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6,7,8-Trimethoxycoumarin from *Cryptocarya bracteolata*

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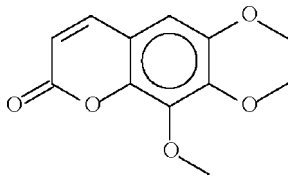
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.118; data-to-parameter ratio = 16.5.

The title compound, $C_{12}H_{12}O_5$, isolated from the bark of *Cryptocarya bracteolata*, has a planar fused-ring system.

Related literature

For the isolation and spectroscopic characterization of 6,7,8-trimethoxycoumarin that has been isolated from other plants, see: Adesina *et al.* (1997); Amaral *et al.* (2001); Anis *et al.* (1999, 2002); Anjaneyulu & Murty (1981); Bohlmann *et al.* (1981, 1985); Boulware & Stermitz (1981); Borges del Castillo *et al.* (1987); Campbell (1996); Campbell *et al.* (1986); Cheng *et al.* (2005); Del Castillo *et al.* (1984); Eshiett & Taylor (1968); Estevez-Braun *et al.* (1995); Facundo *et al.* (2002); Goh *et al.* (1989, 1990); Gonzalez *et al.* (1972, 1975, 1977); Goren *et al.* (1988); Hsiao & Chiang (1995); Iqbal *et al.* (2003); Iossifova *et al.* (1994); Ishi *et al.* (1972); Ishii *et al.* (1981, 1982); Ishikawa *et al.* (1995); Laskaris *et al.* (1995); Macleod & Rasmussen (1998); Mbwambo *et al.* (1996); Oksuz (1990); Qi *et al.* (2004); Ren *et al.* (1991); Rios & Flores (1976); Sang *et al.* (2005); Semple *et al.* (1999); Shi *et al.* (2005, 2006); Wagner & Bladt (1975); Yang & Kinghorn (1985).

For the crystal structures of coumarins having methoxy (and/or hydroxy) groups in the 5 to 8 positions, see: Baures *et al.* (2002); Bhadbhade *et al.* (1984); Gnanaguru *et al.* (1984, 1985); Kimura *et al.* (1980); Li *et al.* (2001); Ramasubbu *et al.* (1981, 1982); Wagner *et al.* (1974); Ye & Fan (2007).



Experimental

Crystal data

$C_{12}H_{12}O_5$
M_r = 236.22
Orthorhombic, *Pbca*
a = 13.9688 (6) Å
b = 8.1164 (4) Å
c = 19.790 (1) Å
V = 2243.7 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 295 (2) K
0.34 × 0.28 × 0.19 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.738, T_{max} = 0.979
19734 measured reflections
2561 independent reflections
1836 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.118
S = 1.04
2561 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2490).

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supplementary materials

Acta Cryst. (2007). E**63**, o3692-o3693 [**doi:10.1107/S1600536807036914**]

