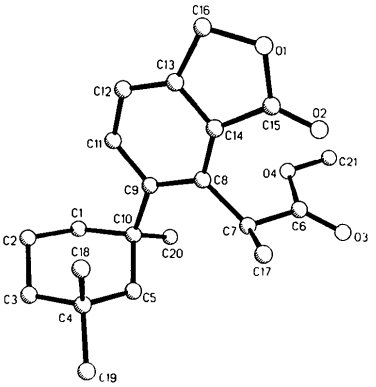


Fig. 1. Perspective drawing of the molecule with the atoms labelled according to Table 1.

0·050, $wR = 0·043$, $S = 1·92$, where $w^{-1} = \sigma^2(F)$. Final $(\Delta/\sigma)_{max} = 0·002$, $\Delta\rho_{max} = 0·20$ and $\Delta\rho_{min} = -0·23$ e Å$^{-3}$ on final difference Fourier map. Atomic scattering factors were taken from *SHELXTL-Plus* (Sheldrick, 1987). A perspective molecular drawing is shown in Fig. 1 with atoms labelled according to the tables. The absolute configuration was not established. The atomic coordinates are given in Table 1.*

**Related literature.** Studies by Dayton, Robilliard, Painc & Dayton (1974) revealed that the sponge *Dendrilla membranosa* has a chemical defence system. Molinski & Faulkner (1987) reported the isolation as an oil and the spectroscopic identification of membranolide, a metabolide related to the spongian group of diterpenes, that is likely to be a defensive constituent of *Dendrilla membranosa*. For studies of toxicity on this sponge see McClintock (1987).

* Lists of bond lengths and angles, atomic coordinates of H atoms, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53186 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* (Å$^2 \times 10^3$)

$U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| O(1) | 3005 (5) | 9054 (2) | 7596 (2) | 66 (1) |
| C(1) | 9781 (7) | 11206 (3) | 5229 (2) | 52 (2) |
| O(2) | 4281 (5) | 7808 (3) | 7060 (2) | 68 (1) |
| C(2) | 8495 (7) | 11840 (3) | 4779 (2) | 61 (2) |
| O(3) | 7696 (5) | 6803 (2) | 6320 (1) | 67 (1) |
| C(3) | 8050 (8) | 11377 (3) | 4071 (2) | 64 (2) |
| O(4) | 8654 (4) | 8059 (2) | 6947 (1) | 50 (1) |
| C(4) | 7101 (7) | 10411 (3) | 4168 (2) | 50 (2) |
| C(5) | 8392 (6) | 9793 (3) | 4634 (2) | 46 (2) |
| C(6) | 7754 (7) | 7634 (3) | 6404 (2) | 45 (2) |
| C(7) | 6976 (6) | 8347 (3) | 5872 (2) | 39 (2) |
| C(8) | 6553 (6) | 9323 (3) | 6179 (2) | 33 (2) |
| C(9) | 7414 (6) | 10159 (3) | 5939 (2) | 36 (1) |
| C(10) | 9000 (6) | 10197 (3) | 5376 (2) | 41 (2) |
| C(11) | 6850 (7) | 11005 (3) | 6275 (2) | 46 (2) |
| C(12) | 5454 (7) | 11060 (3) | 6787 (2) | 52 (2) |
| C(13) | 4623 (7) | 10246 (3) | 7006 (2) | 45 (2) |
| C(14) | 5173 (6) | 9396 (3) | 6714 (2) | 36 (2) |
| C(15) | 4191 (8) | 8645 (4) | 7106 (2) | 53 (2) |
| C(16) | 3131 (7) | 10059 (3) | 7556 (2) | 65 (2) |
| C(17) | 5305 (7) | 7932 (3) | 5457 (2) | 56 (2) |
| C(18) | 5083 (7) | 10509 (3) | 4462 (2) | 61 (2) |
| C(19) | 6957 (8) | 9929 (3) | 3439 (2) | 79 (2) |
| C(20) | 10742 (6) | 9660 (3) | 5673 (2) | 58 (2) |
| C(21) | 9544 (7) | 7448 (3) | 7452 (2) | 60 (2) |


