liaisons dans le Tableau 2.* La Fig. 1 représente l'une des deux molécules de l'unité asymétrique [molécule (1)] et indique les numéros attribués à ses atomes. Pour obtenir les numéros des atomes de la molécule (2), on ajoute le nombre 20 aux numéros des atomes homologues de la molécule (1). Les cycles pyridyl et thiényl sont appelés respectivement cycles *A*1 et *B*1 ou *A*2 et *B*2 selon la molécule à laquelle ils appartiennent.

Littérature associée. Structure du *N*-(4,6-diméthylpyridin-2-yl)benzamide (Rodier, Piessard, Le Baut & Michelet, 1986); structure du *N*-éthyl-*N*-(4,6-diméthylpyridin-2-yl)benzamide (Rodier, Piessard, Le Baut & Brion, 1987); structure du *N*-(4,6-diméthylpyridin-2-yl)(furan-2-yl)carboxamide (Rodier, Cense, Robert & Le Baut, 1991); structure du *N*-(4,6-diméthylpyridin-2-yl)benzènethiocarboxamide (Rodier, Robert & Le Baut, 1992).

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* Les listes des facteurs de structure observés et calculés, des paramètres des atomes d'hydrogène, des coefficients d'agitation thermique anisotrope, des distances C—H et N—H, des angles de torsion, des distances des atomes aux plans moyens et des distances interatomiques intermoléculaires ont été déposées aux archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 54630: 24 pp.). On peut en obtenir des copies en s'adressant à: The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

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Structure of Sesamin

By Paul W. Baures, Mahmut Miski and Drake S. Eggleston*

SmithKline Beecham Pharmaceuticals, Departments of Physical and Structural Chemistry and Biomolecular Discovery, L-950, Box 1539, King of Prussia, PA 19406-0939, USA

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Abstract. Sesamin, 5,5′-(tetrahydro-1*H*,3*H*-furo[3,4-*c*]furan-1,4-diyl)bis(1,3-benzodioxole), a well known tetrahydofuranolignan, was isolated from the hexane extract of *Magnolia grandiflora* var. *rubra* in the course of fractionating its DNA damaging compounds. $C_{20}H_{18}O_6$, $M_r = 354.36$, $P2_1$, $a = 9.976\ (4)$, $b = 6.960\ (4)$, $c = 11.906\ (4)$ Å, $\beta = 93.67\ (3)°$, $V = 824.9\ (7)$ Å^3, $Z = 2$, $D_x = 1.427$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.988$ cm^{-1}, $F(000) = 372$, $T = 295$ K, $R = 0.065$, $wR = 0.086$ for 1701 observations, $I \geq 3\sigma(I)$. The optically active form was crystallized but the absolute configuration was not determined. Stereochemistry at the furan ring junction is *cisoid* and the molecule folds with an angle of 145° between the two best planes through the furan rings. The conformation of each furan ring is similar, both being twisted sofa forms. One benzodioxole ring is nearly coplanar to the attached furan ring [O3—C4—C5—C6 torsion angle 11.2 (5)°] but the other is twisted more out of the plane [O3′—C4′—C5′—C6′ torsion angle 26.7 (5)°].

Experimental. Colorless crystals of (1) were grown by slow evaporation from hexane/ethyl acetate. The data crystal had approximate dimensions 0.70 × 0.45 × 0.35 mm and was mounted with epoxy on a glass fiber. Cell constants were derived from least-squares refinement of the setting angles for 25 reflections ($30 \leq 2\theta \leq 35°$) located using the *SEARCH* routine on an Enraf–Nonius CAD-4 diffractometer equipped with Mo source and graphite monochromator. The only apparent systematic absence was 0*k*0 with *k* odd. Intensity data were collected in an ω–2θ scan mode using variable speeds (2.5–12.4° min^{-1}). Of the 2711 intensities scanned ($0 \leq h \leq 14$, $0 \leq k \leq 9$, $-16 \leq l \leq 16$, $2\theta_{\max} = 60°$), 2580 were unique after aver-

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* Author to whom correspondence should be addressed.

