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# (*E*)-3-[3,4-Bis(methoxymethoxy)phenyl]-1-(7-hydroxy-5-methoxy-2,2-dimethylchroman-8-yl)prop-2-en-1-one

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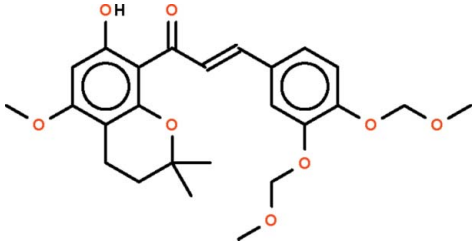
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.032; $wR$ factor = 0.082; data-to-parameter ratio = 8.5.

The reaction of 5,6-(2,2-dimethylchromanyl)-2-hydroxy-4-methoxyacetophenone and 3,4-bis(methoxymethyloxy)-benzaldehyde affords the intense orange title chalcone derivative, $C_{25}H_{30}O_8$. The two benzene rings are connected through a −C(=O)−CH=CH− (propenone) unit, which is in an *E* conformation; the ring with the hydroxy substitutent is aligned at 19.5 (2)° with respect to this unit, whereas the ring with the methoxymethyloxy substituent is aligned at 9.3 (3)°. The dihedral angle between the rings is 19.38 (10)°. The hydroxy group engages in an intramolecular O−H···O hydrogen bond with the carbonyl O atom of the propenone unit, generating an *S*(5) ring.

## Related literature

For background to chalcones, see: Avila *et al.* (2008); Narender *et al.* (2007); Reddy *et al.* (2010).



## Experimental

### Crystal data

$C_{25}H_{30}O_8$
$M_r$ = 458.49
Monoclinic, *Pc*
$a$ = 9.5990 (8) Å
$b$ = 8.3294 (7) Å
$c$ = 14.7444 (12) Å
$\beta$ = 107.684 (1)°
$V$ = 1123.17 (16) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 100 K
0.30 × 0.25 × 0.05 mm

### Data collection

Bruker SMART APEX CCD diffractometer
10316 measured reflections
2576 independent reflections
2433 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.037

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.082
$S$ = 1.06
2576 reflections
302 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O4 | 0.90 (4) | 1.65 (4) | 2.480 (2) | 153 (3) |

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6345).

## References

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# supporting information

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## (*E*)-3-[3,4-Bis(methoxymethoxy)phenyl]-1-(7-hydroxy-5-methoxy-2,2-dimethylchroman-8-yl)prop-2-en-1-one

**Nur Athirah Hashim, Farediah Ahmad, Norazah Basar, Khalijah Awang and Seik Weng Ng**

### S1. Comment

Chalcones or 1,3-diphenyl-2-propen-1-one derivatives flavonoids consist of two aromatic rings that are linked by a three-carbon $\alpha$, $\beta$-unsaturated carbonyl unit (Avila *et al.*, 2008; Reddy *et al.*, 2010), and key precursors for the synthesis of a various flavonoids, some of which are components in food (Narender *et al.*, 2007). We intend to use the intensely-orange title compound, (I), in the synthesis of other compounds. Its two benzene rings are connected through the –C(═O)–CH═CH– unit, which is of an *E* configuration; the ring with the hydroxy substitutent is aligned at 19.5 (2) ° with this unit whereas the ring with the methoxymethyloxy substituents is aligned at 9.3 (3) °. The hydroxy group engages in intramolecular hydrogen bonding with the carbonyl O atom of the unit (Fig.1).

### S2. Experimental

A solution of 2-hydroxy-4-methoxy-5,6-(2,2-dimethylchromane)acetophenone (100 mg, 0.45 mmol) and 3,4-bis(methoxymethyloxy)benzaldehyde (100 mg, 0.45 mmol) in ethanol (10 ml) was treated with 50% potassium hydroxide (1 ml). The mixture was stirred for 48 h. The mixture was poured into iced water (30 ml); this was acidified with 10% hydrochloric acid. The mixture was extracted with dichloromethane (3 *x* 20 ml). The organic layer was washed with water (3 *x* 10 ml) and brine (3 *x* 5 ml) followed by drying over anhydrous magnesium sulfate. The solvent was evaporated under reduced pressure to yield a dark greenish syrup. The syrup was subjected to VLC for purification by using silica gel and eluting with a hexane:ethyl acetate solvent system (9:1) to give the title compound (520 mg, 30%) as orange prisms of (I), m.p. 363–368 K. The formulation was established by $^{1}$H– and $^{13}$C-NMR spectroscopy.

