

2-Methoxynaphthalene-1,4-dione

Bo Jin,^a Zhong-Cheng Song,^b Fu-Sheng Jiang,^a Wen-Hong Liu^b and Zhi-Shan Ding^{a*}^aDepartment of Life Science, Zhejiang Traditional Chinese Medicine University, Hangzhou 310053, People's Republic of China, and ^bBioengineering Department, Zhejiang Traditional Chinese Medicine University, Hangzhou 310053, People's Republic of China

Correspondence e-mail: zjtcmbio@163.com

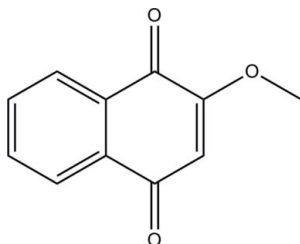
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.056; wR factor = 0.136; data-to-parameter ratio = 16.3.

The title compound, $\text{C}_{11}\text{H}_8\text{O}_3$, was isolated from *Impatiens balsamina* plants (balsam, LIB) grown in our laboratory. The two six-membered rings of the naphthalene-1,4-dione unit are coplanar [maximum deviation = 0.009 (1) Å]. The O and C atoms of the methoxy substituent also lie close to the naphthalene plane, with deviations of 0.0090 (2) and 0.047 (2) Å, respectively.

Related literature

For background to compounds extracted from *Impatiens balsamina*, see: Ding *et al.* (2008). For the antimicrobial activity of flavonol and naphthoquinone derivatives, see: Yang *et al.* (2001). For their anti-anaphylaxis properties, see: Yoshimi *et al.* (2003); Ishiguro *et al.* (1994) and for their use as anti-inflammatories, see: Hisae & Kyoko (2002). For standard bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

| | |
|-------------------------------------|-----------------------------------|
| $\text{C}_{11}\text{H}_8\text{O}_3$ | $V = 860.1$ (12) Å ³ |
| $M_r = 188.17$ | $Z = 4$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a = 3.904$ (3) Å | $\mu = 0.11$ mm ⁻¹ |
| $b = 7.662$ (6) Å | $T = 296$ K |
| $c = 28.81$ (2) Å | $0.20 \times 0.20 \times 0.10$ mm |
| $\beta = 93.562$ (7)° | |

Data collection

| | |
|---|--|
| Enraf–Nonius CAD-4 diffractometer | 2082 independent reflections |
| Absorption correction: ψ scan (North <i>et al.</i> , 1968) | 1458 reflections with $I > 2\sigma(I)$ |
| $T_{\min} = 0.979$, $T_{\max} = 0.989$ | $R_{\text{int}} = 0.032$ |
| 7068 measured reflections | 3 standard reflections every 200 reflections |
| | intensity decay: 1% |

Refinement

| | |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.056$ | 128 parameters |
| $wR(F^2) = 0.136$ | H-atom parameters constrained |
| $S = 1.08$ | $\Delta\rho_{\max} = 0.19$ e Å ⁻³ |
| 2082 reflections | $\Delta\rho_{\min} = -0.25$ e Å ⁻³ |

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5115).

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supplementary materials

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2-Methoxynaphthalene-1,4-dione

B. Jin, Z.-C. Song, F.-S. Jiang, W.-H. Liu and Z.-S. Ding

Comment

Modern chemical and pharmacological studies have identified flavonol and naphthoquinone derivatives, some of which have strong antimicrobial (Yang *et al.*, 2001) anti-anaphylaxis (Yoshimi *et al.*, 2003, Ishiguro *et al.*, 1994) and anti-inflammatory properties (Hisae & Kyoko, 2002). We have purified and identified an active component of the *Impatiens balsamina* plant (balsam, LIB) which was grown in our laboratory (Ding *et al.*, 2008) and authenticated by Professor Yao Zhensheng (Zhejiang Traditional Chinese Medicinal University). The molecular structure of the title compound is shown in Fig. 1. In the crystal, all bond lengths are within normal ranges (Allen *et al.*, 1987). The C1...C4,C9,C10 and C5...C10 rings of the naphthalene-1,4-dione unit are co-planar, maximum deviation 0.009 (1) Å. The O2 and C11 atoms of the methoxy substituent also lie close to the naphthalene plane with deviations of 0.0090 (2) Å and 0.047 (2) Å respectively.

