

## References

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## (2*S*,4*R*)-4-Ethoxy-2-phthalimido- $\gamma$ -butyrolactone

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**Abstract.** C<sub>14</sub>H<sub>13</sub>NO<sub>5</sub>, orthorhombic,  $P2_12_12_1$ ,  $a = 11.940$  (3),  $b = 15.725$  (3),  $c = 7.018$  (3) Å,  $Z = 4$ ,  $V = 1317.68$  Å<sup>3</sup>,  $D_c = 1.135$  Mg m<sup>-3</sup>. The structure has been refined to  $R = 0.071$  for 431 reflexions and 120 parameters. The molecular conformation may be described in terms of three planes: the phthalimido group, the lactone ring and the ethoxy side chain. The phthalimido and ethoxy planes make angles of 86.5 and 57.0° respectively with the main plane of the  $\gamma$ -butyrolactone ring. The almost planar  $\gamma$ -butyrolactone ring has substituents in positions 2 and 4 which are *trans* to each other, thus indicating that in this case the generally less stable *trans* isomer is preferred.

**Introduction.** In the continuation of our work on the stereochemistry of 2,4-disubstituted  $\gamma$ -butyrolactones (Sikirica & Vicković, 1977) we have undertaken the crystal structure analysis of the title compound.

The compound was prepared by cyclization of (2*S*)-3-formyl-2-phthalimidopropionic acid (Deljac, 1978) and recrystallized from ethanol as prismatic crystals.

The cell parameters were determined and refined from diffractometer data. The space group is  $P2_12_12_1$  with four molecules in the unit cell. The intensities were collected, at room temperature, on a Philips PW 1100/10 four-circle diffractometer (Cu  $K\alpha$  radiation,  $\lambda = 1.5418$  Å) using the  $\theta$ - $2\theta$  scanning technique:  $9.3^\circ \leq 2\theta \leq 133.4^\circ$ ; only 431 independent reflexions were measured, the option of skipping the measurement of 'weak' reflexions having been used.

An attempt to solve the structure by means of the *MULTAN* procedure (Declercq, Hull, Germain, Les-

singer, Main & Woolfson, 1976), using the measured reflexions only, was unsuccessful. The observed amplitudes were then completed by introducing the unobserved reflexions, within  $\sin \theta/\lambda = 0.6$  Å<sup>-1</sup> (66.9% of the total), with statistically evaluated amplitudes (Vicković & Viterbo, 1979) and the solution was straightforward. With 199  $E$ 's ( $E_{\min} = 1.24$ ) and 2000  $\sum_2$  relationships, 32 sets of phases were generated. The  $E$  map computed with the set having the best figures of merit revealed all 20 non-hydrogen atoms. The isotropic refinement was carried out by full-matrix least squares, using the *CRYLSQ* program of the XRAY 72 system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Following the suggestion of Hamilton (1955), the statistically evaluated reflexions were also used in the refinement, being treated as 'less-thans'. The H atoms were located in a difference map computed in the final stages of the refinement. Their positions, but not their thermal parameters, were allowed to vary in the subsequent refinement. The final discrepancy factor was  $R = 0.071$ . The weighting function  $w = 1/\sigma^2(|F_o|)$  was used, where  $\sigma^2(|F_o|)$  is the square of the standard deviation of the observed structure factor.\*

The atomic coordinates and isotropic thermal parameters as obtained from the final refinement are given in Table 1.

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34376 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates ( $\times 10^4$ , for H  $\times 10^3$ ) and isotropic temperature factors ( $\times 10^3$ ) with e.s.d.'s in parentheses