### S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with $U$(H) set to 1.2 to 1.5$U_{eq}$(C).

The hydroxy H-atom was located in a difference Fourier map, and was freely refined.

In the absence of heavy scatters, 2245 Friedel pairs were merged. Omitted from the refinement were (-3 3 - 8), (-2 8 - 1), (1 1 - 4), (-4 9 3) and (-3 0 16).

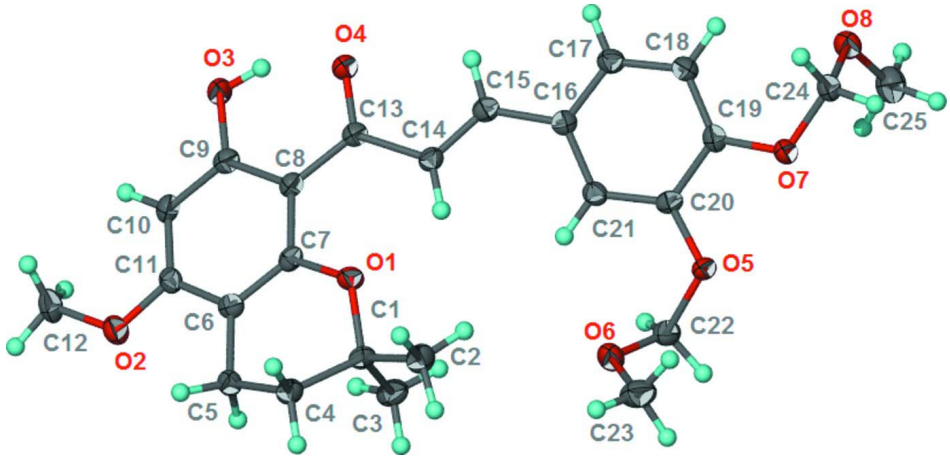


**Figure 1**

The molecular structure of (I) at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