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Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *for sesamin, with e.s.d.'s in parentheses*

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

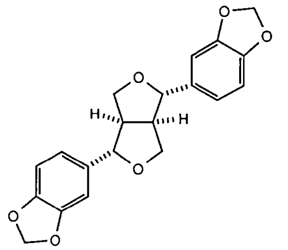
	x	y	z	B_{eq}
O3′	0.3843 (4)	−0.090	0.1059 (2)	7.52 (8)
O3	0.6274 (3)	0.0830 (5)	0.3940 (2)	6.59 (7)
O8′	0.0842 (3)	−0.6828 (5)	0.0692 (2)	4.82 (6)
O8	0.9181 (3)	0.6811 (5)	0.4305 (2)	4.63 (6)
O10′	0.0614 (3)	−0.8085 (5)	0.2471 (2)	4.91 (6)
O10	0.9194 (3)	0.8301 (5)	0.2570 (2)	4.80 (6)
C1′	0.4898 (4)	0.1338 (6)	0.2308 (3)	3.40 (6)
C1	0.4281 (3)	−0.0327 (6)	0.2967 (3)	3.36 (6)
C2	0.5331 (5)	−0.0717 (8)	0.3957 (3)	6.0 (1)
C2′	0.4684 (5)	0.0744 (8)	0.1089 (3)	6.7 (1)
C4	0.6346 (3)	0.1382 (6)	0.2797 (3)	3.43 (6)
C4′	0.4164 (4)	−0.1920 (6)	0.2089 (3)	3.48 (6)
C5′	0.3140 (3)	−0.3461 (5)	0.2247 (3)	3.07 (6)
C5	0.7068 (3)	0.3271 (6)	0.2725 (3)	3.20 (6)
C6	0.7761 (3)	0.4067 (6)	0.3673 (3)	3.38 (7)
C6′	0.2433 (3)	−0.4273 (6)	0.1289 (3)	3.53 (7)
C7	0.8440 (3)	0.5760 (6)	0.3519 (3)	3.16 (6)
C7′	0.1620 (4)	−0.5794 (6)	0.1475 (3)	3.52 (7)
C9	0.9690 (4)	0.8433 (7)	0.3712 (3)	4.52 (8)
C9′	0.0294 (4)	−0.8397 (8)	0.1299 (3)	5.00 (9)
C11′	0.1460 (4)	−0.6539 (6)	0.2538 (3)	3.41 (7)
C11	0.8463 (3)	0.6633 (6)	0.2475 (3)	3.35 (7)
C12′	0.2104 (4)	−0.5776 (6)	0.3474 (3)	3.91 (7)
C12	0.7788 (4)	0.5880 (6)	0.1538 (3)	3.98 (8)
C13′	0.2961 (4)	−0.4200 (6)	0.3310 (3)	3.40 (7)
C13	0.7087 (4)	0.4191 (7)	0.1686 (3)	4.00 (8)

Table 2. *Principal bond distances* (Å) *and angles* (°) *for sesamin, with e.s.d.'s in parentheses*

O3′—C2′	1.418 (6)	C1—C4′	1.523 (5)
O3′—C4′	1.435 (4)	C4—C5	1.505 (6)
O3—C2	1.431 (6)	C4′—C5′	1.501 (5)
O3—C4	1.420 (4)	C5′—C6′	1.419 (5)
O8′—C7′	1.377 (4)	C5′—C13′	1.388 (5)
O8′—C9′	1.437 (6)	C5—C6	1.399 (5)
O8—C7	1.367 (4)	C5—C13	1.394 (5)
O8—C9	1.441 (5)	C6—C7	1.378 (6)
O10′—C9′	1.428 (5)	C6′—C7′	1.360 (6)
O10′—C11′	1.367 (5)	C7—C11	1.385 (5)
O10—C9	1.420 (4)	C7′—C11′	1.386 (5)
O10—C11	1.371 (5)	C11′—C12′	1.358 (5)
C1′—C1	1.549 (5)	C11—C12	1.370 (5)
C1′—C2′	1.511 (5)	C12′—C13′	1.412 (6)
C1′—C4	1.523 (5)	C12—C13	1.385 (6)
C1—C2	1.550 (5)		

