The structures were solved by direct methods and refined by full-matrix least-squares techniques. The H atoms were located from difference Fourier maps and refined isotropically.

Data collection, cell refinement and data reduction: *XSCANS* (Siemens, 1994). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Geometrical calculations: *PARST* (Nardelli, 1983).

The authors would like to thank the Malaysian Government and the Universiti Sains Malaysia for research grant R&D No. 190-9609-2801 and short-term grant No. 131-0250-0530. KC thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1334). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). **C53**, 1859–1862

Substituted Methoxybenzene Derivatives: $C_8H_9NO_4$, $C_9H_{11}NO_5$ and $C_{13}H_{18}O_4$

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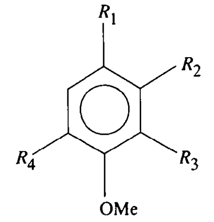
(*Received 14 April 1997; accepted 2 July 1997*)

Abstract

The structures of three methoxybenzenes, namely 1,2-dimethoxy-4-nitrobenzene, $C_8H_9NO_4$, (I), 1,2,3-trimethoxy-5-nitrobenzene, $C_9H_{11}NO_5$, (II), and 1-(2,4,5-trimethoxyphenyl)-1-butanone, $C_{13}H_{18}O_4$, (III), are reported. Molecules of (I) and (III) are planar, but one of the three methoxy groups of (II) is twisted out of the phenyl ring plane as a result of steric hindrance. In all three solids, the molecules are linked to form centrosymmetrically hydrogen-bonded dimers; they are packed in parallel layers in (I) and (II), but in zigzag layers in (III).

Comment

This paper reports an investigation of a series of methoxybenzenes, (I), (II) and (III), which was carried out since such moieties often occur in natural products.



	R_1	R_2	R_3	R_4
(I)	NO_2	H	OMe	H
(II)	NO_2	H	OMe	OMe
(III)	Butanone	OMe	H	OMe

The mean lengths of the C_{sp^2}—O [1.361 (3) Å] and O—CH_3 [1.425 (3) Å] bonds in the methoxy groups of these compounds agree with values observed for related structures (Bryan & White, 1982). Molecules of (I) and (III) are essentially planar; the steric interactions of the methoxy groups in (II) cause C8 to deviate by 1.173 (2) Å from the mean plane formed by the remaining non-H atoms.

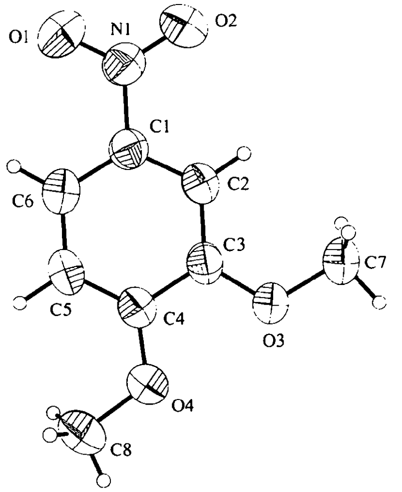


Fig. 1. View of (I) showing the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level.

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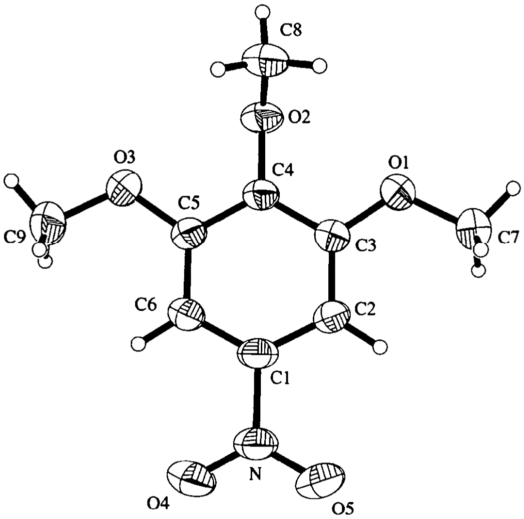

Fig. 2. View of (II) showing the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level.