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# Structure of 2-(2,4-Dimethoxybenzoyl)furan-3-carboxylic Acid


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**Abstract.** $C_{14}H_{12}O_6$, $M_r = 276·24$, orthorhombic, $P2_12_12_1$, $a = 18·48$ (1), $b = 9·52$ (1), $c = 7·55$ (1) Å, $V = 1328·27$ Å$^3$, $Z = 4$, $D_x = 1·380$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·7107 Å, $\mu = 0·69$ cm$^{-1}$, $F(000) = 576$, $T = 290$ K, $R = 0·061$ for 1330 unique observed [$I/\sigma(I) > 3·0$] reflexions. Internal hydrogen bonding between


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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and* $U_{eq}$ (Å$^2 \times 10^3$)

| | $x$ | $y$ | $z$ | $U_{eq}$* |
|---|---|---|---|---|
| C(2) | 6964 (2) | 3346 (5) | 1586 (5) | 56 (6) |
| C(3) | 6354 (2) | 2493 (5) | 1435 (5) | 59 (6) |
| C(4) | 5731 (2) | 3004 (5) | 611 (5) | 63 (8) |
| C(5) | 5720 (2) | 4368 (5) | −63 (5) | 54 (6) |
| C(6) | 6331 (2) | 5221 (5) | 88 (5) | 53 (6) |
| C(1) | 6953 (2) | 4710 (5) | 912 (5) | 52 (6) |
| C(7) | 7591 (3) | 5617 (7) | 1224 (7) | 48 (6) |
| O(1) | 8210 (2) | 5209 (5) | 838 (6) | 68 (5) |
| C(8) | 7500 (3) | 7013 (7) | 1973 (7) | 49 (6) |
| O(2) | 6845 (2) | 7254 (5) | 2795 (5) | 62 (4) |
| C(9) | 6871 (5) | 8578 (9) | 3476 (10) | 76 (10) |
| C(10) | 7510 (5) | 9175 (8) | 3125 (11) | 78 (9) |
| C(11) | 7929 (3) | 8162 (7) | 2162 (7) | 53 (6) |
| O(5) | 6389 (2) | 6515 (5) | −632 (5) | 61 (5) |
| O(3) | 8954 (3) | 9577 (6) | 1613 (8) | 104 (7) |
| C(13) | 5777 (4) | 7119 (8) | −1519 (12) | 94 (10) |
| O(6) | 5094 (3) | 2312 (6) | 484 (6) | 82 (5) |
| O(4) | 9052 (3) | 7364 (8) | 885 (7) | 80 (6) |
| C(12) | 8685 (4) | 8423 (10) | 1547 (10) | 67 (8) |
| C(14) | 5066 (5) | 902 (9) | 1132 (11) | 108 (11) |

*$U_{eq} = \frac{1}{3}(U_{111} + U_{222} + U_{333})$.