Experimental

Dried leaves (200 g) of *Impatiens balsamina* were crushed, soaked with 55% alcohol (1500 ml) for 24 h and then reflux extracted for 40 min (1500 ml³). Extracts were filtered and vacuum evaporated. In addition, 200 g of dried leaves were directly reflux extracted using chloroform (3000 ml²). Next these extracts were filtered, combined, vacuum evaporated and the residue dried for further use. A portion of residue was re-chromatographed on silica gel using a petroleum ether-acetone (8:2) system and the isolated product was recrystallized from chloroform to yield the active component as light yellow crystals.

Refinement

H atoms were positioned geometrically and refined using the riding-model approximation, with C—H = 0.93–0.97 Å, O—H = 0.82 Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Figures

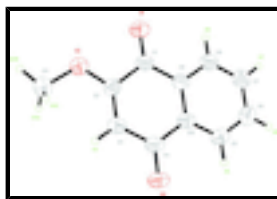


Fig. 1. The molecular structure of the title compounds with atom labels and 50% probability displacement ellipsoids for non-hydrogen atoms.

2-Methoxynaphthalene-1,4-dione

Crystal data

C₁₁H₈O₃

$F(000) = 392$

supplementary materials

$$M_r = 188.17$$

Monoclinic, $P2_1/c$

Hall symbol: -p 2ybc

$$a = 3.904 (3) \text{ \AA}$$

$$b = 7.662 (6) \text{ \AA}$$

$$c = 28.81 (2) \text{ \AA}$$

$$\beta = 93.562 (7)^\circ$$

$$V = 860.1 (12) \text{ \AA}^3$$

$$Z = 4$$

$$D_x = 1.453 \text{ Mg m}^{-3}$$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1421 reflections

$$\theta = 3.0\text{--}26.7^\circ$$

$$\mu = 0.11 \text{ mm}^{-1}$$

$$T = 296 \text{ K}$$

Block, yellow

$$0.20 \times 0.20 \times 0.10 \text{ mm}$$

Data collection

Enraf-Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube
graphite

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968.

$$T_{\min} = 0.979, T_{\max} = 0.989$$

7068 measured reflections

2082 independent reflections

1458 reflections with $I > 2\sigma(I)$

$$R_{\text{int}} = 0.032$$

$$\theta_{\max} = 28.3^\circ, \theta_{\min} = 2.8^\circ$$

$$h = -5 \rightarrow 5$$

$$k = -10 \rightarrow 10$$

$$l = -38 \rightarrow 36$$

3 standard reflections every 200 reflections

intensity decay: 1%

Refinement

Refinement on F^2

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.056$$

$$wR(F^2) = 0.136$$

$$S = 1.08$$

2082 reflections

128 parameters

0 restraints

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

$$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 0.5448P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.19 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\min} = -0.25 \text{ e \AA}^{-3}$$