**(*E*)-3-[3,4-Bis(methoxymethoxy)phenyl]-1-(7-hydroxy-5-methoxy- 2,2-dimethylchroman-8-yl)prop-2-en-1-one**

*Crystal data*

$C_{25}H_{30}O_8$
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Monoclinic, *Pc*
Hall symbol: P -2yc
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$c = 14.7444\ (12)$ Å
$\beta = 107.684\ (1)°$
$V = 1123.17\ (16)$ Å$^3$
$Z = 2$
$F(000) = 488$
$D_x = 1.356$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3857 reflections
$\theta = 2.2–28.2°$
$\mu = 0.10$ mm$^{-1}$
$T = 100$ K
Prism, orange
0.30 × 0.25 × 0.05 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
10316 measured reflections
2576 independent reflections
2433 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.2°$
$h = -12\rightarrow12$
$k = -10\rightarrow10$
$l = -19\rightarrow19$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.082$
$S = 1.06$
2576 reflections
302 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 0.1273P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å$^{-3}$
$\Delta\rho_{min} = -0.20$ e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.50254 (17) | 0.07085 (17) | 0.49935 (10) | 0.0174 (3) |
| O2 | 0.69328 (17) | 0.04190 (19) | 0.83437 (11) | 0.0204 (3) |
| O3 | 0.79441 (18) | 0.4985 (2) | 0.67354 (12) | 0.0221 (3) |
| H3 | 0.777 (4) | 0.526 (4) | 0.612 (3) | 0.042 (9)* |
| O4 | 0.68542 (17) | 0.51474 (19) | 0.49826 (11) | 0.0198 (3) |
| O5 | 0.01705 (16) | 0.23344 (18) | 0.10555 (10) | 0.0178 (3) |
| O6 | 0.06369 (19) | 0.0284 (2) | 0.22016 (12) | 0.0251 (4) |
| O7 | 0.05288 (16) | 0.41671 (18) | −0.02881 (10) | 0.0187 (3) |
| O8 | 0.05354 (17) | 0.66810 (19) | −0.10124 (11) | 0.0210 (3) |
| C1 | 0.4074 (2) | −0.0676 (2) | 0.49859 (16) | 0.0181 (4) |
| C2 | 0.3732 (3) | −0.1334 (3) | 0.39786 (16) | 0.0237 (5) |
| H2A | 0.4635 | −0.1720 | 0.3874 | 0.036* |
| H2B | 0.3304 | −0.0482 | 0.3520 | 0.036* |
| H2C | 0.3036 | −0.2224 | 0.3894 | 0.036* |
| C3 | 0.2692 (2) | −0.0069 (3) | 0.51717 (18) | 0.0228 (5) |
| H3A | 0.2199 | 0.0701 | 0.4675 | 0.034* |
| H3B | 0.2949 | 0.0460 | 0.5795 | 0.034* |
| H3C | 0.2038 | −0.0975 | 0.5165 | 0.034* |
| C4 | 0.4918 (2) | −0.1879 (3) | 0.57238 (15) | 0.0194 (4) |
| H4A | 0.5793 | −0.2241 | 0.5559 | 0.023* |
| H4B | 0.4295 | −0.2829 | 0.5718 | 0.023* |
| C5 | 0.5390 (2) | −0.1138 (3) | 0.67197 (15) | 0.0193 (4) |
| H5A | 0.4542 | −0.1096 | 0.6969 | 0.023* |
| H5B | 0.6152 | −0.1819 | 0.7153 | 0.023* |
| C6 | 0.5981 (2) | 0.0529 (2) | 0.66954 (15) | 0.0160 (4) |
| C7 | 0.5763 (2) | 0.1365 (3) | 0.58478 (15) | 0.0149 (4) |
| C8 | 0.6366 (2) | 0.2927 (3) | 0.58144 (15) | 0.0148 (4) |
| C9 | 0.7257 (2) | 0.3568 (3) | 0.66940 (15) | 0.0166 (4) |
| C10 | 0.7437 (2) | 0.2780 (3) | 0.75539 (15) | 0.0174 (4) |
| H10 | 0.7988 | 0.3259 | 0.8137 | 0.021* |
| C11 | 0.6798 (2) | 0.1281 (3) | 0.75453 (15) | 0.0161 (4) |
| C12 | 0.7820 (3) | 0.1083 (3) | 0.92334 (15) | 0.0225 (5) |
| H12A | 0.7826 | 0.0346 | 0.9753 | 0.034* |
| H12B | 0.7416 | 0.2120 | 0.9342 | 0.034* |
| H12C | 0.8822 | 0.1233 | 0.9211 | 0.034* |
| C13 | 0.6066 (2) | 0.3942 (3) | 0.49629 (14) | 0.0150 (4) |
| C14 | 0.4826 (2) | 0.3661 (3) | 0.41004 (15) | 0.0163 (4) |
| H14 | 0.4162 | 0.2807 | 0.4081 | 0.020* |
| C15 | 0.4642 (2) | 0.4620 (3) | 0.33424 (15) | 0.0167 (4) |
| H15 | 0.5348 | 0.5446 | 0.3407 | 0.020* |
| C16 | 0.3495 (2) | 0.4553 (3) | 0.24339 (15) | 0.0160 (4) |
| C17 | 0.3573 (2) | 0.5633 (2) | 0.17258 (15) | 0.0178 (4) |
| H17 | 0.4318 | 0.6428 | 0.1869 | 0.021* |
| C18 | 0.2588 (2) | 0.5565 (3) | 0.08200 (15) | 0.0174 (4) |
| H18 | 0.2660 | 0.6310 | 0.0349 | 0.021* |

