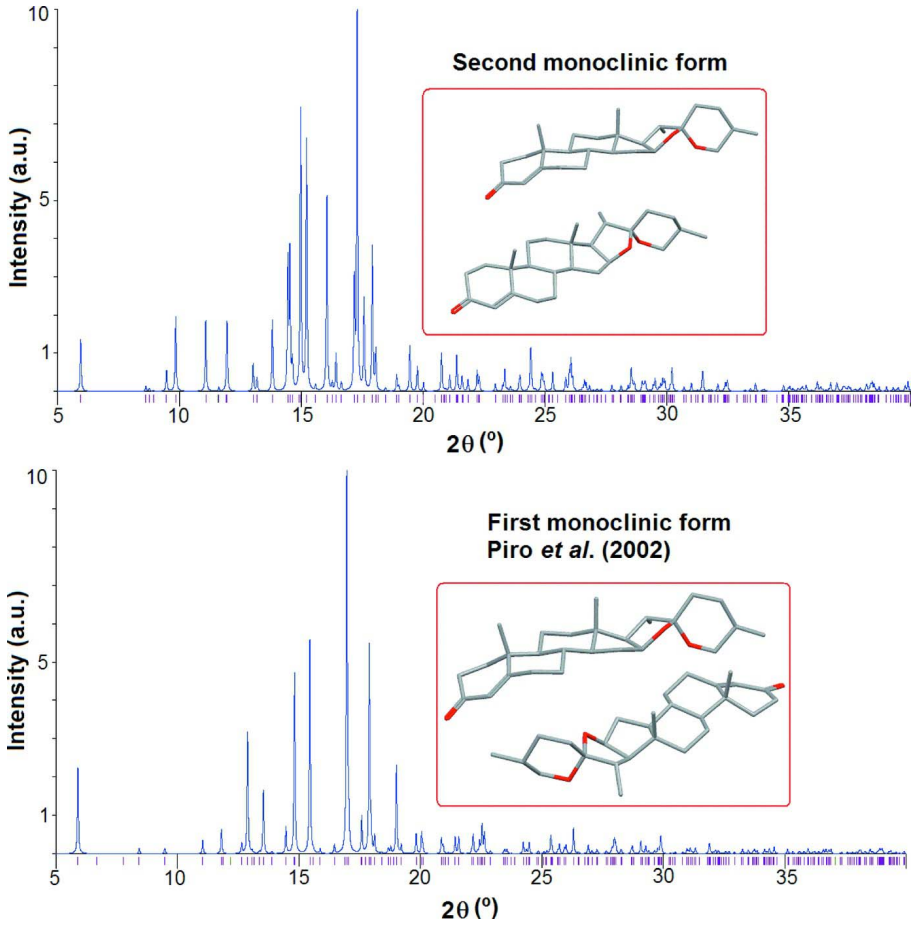


**Figure 2**

Simulated powder diffraction patterns for both $P2_1$ polymorphs of the title compound. Spectra were calculated with *Mercury* (Macrae *et al.*, 2008) with $\lambda$ = 1.5418 Å, by steps of 0.002° and peak shape defined by a FWHM of 0.05° in 2$\theta$. The insets for each polymorph represent the asymmetric unit for the crystal, omitting H atoms for clarity. The asymmetric unit for the previously reported form (Piro *et al.*, 2002) has been regrouped in order to be comparable to the unit used for the refinement of the new polymorph.

**(20*S*,22*R*,25*R*)-Spirost-4-en-3-one**

*Crystal data*

$C_{27}H_{40}O_3$
$M_r$ = 412.59
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a$ = 10.3396 (6) Å
$b$ = 7.6466 (4) Å
$c$ = 29.9511 (17) Å
$\beta$ = 97.207 (5)°
$V$ = 2349.3 (2) Å$^3$
$Z$ = 4

$F(000)$ = 904
$D_x$ = 1.167 Mg m$^{-3}$
Melting point: 432 K
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2248 reflections
$\theta$ = 3.4–26.0°
$\mu$ = 0.07 mm$^{-1}$
$T$ = 136 K
Prism, colourless
0.44 × 0.19 × 0.05 mm

*Data collection*

Oxford Diffraction Xcalibur (Atlas, Gemini) diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 10.4685 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: analytical [*CrysAlis PRO* (Oxford Diffraction, 2009); based on expressions derived by Clark & Reid (1995)]
$T_{min}$ = 0.984, $T_{max}$ = 0.996
11305 measured reflections
5346 independent reflections
3908 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.042
$\theta_{max}$ = 26.7°, $\theta_{min}$ = 3.4°
$h$ = −13→9
$k$ = −9→9
$l$ = −37→34

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.099
$S$ = 1.03
5346 reflections
549 parameters
1 restraint
0 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2 + 0.2215P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.5754 (3) | 0.1116 (4) | 0.69355 (10) | 0.0372 (8) |
| H1A | 0.5868 | 0.0139 | 0.7154 | 0.045* |
| H1B | 0.4900 | 0.1666 | 0.6960 | 0.045* |
| C2 | 0.5743 (3) | 0.0383 (4) | 0.64620 (10) | 0.0406 (8) |
| H2A | 0.6559 | −0.0275 | 0.6443 | 0.049* |
| H2B | 0.5003 | −0.0439 | 0.6397 | 0.049* |
| C3 | 0.5620 (3) | 0.1816 (4) | 0.61200 (11) | 0.0368 (8) |
| O3 | 0.5019 (2) | 0.1613 (3) | 0.57407 (8) | 0.0489 (6) |
| C4 | 0.6307 (3) | 0.3437 (4) | 0.62540 (11) | 0.0339 (7) |
| H4A | 0.6329 | 0.4333 | 0.6035 | 0.041* |
| C5 | 0.6909 (3) | 0.3724 (4) | 0.66703 (10) | 0.0283 (7) |
| C6 | 0.7742 (3) | 0.5326 (4) | 0.67710 (10) | 0.0341 (7) |
| H6A | 0.7613 | 0.6125 | 0.6509 | 0.041* |
| H6B | 0.8671 | 0.4974 | 0.6816 | 0.041* |
| C7 | 0.7426 (3) | 0.6299 (4) | 0.71874 (10) | 0.0319 (7) |
| H7A | 0.6565 | 0.6871 | 0.7119 | 0.038* |
| H7B | 0.8085 | 0.7228 | 0.7263 | 0.038* |
| C8 | 0.7402 (3) | 0.5111 (4) | 0.75955 (9) | 0.0251 (6) |
| H8A | 0.8305 | 0.4669 | 0.7691 | 0.030* |
| C9 | 0.6494 (3) | 0.3532 (4) | 0.74742 (10) | 0.0274 (7) |
| H9A | 0.5608 | 0.4036 | 0.7382 | 0.033* |
| C10 | 0.6836 (3) | 0.2473 (4) | 0.70624 (10) | 0.0291 (7) |
| C11 | 0.6363 (3) | 0.2383 (4) | 0.78883 (10) | 0.0337 (8) |