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|------------|--------------|-------------|----------------------------------|
| O2 | 0.5248 (4) | 1.29754 (19) | 0.09145 (5) | 0.0460 (4) |
| C10 | 0.8859 (5) | 0.7924 (3) | 0.12534 (7) | 0.0351 (5) |
| C1 | 0.6106 (5) | 1.0740 (3) | 0.14687 (7) | 0.0389 (5) |
| C9 | 0.7444 (5) | 0.8979 (3) | 0.15900 (7) | 0.0347 (4) |
| C2 | 0.6487 (5) | 1.1357 (3) | 0.09790 (7) | 0.0369 (5) |
| O3 | 1.0374 (5) | 0.7687 (2) | 0.04711 (5) | 0.0549 (5) |
| C5 | 1.0035 (6) | 0.6270 (3) | 0.13751 (8) | 0.0423 (5) |
| H5 | 1.0976 | 0.5566 | 0.1153 | 0.051* |
| O1 | 0.4735 (5) | 1.1666 (2) | 0.17443 (6) | 0.0636 (5) |
| C3 | 0.7874 (5) | 1.0345 (3) | 0.06610 (7) | 0.0393 (5) |
| H3 | 0.8047 | 1.0774 | 0.0361 | 0.047* |
| C6 | 0.9824 (6) | 0.5657 (3) | 0.18238 (8) | 0.0483 (6) |
| H6 | 1.0607 | 0.4542 | 0.1902 | 0.058* |
| C4 | 0.9122 (5) | 0.8589 (3) | 0.07713 (7) | 0.0384 (5) |
| C7 | 0.8444 (6) | 0.6705 (3) | 0.21567 (8) | 0.0482 (6) |
| H7 | 0.8321 | 0.6295 | 0.2459 | 0.058* |
| C8 | 0.7251 (6) | 0.8354 (3) | 0.20418 (7) | 0.0426 (5) |
| H8 | 0.6317 | 0.9049 | 0.2266 | 0.051* |
| C11 | 0.5484 (6) | 1.3721 (3) | 0.04595 (8) | 0.0508 (6) |
| H11A | 0.7849 | 1.3784 | 0.0388 | 0.076* |
| H11B | 0.4516 | 1.4874 | 0.0454 | 0.076* |
| H11C | 0.4243 | 1.3005 | 0.0233 | 0.076* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|-------------|
| O2 | 0.0583 (10) | 0.0385 (8) | 0.0422 (8) | 0.0075 (7) | 0.0122 (7) | -0.0003 (7) |
| C10 | 0.0342 (11) | 0.0366 (11) | 0.0347 (10) | -0.0011 (8) | 0.0035 (8) | -0.0051 (8) |
| C1 | 0.0426 (12) | 0.0375 (11) | 0.0379 (11) | -0.0023 (9) | 0.0124 (9) | -0.0080 (9) |
| C9 | 0.0341 (10) | 0.0352 (10) | 0.0354 (10) | -0.0059 (8) | 0.0057 (8) | -0.0060 (8) |
| C2 | 0.0383 (11) | 0.0331 (11) | 0.0395 (11) | -0.0010 (8) | 0.0053 (9) | -0.0030 (8) |
| O3 | 0.0747 (12) | 0.0500 (10) | 0.0418 (9) | 0.0127 (9) | 0.0185 (8) | -0.0096 (7) |
| C5 | 0.0440 (12) | 0.0378 (11) | 0.0453 (12) | 0.0035 (9) | 0.0037 (9) | -0.0076 (9) |
| O1 | 0.0953 (14) | 0.0488 (10) | 0.0502 (10) | 0.0166 (9) | 0.0325 (9) | -0.0034 (8) |
| C3 | 0.0444 (12) | 0.0416 (11) | 0.0324 (10) | 0.0007 (9) | 0.0070 (9) | -0.0023 (9) |
| C6 | 0.0534 (14) | 0.0383 (12) | 0.0528 (14) | 0.0013 (10) | -0.0003 (11) | 0.0024 (10) |
| C4 | 0.0372 (11) | 0.0406 (12) | 0.0379 (11) | -0.0007 (9) | 0.0076 (9) | -0.0091 (9) |
| C7 | 0.0553 (14) | 0.0495 (13) | 0.0400 (12) | -0.0014 (11) | 0.0037 (10) | 0.0053 (10) |
| C8 | 0.0473 (13) | 0.0440 (12) | 0.0375 (11) | -0.0036 (10) | 0.0102 (9) | -0.0049 (9) |
| C11 | 0.0607 (15) | 0.0482 (13) | 0.0442 (13) | 0.0094 (11) | 0.0090 (11) | 0.0078 (10) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|--------|-----------|-------|-----------|
| O2—C2 | 1.340 (3) | C5—H5 | 0.9300 |
| O2—C11 | 1.438 (3) | C3—C4 | 1.459 (3) |