| | | | | |
|---|---|---|---|---|
| C19 | 0.1493 (2) | 0.4408 (3) | 0.05985 (14) | 0.0163 (4) |
| C20 | 0.1330 (2) | 0.3372 (2) | 0.13222 (15) | 0.0147 (4) |
| C21 | 0.2341 (2) | 0.3429 (2) | 0.22193 (15) | 0.0159 (4) |
| H21 | 0.2257 | 0.2703 | 0.2697 | 0.019* |
| C22 | −0.0247 (2) | 0.1575 (3) | 0.18001 (15) | 0.0201 (4) |
| H22A | −0.0210 | 0.2375 | 0.2303 | 0.024* |
| H22B | −0.1270 | 0.1199 | 0.1543 | 0.024* |
| C23 | 0.0360 (3) | −0.1112 (3) | 0.16131 (19) | 0.0282 (5) |
| H23A | 0.1017 | −0.1979 | 0.1934 | 0.042* |
| H23B | 0.0531 | −0.0866 | 0.1006 | 0.042* |
| H23C | −0.0657 | −0.1451 | 0.1496 | 0.042* |
| C24 | 0.0800 (2) | 0.5048 (3) | −0.10506 (15) | 0.0178 (4) |
| H24A | 0.1831 | 0.4887 | −0.1030 | 0.021* |
| H24B | 0.0171 | 0.4616 | −0.1664 | 0.021* |
| C25 | −0.0952 (3) | 0.7036 (3) | −0.10670 (18) | 0.0271 (5) |
| H25A | −0.1074 | 0.8200 | −0.1034 | 0.041* |
| H25B | −0.1188 | 0.6520 | −0.0535 | 0.041* |
| H25C | −0.1607 | 0.6630 | −0.1670 | 0.041* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0221 (8) | 0.0131 (7) | 0.0157 (7) | −0.0047 (6) | 0.0039 (6) | −0.0013 (6) |
| O2 | 0.0239 (8) | 0.0202 (8) | 0.0156 (7) | −0.0001 (6) | 0.0038 (6) | 0.0013 (6) |
| O3 | 0.0244 (8) | 0.0185 (8) | 0.0178 (8) | −0.0070 (6) | −0.0018 (6) | 0.0009 (6) |
| O4 | 0.0205 (8) | 0.0183 (8) | 0.0175 (7) | −0.0055 (6) | 0.0013 (6) | 0.0006 (6) |
| O5 | 0.0174 (7) | 0.0182 (8) | 0.0161 (7) | −0.0057 (6) | 0.0026 (6) | 0.0007 (6) |
| O6 | 0.0279 (9) | 0.0205 (8) | 0.0221 (8) | −0.0044 (7) | 0.0004 (7) | 0.0051 (6) |
| O7 | 0.0202 (8) | 0.0189 (8) | 0.0149 (7) | −0.0030 (6) | 0.0023 (6) | 0.0022 (6) |
| O8 | 0.0227 (8) | 0.0182 (8) | 0.0206 (8) | 0.0002 (6) | 0.0044 (7) | 0.0026 (6) |
| C1 | 0.0199 (11) | 0.0120 (10) | 0.0202 (10) | −0.0048 (8) | 0.0030 (8) | −0.0014 (8) |
| C2 | 0.0278 (12) | 0.0180 (11) | 0.0216 (11) | −0.0053 (9) | 0.0019 (10) | −0.0033 (9) |
| C3 | 0.0165 (10) | 0.0197 (10) | 0.0313 (12) | 0.0002 (8) | 0.0061 (9) | 0.0020 (9) |
| C4 | 0.0200 (11) | 0.0128 (10) | 0.0226 (11) | −0.0019 (8) | 0.0024 (9) | 0.0000 (8) |
| C5 | 0.0209 (11) | 0.0150 (10) | 0.0191 (11) | −0.0016 (8) | 0.0020 (9) | 0.0025 (8) |
| C6 | 0.0150 (10) | 0.0137 (10) | 0.0192 (10) | 0.0007 (8) | 0.0050 (8) | 0.0002 (8) |
| C7 | 0.0116 (9) | 0.0144 (10) | 0.0179 (10) | 0.0010 (7) | 0.0032 (8) | −0.0017 (8) |
| C8 | 0.0133 (10) | 0.0140 (10) | 0.0165 (9) | 0.0000 (8) | 0.0035 (8) | −0.0017 (7) |
| C9 | 0.0146 (10) | 0.0151 (10) | 0.0194 (11) | 0.0007 (8) | 0.0039 (8) | −0.0004 (8) |
| C10 | 0.0149 (10) | 0.0189 (10) | 0.0161 (10) | 0.0004 (8) | 0.0010 (8) | −0.0016 (8) |
| C11 | 0.0134 (10) | 0.0181 (10) | 0.0167 (10) | 0.0031 (8) | 0.0044 (8) | 0.0030 (8) |
| C12 | 0.0289 (12) | 0.0225 (11) | 0.0134 (10) | 0.0044 (10) | 0.0022 (9) | 0.0010 (9) |