| | | | | |
|---|---|---|---|---|
| H11A | 0.5728 | 0.1436 | 0.7801 | 0.040* |
| H11B | 0.7215 | 0.1832 | 0.7991 | 0.040* |
| C12 | 0.5917 (3) | 0.3425 (4) | 0.82764 (10) | 0.0309 (7) |
| H12A | 0.5026 | 0.3880 | 0.8185 | 0.037* |
| H12B | 0.5886 | 0.2646 | 0.8539 | 0.037* |
| C13 | 0.6838 (3) | 0.4950 (4) | 0.84101 (9) | 0.0261 (7) |
| C14 | 0.6932 (3) | 0.6075 (4) | 0.79873 (10) | 0.0253 (7) |
| H14A | 0.6020 | 0.6450 | 0.7880 | 0.030* |
| C15 | 0.7635 (3) | 0.7722 (4) | 0.81842 (10) | 0.0302 (7) |
| H15A | 0.8581 | 0.7510 | 0.8264 | 0.036* |
| H15B | 0.7508 | 0.8716 | 0.7971 | 0.036* |
| C16 | 0.6966 (3) | 0.8054 (4) | 0.86036 (10) | 0.0294 (7) |
| H16A | 0.6302 | 0.9004 | 0.8544 | 0.035* |
| C17 | 0.6305 (3) | 0.6331 (4) | 0.87178 (10) | 0.0263 (7) |
| H17A | 0.5342 | 0.6444 | 0.8633 | 0.032* |
| C18 | 0.8184 (3) | 0.4264 (4) | 0.86118 (10) | 0.0326 (7) |
| H18A | 0.8629 | 0.3752 | 0.8373 | 0.049* |
| H18B | 0.8075 | 0.3370 | 0.8839 | 0.049* |
| H18C | 0.8705 | 0.5232 | 0.8753 | 0.049* |
| C19 | 0.8168 (3) | 0.1547 (4) | 0.71607 (10) | 0.0339 (7) |
| H19A | 0.8844 | 0.2412 | 0.7258 | 0.051* |
| H19B | 0.8376 | 0.0964 | 0.6887 | 0.051* |
| H19C | 0.8130 | 0.0676 | 0.7399 | 0.051* |
| C20 | 0.6594 (3) | 0.6163 (4) | 0.92306 (10) | 0.0313 (7) |
| H20A | 0.7254 | 0.5214 | 0.9299 | 0.038* |
| C21 | 0.5398 (3) | 0.5704 (4) | 0.94613 (11) | 0.0421 (9) |
| H21A | 0.5074 | 0.4546 | 0.9361 | 0.063* |
| H21B | 0.4714 | 0.6577 | 0.9382 | 0.063* |
| H21C | 0.5640 | 0.5695 | 0.9788 | 0.063* |
| C22 | 0.7243 (3) | 0.7909 (4) | 0.93750 (10) | 0.0290 (7) |
| O22 | 0.78615 (18) | 0.8456 (3) | 0.90003 (6) | 0.0311 (5) |
| C23 | 0.8256 (3) | 0.7808 (4) | 0.97853 (10) | 0.0339 (8) |
| H23A | 0.9006 | 0.7109 | 0.9711 | 0.041* |
| H23B | 0.7877 | 0.7195 | 1.0029 | 0.041* |
| C24 | 0.8740 (3) | 0.9597 (4) | 0.99535 (11) | 0.0367 (8) |
| H24A | 0.9255 | 1.0138 | 0.9733 | 0.044* |
| H24B | 0.9313 | 0.9470 | 1.0242 | 0.044* |
| C25 | 0.7585 (3) | 1.0770 (4) | 1.00196 (10) | 0.0340 (8) |
| H25A | 0.7112 | 1.0230 | 1.0257 | 0.041* |
| C26 | 0.6669 (3) | 1.0798 (4) | 0.95837 (11) | 0.0359 (8) |
| H26A | 0.5909 | 1.1546 | 0.9622 | 0.043* |
| H26B | 0.7124 | 1.1321 | 0.9344 | 0.043* |
| O26 | 0.62185 (19) | 0.9090 (3) | 0.94460 (7) | 0.0328 (5) |
| C27 | 0.7991 (4) | 1.2607 (5) | 1.01715 (12) | 0.0480 (9) |
| H27A | 0.7217 | 1.3284 | 1.0221 | 0.072* |
| H27B | 0.8430 | 1.3176 | 0.9939 | 0.072* |
| H27C | 0.8588 | 1.2545 | 1.0452 | 0.072* |
| C51 | 0.2234 (4) | 0.4065 (5) | 0.50640 (11) | 0.0479 (9) |

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|---|---|---|---|---|
| H51A | 0.2770 | 0.3160 | 0.5236 | 0.058* |
| H51B | 0.1309 | 0.3823 | 0.5094 | 0.058* |
| C52 | 0.2432 (4) | 0.3930 (5) | 0.45691 (12) | 0.0513 (10) |
| H52A | 0.3374 | 0.4016 | 0.4542 | 0.062* |
| H52B | 0.2122 | 0.2773 | 0.4452 | 0.062* |
| C53 | 0.1716 (3) | 0.5336 (5) | 0.42910 (12) | 0.0475 (9) |
| O53 | 0.1312 (2) | 0.5121 (4) | 0.38911 (8) | 0.0611 (8) |
| C54 | 0.1587 (3) | 0.7000 (5) | 0.45163 (11) | 0.0421 (9) |
| H54A | 0.1151 | 0.7927 | 0.4348 | 0.051* |
| C55 | 0.2052 (3) | 0.7301 (5) | 0.49495 (11) | 0.0386 (8) |
| C56 | 0.2100 (4) | 0.9128 (5) | 0.51395 (11) | 0.0471 (9) |
| H56A | 0.1587 | 0.9914 | 0.4922 | 0.056* |
| H56B | 0.3015 | 0.9542 | 0.5177 | 0.056* |
| C57 | 0.1576 (4) | 0.9241 (5) | 0.55872 (11) | 0.0462 (9) |
| H57A | 0.0622 | 0.9047 | 0.5540 | 0.055* |
| H57B | 0.1734 | 1.0432 | 0.5712 | 0.055* |
| C58 | 0.2201 (3) | 0.7912 (4) | 0.59259 (11) | 0.0351 (8) |
| H58A | 0.3158 | 0.8158 | 0.5984 | 0.042* |
| C59 | 0.2006 (3) | 0.6060 (4) | 0.57195 (11) | 0.0345 (8) |
| H59A | 0.1044 | 0.5920 | 0.5639 | 0.041* |
| C60 | 0.2604 (3) | 0.5871 (4) | 0.52681 (11) | 0.0354 (8) |
| C61 | 0.2428 (4) | 0.4588 (4) | 0.60576 (11) | 0.0426 (9) |
| H61A | 0.2140 | 0.3452 | 0.5921 | 0.051* |
| H61B | 0.3391 | 0.4569 | 0.6117 | 0.051* |
| C62 | 0.1873 (3) | 0.4792 (4) | 0.65045 (11) | 0.0402 (8) |
| H62A | 0.0915 | 0.4641 | 0.6454 | 0.048* |
| H62B | 0.2238 | 0.3870 | 0.6715 | 0.048* |
| C63 | 0.2195 (3) | 0.6582 (4) | 0.67138 (11) | 0.0307 (7) |
| C64 | 0.1640 (3) | 0.7983 (4) | 0.63715 (10) | 0.0318 (7) |
| H64A | 0.0688 | 0.7727 | 0.6300 | 0.038* |
| C65 | 0.1743 (3) | 0.9677 (4) | 0.66473 (10) | 0.0383 (8) |
| H65A | 0.2647 | 1.0133 | 0.6687 | 0.046* |
| H65B | 0.1149 | 1.0588 | 0.6504 | 0.046* |
| C66 | 0.1328 (3) | 0.9077 (4) | 0.70958 (11) | 0.0349 (8) |
| H66A | 0.0402 | 0.9414 | 0.7114 | 0.042* |
| C67 | 0.1481 (3) | 0.7065 (4) | 0.71224 (11) | 0.0333 (8) |
| H67A | 0.0595 | 0.6518 | 0.7082 | 0.040* |
| C68 | 0.3673 (3) | 0.6767 (5) | 0.68339 (11) | 0.0392 (8) |
| H68A | 0.4007 | 0.5776 | 0.7021 | 0.059* |
| H68B | 0.3868 | 0.7859 | 0.7000 | 0.059* |
| H68C | 0.4089 | 0.6783 | 0.6557 | 0.059* |
| C69 | 0.4108 (3) | 0.6055 (5) | 0.53395 (11) | 0.0443 (9) |
| H69A | 0.4436 | 0.6093 | 0.5047 | 0.066* |
| H69B | 0.4490 | 0.5052 | 0.5512 | 0.066* |
| H69C | 0.4349 | 0.7136 | 0.5505 | 0.066* |
| C70 | 0.2126 (3) | 0.6711 (4) | 0.76027 (11) | 0.0357 (8) |
| H70A | 0.3058 | 0.6409 | 0.7584 | 0.043* |
| C71 | 0.1534 (4) | 0.5204 (5) | 0.78430 (13) | 0.0572 (11) |