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| | | | |
|--------------|--------------|---------------|-------------|
| C10—C5 | 1.386 (3) | C3—H3 | 0.9300 |
| C10—C9 | 1.402 (3) | C6—C7 | 1.385 (3) |
| C10—C4 | 1.489 (3) | C6—H6 | 0.9300 |
| C1—O1 | 1.213 (2) | C7—C8 | 1.380 (3) |
| C1—C9 | 1.481 (3) | C7—H7 | 0.9300 |
| C1—C2 | 1.504 (3) | C8—H8 | 0.9300 |
| C9—C8 | 1.394 (3) | C11—H11A | 0.9600 |
| C2—C3 | 1.340 (3) | C11—H11B | 0.9600 |
| O3—C4 | 1.232 (2) | C11—H11C | 0.9600 |
| C5—C6 | 1.383 (3) | | |
| C2—O2—C11 | 116.82 (16) | C5—C6—C7 | 119.9 (2) |
| C5—C10—C9 | 119.40 (19) | C5—C6—H6 | 120.0 |
| C5—C10—C4 | 120.52 (18) | C7—C6—H6 | 120.0 |
| C9—C10—C4 | 120.07 (19) | O3—C4—C3 | 120.4 (2) |
| O1—C1—C9 | 122.80 (19) | O3—C4—C10 | 121.1 (2) |
| O1—C1—C2 | 120.2 (2) | C3—C4—C10 | 118.50 (17) |
| C9—C1—C2 | 116.96 (16) | C8—C7—C6 | 120.3 (2) |
| C8—C9—C10 | 119.6 (2) | C8—C7—H7 | 119.8 |
| C8—C9—C1 | 119.66 (18) | C6—C7—H7 | 119.8 |
| C10—C9—C1 | 120.70 (18) | C7—C8—C9 | 120.1 (2) |
| O2—C2—C3 | 126.77 (19) | C7—C8—H8 | 119.9 |
| O2—C2—C1 | 111.40 (16) | C9—C8—H8 | 119.9 |
| C3—C2—C1 | 121.83 (19) | O2—C11—H11A | 109.5 |
| C6—C5—C10 | 120.6 (2) | O2—C11—H11B | 109.5 |
| C6—C5—H5 | 119.7 | H11A—C11—H11B | 109.5 |
| C10—C5—H5 | 119.7 | O2—C11—H11C | 109.5 |
| C2—C3—C4 | 121.89 (19) | H11A—C11—H11C | 109.5 |
| C2—C3—H3 | 119.1 | H11B—C11—H11C | 109.5 |
| C4—C3—H3 | 119.1 | | |
| C5—C10—C9—C8 | 0.2 (3) | C4—C10—C5—C6 | 179.4 (2) |
| C4—C10—C9—C8 | -179.15 (19) | O2—C2—C3—C4 | -179.6 (2) |
| C5—C10—C9—C1 | -178.86 (19) | C1—C2—C3—C4 | -0.5 (3) |
| C4—C10—C9—C1 | 1.8 (3) | C10—C5—C6—C7 | -0.4 (3) |
| O1—C1—C9—C8 | -2.2 (3) | C2—C3—C4—O3 | 179.7 (2) |
| C2—C1—C9—C8 | 178.25 (19) | C2—C3—C4—C10 | -0.5 (3) |
| O1—C1—C9—C10 | 176.9 (2) | C5—C10—C4—O3 | 0.2 (3) |
| C2—C1—C9—C10 | -2.7 (3) | C9—C10—C4—O3 | 179.6 (2) |
| C11—O2—C2—C3 | -1.2 (3) | C5—C10—C4—C3 | -179.5 (2) |
| C11—O2—C2—C1 | 179.59 (18) | C9—C10—C4—C3 | -0.1 (3) |
| O1—C1—C2—O2 | 1.7 (3) | C5—C6—C7—C8 | 0.6 (4) |
| C9—C1—C2—O2 | -178.74 (17) | C6—C7—C8—C9 | -0.3 (3) |
| O1—C1—C2—C3 | -177.6 (2) | C10—C9—C8—C7 | -0.1 (3) |
| C9—C1—C2—C3 | 2.0 (3) | C1—C9—C8—C7 | 179.0 (2) |
| C9—C10—C5—C6 | 0.0 (3) | | |

Fig. 1