| C13 | 0.0143 (10) | 0.0139 (10) | 0.0167 (10) | 0.0004 (8) | 0.0046 (8) | −0.0009 (7) |
| C14 | 0.0162 (10) | 0.0134 (10) | 0.0179 (10) | −0.0016 (8) | 0.0030 (8) | −0.0014 (8) |
| C15 | 0.0178 (10) | 0.0138 (10) | 0.0188 (10) | −0.0012 (8) | 0.0058 (8) | −0.0023 (8) |
| C16 | 0.0167 (10) | 0.0150 (10) | 0.0169 (10) | −0.0001 (8) | 0.0058 (8) | −0.0005 (8) |
| C17 | 0.0203 (11) | 0.0139 (10) | 0.0189 (10) | −0.0024 (8) | 0.0055 (8) | −0.0005 (8) |
| C18 | 0.0190 (10) | 0.0162 (11) | 0.0172 (10) | −0.0007 (8) | 0.0059 (8) | 0.0018 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C19 | 0.0189 (10) | 0.0152 (10) | 0.0140 (10) | 0.0018 (8) | 0.0037 (8) | −0.0006 (8) |
| C20 | 0.0156 (10) | 0.0106 (9) | 0.0189 (10) | −0.0005 (8) | 0.0065 (8) | −0.0020 (8) |
| C21 | 0.0166 (10) | 0.0143 (10) | 0.0170 (10) | 0.0006 (8) | 0.0054 (8) | 0.0016 (8) |
| C22 | 0.0206 (11) | 0.0229 (11) | 0.0169 (10) | −0.0047 (9) | 0.0057 (9) | −0.0004 (8) |
| C23 | 0.0282 (13) | 0.0222 (12) | 0.0345 (14) | −0.0057 (10) | 0.0099 (11) | −0.0002 (10) |
| C24 | 0.0186 (10) | 0.0193 (11) | 0.0149 (10) | 0.0002 (8) | 0.0043 (8) | 0.0020 (8) |
| C25 | 0.0267 (13) | 0.0280 (12) | 0.0260 (12) | 0.0072 (10) | 0.0070 (10) | 0.0006 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C7 | 1.358 (3) | C8—C9 | 1.423 (3) |
| O1—C1 | 1.469 (2) | C8—C13 | 1.467 (3) |
| O2—C11 | 1.351 (3) | C9—C10 | 1.391 (3) |
| O2—C12 | 1.440 (3) | C10—C11 | 1.390 (3) |
| O3—C9 | 1.344 (3) | C10—H10 | 0.9500 |
| O3—H3 | 0.90 (4) | C12—H12A | 0.9800 |
| O4—C13 | 1.253 (3) | C12—H12B | 0.9800 |
| O5—C20 | 1.369 (2) | C12—H12C | 0.9800 |
| O5—C22 | 1.426 (2) | C13—C14 | 1.472 (3) |
| O6—C22 | 1.387 (3) | C14—C15 | 1.341 (3) |
| O6—C23 | 1.427 (3) | C14—H14 | 0.9500 |
| O7—C19 | 1.368 (3) | C15—C16 | 1.454 (3) |
| O7—C24 | 1.431 (2) | C15—H15 | 0.9500 |
| O8—C24 | 1.388 (3) | C16—C17 | 1.397 (3) |
| O8—C25 | 1.436 (3) | C16—C21 | 1.410 (3) |
| C1—C4 | 1.519 (3) | C17—C18 | 1.384 (3) |
| C1—C3 | 1.520 (3) | C17—H17 | 0.9500 |
| C1—C2 | 1.523 (3) | C18—C19 | 1.389 (3) |
| C2—H2A | 0.9800 | C18—H18 | 0.9500 |
| C2—H2B | 0.9800 | C19—C20 | 1.418 (3) |
| C2—H2C | 0.9800 | C20—C21 | 1.383 (3) |
| C3—H3A | 0.9800 | C21—H21 | 0.9500 |
| C3—H3B | 0.9800 | C22—H22A | 0.9900 |
| C3—H3C | 0.9800 | C22—H22B | 0.9900 |
| C4—C5 | 1.529 (3) | C23—H23A | 0.9800 |
| C4—H4A | 0.9900 | C23—H23B | 0.9800 |
| C4—H4B | 0.9900 | C23—H23C | 0.9800 |
| C5—C6 | 1.504 (3) | C24—H24A | 0.9900 |
| C5—H5A | 0.9900 | C24—H24B | 0.9900 |
| C5—H5B | 0.9900 | C25—H25A | 0.9800 |
| C6—C7 | 1.390 (3) | C25—H25B | 0.9800 |
| C6—C11 | 1.407 (3) | C25—H25C | 0.9800 |
| C7—C8 | 1.431 (3) | | |
| | | | |
| C7—O1—C1 | 118.07 (16) | O2—C12—H12B | 109.5 |
| C11—O2—C12 | 117.60 (17) | H12A—C12—H12B | 109.5 |
| C9—O3—H3 | 104 (2) | O2—C12—H12C | 109.5 |
| C20—O5—C22 | 116.93 (16) | H12A—C12—H12C | 109.5 |

