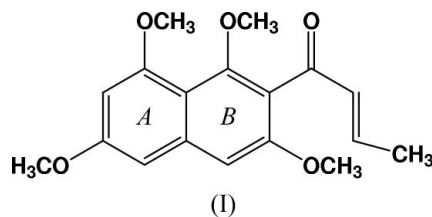


**(E)-1-(1,3,6,8-Tetramethoxynaphthalen-2-yl)-
but-2-en-1-one****M. Iqbal Choudhary,^a Alain Meli Lannang,^b Shamsher Ali,^a Shazia Anjum^{a*} and Hoong-Kun Fun^c**^aHEJ Research Institute of Chemistry, International Centre for Chemical Sciences, University of Karachi, Karachi 75270, Pakistan, ^bDepartment of Organic Chemistry, Faculty of Science, University of Yaounde I, PO Box 812, Cameroon, and ^cX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, MalaysiaCorrespondence e-mail:
anjumshazia@yahoo.com**Key indicators**Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.042
 wR factor = 0.126
Data-to-parameter ratio = 13.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.In the title compound, $\text{C}_{18}\text{H}_{20}\text{O}_5$, one of the four methoxy groups is twisted out of the naphthalene ring plane as a result of steric hindrance. The orientation of the butenone substituent with respect to the attached ring is anticlinal.

Received 1 March 2005

Accepted 9 March 2005

Online 18 March 2005

Comment*Guiera senegalensis* Lam. (Combretaceae) is a shrub of up to 1.5 m in height that grows in African savannah regions from Sudan to Senegal. It is commonly used in African folk medicines for the treatment of various ailments and it is also used for the prevention of leprosy, fevers and diarrhoea, as well as for the treatment of dysentery and skin diseases (Iwu, 1993). Pharmacological investigations of *G. senegalensis* revealed antiviral (Kudi, 1999) and antioxidant (Bouchet *et al.*, 1998) activities. Previous phytochemical studies resulted in the isolation of flavonoids (Ficarra *et al.*, 1997), quinic acid esters (Bouchet *et al.*, 2000) and naphthalene derivatives (Moahmoud & Khalid, 1997; Bucar *et al.*, 1998). In our systematic search for secondary metabolites from Cameroonian medicinal plants, we investigated different parts of this taxon. In this paper, we report the crystal structure of title compound, (I), isolated from the leaves of *G. senegalensis*.The bond lengths in compound (I) show normal values (Allen *et al.*, 1987). The naphthalene ring system is planar within 0.071 (1) Å. The methoxy substituents at the C1, C3 and C6 positions are nearly coplanar with the attached aromatic rings [$\text{C}15-\text{O}1-\text{C}1-\text{C}2 = -4.3$ (2)°, $\text{C}16-\text{O}2-\text{C}3-\text{C}4 = 10.2$ (2)° and $\text{C}17-\text{O}3-\text{C}6-\text{C}5 = -0.2$ (2)°]. As a result of steric interactions, the methoxy group at C8 position is twisted away from the C5–C10 ring [$\text{C}18-\text{O}4-\text{C}8-\text{C}9 = 78.3$ (2)°]. The C8–O4–C18 angle [114.38 (13)°] is narrower than the C–O–C angles observed in other methoxy groups (Table 1). The butenone substituent at C7 is anticlinal to the C5–C10 plane, as shown by the C6–C7–C11–C12 torsion angle of 92.6 (2)°. A weak $\text{C}16 \cdots \text{O}2(x, \frac{3}{2} - y, z - \frac{1}{2})$ interaction [3.443 (3) Å] is observed in the crystal structure. A view of the molecular packing along the c axis is shown in Fig. 2.

Experimental

Powdered leaves of *Guiera senegalensis* (2.4 kg) were soaked and extracted with MeOH (10 l) for one week. The combined methanol extracts were dried under vacuum to afford a green gum (150 g). Subsequent extraction of this methanolic extract yielded a hexane-soluble fraction (10 g) and an ethyl acetate-soluble fraction (40 g). The ethyl-acetate-soluble fraction was subjected to VLC, using a hexane–ethyl acetate mixture of increasing polarity. The less polar fractions afforded chlorophyll, α -amyryn and a mixture of sterols, while the more polar fraction was further subjected to column chromatography (hexane–ethyl acetate, 85:15) to yield compound (I) as light yellow crystals (m.p. 372–374 K).