| | | | | |
|---|---|---|---|---|
| H71A | 0.1959 | 0.5130 | 0.8154 | 0.086* |
| H71B | 0.1665 | 0.4106 | 0.7686 | 0.086* |
| H71C | 0.0599 | 0.5409 | 0.7843 | 0.086* |
| C72 | 0.2107 (3) | 0.8498 (4) | 0.78344 (11) | 0.0325 (7) |
| O72 | 0.2163 (2) | 0.9736 (3) | 0.74834 (7) | 0.0345 (5) |
| C73 | 0.3239 (3) | 0.8823 (5) | 0.81953 (11) | 0.0421 (9) |
| H73A | 0.4047 | 0.8930 | 0.8052 | 0.051* |
| H73B | 0.3339 | 0.7799 | 0.8399 | 0.051* |
| C74 | 0.3084 (3) | 1.0466 (5) | 0.84749 (11) | 0.0442 (9) |
| H74A | 0.3787 | 1.0510 | 0.8731 | 0.053* |
| H74B | 0.3158 | 1.1518 | 0.8287 | 0.053* |
| C75 | 0.1776 (3) | 1.0458 (5) | 0.86483 (10) | 0.0397 (8) |
| H75A | 0.1745 | 0.9421 | 0.8850 | 0.048* |
| C76 | 0.0728 (3) | 1.0224 (4) | 0.82481 (11) | 0.0367 (8) |
| H76A | −0.0139 | 1.0226 | 0.8355 | 0.044* |
| H76B | 0.0761 | 1.1223 | 0.8040 | 0.044* |
| O76 | 0.08903 (19) | 0.8629 (3) | 0.80108 (7) | 0.0373 (5) |
| C77 | 0.1522 (4) | 1.2083 (5) | 0.89150 (12) | 0.0548 (10) |
| H77A | 0.2242 | 1.2256 | 0.9156 | 0.082* |
| H77B | 0.0706 | 1.1940 | 0.9046 | 0.082* |
| H77C | 0.1453 | 1.3102 | 0.8715 | 0.082* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0408 (19) | 0.0340 (18) | 0.0368 (19) | −0.0103 (16) | 0.0045 (15) | −0.0027 (16) |
| C2 | 0.0393 (19) | 0.0381 (19) | 0.044 (2) | −0.0111 (16) | 0.0023 (16) | −0.0079 (17) |
| C3 | 0.0295 (17) | 0.043 (2) | 0.037 (2) | 0.0026 (15) | 0.0015 (15) | −0.0086 (17) |
| O3 | 0.0464 (14) | 0.0565 (16) | 0.0406 (14) | 0.0030 (13) | −0.0077 (12) | −0.0125 (13) |
| C4 | 0.0335 (17) | 0.0310 (17) | 0.0366 (19) | 0.0048 (15) | 0.0017 (15) | 0.0014 (16) |
| C5 | 0.0225 (15) | 0.0267 (16) | 0.0353 (18) | 0.0033 (13) | 0.0022 (13) | −0.0018 (15) |
| C6 | 0.0373 (18) | 0.0281 (17) | 0.0368 (18) | −0.0049 (15) | 0.0037 (15) | 0.0009 (15) |
| C7 | 0.0304 (16) | 0.0256 (16) | 0.0391 (19) | −0.0055 (14) | 0.0021 (14) | 0.0025 (15) |
| C8 | 0.0226 (15) | 0.0221 (15) | 0.0303 (16) | −0.0017 (13) | 0.0023 (12) | 0.0017 (14) |
| C9 | 0.0236 (15) | 0.0218 (15) | 0.0360 (17) | −0.0023 (12) | 0.0003 (13) | 0.0032 (14) |
| C10 | 0.0275 (16) | 0.0245 (15) | 0.0347 (18) | −0.0040 (13) | 0.0015 (14) | 0.0027 (14) |
| C11 | 0.0376 (18) | 0.0217 (15) | 0.041 (2) | −0.0076 (14) | 0.0032 (15) | 0.0004 (15) |
| C12 | 0.0297 (16) | 0.0270 (16) | 0.0363 (18) | −0.0068 (14) | 0.0054 (14) | 0.0041 (15) |
| C13 | 0.0216 (15) | 0.0259 (15) | 0.0309 (17) | −0.0024 (13) | 0.0034 (13) | 0.0023 (14) |
| C14 | 0.0197 (14) | 0.0225 (15) | 0.0329 (17) | −0.0022 (12) | 0.0004 (13) | 0.0020 (14) |
| C15 | 0.0347 (17) | 0.0219 (16) | 0.0345 (18) | −0.0051 (14) | 0.0065 (15) | 0.0028 (14) |
| C16 | 0.0286 (16) | 0.0286 (16) | 0.0310 (17) | 0.0015 (14) | 0.0038 (13) | 0.0041 (14) |
| C17 | 0.0184 (14) | 0.0240 (15) | 0.0367 (18) | 0.0006 (13) | 0.0039 (13) | 0.0037 (15) |
| C18 | 0.0302 (17) | 0.0318 (17) | 0.0359 (18) | 0.0061 (14) | 0.0053 (14) | 0.0028 (15) |
| C19 | 0.0386 (18) | 0.0293 (17) | 0.0333 (18) | 0.0044 (16) | 0.0028 (14) | 0.0003 (15) |
| C20 | 0.0273 (16) | 0.0302 (17) | 0.0377 (18) | 0.0043 (14) | 0.0092 (14) | 0.0042 (15) |
| C21 | 0.041 (2) | 0.0377 (19) | 0.051 (2) | −0.0022 (16) | 0.0211 (17) | 0.0015 (17) |
| C22 | 0.0275 (16) | 0.0313 (17) | 0.0297 (17) | 0.0058 (14) | 0.0093 (14) | 0.0024 (15) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O22 | 0.0297 (11) | 0.0341 (12) | 0.0302 (11) | −0.0033 (10) | 0.0061 (9) | −0.0011 (10) |
| C23 | 0.0325 (18) | 0.0353 (18) | 0.0340 (18) | 0.0090 (15) | 0.0043 (15) | 0.0007 (16) |
| C24 | 0.0354 (18) | 0.043 (2) | 0.0316 (18) | 0.0026 (16) | 0.0053 (15) | −0.0024 (16) |
| C25 | 0.0391 (18) | 0.0338 (18) | 0.0303 (17) | −0.0016 (15) | 0.0088 (15) | 0.0002 (15) |
| C26 | 0.0363 (18) | 0.0296 (18) | 0.044 (2) | 0.0050 (14) | 0.0132 (16) | −0.0007 (16) |
| O26 | 0.0270 (11) | 0.0319 (12) | 0.0403 (13) | 0.0046 (10) | 0.0073 (10) | 0.0001 (11) |
| C27 | 0.050 (2) | 0.043 (2) | 0.053 (2) | −0.0026 (18) | 0.0111 (18) | −0.0088 (19) |
| C51 | 0.063 (3) | 0.040 (2) | 0.041 (2) | −0.0044 (19) | 0.0073 (19) | −0.0030 (18) |
| C52 | 0.068 (3) | 0.040 (2) | 0.048 (2) | −0.010 (2) | 0.014 (2) | −0.0079 (19) |
| C53 | 0.039 (2) | 0.063 (3) | 0.041 (2) | −0.019 (2) | 0.0092 (17) | −0.003 (2) |
| O53 | 0.0594 (16) | 0.087 (2) | 0.0365 (15) | −0.0273 (16) | 0.0049 (13) | −0.0063 (15) |
| C54 | 0.0335 (19) | 0.055 (2) | 0.037 (2) | 0.0004 (17) | −0.0001 (16) | 0.0055 (18) |
| C55 | 0.0303 (18) | 0.047 (2) | 0.038 (2) | −0.0033 (16) | 0.0013 (15) | 0.0010 (17) |
| C56 | 0.061 (2) | 0.0367 (19) | 0.039 (2) | 0.0054 (18) | −0.0082 (18) | 0.0119 (18) |
| C57 | 0.063 (2) | 0.0325 (19) | 0.040 (2) | 0.0067 (18) | −0.0077 (18) | −0.0005 (17) |
| C58 | 0.0340 (18) | 0.0296 (17) | 0.0393 (19) | −0.0023 (15) | −0.0045 (15) | 0.0047 (16) |
| C59 | 0.0317 (17) | 0.0293 (17) | 0.0409 (19) | −0.0041 (15) | −0.0016 (15) | 0.0016 (16) |
| C60 | 0.0352 (18) | 0.0341 (18) | 0.0361 (19) | −0.0007 (15) | 0.0009 (15) | 0.0024 (16) |
| C61 | 0.056 (2) | 0.0251 (18) | 0.049 (2) | −0.0011 (16) | 0.0124 (18) | −0.0003 (16) |
| C62 | 0.045 (2) | 0.0276 (17) | 0.048 (2) | −0.0004 (16) | 0.0076 (17) | 0.0036 (17) |
| C63 | 0.0244 (16) | 0.0280 (16) | 0.0401 (19) | −0.0008 (14) | 0.0046 (14) | 0.0058 (15) |
| C64 | 0.0298 (16) | 0.0240 (16) | 0.0386 (19) | 0.0010 (14) | −0.0067 (14) | 0.0022 (15) |
| C65 | 0.044 (2) | 0.0308 (18) | 0.0369 (19) | 0.0047 (16) | −0.0083 (16) | 0.0069 (16) |
| C66 | 0.0290 (17) | 0.0326 (17) | 0.0405 (19) | 0.0017 (14) | −0.0060 (15) | −0.0038 (16) |
| C67 | 0.0227 (16) | 0.0305 (18) | 0.046 (2) | −0.0039 (14) | 0.0008 (14) | 0.0036 (16) |
| C68 | 0.0287 (17) | 0.048 (2) | 0.041 (2) | 0.0065 (16) | 0.0050 (15) | 0.0124 (17) |
| C69 | 0.0361 (18) | 0.053 (2) | 0.043 (2) | 0.0024 (17) | 0.0017 (16) | 0.0069 (18) |
| C70 | 0.0302 (17) | 0.0338 (18) | 0.043 (2) | 0.0029 (15) | 0.0058 (15) | 0.0086 (17) |
| C71 | 0.079 (3) | 0.036 (2) | 0.060 (3) | −0.001 (2) | 0.021 (2) | 0.012 (2) |
| C72 | 0.0230 (16) | 0.0364 (18) | 0.0382 (19) | −0.0005 (14) | 0.0039 (14) | 0.0108 (16) |
| O72 | 0.0356 (12) | 0.0330 (12) | 0.0330 (12) | −0.0068 (10) | −0.0031 (10) | 0.0020 (10) |
| C73 | 0.0271 (17) | 0.060 (2) | 0.038 (2) | −0.0022 (17) | −0.0009 (15) | 0.0130 (19) |
| C74 | 0.0326 (18) | 0.064 (2) | 0.0323 (18) | −0.0096 (18) | −0.0087 (15) | 0.0074 (19) |
| C75 | 0.045 (2) | 0.043 (2) | 0.0304 (18) | −0.0048 (17) | 0.0014 (15) | 0.0095 (17) |
| C76 | 0.0329 (18) | 0.0352 (19) | 0.0417 (19) | −0.0019 (16) | 0.0030 (15) | 0.0005 (17) |
| O76 | 0.0275 (11) | 0.0351 (13) | 0.0499 (14) | −0.0038 (10) | 0.0080 (10) | −0.0017 (11) |
| C77 | 0.064 (3) | 0.063 (3) | 0.035 (2) | −0.007 (2) | −0.0005 (18) | 0.0020 (19) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.524 (4) | C51—C52 | 1.525 (5) |
| C1—C10 | 1.538 (4) | C51—C60 | 1.539 (5) |
| C1—H1A | 0.9900 | C51—H51A | 0.9900 |
| C1—H1B | 0.9900 | C51—H51B | 0.9900 |
| C2—C3 | 1.495 (5) | C52—C53 | 1.497 (5) |
| C2—H2A | 0.9900 | C52—H52A | 0.9900 |
| C2—H2B | 0.9900 | C52—H52B | 0.9900 |
| C3—O3 | 1.234 (4) | C53—O53 | 1.229 (4) |