Table 2. *Bond distances* (Å) *and angles* (°), *including refined* H *atoms*

| | | | |
|---|---|---|---|
| C(12)—O(3) | 1·206 (8) | O(3)—C(12)—O(4) | 120·1 (8) |
| C(12)—O(4) | 1·314 (8) | O(3)—C(12)—C(11) | 121·6 (8) |
| C(12)—C(11) | 1·493 (10) | O(4)—C(12)—C(11) | 118·3 (7) |
| C(11)—C(10) | 1·434 (9) | C(12)—C(11)—C(10) | 123·4 (7) |
| C(11)—C(8) | 1·358 (8) | C(12)—C(11)—C(8) | 130·3 (6) |
| C(10)—C(9) | 1·338 (11) | C(8)—C(11)—C(10) | 106·2 (6) |
| C(9)—O(2) | 1·362 (8) | C(11)—C(10)—C(9) | 107·0 (7) |
| O(2)—C(8) | 1·380 (7) | C(10)—C(9)—O(2) | 110·5 (7) |
| C(8)—C(7) | 1·455 (8) | C(9)—O(2)—C(8) | 107·0 (6) |
| C(7)—O(1) | 1·243 (6) | O(2)—C(8)—C(11) | 109·3 (5) |
| C(7)—C(1) | 1·481 (8) | O(2)—C(8)—C(7) | 115·3 (5) |
| C(4)—O(6) | 1·353 (6) | C(11)—C(8)—C(7) | 135·2 (6) |
| O(6)—C(14) | 1·429 (8) | C(8)—C(7)—O(1) | 118·9 (6) |
| C(6)—O(5) | 1·351 (6) | C(8)—C(7)—C(1) | 120·2 (5) |
| O(5)—C(13) | 1·434 (7) | C(1)—C(7)—O(1) | 120·9 (5) |
| O(4)—H(1) | 0·88 (9) | C(4)—O(6)—C(14) | 117·7 (5) |
| C(10)—H(2) | 0·97 (7) | C(6)—O(5)—C(13) | 119·3 (4) |
| C(9)—H(3) | 1·00 (8) | | |
| O(4)···O(1) | 2·575 (9) | | |
| H(1)···O(1) | 1·72 (10) | | |

Table 3. *Deviations* (Å) *from the plane defined by* O(1), C(7), C(8), C(11), C(12) *and* O(4)

| | | | | | |
|---|---|---|---|---|---|
| C(1) | −0·4029 | C(7) | −0·0909 | O(1) | 0·0791 |
| C(8) | 0·0071 | C(11) | 0·0582 | O(3) | −0·1562 |
| O(4) | −0·0175 | H(1) | −0·1287 | C(12) | −0·0360 |

carboxyl O—H(1) and ketonic O(1) [O···O = 2·575 (9) Å] causes distortion of the exocyclic angles of the furan ring at C(8) and C(1). The molecule is twisted about the C(1)—C(7) bond such that O(2) and O(5) are separated by 2·811 (6) Å, slightly more than the sum of their van der Waals radii.

**Experimental.** The compound, prepared by reacting 3-furoic acid with 2,4-dimethoxybenzaldehyde and supplied by Dr I. G. C. Coutts in an impure form, was recrystallized from a cyclohexane/tetrahydrofuran mixture. On cooling the solution, the first crystals were fine pale yellow fibres, which on warming and standing transformed into deep yellow plates used for the structure determination. Cell dimensions were initially obtained from Weissenberg photographs [$\lambda$(Cu $K\alpha$) = 1·542 Å] and refined using 20 reflexions in the range $2\theta$ 5–40° on a Stoe Stadi-2 two-circle diffractometer (graphite-monochromated Mo $K\alpha$). The same instrument was used with a crystal of size 0·1 × 0·15 × 0·2 mm to measure 3137 intensities for the layers $hk0$ to $hk7$, $h$ −18 to 18, $k$ 0 to 10, max. $\sin\theta/\lambda$ 0·65 Å$^{-1}$. A variable $\omega$ step scan with $2\theta'$ fixed and a fixed background count of 25 s was employed. A separate standard for each layer was measured every 20 reflexions, maximum variation < 1%. Lp correction but no absorption correction. 1754 unique reflections were measured of which 1330 with $I > 3\sigma(I)$ were used in the refinement. All calculations, including the structure solution (*TANG*), were performed using *SHELX*76 (Sheldrick, 1976). H positions from $\Delta F$ map, least-squares refinement (based on $F$) of positions and $U_{ij}$ of all non-H atoms, and positions and isotropic $U$ of H(1), H(2) and H(3). Methyl H positions were refined as rigid groups with C—H = 1·08 Å and common isotropic $U$ for each group. Phenyl H atoms were included in calculated positions with C—H = 1·08 Å and refined common isotropic $U$. Interlayer scale factors were refined at an intermediate stage. Largest $\Delta/\sigma$ = 0·11 [$x$ of H(1)], variation in final $\Delta F$ map +0·32 to −0·34 e Å$^{-3}$. Final $R$ = 0·061, $wR$ = 0·067, $w = 4{\cdot}744/[\sigma^2(F) + 0{\cdot}000193F^2]$, $S$ = 2·56, 191 parameters refined, $m/n$ = 6·86. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic parameters are given in Table 1, bond distances and angles in Table 2, and deviations from the least-squares plane defined by O(1), C(7), C(8), C(11), C(12) and O(4) in