6,7,8-Trimethoxycoumarin from *Cryptocarya bracteolata*

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Comment

6,7,8-Trimethoxycoumarin has been isolated from a wide range plants such as, for example, *Acmadenia sheilae* (Rutaceae) (Campbell, 1996), *Artemisia adamsii* (Bohlmann *et al.*, 1985), *Aralia bipinnata* (Hsiao & Chiang, 1995), *Baccharis* species (Bohlmann *et al.*, 1981), *Bupleurum fruiticescens* (Gonzalez *et al.*, 1975), *Bupleurum salicifolium* (Estevez-Braun *et al.*, 1995), *Cedrelopsis grevei* (Eshiett & Taylor, 1968), *Cuscuta reflexa* (Anis *et al.*, 1999; Anis *et al.*, 2002), *Duranta repens* (Iqbal *et al.*, 2003), *Euodia latifolia* (Goh *et al.*, 1990), *Euodia* (Rutaceae) (Goh *et al.*, 1989), *Euphorbia antiquorum* (Sang *et al.*, 2005), *Euphorbia quinquecostata* (Mbwambo *et al.*, 1996), *Fraxinus ornus* (Iossifova *et al.*, 1994), *Guazuma tomentosa* Kunth (Anjaneyulu & Murty, 1981), *Pelargonium reniforme* (Wagner, & Bladt, 1975), *Picnomon acarna* (Laskaris *et al.*, 1995), *Platypodium elegans* (Amaral *et al.*, 2001), *Pterocaulon sphacelatum* (Semple *et al.*, 1999), *Pterocaulon serrulatum* (Macleod & Rasmussen, 1998), *Ruta angustifolia* (Del Castillo *et al.*, 1984), *Ruta angustifolia* Pers (Borges del Castillo *et al.*, 1987), *Ruta oerojasme* (Gonzalez *et al.*, 1972), *Ruta sp*. Tene. 29662 (Gonzalez *et al.*, 1977), *Rutoideae*: tribe Diosmeae (Campbell *et al.*, 1986), *Sapium chihsinianum* S. Lee (Qi *et al.*, 2004), *Sapium sebiferum* (Yang & Kinghorn, 1985; Shi *et al.*, 2006), *Tanacetum cilicium* (Oksuz, 1990), *Tanacetum heterotomum* (Goren *et al.*, 1988), *Tagetes florida* (Rios & Flores, 1976), *Xanthoxylum inerme* (*Fagara boninensis*) (Ishi *et al.*, 1972), *Xanthoxylum inerme* Koidz. (*Fagara boninensis* Koidz.) (Ishii *et al.*, 1981), *Xanthoxylum integrifoliolum* (Merr.) Merr. (*Fagara integrifoliola* Merr.) (Ishii *et al.*, 1982), *Zanthoxylum microcarpum*, *Zanthoxylum procerum* (Boulware & Stermitz, 1981), *Xanthoxylum nitidum* (Roxb.) D. C. (*Fagara nitida* Roxb.) (Ishikawa *et al.*, 1995), *Zanthoxylum ailanthoides* (Cheng *et al.*, 2005; Shi *et al.*, 2005), *Zanthoxylum lemairie* (Adesina *et al.*, 1997), *Zanthoxylum rugosum* A. St. Hill & Tul (Facundo *et al.*, 2002) and *Zanthoxylum utile* Huang (Ren *et al.*, 1991). The compound (I) has been evaluated for a variety of biological activities and almost all spectrosopic analyses already have been carried out on it.

The compound has been isolated from *Cryptocarya bracteolata*; its crystal structure shows a planar conformation for the coumarin (Fig. 1). The compound complements the list of coumarins having methoxy (and/or hydroxy) groups in the 5 to 8 positions of the coumarin fused-ring, *i.e.*, 6-methoxy- (Bauers *et al.*, 2002; Gnanaguru *et al.*, 1985), 7-methoxy- (Bhadbhade *et al.*, 1984; Gnanaguru *et al.*, 1984; Gnanaguru *et al.*, 1985; Ramasubbu, *et al.*, 1981; Ramasubbu *et al.*, 1982), 8-methoxy- (Baures *et al.*, 2002; Gnanaguru *et al.*, 1985), 5,7-dimethoxy- (Ye & Fan, 2007), 7-hydroxy-6-methoxy- (Kimura *et al.*, 1980), 7-hydroxy-5,6-dimethoxy- (Wagner *et al.*, 1974) and 7-hydroxy-6,8-dimethoxy- (Li *et al.*, 2001) coumarins.

Experimental

Cryptocarya bracteolata was collected in Mersing, Johor in 1997. Specimens were deposited at the Forest Research Institute of Malaysia herbarium and the Department of Chemistry, University of Malaya.