| | | | |
|---|---|---|---|
| C3—C4 | 1.460 (5) | C53—C54 | 1.455 (5) |
| C4—C5 | 1.340 (4) | C54—C55 | 1.346 (4) |
| C4—H4A | 0.9500 | C54—H54A | 0.9500 |
| C5—C6 | 1.506 (4) | C55—C56 | 1.507 (5) |
| C5—C10 | 1.524 (4) | C55—C60 | 1.515 (4) |
| C6—C7 | 1.522 (4) | C56—C57 | 1.511 (5) |
| C6—H6A | 0.9900 | C56—H56A | 0.9900 |
| C6—H6B | 0.9900 | C56—H56B | 0.9900 |
| C7—C8 | 1.526 (4) | C57—C58 | 1.522 (4) |
| C7—H7A | 0.9900 | C57—H57A | 0.9900 |
| C7—H7B | 0.9900 | C57—H57B | 0.9900 |
| C8—C14 | 1.517 (4) | C58—C64 | 1.521 (4) |
| C8—C9 | 1.544 (4) | C58—C59 | 1.548 (4) |
| C8—H8A | 1.0000 | C58—H58A | 1.0000 |
| C9—C11 | 1.540 (4) | C59—C61 | 1.540 (4) |
| C9—C10 | 1.553 (4) | C59—C60 | 1.562 (4) |
| C9—H9A | 1.0000 | C59—H59A | 1.0000 |
| C10—C19 | 1.544 (4) | C60—C69 | 1.549 (4) |
| C11—C12 | 1.528 (4) | C61—C62 | 1.529 (4) |
| C11—H11A | 0.9900 | C61—H61A | 0.9900 |
| C11—H11B | 0.9900 | C61—H61B | 0.9900 |
| C12—C13 | 1.527 (4) | C62—C63 | 1.525 (4) |
| C12—H12A | 0.9900 | C62—H62A | 0.9900 |
| C12—H12B | 0.9900 | C62—H62B | 0.9900 |
| C13—C18 | 1.538 (4) | C63—C68 | 1.531 (4) |
| C13—C14 | 1.544 (4) | C63—C64 | 1.543 (4) |
| C13—C17 | 1.548 (4) | C63—C67 | 1.552 (4) |
| C14—C15 | 1.535 (4) | C64—C65 | 1.533 (4) |
| C14—H14A | 1.0000 | C64—H64A | 1.0000 |
| C15—C16 | 1.529 (4) | C65—C66 | 1.531 (4) |
| C15—H15A | 0.9900 | C65—H65A | 0.9900 |
| C15—H15B | 0.9900 | C65—H65B | 0.9900 |
| C16—O22 | 1.445 (3) | C66—O72 | 1.448 (4) |
| C16—C17 | 1.543 (4) | C66—C67 | 1.547 (4) |
| C16—H16A | 1.0000 | C66—H66A | 1.0000 |
| C17—C20 | 1.533 (4) | C67—C70 | 1.532 (4) |
| C17—H17A | 1.0000 | C67—H67A | 1.0000 |
| C18—H18A | 0.9800 | C68—H68A | 0.9800 |
| C18—H18B | 0.9800 | C68—H68B | 0.9800 |
| C18—H18C | 0.9800 | C68—H68C | 0.9800 |
| C19—H19A | 0.9800 | C69—H69A | 0.9800 |
| C19—H19B | 0.9800 | C69—H69B | 0.9800 |
| C19—H19C | 0.9800 | C69—H69C | 0.9800 |
| C20—C21 | 1.531 (4) | C70—C71 | 1.526 (5) |
| C20—C22 | 1.532 (4) | C70—C72 | 1.534 (5) |
| C20—H20A | 1.0000 | C70—H70A | 1.0000 |
| C21—H21A | 0.9800 | C71—H71A | 0.9800 |
| C21—H21B | 0.9800 | C71—H71B | 0.9800 |