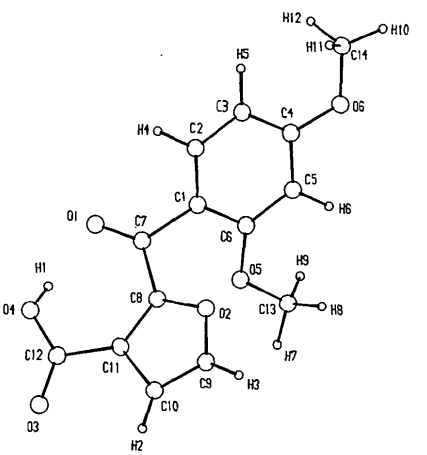
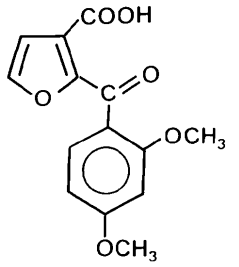



Fig. 1. One molecule of 2-(2,4-dimethoxybenzoyl)furan-3-carboxylic acid, showing the atom-numbering scheme.

Table 3.* A view of one molecule showing the atom-numbering scheme is given in Fig. 1.




**Related literature.** One other keto–acid group with chelated internal unsymmetrical hydrogen bonding has been reported in 4-(*o*-chloro-*N*-β-cyanoethylanilino)-4-oxo-but-2-enoic acid (Gonzalez-Rodriguez, Canoira, Esteban-Calderon, Martinez-Ripoll & Garcia-Blanco, 1986). There are numerous examples of symmetrical or near symmetrical internal hydrogen bonding in similar systems in dicarboxylic acid salts, *e.g.* potassium hydrogen maleate (Darlow & Cochran, 1961) and magnesium bis(hydrogen maleate) hexahydrate (Vanhouteghem, Lenstra & Schweiss, 1987).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53257 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# 1-Cyclohexyl-6-(cyclohexylimino)-1a-phenylindano[1,2-*b*]aziridine

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**Abstract.** $C_{27}H_{32}N_2$, $M_r = 384{\cdot}53$, monoclinic, $P2_1/n$, $a = 13{\cdot}519$ (1), $b = 15{\cdot}896$ (1), $c = 10{\cdot}3856$ (9) Å, $\beta = 91{\cdot}608$ (9)°, $V = 2231{\cdot}0$ (3) Å$^3$, $Z = 4$, $D_x = 1{\cdot}145$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}34$ cm$^{-1}$, $F(000) = 832$, $T = 293$ K, $R = 0{\cdot}048$ for 3380 unique observed reflections. The title compound was reported to exhibit photo- and thermochromism, and the colored species was revealed to be the tautomeric isoquinolinium imine. The indane moiety is planar and makes an angle of 111·32 (9)° with the aziridine ring. The two cyclohexyl groups adopt chair conformations. The aziridine C—C bond [1·515 (2) Å], which is cleaved on valence tautomerism, has a normal bond length.

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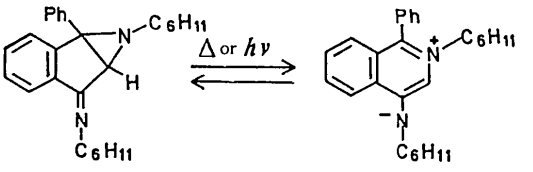
**Experimental.** The title compound was prepared by the method in the literature (Cromwell & McMaster, 1967). Recrystallization from petroleum ether gave




colorless plate-like crystals; crystal dimensions 0·5 × 0·4 × 0·3 mm, Rigaku AFC-4 diffractometer; cell parameters were determined from 21 independent $2\theta$ values ($50 < 2\theta < 63°$) with graphite-monochromated Cu $K\alpha$ radiation ($\lambda = 1{\cdot}54184$ Å); intensity