C2′—O3′—C4′	106.2 (3)	C4—C5—C6	120.9 (3)
C2—O3—C4	106.9 (3)	C4—C5—C13	119.1 (3)
C7′—O8′—C9′	105.8 (3)	C6—C5—C13	120.0 (4)
C7—O8—C9	106.0 (3)	C5—C6—C7	116.9 (3)
C9′—O10′—C11′	106.0 (3)	C5′—C6′—C7′	116.7 (3)
C9—O10—C11	106.5 (3)	O8—C7—C6	128.0 (3)
C1—C1′—C2′	104.2 (3)	O8—C7—C11	109.9 (3)
C1—C1′—C4	102.6 (3)	C6—C7—C11	122.1 (3)
C2′—C1′—C4	116.4 (3)	O8′—C7′—C6′	127.7 (3)
C1′—C1—C2	104.2 (3)	O8′—C7′—C11′	109.3 (3)
C1′—C1—C4′	102.2 (3)	C6′—C7′—C11′	123.0 (3)
C2—C1—C4′	114.4 (3)	O8—C9—O10	107.8 (3)
O3—C2—C1	105.7 (4)	O8′—C9′—O10′	107.9 (4)
O3′—C2′—C1′	106.9 (3)	O10′—C11′—C7′	110.4 (3)
O3—C4—C1′	104.9 (3)	O10′—C11′—C12′	127.8 (3)
O3—C4—C5	110.2 (3)	C7′—C11′—C12′	121.8 (4)
C1′—C4—C5	116.2 (3)	O10—C11—C7	109.7 (3)
O3′—C4′—C1	103.3 (3)	O10—C11—C12	128.4 (3)
O3′—C4′—C5′	110.1 (3)	C7—C11—C12	121.8 (4)
C1—C4′—C5′	117.2 (3)	C11′—C12′—C13′	116.6 (3)
C4′—C5′—C6′	119.4 (3)	C11—C12—C13	116.5 (3)
C4′—C5′—C13′	120.5 (3)	C5′—C13′—C12	121.9 (3)
C6′—C5′—C13′	119.9 (3)	C5—C13—C12	122.6 (3)

aging, $R_{int} = 0.038$. Data were corrected for Lorentz and polarization effects but not for absorption. Intensities of three standard reflections (632, 616, 318) measured every 3 h of exposure (11 times) showed a non-systematic overall decrease of 2.2%. No correction was made for deterioration.



(1)

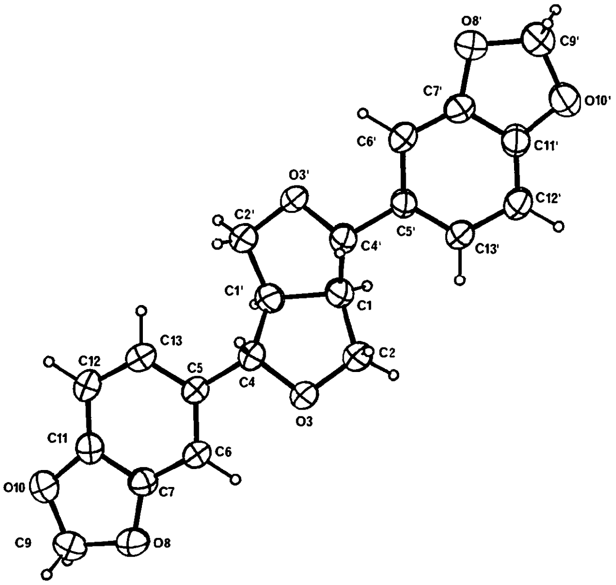


Fig. 1. Diagram of the sesamin molecule; principal ellipses are shown at the 50% probability level, H atoms as small spheres of arbitrary size.