| | | | |
|---|---|---|---|
| C21—H21C | 0.9800 | C71—H71C | 0.9800 |
| C22—O22 | 1.423 (3) | C72—O72 | 1.421 (4) |
| C22—O26 | 1.428 (3) | C72—O76 | 1.428 (3) |
| C22—C23 | 1.513 (4) | C72—C73 | 1.510 (4) |
| C23—C24 | 1.521 (5) | C73—C74 | 1.529 (5) |
| C23—H23A | 0.9900 | C73—H73A | 0.9900 |
| C23—H23B | 0.9900 | C73—H73B | 0.9900 |
| C24—C25 | 1.526 (4) | C74—C75 | 1.508 (4) |
| C24—H24A | 0.9900 | C74—H74A | 0.9900 |
| C24—H24B | 0.9900 | C74—H74B | 0.9900 |
| C25—C26 | 1.514 (4) | C75—C77 | 1.518 (5) |
| C25—C27 | 1.520 (5) | C75—C76 | 1.522 (4) |
| C25—H25A | 1.0000 | C75—H75A | 1.0000 |
| C26—O26 | 1.429 (4) | C76—O76 | 1.432 (4) |
| C26—H26A | 0.9900 | C76—H76A | 0.9900 |
| C26—H26B | 0.9900 | C76—H76B | 0.9900 |
| C27—H27A | 0.9800 | C77—H77A | 0.9800 |
| C27—H27B | 0.9800 | C77—H77B | 0.9800 |
| C27—H27C | 0.9800 | C77—H77C | 0.9800 |
| | | | |
| C2—C1—C10 | 113.5 (2) | C52—C51—C60 | 112.9 (3) |
| C2—C1—H1A | 108.9 | C52—C51—H51A | 109.0 |
| C10—C1—H1A | 108.9 | C60—C51—H51A | 109.0 |
| C2—C1—H1B | 108.9 | C52—C51—H51B | 109.0 |
| C10—C1—H1B | 108.9 | C60—C51—H51B | 109.0 |
| H1A—C1—H1B | 107.7 | H51A—C51—H51B | 107.8 |
| C3—C2—C1 | 111.0 (3) | C53—C52—C51 | 112.0 (3) |
| C3—C2—H2A | 109.4 | C53—C52—H52A | 109.2 |
| C1—C2—H2A | 109.4 | C51—C52—H52A | 109.2 |
| C3—C2—H2B | 109.4 | C53—C52—H52B | 109.2 |
| C1—C2—H2B | 109.4 | C51—C52—H52B | 109.2 |
| H2A—C2—H2B | 108.0 | H52A—C52—H52B | 107.9 |
| O3—C3—C4 | 122.1 (3) | O53—C53—C54 | 122.0 (4) |
| O3—C3—C2 | 121.9 (3) | O53—C53—C52 | 122.0 (4) |
| C4—C3—C2 | 116.0 (3) | C54—C53—C52 | 116.0 (3) |
| C5—C4—C3 | 123.2 (3) | C55—C54—C53 | 123.7 (3) |
| C5—C4—H4A | 118.4 | C55—C54—H54A | 118.2 |
| C3—C4—H4A | 118.4 | C53—C54—H54A | 118.2 |
| C4—C5—C6 | 120.4 (3) | C54—C55—C56 | 120.9 (3) |
| C4—C5—C10 | 123.5 (3) | C54—C55—C60 | 123.0 (3) |
| C6—C5—C10 | 116.0 (3) | C56—C55—C60 | 116.0 (3) |
| C5—C6—C7 | 112.7 (2) | C55—C56—C57 | 112.9 (3) |
| C5—C6—H6A | 109.1 | C55—C56—H56A | 109.0 |
| C7—C6—H6A | 109.1 | C57—C56—H56A | 109.0 |
| C5—C6—H6B | 109.1 | C55—C56—H56B | 109.0 |
| C7—C6—H6B | 109.1 | C57—C56—H56B | 109.0 |
| H6A—C6—H6B | 107.8 | H56A—C56—H56B | 107.8 |
| C6—C7—C8 | 113.1 (2) | C56—C57—C58 | 112.6 (3) |