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| C22—O6—C23 | 113.33 (18) | H12B—C12—H12C | 109.5 |
| C19—O7—C24 | 116.34 (16) | O4—C13—C8 | 118.78 (18) |
| C24—O8—C25 | 113.00 (18) | O4—C13—C14 | 118.10 (18) |
| O1—C1—C4 | 108.51 (17) | C8—C13—C14 | 123.03 (18) |
| O1—C1—C3 | 108.19 (17) | C15—C14—C13 | 119.54 (19) |
| C4—C1—C3 | 113.25 (18) | C15—C14—H14 | 120.2 |
| O1—C1—C2 | 103.86 (16) | C13—C14—H14 | 120.2 |
| C4—C1—C2 | 111.78 (18) | C14—C15—C16 | 128.0 (2) |
| C3—C1—C2 | 110.74 (19) | C14—C15—H15 | 116.0 |
| C1—C2—H2A | 109.5 | C16—C15—H15 | 116.0 |
| C1—C2—H2B | 109.5 | C17—C16—C21 | 118.45 (19) |
| H2A—C2—H2B | 109.5 | C17—C16—C15 | 117.75 (19) |
| C1—C2—H2C | 109.5 | C21—C16—C15 | 123.77 (18) |
| H2A—C2—H2C | 109.5 | C18—C17—C16 | 121.24 (19) |
| H2B—C2—H2C | 109.5 | C18—C17—H17 | 119.4 |
| C1—C3—H3A | 109.5 | C16—C17—H17 | 119.4 |
| C1—C3—H3B | 109.5 | C17—C18—C19 | 120.12 (19) |
| H3A—C3—H3B | 109.5 | C17—C18—H18 | 119.9 |
| C1—C3—H3C | 109.5 | C19—C18—H18 | 119.9 |
| H3A—C3—H3C | 109.5 | O7—C19—C18 | 124.78 (18) |
| H3B—C3—H3C | 109.5 | O7—C19—C20 | 115.63 (18) |
| C1—C4—C5 | 111.09 (18) | C18—C19—C20 | 119.59 (19) |
| C1—C4—H4A | 109.4 | O5—C20—C21 | 124.72 (18) |
| C5—C4—H4A | 109.4 | O5—C20—C19 | 115.73 (18) |
| C1—C4—H4B | 109.4 | C21—C20—C19 | 119.53 (18) |
| C5—C4—H4B | 109.4 | C20—C21—C16 | 120.83 (18) |
| H4A—C4—H4B | 108.0 | C20—C21—H21 | 119.6 |
| C6—C5—C4 | 110.70 (17) | C16—C21—H21 | 119.6 |
| C6—C5—H5A | 109.5 | O6—C22—O5 | 113.22 (18) |
| C4—C5—H5A | 109.5 | O6—C22—H22A | 108.9 |
| C6—C5—H5B | 109.5 | O5—C22—H22A | 108.9 |
| C4—C5—H5B | 109.5 | O6—C22—H22B | 108.9 |
| H5A—C5—H5B | 108.1 | O5—C22—H22B | 108.9 |
| C7—C6—C11 | 117.93 (19) | H22A—C22—H22B | 107.7 |
| C7—C6—C5 | 121.93 (19) | O6—C23—H23A | 109.5 |
| C11—C6—C5 | 120.13 (18) | O6—C23—H23B | 109.5 |
| O1—C7—C6 | 121.74 (18) | H23A—C23—H23B | 109.5 |
| O1—C7—C8 | 115.87 (17) | O6—C23—H23C | 109.5 |
| C6—C7—C8 | 122.31 (19) | H23A—C23—H23C | 109.5 |
| C9—C8—C7 | 116.57 (18) | H23B—C23—H23C | 109.5 |
| C9—C8—C13 | 118.11 (18) | O8—C24—O7 | 113.05 (17) |
| C7—C8—C13 | 125.21 (18) | O8—C24—H24A | 109.0 |
| O3—C9—C10 | 116.71 (19) | O7—C24—H24A | 109.0 |
| O3—C9—C8 | 121.45 (19) | O8—C24—H24B | 109.0 |
| C10—C9—C8 | 121.84 (19) | O7—C24—H24B | 109.0 |
| C9—C10—C11 | 118.90 (19) | H24A—C24—H24B | 107.8 |
| C9—C10—H10 | 120.5 | O8—C25—H25A | 109.5 |
| C11—C10—H10 | 120.5 | O8—C25—H25B | 109.5 |