The bark of *C. bracteolata* (2.7 kg) was extracted with *n*-hexane; the solvent was then removed under vacuum. The plant material was moistened with 10% ammonium hydroxide overnight. It was extracted with dichloromethane (8 *L*) for 17 h to yield the crude extract (89 g). A portion (30 g) was subjected to column chromatography on silica gel 60 GF_{254} by using a

step gradient of dichloromethane and methanol. The separation afforded 15 fractions; the second (100% dichloromethane) gave 5,6,7-trimethoxy-*2H*-chromen-2-one (24.5 g), whose formulation was established by spectroscopic analysis.

Refinement

The carbon-bound hydrogen atoms were placed at calculated positions (C–H 0.93 to 0.98 Å) and were inlcuded in the refinement in the riding model approximation, with U(H) set to 1.2 to 1.5 U_{eq}(C).

Figures

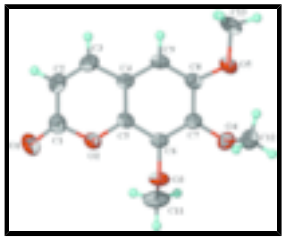

Fig. 1. View of (I); displacement ellipsoids are drawn at the 50% probability level, and hydrogen atoms as spheres of arbitrary radius.

6,7,8-Trimethoxycoumarin

Crystal data

$C_{12}H_{12}O_5$	F_{000} = 992
M_r = 236.22	D_x = 1.399 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 11077 reflections
a = 13.9688 (6) Å	θ = 3.1–27.4º
b = 8.1164 (4) Å	μ = 0.11 mm^{-1}
c = 19.790 (1) Å	*T* = 295 (2) K
V = 2243.7 (2) Å^3	Prism, colourless
Z = 8	0.34 × 0.28 × 0.19 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer	2561 independent reflections
Radiation source: fine-focus sealed tube	1836 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.044
T = 295(2) K	θ_{max} = 27.4º
ω scans	θ_{min} = 3.1º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	*h* = −18→18
T_{min} = 0.738, T_{max} = 0.979	*k* = −10→10
19734 measured reflections	*l* = −25→25

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.118$

$S = 1.04$

2561 reflections

155 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.3525P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.18$ e Å^{-3}

$\Delta\rho_{min} = -0.14$ e Å^{-3}

Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0102 (13)

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.65727 (10)	0.89131 (17)	0.55051 (7)	0.0721 (4)
O2	0.61944 (7)	0.74494 (13)	0.46074 (5)	0.0488 (3)
O3	0.67995 (7)	0.60631 (14)	0.34306 (6)	0.0526 (3)
O4	0.55894 (8)	0.41023 (14)	0.26964 (6)	0.0555 (3)
O5	0.37230 (8)	0.38596 (15)	0.30093 (6)	0.0603 (3)
C1	0.59401 (13)	0.8219 (2)	0.52022 (8)	0.0522 (4)
C2	0.49443 (13)	0.8134 (2)	0.53901 (8)	0.0550 (4)
H2	0.4745	0.8655	0.5784	0.066*
C3	0.42979 (12)	0.7333 (2)	0.50165 (8)	0.0503 (4)
H3	0.3660	0.7320	0.5151	0.060*
C4	0.45761 (10)	0.64940 (18)	0.44103 (7)	0.0412 (3)
C5	0.55343 (10)	0.65873 (17)	0.42246 (7)	0.0399 (3)
C6	0.58711 (9)	0.58398 (18)	0.36371 (7)	0.0409 (3)
C7	0.52399 (10)	0.49282 (18)	0.32449 (7)	0.0426 (4)
C8	0.42667 (10)	0.48059 (19)	0.34304 (8)	0.0447 (4)
C9	0.39418 (10)	0.55879 (19)	0.40050 (8)	0.0452 (4)
H9	0.3299	0.5514	0.4124	0.054*
C11	0.74647 (13)	0.4929 (3)	0.37015 (11)	0.0789 (6)
H11A	0.8092	0.5169	0.3529	0.118*
H11B	0.7285	0.3831	0.3574	0.118*
H11C	0.7469	0.5018	0.4185	0.118*