| | | | |
|---|---|---|---|
| C6—C7—H7A | 109.0 | C56—C57—H57A | 109.1 |
| C8—C7—H7A | 109.0 | C58—C57—H57A | 109.1 |
| C6—C7—H7B | 109.0 | C56—C57—H57B | 109.1 |
| C8—C7—H7B | 109.0 | C58—C57—H57B | 109.1 |
| H7A—C7—H7B | 107.8 | H57A—C57—H57B | 107.8 |
| C14—C8—C7 | 111.7 (2) | C64—C58—C57 | 112.6 (3) |
| C14—C8—C9 | 108.5 (2) | C64—C58—C59 | 109.8 (3) |
| C7—C8—C9 | 110.3 (2) | C57—C58—C59 | 108.8 (3) |
| C14—C8—H8A | 108.7 | C64—C58—H58A | 108.6 |
| C7—C8—H8A | 108.7 | C57—C58—H58A | 108.6 |
| C9—C8—H8A | 108.7 | C59—C58—H58A | 108.6 |
| C11—C9—C8 | 111.7 (2) | C61—C59—C58 | 113.2 (3) |
| C11—C9—C10 | 113.2 (2) | C61—C59—C60 | 113.0 (3) |
| C8—C9—C10 | 113.7 (2) | C58—C59—C60 | 112.6 (3) |
| C11—C9—H9A | 105.8 | C61—C59—H59A | 105.7 |
| C8—C9—H9A | 105.8 | C58—C59—H59A | 105.7 |
| C10—C9—H9A | 105.8 | C60—C59—H59A | 105.7 |
| C5—C10—C1 | 109.8 (2) | C55—C60—C51 | 110.0 (3) |
| C5—C10—C19 | 107.8 (2) | C55—C60—C69 | 108.3 (3) |
| C1—C10—C19 | 110.0 (3) | C51—C60—C69 | 109.3 (3) |
| C5—C10—C9 | 108.8 (2) | C55—C60—C59 | 108.6 (3) |
| C1—C10—C9 | 108.5 (2) | C51—C60—C59 | 108.8 (3) |
| C19—C10—C9 | 112.0 (2) | C69—C60—C59 | 111.8 (3) |
| C12—C11—C9 | 112.4 (2) | C62—C61—C59 | 113.2 (3) |
| C12—C11—H11A | 109.1 | C62—C61—H61A | 108.9 |
| C9—C11—H11A | 109.1 | C59—C61—H61A | 108.9 |
| C12—C11—H11B | 109.1 | C62—C61—H61B | 108.9 |
| C9—C11—H11B | 109.1 | C59—C61—H61B | 108.9 |
| H11A—C11—H11B | 107.9 | H61A—C61—H61B | 107.7 |
| C13—C12—C11 | 111.1 (2) | C63—C62—C61 | 111.4 (3) |
| C13—C12—H12A | 109.4 | C63—C62—H62A | 109.3 |
| C11—C12—H12A | 109.4 | C61—C62—H62A | 109.3 |
| C13—C12—H12B | 109.4 | C63—C62—H62B | 109.3 |
| C11—C12—H12B | 109.4 | C61—C62—H62B | 109.3 |
| H12A—C12—H12B | 108.0 | H62A—C62—H62B | 108.0 |
| C12—C13—C18 | 110.3 (2) | C62—C63—C68 | 109.8 (3) |
| C12—C13—C14 | 108.2 (2) | C62—C63—C64 | 107.8 (3) |
| C18—C13—C14 | 111.4 (2) | C68—C63—C64 | 111.7 (3) |
| C12—C13—C17 | 114.8 (2) | C62—C63—C67 | 115.9 (3) |
| C18—C13—C17 | 111.8 (2) | C68—C63—C67 | 110.6 (3) |
| C14—C13—C17 | 99.9 (2) | C64—C63—C67 | 100.6 (2) |
| C8—C14—C15 | 120.9 (2) | C58—C64—C65 | 119.7 (3) |
| C8—C14—C13 | 115.0 (2) | C58—C64—C63 | 114.0 (2) |
| C15—C14—C13 | 102.6 (2) | C65—C64—C63 | 103.5 (2) |
| C8—C14—H14A | 105.7 | C58—C64—H64A | 106.2 |
| C15—C14—H14A | 105.7 | C65—C64—H64A | 106.2 |
| C13—C14—H14A | 105.7 | C63—C64—H64A | 106.2 |
| C16—C15—C14 | 102.2 (2) | C66—C65—C64 | 102.2 (3) |

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| C16—C15—H15A | 111.3 | C66—C65—H65A | 111.3 |
| C14—C15—H15A | 111.3 | C64—C65—H65A | 111.3 |
| C16—C15—H15B | 111.3 | C66—C65—H65B | 111.3 |
| C14—C15—H15B | 111.3 | C64—C65—H65B | 111.3 |
| H15A—C15—H15B | 109.2 | H65A—C65—H65B | 109.2 |
| O22—C16—C15 | 113.7 (2) | O72—C66—C65 | 113.3 (3) |
| O22—C16—C17 | 104.8 (2) | O72—C66—C67 | 105.0 (3) |
| C15—C16—C17 | 107.4 (2) | C65—C66—C67 | 107.8 (3) |
| O22—C16—H16A | 110.2 | O72—C66—H66A | 110.2 |
| C15—C16—H16A | 110.2 | C65—C66—H66A | 110.2 |
| C17—C16—H16A | 110.2 | C67—C66—H66A | 110.2 |
| C20—C17—C16 | 104.9 (2) | C70—C67—C66 | 104.7 (3) |
| C20—C17—C13 | 120.0 (2) | C70—C67—C63 | 120.3 (3) |
| C16—C17—C13 | 104.5 (2) | C66—C67—C63 | 104.6 (3) |
| C20—C17—H17A | 109.0 | C70—C67—H67A | 108.9 |
| C16—C17—H17A | 109.0 | C66—C67—H67A | 108.9 |
| C13—C17—H17A | 109.0 | C63—C67—H67A | 108.9 |
| C13—C18—H18A | 109.5 | C63—C68—H68A | 109.5 |
| C13—C18—H18B | 109.5 | C63—C68—H68B | 109.5 |
| H18A—C18—H18B | 109.5 | H68A—C68—H68B | 109.5 |
| C13—C18—H18C | 109.5 | C63—C68—H68C | 109.5 |
| H18A—C18—H18C | 109.5 | H68A—C68—H68C | 109.5 |
| H18B—C18—H18C | 109.5 | H68B—C68—H68C | 109.5 |
| C10—C19—H19A | 109.5 | C60—C69—H69A | 109.5 |
| C10—C19—H19B | 109.5 | C60—C69—H69B | 109.5 |
| H19A—C19—H19B | 109.5 | H69A—C69—H69B | 109.5 |
| C10—C19—H19C | 109.5 | C60—C69—H69C | 109.5 |
| H19A—C19—H19C | 109.5 | H69A—C69—H69C | 109.5 |
| H19B—C19—H19C | 109.5 | H69B—C69—H69C | 109.5 |
| C21—C20—C22 | 115.0 (3) | C71—C70—C67 | 115.0 (3) |
| C21—C20—C17 | 113.9 (3) | C71—C70—C72 | 115.5 (3) |
| C22—C20—C17 | 103.5 (2) | C67—C70—C72 | 103.8 (2) |
| C21—C20—H20A | 108.1 | C71—C70—H70A | 107.3 |
| C22—C20—H20A | 108.1 | C67—C70—H70A | 107.3 |
| C17—C20—H20A | 108.1 | C72—C70—H70A | 107.3 |
| C20—C21—H21A | 109.5 | C70—C71—H71A | 109.5 |
| C20—C21—H21B | 109.5 | C70—C71—H71B | 109.5 |
| H21A—C21—H21B | 109.5 | H71A—C71—H71B | 109.5 |
| C20—C21—H21C | 109.5 | C70—C71—H71C | 109.5 |
| H21A—C21—H21C | 109.5 | H71A—C71—H71C | 109.5 |
| H21B—C21—H21C | 109.5 | H71B—C71—H71C | 109.5 |
| O22—C22—O26 | 110.3 (2) | O72—C72—O76 | 110.2 (2) |
| O22—C22—C23 | 108.8 (2) | O72—C72—C73 | 108.7 (2) |
| O26—C22—C23 | 111.2 (2) | O76—C72—C73 | 111.3 (3) |
| O22—C22—C20 | 105.0 (2) | O72—C72—C70 | 104.8 (2) |
| O26—C22—C20 | 106.7 (2) | O76—C72—C70 | 106.9 (2) |
| C23—C22—C20 | 114.8 (3) | C73—C72—C70 | 114.7 (3) |
| C22—O22—C16 | 106.3 (2) | C72—O72—C66 | 106.6 (2) |