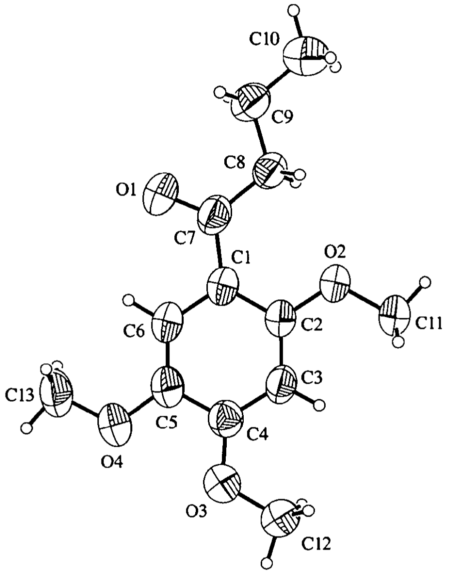

Fig. 3. View of (III) showing the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level.

In all three solids, centrosymmetrically related molecules form dimeric pairs through intermolecular C—H···O hydrogen bonds involving methoxy groups. In (I) and (II), the molecules are packed in two-dimensional layers, parallel to the ($\bar{1}02$) and (010) planes, respectively; the molecules form zigzag layers parallel to the (120) and ($\bar{1}21$) planes in (III).

Experimental

Compounds (I) and (III) were prepared from their hydroxy precursors by methylation with dimethyl sulfate in the presence of anhydrous potassium carbonate, with dry acetone as solvent. Compound (II) was prepared by nitration (HNO_3/H_2SO_4) of 3,4,5-trimethoxybenzoic acid. Single crystals of (I) were obtained by slow evaporation from a chloroform–tetrachloromethane mixture; crystals of (II) and (III) were obtained by slow evaporation from ethanol solutions.

Compound (I)

Crystal data

$C_8H_9NO_4$
$M_r = 183.16$
Monoclinic
$P2_1/c$
$a = 10.381\ (1)$ Å
$b = 12.556\ (1)$ Å
$c = 6.883\ (1)$ Å
$\beta = 102.07\ (1)°$
$V = 877.3\ (2)$ Å^3
$Z = 4$
$D_x = 1.387$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 38 reflections
$\theta = 5.27–12.54°$
$\mu = 0.113$ mm^{-1}
$T = 293\ (2)$ K
Rectangular
$0.66 \times 0.34 \times 0.28$ mm
Light yellow

Data collection

Siemens $P4$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2719 measured reflections
2014 independent reflections
1165 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$

$\theta_{\rm max} = 27.50°$
$h = -13 \rightarrow 13$
$k = -16 \rightarrow 1$
$l = -1 \rightarrow 8$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.140$
$S = 0.957$
2014 reflections
155 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0725P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$

$\Delta\rho_{\rm max} = 0.19$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.007 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °) and hydrogen-bonding geometry (Å, °) for (I)*

O1—N1	1.218 (2)	O4—C4	1.350 (2)
O2—N1	1.219 (2)	O4—C8	1.430 (3)
O3—C3	1.352 (2)	N1—C1	1.463 (2)
O3—C7	1.429 (2)		
C3—O3—C7	117.4 (2)	O1—N1—C1	118.8 (2)
C4—O4—C8	117.8 (2)	O2—N1—C1	118.6 (2)
O1—N1—O2	122.6 (2)		

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O2$^{\rm i}$	0.97 (2)	2.52 (2)	3.296 (2)	137 (1)
C7—H7*A*···O3$^{\rm ii}$	0.99 (2)	2.58 (2)	3.383 (3)	139 (2)
C7—H7*A*···O4$^{\rm ii}$	0.99 (2)	2.58 (2)	3.513 (3)	157 (2)

Symmetry codes: (i) $-x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $-x, 1 - y, -z$.