Crystal data

$C_{18}H_{20}O_5$ $D_x = 1.253 \text{ Mg m}^{-3}$
 $M_r = 316.34$ Mo $K\alpha$ radiation
 Monoclinic, $P2_1/c$ Cell parameters from 6949 reflections
 $a = 8.2731 (18) \text{ \AA}$ $\theta = 1.6\text{--}25.0^\circ$
 $b = 25.493 (6) \text{ \AA}$ $\mu = 0.09 \text{ mm}^{-1}$
 $c = 8.1129 (18) \text{ \AA}$ $T = 293 (2) \text{ K}$
 $\beta = 101.443 (3)^\circ$ Block, colourless
 $V = 1677.0 (7) \text{ \AA}^3$ $Z = 4$
 $0.53 \times 0.32 \times 0.29 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer 2950 independent reflections
 ω scans 2622 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $R_{int} = 0.019$
 $T_{min} = 0.953$, $T_{max} = 0.974$ $\theta_{max} = 25.0^\circ$
 12 810 measured reflections $h = -9 \rightarrow 9$
 $k = -30 \rightarrow 28$
 $l = -9 \rightarrow 9$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 0.2665P]$
 $R[F^2 > 2\sigma(F^2)] = 0.042$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.126$ $(\Delta/\sigma)_{max} = 0.002$
 $S = 1.06$ $\Delta\rho_{max} = 0.19 \text{ e \AA}^{-3}$
 2950 reflections $\Delta\rho_{min} = -0.19 \text{ e \AA}^{-3}$
 214 parameters Extinction correction: *SHELXTL*
 H-atom parameters constrained Extinction coefficient: 0.020 (3)

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1–C1	1.3602 (17)	O3–C17	1.4198 (18)
O1–C15	1.421 (2)	O4–C8	1.3781 (16)
O2–C3	1.3657 (17)	O4–C18	1.432 (2)
O2–C16	1.421 (2)	O5–C11	1.2132 (19)
O3–C6	1.3637 (17)	C12–C13	1.299 (2)
C1–O1–C15	117.75 (12)	O3–C6–C7	114.40 (12)
C3–O2–C16	117.50 (12)	C7–C8–O4	115.94 (12)
C6–O3–C17	117.20 (11)	O4–C8–C9	122.12 (12)
C8–O4–C18	114.38 (13)	C8–C9–C1	126.10 (12)
C4–C3–O2	125.01 (13)	C13–C12–C11	124.98 (14)
O2–C3–C2	113.80 (12)	C12–C13–C14	126.39 (16)
O3–C6–C5	125.33 (13)		

All H atoms were positioned geometrically and allowed to ride on the parent C atoms, with aromatic C–H = 0.93 \AA and methyl C–H = 0.96 \AA , and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H and $1.2U_{eq}(C)$ for the other H atoms. A rotating group model was used for the methyl groups.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve

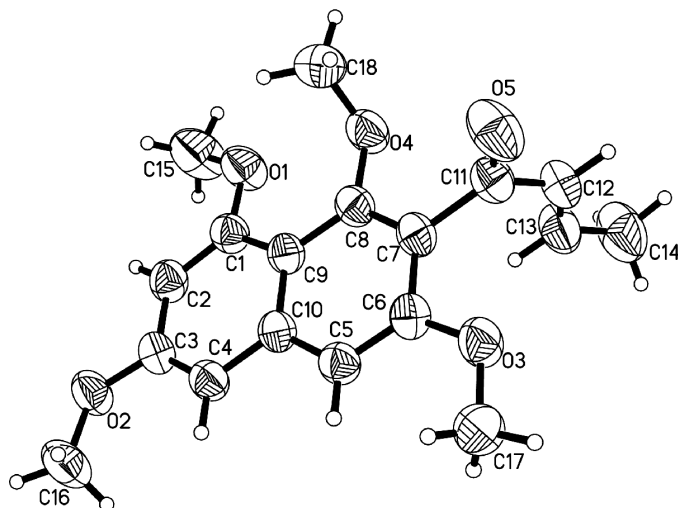


Figure 1 The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

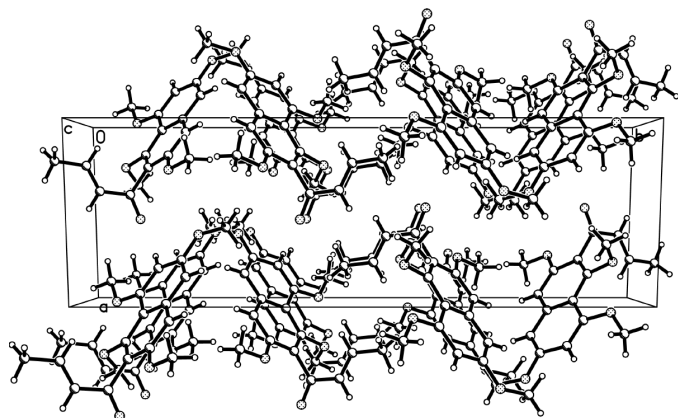


Figure 2 The molecular packing of (I), viewed along the c axis.

structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

AML thanks the TWAS for financially supporting his training in ICCS HEJ RIC, University of Karachi, Karachi, Pakistan.

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