C12	0.52936 (13)	0.4738 (3)	0.20538 (8)	0.0633 (5)
H12A	0.5565	0.4078	0.1700	0.095*
H12B	0.5510	0.5855	0.2008	0.095*
H12C	0.4608	0.4706	0.2024	0.095*
C13	0.27263 (12)	0.3741 (2)	0.31493 (11)	0.0676 (5)
H13A	0.2426	0.3051	0.2817	0.101*
H13B	0.2445	0.4820	0.3135	0.101*
H13C	0.2635	0.3274	0.3590	0.101*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0684 (8)	0.0789 (9)	0.0690 (8)	−0.0085 (7)	−0.0197 (7)	−0.0105 (7)
O2	0.0417 (6)	0.0543 (6)	0.0503 (6)	−0.0043 (5)	−0.0045 (5)	0.0004 (5)
O3	0.0335 (5)	0.0614 (7)	0.0628 (7)	−0.0001 (5)	0.0059 (5)	0.0087 (6)
O4	0.0522 (6)	0.0630 (7)	0.0513 (6)	0.0108 (5)	0.0005 (5)	−0.0067 (5)
O5	0.0433 (6)	0.0690 (8)	0.0684 (8)	−0.0092 (5)	−0.0064 (5)	−0.0122 (6)
C1	0.0588 (10)	0.0503 (9)	0.0476 (9)	0.0013 (8)	−0.0091 (8)	0.0028 (7)
C2	0.0639 (10)	0.0571 (9)	0.0440 (8)	0.0041 (8)	0.0015 (8)	−0.0010 (8)
C3	0.0486 (8)	0.0547 (9)	0.0476 (8)	0.0055 (7)	0.0065 (7)	0.0078 (7)
C4	0.0377 (7)	0.0440 (8)	0.0418 (7)	0.0039 (6)	0.0014 (6)	0.0088 (6)
C5	0.0365 (7)	0.0401 (7)	0.0432 (8)	−0.0013 (6)	−0.0045 (6)	0.0079 (6)
C6	0.0328 (7)	0.0434 (8)	0.0465 (8)	0.0026 (6)	0.0011 (6)	0.0093 (6)
C7	0.0378 (7)	0.0453 (8)	0.0446 (8)	0.0051 (6)	−0.0006 (6)	0.0039 (6)
C8	0.0381 (7)	0.0452 (8)	0.0507 (8)	−0.0006 (6)	−0.0057 (6)	0.0033 (7)
C9	0.0325 (7)	0.0497 (8)	0.0536 (9)	0.0011 (6)	0.0012 (6)	0.0079 (7)
C11	0.0417 (9)	0.0966 (15)	0.0984 (14)	0.0217 (10)	0.0040 (10)	0.0124 (13)
C12	0.0645 (11)	0.0784 (12)	0.0471 (9)	−0.0005 (10)	−0.0010 (8)	−0.0051 (9)
C13	0.0424 (9)	0.0710 (12)	0.0894 (13)	−0.0121 (8)	−0.0108 (9)	−0.0008 (10)

Geometric parameters (Å, °)

O1—C1	1.207 (2)	C4—C9	1.403 (2)
O2—C1	1.379 (2)	C5—C6	1.393 (2)
O2—C5	1.3834 (17)	C6—C7	1.388 (2)
O3—C6	1.3718 (17)	C7—C8	1.412 (2)
O3—C11	1.413 (2)	C8—C9	1.379 (2)
O4—C7	1.3661 (18)	C9—H9	0.9300
O4—C12	1.433 (2)	C11—H11A	0.9600
O5—C8	1.3643 (18)	C11—H11B	0.9600
O5—C13	1.423 (2)	C11—H11C	0.9600
C1—C2	1.441 (2)	C12—H12A	0.9600
C2—C3	1.336 (2)	C12—H12B	0.9600
C2—H2	0.9300	C12—H12C	0.9600
C3—C4	1.433 (2)	C13—H13A	0.9600
C3—H3	0.9300	C13—H13B	0.9600
C4—C5	1.3902 (19)	C13—H13C	0.9600
C1—O2—C5	121.66 (12)	O5—C8—C9	125.42 (13)