Compound (II)

Crystal data

$C_9H_{11}NO_5$
$M_r = 213.19$
Monoclinic
$P2_1/n$
$a = 7.025$ (1) Å
$b = 13.890$ (2) Å
$c = 10.356$ (1) Å
$\beta = 99.03$ (1)°
$V = 998.0$ (2) Å^3
$Z = 4$
$D_x = 1.419$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 38 reflections
$\theta = 5.40–12.50$°
$\mu = 0.117$ mm^{-1}
$T = 293$ (2) K
Needle
1.36 × 0.44 × 0.32 mm
Colourless

Data collection

Siemens $P4$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3068 measured reflections
2290 independent reflections
1606 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.015$

$\theta_{\rm max} = 27.50$°
$h = -1 \rightarrow 9$
$k = -1 \rightarrow 18$
$l = -13 \rightarrow 13$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.132$
$S = 1.013$
2290 reflections
181 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0826P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$

$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.043 (6)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 2. *Selected geometric parameters (Å, °) and hydrogen-bonding geometry (Å, °) for (II)*

O1—C3	1.3584 (14)	O3—C9	1.423 (2)
O1—C7	1.430 (2)	O4—N	1.218 (2)
O2—C4	1.3705 (14)	O5—N	1.220 (2)
O2—C8	1.418 (2)	N—C1	1.471 (2)
O3—C5	1.3560 (14)		
C3—O1—C7	117.61 (10)	O4—N—O5	123.22 (12)
C4—O2—C8	113.83 (12)	O4—N—C1	118.47 (11)
C5—O3—C9	117.41 (11)	O5—N—C1	118.32 (11)

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7A···O3$^{\rm i}$	0.96 (2)	2.58 (2)	3.411 (2)	145 (1)
C8—H8C···O5$^{\rm ii}$	0.96 (2)	2.71 (2)	3.516 (3)	141 (1)
C9—H9A···O5$^{\rm iii}$	0.98 (2)	2.68 (2)	3.398 (2)	129 (1)
C9—H9B···O1$^{\rm iv}$	0.96 (2)	2.61 (2)	3.452 (2)	146 (2)

Symmetry codes: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $1 - x, 1 - y, 2 - z$; (iii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{5}{2} - z$; (iv) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$.

Compound (III)

Crystal data

$C_{13}H_{18}O_4$
$M_r = 238.27$
Monoclinic
$P2_1/n$
$a = 12.1275$ (12) Å
$b = 7.5965$ (7) Å
$c = 13.9890$ (14) Å
$\beta = 96.006$ (9)°
$V = 1281.7$ (2) Å^3
$Z = 4$
$D_x = 1.235$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 5.37–12.54$°
$\mu = 0.091$ mm^{-1}
$T = 293$ (2) K
Block
0.68 × 0.48 × 0.42 mm
Colourless

Data collection

Siemens $P4$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3812 measured reflections
2930 independent reflections
1759 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$

$\theta_{\rm max} = 27.50$°
$h = -1 \rightarrow 15$
$k = -1 \rightarrow 9$
$l = -18 \rightarrow 18$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.132$
$S = 0.965$
2930 reflections
227 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0745P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$

$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.016 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 3. *Selected geometric parameters (Å, °) and hydrogen-bonding geometry (Å, °) for (III)*

O1—C7	1.218 (2)	O4—C13	1.421 (2)
O2—C2	1.363 (2)	C1—C7	1.498 (2)
O2—C11	1.421 (2)	C7—C8	1.500 (2)
O3—C4	1.358 (2)	C8—C9	1.517 (2)
O3—C12	1.431 (2)	C9—C10	1.504 (3)
O4—C5	1.378 (2)		
C2—O2—C11	118.49 (12)	O1—C7—C8	119.56 (14)
C4—O3—C12	117.68 (12)	C1—C7—C8	121.87 (11)
C5—O4—C13	116.57 (14)	C7—C8—C9	113.19 (12)
O1—C7—C1	118.57 (14)	C10—C9—C8	112.91 (15)

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12C···O3$^{\rm i}$	0.99 (2)	2.54 (2)	3.415 (2)	147 (2)

Symmetry code: (i) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$.

All three structures were solved by direct methods and refined by full-matrix least-squares techniques. The H atoms were located from difference Fourier maps and refined isotropically.

Data collection, cell refinement and data reduction: *XSCANS* (Siemens, 1994). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Geometrical calculations: *PARST* (Nardelli, 1983).