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| C22—C23—C24 | 112.9 (3) | C72—C73—C74 | 113.7 (3) |
| C22—C23—H23A | 109.0 | C72—C73—H73A | 108.8 |
| C24—C23—H23A | 109.0 | C74—C73—H73A | 108.8 |
| C22—C23—H23B | 109.0 | C72—C73—H73B | 108.8 |
| C24—C23—H23B | 109.0 | C74—C73—H73B | 108.8 |
| H23A—C23—H23B | 107.8 | H73A—C73—H73B | 107.7 |
| C23—C24—C25 | 109.9 (3) | C75—C74—C73 | 110.2 (3) |
| C23—C24—H24A | 109.7 | C75—C74—H74A | 109.6 |
| C25—C24—H24A | 109.7 | C73—C74—H74A | 109.6 |
| C23—C24—H24B | 109.7 | C75—C74—H74B | 109.6 |
| C25—C24—H24B | 109.7 | C73—C74—H74B | 109.6 |
| H24A—C24—H24B | 108.2 | H74A—C74—H74B | 108.1 |
| C26—C25—C27 | 111.4 (3) | C74—C75—C77 | 113.6 (3) |
| C26—C25—C24 | 108.2 (2) | C74—C75—C76 | 108.0 (3) |
| C27—C25—C24 | 113.0 (3) | C77—C75—C76 | 111.0 (3) |
| C26—C25—H25A | 108.1 | C74—C75—H75A | 108.0 |
| C27—C25—H25A | 108.1 | C77—C75—H75A | 108.0 |
| C24—C25—H25A | 108.1 | C76—C75—H75A | 108.0 |
| O26—C26—C25 | 112.4 (3) | O76—C76—C75 | 112.0 (3) |
| O26—C26—H26A | 109.1 | O76—C76—H76A | 109.2 |
| C25—C26—H26A | 109.1 | C75—C76—H76A | 109.2 |
| O26—C26—H26B | 109.1 | O76—C76—H76B | 109.2 |
| C25—C26—H26B | 109.1 | C75—C76—H76B | 109.2 |
| H26A—C26—H26B | 107.8 | H76A—C76—H76B | 107.9 |
| C22—O26—C26 | 113.6 (2) | C72—O76—C76 | 114.0 (2) |
| C25—C27—H27A | 109.5 | C75—C77—H77A | 109.5 |
| C25—C27—H27B | 109.5 | C75—C77—H77B | 109.5 |
| H27A—C27—H27B | 109.5 | H77A—C77—H77B | 109.5 |
| C25—C27—H27C | 109.5 | C75—C77—H77C | 109.5 |
| H27A—C27—H27C | 109.5 | H77A—C77—H77C | 109.5 |
| H27B—C27—H27C | 109.5 | H77B—C77—H77C | 109.5 |

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| C10—C1—C2—C3 | −56.6 (4) | C60—C51—C52—C53 | −55.0 (4) |
| C1—C2—C3—O3 | −146.1 (3) | C51—C52—C53—O53 | −150.9 (3) |
| C1—C2—C3—C4 | 36.8 (4) | C51—C52—C53—C54 | 32.2 (4) |
| O3—C3—C4—C5 | 176.5 (3) | O53—C53—C54—C55 | −177.9 (3) |
| C2—C3—C4—C5 | −6.5 (5) | C52—C53—C54—C55 | −1.0 (5) |
| C3—C4—C5—C6 | 172.2 (3) | C53—C54—C55—C56 | 169.0 (3) |
| C3—C4—C5—C10 | −6.2 (5) | C53—C54—C55—C60 | −8.3 (5) |
| C4—C5—C6—C7 | 131.3 (3) | C54—C55—C56—C57 | 133.2 (3) |
| C10—C5—C6—C7 | −50.2 (3) | C60—C55—C56—C57 | −49.2 (4) |
| C5—C6—C7—C8 | 50.3 (3) | C55—C56—C57—C58 | 51.8 (4) |
| C6—C7—C8—C14 | −173.3 (2) | C56—C57—C58—C64 | −178.0 (3) |
| C6—C7—C8—C9 | −52.5 (3) | C56—C57—C58—C59 | −56.1 (4) |
| C14—C8—C9—C11 | −52.9 (3) | C64—C58—C59—C61 | −48.2 (3) |
| C7—C8—C9—C11 | −175.5 (2) | C57—C58—C59—C61 | −171.8 (3) |
| C14—C8—C9—C10 | 177.5 (2) | C64—C58—C59—C60 | −178.0 (3) |
| C7—C8—C9—C10 | 54.9 (3) | C57—C58—C59—C60 | 58.4 (3) |