The structure was determined using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The y coordinate of atom O3′ was held fixed to define the origin. Refinement by full-matrix least squares minimized the function $\sum w(|F_o| - |F_c|)^2$ where the weights, w, eventually were defined as $4F_o^2/\sigma^2(I)$ with $\sigma^2(I)$ defined as $[\sigma^2(I_c) + (0.071)^2]$. Non-H atoms were refined with anisotropic displacement parameters. H-atom positions were assigned based on geometric considerations assuming a C—H bond distance of 1 Å and were held fixed along with isotropic temperature factors assigned as 1.3 times the value of B_{eq} for the attached atom. The refinement converged (maximum $\Delta/\sigma = 0.01$) to values of the standard crystallographic residuals $R = 0.0646$, $wR = 0.0864$, $S = 1.724$ for 1701 observations with $I > 3\sigma(I)$, 235 variables. A correction for secondary extinction of the type described by

Zachariasen (1963) refined in the latter stages to 5.646 (1) × 10^{-6}. A final difference Fourier map showed maximum excursions of ±0.340 e Å^{-3}. Neutral atom scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV), for H atoms from Stewart, Davidson & Simpson (1965), were used as incorporated in a locally modified version of the Enraf–Nonius *SDP-Plus* (Frenz, 1987) which was the source of all programs. A refinement using all 2222 observations, not marked as weak in a prescan, led to $R = 0.082$, $wR = 0.091$, $S = 1.571$. Atomic coordinates are presented in Table 1,* bond distances and angles are listed in Table 2. A view of the molecule is provided in Fig. 1.

Related literature. Sesamin was first isolated from sesame oil. The structure and properties of sesamin have been the subject of a review article (Budowski, 1964). The structure of the related (—)-dihydrosesamin has been determined crystallographically (Lin-Gen, Seligmann, Lotter & Wagner, 1983). The molecule is known to enhance the effects of pyrethroid insecticides.

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* Lists of structure factors, anisotropic displacement parameters, bond distances and angles involving H atoms, and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54632 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0541]

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Structure of 4-Methoxybenzophenone

BY GUO SHOUWU, SU GENBO, PAN FENG AND HE YOUPING

Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian, People's Republic of China

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Abstract. $C_{14}H_{12}O_2$, $M_r = 212.25$, triclinic, $P\bar{1}$, $a = 9.636$ (5), $b = 13.906$ (4), $c = 9.293$ (4) Å, $\alpha = 108.25$ (2), $\beta = 109.91$ (2), $\gamma = 88.17$ (3)°, $V = 1108.0$ (8) Å^3, $Z = 4$, $D_x = 1.27$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.79$ cm^{-1}, $F(000) = 448$, $T = 296$ K, $R = 0.046$ for 2504 observed unique reflections with $I > 3\sigma(I)$. In one of the two crystallographically independent 4-methoxybenzophenone (MOBP) molecules the plane of the carbonyl group makes dihedral angles of 21.0 and 34.2° with the *p*-methoxyphenyl and phenyl ring planes; corresponding angles in the other MOBP molecule are slightly different: −38.5 and 24.9°.

Experimental. A colorless transparent crystal, with dimensions 1.5 × 0.7 × 0.5 mm, was grown from alcohol solution and mounted in a random orientation on a glass fiber. Data were collected with a Rigaku AFC5R diffractometer (*CONTROL* software; Molecular Structure Corporation, 1988). Cell constants were obtained by least-squares analysis of 20 diffraction maxima ($3 \leq 2\theta \leq 50°$). $\omega/2\theta$ scans, scan width $(1.260 + 0.35\tan\theta)°$, $2\theta \leq 50°$ ($0 \leq h \leq 11$, $-16 \leq k \leq 16$, $-10 \leq l \leq 10$). The intensities of 3915 unique reflections were measured. Three standard reflections were measured periodically; corrections were applied to the intensities to allow for the drop of 1.0% in the mean standard intensities during data collection. An empirical absorption correction, based on azimuthal scans of three reflections, was applied (range of transmission factors from 0.9152 to 1.0000). The data were corrected for Lorentz and polarization factors. 2504 reflections with $I \geq 3\sigma(I)$ were obtained and used in the refinement.

The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983), the C and O atoms being located in the *E* map. H atoms were placed in geometrically calculated positions with C—H = 0.95 Å, but were not included in the refinement. The structure was refined on *F* by the full-matrix least-squares technique with anisotropic thermal parameters for C, N and O atoms. Final $R = 0.046$, $wR = 0.055$ and $S = 1.35$, $w = 1/\sigma^2(F_o)$. $(\Delta/\sigma)_{\max} = 0.006$, 289 parameters; in the final difference synthesis the