The authors would like to thank the Malaysian Government and the Universiti Sains Malaysia for research grant R&D No. 160-9609-2801 and short-term grant No. 131-0250-0530. KC thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1333). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1862–1864

5,6,8-Trichloro-4-phenyl-3,4-dihydroquinolin-2(1*H*)-one

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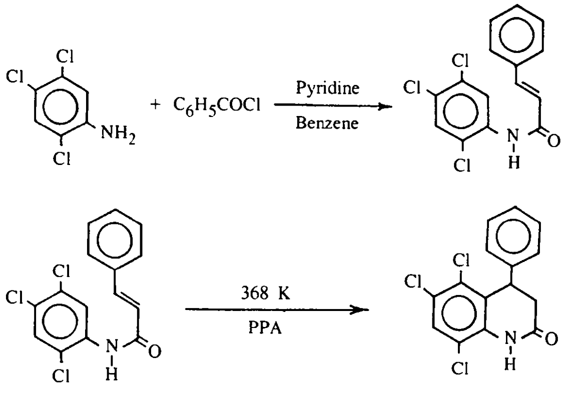
(*Received 18 November 1996; accepted 13 June 1997*)

Abstract

In the quinolone ring system of the title compound, $C_{15}H_{10}Cl_3NO$, the pyridone moiety assumes a slightly twisted boat conformation. The plane of the benzene ring is twisted at an angle of 11.9 (1)° with respect to the pyridone ring, indicating the slight non-planarity of the system. The molecules are stabilized by N—H···O-type hydrogen bonds in addition to van der Waals interactions.

Comment

The quinolones are a new class of antibiotics with therapeutic properties and potential attributable to their broad spectrum of antimicrobial activity (Fitton, 1992; McCarter, Mazens Sullivan & Bartlet, 1992). Quinolone antibacterial agents have been shown to be inhibitors of DNA gyrase, a bacterial topoisomerase II enzyme. Quinolones possess good activity *in vivo* against both systemic tumour and subcutaneously implanted human tumour xenografts (Chu *et al.*, 1992). In the treatment of asthma, urinary infection and tuberculosis, quinolones play an important role (Dahl & Haahela, 1992; Reeves, Bywater, Holt & White, 1984).



An *ORTEP*II (Johnson, 1976) plot of the molecule with 50% probability displacement ellipsoids and the atomic numbering is shown in Fig. 1. When compared with the N—C_{sp^3} single-bond distance of 1.452 Å (Ammon, Mazzochi, Regan & Colicelli, 1979), the bond distances N1—C2 [1.394 (4) Å] and N1—C9 [1.397 (4) Å] are shorter, exhibiting the partial double-bond character (Chinnakali, Sivakumar, Natarajan, McGuire & Clearfield, 1991; Kido & Nakagawa, 1982). The C2—O1 bond length is 1.213 (5) Å, indicative of a double bond, which is a unique feature in quinolone compounds (Baenzier & Wawzonek, 1984).

In the quinolone ring system, the pyridone moiety assumes a slightly twisted boat conformation. This kind of feature is uncommon in quinolone ring systems.

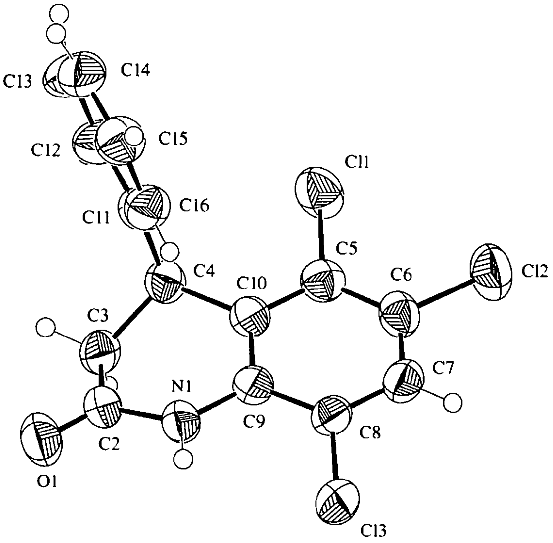


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the title molecule showing 50% probability displacement ellipsoids.

Acta Crystallographica Section C
ISSN 0108-2701 © 1997