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| C4—C5—C10—C1 | −12.7 (4) | C54—C55—C60—C51 | −14.3 (4) |
| C6—C5—C10—C1 | 168.9 (2) | C56—C55—C60—C51 | 168.3 (3) |
| C4—C5—C10—C19 | 107.1 (3) | C54—C55—C60—C69 | 105.1 (4) |
| C6—C5—C10—C19 | −71.4 (3) | C56—C55—C60—C69 | −72.3 (4) |
| C4—C5—C10—C9 | −131.3 (3) | C54—C55—C60—C59 | −133.2 (3) |
| C6—C5—C10—C9 | 50.3 (3) | C56—C55—C60—C59 | 49.3 (4) |
| C2—C1—C10—C5 | 43.5 (4) | C52—C51—C60—C55 | 45.0 (4) |
| C2—C1—C10—C19 | −74.9 (3) | C52—C51—C60—C69 | −73.8 (4) |
| C2—C1—C10—C9 | 162.3 (3) | C52—C51—C60—C59 | 163.9 (3) |
| C11—C9—C10—C5 | 178.6 (2) | C61—C59—C60—C55 | 175.9 (3) |
| C8—C9—C10—C5 | −52.5 (3) | C58—C59—C60—C55 | −54.2 (3) |
| C11—C9—C10—C1 | 59.2 (3) | C61—C59—C60—C51 | 56.2 (4) |
| C8—C9—C10—C1 | −171.9 (2) | C58—C59—C60—C51 | −174.0 (3) |
| C11—C9—C10—C19 | −62.3 (3) | C61—C59—C60—C69 | −64.6 (3) |
| C8—C9—C10—C19 | 66.5 (3) | C58—C59—C60—C69 | 65.2 (3) |
| C8—C9—C11—C12 | 54.9 (3) | C58—C59—C61—C62 | 49.0 (4) |
| C10—C9—C11—C12 | −175.2 (2) | C60—C59—C61—C62 | 178.5 (3) |
| C9—C11—C12—C13 | −56.5 (3) | C59—C61—C62—C63 | −54.2 (4) |
| C11—C12—C13—C18 | −66.5 (3) | C61—C62—C63—C68 | −64.2 (3) |
| C11—C12—C13—C14 | 55.5 (3) | C61—C62—C63—C64 | 57.7 (3) |
| C11—C12—C13—C17 | 166.1 (2) | C61—C62—C63—C67 | 169.5 (3) |
| C7—C8—C14—C15 | −57.8 (3) | C57—C58—C64—C65 | −60.1 (4) |
| C9—C8—C14—C15 | −179.6 (2) | C59—C58—C64—C65 | 178.6 (3) |
| C7—C8—C14—C13 | 178.2 (2) | C57—C58—C64—C63 | 176.7 (3) |
| C9—C8—C14—C13 | 56.4 (3) | C59—C58—C64—C63 | 55.4 (3) |
| C12—C13—C14—C8 | −58.0 (3) | C62—C63—C64—C58 | −60.4 (3) |
| C18—C13—C14—C8 | 63.4 (3) | C68—C63—C64—C58 | 60.4 (4) |
| C17—C13—C14—C8 | −178.4 (2) | C67—C63—C64—C58 | 177.8 (2) |
| C12—C13—C14—C15 | 168.7 (2) | C62—C63—C64—C65 | 168.0 (3) |
| C18—C13—C14—C15 | −69.9 (3) | C68—C63—C64—C65 | −71.2 (3) |
| C17—C13—C14—C15 | 48.3 (3) | C67—C63—C64—C65 | 46.2 (3) |
| C8—C14—C15—C16 | −171.8 (3) | C58—C64—C65—C66 | −169.1 (3) |
| C13—C14—C15—C16 | −42.0 (3) | C63—C64—C65—C66 | −40.9 (3) |
| C14—C15—C16—O22 | 134.7 (2) | C64—C65—C66—O72 | 135.2 (3) |
| C14—C15—C16—C17 | 19.3 (3) | C64—C65—C66—C67 | 19.6 (3) |
| O22—C16—C17—C20 | 16.4 (3) | O72—C66—C67—C70 | 15.0 (3) |
| C15—C16—C17—C20 | 137.6 (2) | C65—C66—C67—C70 | 136.0 (3) |
| O22—C16—C17—C13 | −110.8 (2) | O72—C66—C67—C63 | −112.4 (3) |
| C15—C16—C17—C13 | 10.5 (3) | C65—C66—C67—C63 | 8.6 (3) |
| C12—C13—C17—C20 | 91.8 (3) | C62—C63—C67—C70 | 94.0 (3) |
| C18—C13—C17—C20 | −34.7 (3) | C68—C63—C67—C70 | −31.9 (4) |
| C14—C13—C17—C20 | −152.7 (2) | C64—C63—C67—C70 | −150.1 (3) |
| C12—C13—C17—C16 | −151.0 (2) | C62—C63—C67—C66 | −148.9 (3) |
| C18—C13—C17—C16 | 82.4 (3) | C68—C63—C67—C66 | 85.2 (3) |
| C14—C13—C17—C16 | −35.6 (3) | C64—C63—C67—C66 | −33.0 (3) |
| C16—C17—C20—C21 | 132.5 (3) | C66—C67—C70—C71 | 135.3 (3) |
| C13—C17—C20—C21 | −110.5 (3) | C63—C67—C70—C71 | −107.7 (3) |
| C16—C17—C20—C22 | 7.0 (3) | C66—C67—C70—C72 | 8.1 (3) |

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| C13—C17—C20—C22 | 123.9 (3) | C63—C67—C70—C72 | 125.1 (3) |
| C21—C20—C22—O22 | −153.4 (3) | C71—C70—C72—O72 | −156.0 (3) |
| C17—C20—C22—O22 | −28.6 (3) | C67—C70—C72—O72 | −29.1 (3) |
| C21—C20—C22—O26 | −36.4 (3) | C71—C70—C72—O76 | −39.0 (4) |
| C17—C20—C22—O26 | 88.4 (3) | C67—C70—C72—O76 | 87.9 (3) |
| C21—C20—C22—C23 | 87.3 (3) | C71—C70—C72—C73 | 84.9 (4) |
| C17—C20—C22—C23 | −147.9 (2) | C67—C70—C72—C73 | −148.2 (3) |
| O26—C22—O22—C16 | −73.8 (3) | O76—C72—O72—C66 | −74.4 (3) |
| C23—C22—O22—C16 | 164.0 (2) | C73—C72—O72—C66 | 163.4 (2) |
| C20—C22—O22—C16 | 40.7 (3) | C70—C72—O72—C66 | 40.4 (3) |
| C15—C16—O22—C22 | −152.6 (2) | C65—C66—O72—C72 | −152.0 (2) |
| C17—C16—O22—C22 | −35.7 (3) | C67—C66—O72—C72 | −34.7 (3) |
| O22—C22—C23—C24 | 70.5 (3) | O72—C72—C73—C74 | 73.0 (3) |
| O26—C22—C23—C24 | −51.1 (3) | O76—C72—C73—C74 | −48.6 (4) |
| C20—C22—C23—C24 | −172.3 (3) | C70—C72—C73—C74 | −170.2 (3) |
| C22—C23—C24—C25 | 52.3 (3) | C72—C73—C74—C75 | 51.0 (4) |
| C23—C24—C25—C26 | −54.1 (3) | C73—C74—C75—C77 | −177.8 (3) |
| C23—C24—C25—C27 | −177.9 (3) | C73—C74—C75—C76 | −54.2 (4) |
| C27—C25—C26—O26 | −176.9 (3) | C74—C75—C76—O76 | 59.4 (4) |
| C24—C25—C26—O26 | 58.3 (3) | C77—C75—C76—O76 | −175.6 (3) |
| O22—C22—O26—C26 | −66.5 (3) | O72—C72—O76—C76 | −67.9 (3) |
| C23—C22—O26—C26 | 54.2 (3) | C73—C72—O76—C76 | 52.8 (3) |
| C20—C22—O26—C26 | −180.0 (2) | C70—C72—O76—C76 | 178.8 (2) |
| C25—C26—O26—C22 | −59.6 (3) | C75—C76—O76—C72 | −59.8 (3) |