C6—O3—C11	115.00 (13)	O5—C8—C7	114.64 (14)
C7—O4—C12	115.18 (12)	C9—C8—C7	119.93 (14)
C8—O5—C13	117.63 (13)	C8—C9—C4	120.31 (13)
O1—C1—O2	116.53 (16)	C8—C9—H9	119.8
O1—C1—C2	126.89 (17)	C4—C9—H9	119.8
O2—C1—C2	116.57 (14)	O3—C11—H11A	109.5
C3—C2—C1	122.18 (16)	O3—C11—H11B	109.5
C3—C2—H2	118.9	H11A—C11—H11B	109.5
C1—C2—H2	118.9	O3—C11—H11C	109.5
C2—C3—C4	120.75 (15)	H11A—C11—H11C	109.5
C2—C3—H3	119.6	H11B—C11—H11C	109.5
C4—C3—H3	119.6	O4—C12—H12A	109.5
C5—C4—C9	119.03 (14)	O4—C12—H12B	109.5
C5—C4—C3	117.17 (14)	H12A—C12—H12B	109.5
C9—C4—C3	123.80 (13)	O4—C12—H12C	109.5
O2—C5—C4	121.63 (13)	H12A—C12—H12C	109.5
O2—C5—C6	116.89 (12)	H12B—C12—H12C	109.5
C4—C5—C6	121.47 (13)	O5—C13—H13A	109.5
O3—C6—C7	120.29 (13)	O5—C13—H13B	109.5
O3—C6—C5	120.68 (13)	H13A—C13—H13B	109.5
C7—C6—C5	118.95 (13)	O5—C13—H13C	109.5
O4—C7—C6	118.60 (13)	H13A—C13—H13C	109.5
O4—C7—C8	121.09 (13)	H13B—C13—H13C	109.5
C6—C7—C8	120.25 (13)		
C5—O2—C1—O1	178.68 (14)	C4—C5—C6—C7	−2.6 (2)
C5—O2—C1—C2	−2.4 (2)	C12—O4—C7—C6	−110.92 (16)
O1—C1—C2—C3	179.80 (17)	C12—O4—C7—C8	71.85 (19)
O2—C1—C2—C3	1.0 (2)	O3—C6—C7—O4	7.7 (2)
C1—C2—C3—C4	0.9 (3)	C5—C6—C7—O4	−175.38 (13)
C2—C3—C4—C5	−1.5 (2)	O3—C6—C7—C8	−175.06 (13)
C2—C3—C4—C9	177.99 (15)	C5—C6—C7—C8	1.9 (2)
C1—O2—C5—C4	1.9 (2)	C13—O5—C8—C9	3.9 (2)
C1—O2—C5—C6	−179.19 (13)	C13—O5—C8—C7	−177.30 (14)
C9—C4—C5—O2	−179.40 (12)	O4—C7—C8—O5	−2.0 (2)
C3—C4—C5—O2	0.1 (2)	C6—C7—C8—O5	−179.21 (13)
C9—C4—C5—C6	1.7 (2)	O4—C7—C8—C9	176.87 (13)
C3—C4—C5—C6	−178.74 (13)	C6—C7—C8—C9	−0.3 (2)
C11—O3—C6—C7	−95.09 (18)	O5—C8—C9—C4	178.20 (14)
C11—O3—C6—C5	88.04 (18)	C7—C8—C9—C4	−0.6 (2)
O2—C5—C6—O3	−4.60 (19)	C5—C4—C9—C8	−0.1 (2)
C4—C5—C6—O3	174.30 (13)	C3—C4—C9—C8	−179.61 (14)
O2—C5—C6—C7	178.49 (12)		

Fig. 1