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| O2—C11—C10 | 122.98 (19) | H25A—C25—H25B | 109.5 |
| O2—C11—C6 | 114.77 (18) | O8—C25—H25C | 109.5 |
| C10—C11—C6 | 122.24 (19) | H25A—C25—H25C | 109.5 |
| O2—C12—H12A | 109.5 | H25B—C25—H25C | 109.5 |
| | | | |
| C7—O1—C1—C4 | −47.8 (2) | C5—C6—C11—C10 | 176.19 (19) |
| C7—O1—C1—C3 | 75.5 (2) | C9—C8—C13—O4 | 16.7 (3) |
| C7—O1—C1—C2 | −166.83 (18) | C7—C8—C13—O4 | −167.3 (2) |
| O1—C1—C4—C5 | 60.1 (2) | C9—C8—C13—C14 | −159.84 (19) |
| C3—C1—C4—C5 | −60.0 (2) | C7—C8—C13—C14 | 16.2 (3) |
| C2—C1—C4—C5 | 174.09 (18) | O4—C13—C14—C15 | 4.9 (3) |
| C1—C4—C5—C6 | −43.8 (2) | C8—C13—C14—C15 | −178.5 (2) |
| C4—C5—C6—C7 | 14.6 (3) | C13—C14—C15—C16 | −179.6 (2) |
| C4—C5—C6—C11 | −164.52 (19) | C14—C15—C16—C17 | −176.5 (2) |
| C1—O1—C7—C6 | 18.7 (3) | C14—C15—C16—C21 | 1.3 (3) |
| C1—O1—C7—C8 | −164.55 (17) | C21—C16—C17—C18 | −2.9 (3) |
| C11—C6—C7—O1 | 177.99 (18) | C15—C16—C17—C18 | 175.06 (19) |
| C5—C6—C7—O1 | −1.2 (3) | C16—C17—C18—C19 | 0.0 (3) |
| C11—C6—C7—C8 | 1.5 (3) | C24—O7—C19—C18 | 7.9 (3) |
| C5—C6—C7—C8 | −177.66 (19) | C24—O7—C19—C20 | −171.75 (17) |
| O1—C7—C8—C9 | −174.29 (17) | C17—C18—C19—O7 | −175.3 (2) |
| C6—C7—C8—C9 | 2.4 (3) | C17—C18—C19—C20 | 4.3 (3) |
| O1—C7—C8—C13 | 9.6 (3) | C22—O5—C20—C21 | 17.4 (3) |
| C6—C7—C8—C13 | −173.71 (19) | C22—O5—C20—C19 | −164.53 (18) |
| C7—C8—C9—O3 | 176.20 (19) | O7—C19—C20—O5 | −4.1 (3) |
| C13—C8—C9—O3 | −7.4 (3) | C18—C19—C20—O5 | 176.19 (18) |
| C7—C8—C9—C10 | −5.1 (3) | O7—C19—C20—C21 | 174.01 (18) |
| C13—C8—C9—C10 | 171.26 (19) | C18—C19—C20—C21 | −5.7 (3) |
| O3—C9—C10—C11 | −177.43 (19) | O5—C20—C21—C16 | −179.29 (19) |
| C8—C9—C10—C11 | 3.8 (3) | C19—C20—C21—C16 | 2.7 (3) |
| C12—O2—C11—C10 | −1.9 (3) | C17—C16—C21—C20 | 1.5 (3) |
| C12—O2—C11—C6 | 176.82 (18) | C15—C16—C21—C20 | −176.33 (19) |
| C9—C10—C11—O2 | 179.01 (19) | C23—O6—C22—O5 | −75.0 (2) |
| C9—C10—C11—C6 | 0.4 (3) | C20—O5—C22—O6 | −79.4 (2) |
| C7—C6—C11—O2 | 178.28 (17) | C25—O8—C24—O7 | −60.4 (2) |
| C5—C6—C11—O2 | −2.6 (3) | C19—O7—C24—O8 | −70.3 (2) |
| C7—C6—C11—C10 | −3.0 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O4 | 0.90 (4) | 1.65 (4) | 2.480 (2) | 153 (3) |