|         | x         | y         | z          | $U$ ( $\text{\AA}^2$ ) |
|---------|-----------|-----------|------------|------------------------|
| C(1)    | 7220 (9)  | 9496 (7)  | 205 (20)   | 42 (3)                 |
| C(2)    | 8135 (11) | 10008 (8) | 131 (25)   | 60 (4)                 |
| C(3)    | 7915 (12) | 10880 (9) | 166 (25)   | 64 (4)                 |
| C(4)    | 6836 (12) | 11202 (9) | 120 (26)   | 66 (4)                 |
| C(5)    | 5925 (12) | 10672 (9) | 149 (29)   | 68 (5)                 |
| C(6)    | 6133 (9)  | 9791 (6)  | 200 (21)   | 41 (3)                 |
| C(7)    | 7137 (12) | 8544 (7)  | 192 (23)   | 50 (4)                 |
| C(8)    | 5362 (11) | 9089 (8)  | 275 (23)   | 62 (4)                 |
| C(9)    | 5542 (11) | 7487 (8)  | 363 (24)   | 51 (4)                 |
| C(10)   | 5901 (13) | 7086 (8)  | 2150 (22)  | 55 (4)                 |
| C(11)   | 6509 (12) | 6185 (8)  | -175 (23)  | 57 (4)                 |
| C(12)   | 5891 (15) | 6882 (10) | -1255 (28) | 71 (5)                 |
| C(13)   | 6773 (14) | 4708 (11) | 79 (31)    | 83 (6)                 |
| C(14)   | 6258 (17) | 3858 (12) | -439 (33)  | 90 (6)                 |
| N(1)    | 5992 (8)  | 8345 (5)  | 261 (17)   | 46 (3)                 |
| O(1)    | 7859 (7)  | 8008 (5)  | 274 (15)   | 63 (3)                 |
| O(2)    | 4331 (8)  | 9075 (6)  | 369 (15)   | 75 (3)                 |
| O(3)    | 5833 (9)  | 7334 (6)  | 3761 (17)  | 84 (4)                 |
| O(4)    | 6369 (8)  | 6316 (5)  | 1809 (14)  | 67 (3)                 |
| O(5)    | 6069 (7)  | 5384 (5)  | -660 (13)  | 64 (3)                 |
| H(C2)   | 888 (9)   | 983 (6)   | 6 (18)     | 60                     |
| H(C3)   | 856 (9)   | 1124 (6)  | 38 (18)    | 64                     |
| H(C4)   | 685 (8)   | 1189 (6)  | 25 (18)    | 68                     |
| H(C5)   | 528 (9)   | 1075 (7)  | 10 (21)    | 66                     |
| H1(C12) | 529 (10)  | 671 (7)   | -139 (19)  | 66                     |
| H2(C12) | 658 (10)  | 712 (6)   | -258 (16)  | 66                     |
| H1(C13) | 668 (10)  | 466 (7)   | 190 (19)   | 78                     |
| H2(C13) | 734 (11)  | 474 (7)   | -77 (18)   | 78                     |
| H(C11)  | 750 (9)   | 621 (6)   | -82 (15)   | 58                     |
| H(C9)   | 480 (9)   | 758 (6)   | 71 (15)    | 49                     |
| H1(C14) | 688 (11)  | 353 (7)   | 55 (19)    | 88                     |
| H2(C14) | 550 (11)  | 392 (7)   | -50 (23)   | 88                     |
| H3(C14) | 605 (11)  | 383 (7)   | -219 (20)  | 88                     |

**Discussion.** Bond lengths and angles are shown in Figs. 1 and 2 and Table 2. The crystals consist of isolated molecules and the overall configuration is determined by a combination of intramolecular repulsion and crystal-packing factors. There is no interaction between the phthalimido and the ethoxy groups of different molecules. The molecular conformation (Fig. 3) is best described in terms of three planes: the phthalimido group, the  $\gamma$ -butyrolactone ring and the ethoxy side chain. The plane of the phthalimido group and the main plane through the  $\gamma$ -butyrolactone ring make angles of 32.8 and 57.0° respectively with the plane of the ethoxy side chain, while the plane of the phthalimido group and the main plane through the  $\gamma$ -butyrolactone ring make an angle of 86.5°.

The ethoxy side chain, consisting of atoms O(5), C(13), C(14) and C(11), is planar with a standard deviation of 0.008 Å. The phthalimido N atom is slightly pyramidal, being 0.013 Å out of the C(7)–C(8)–C(9) plane; the C(6)–C(8)=[O(2)]–N(1) and C(1)–C(7)=[O(1)]–N(1) fragments, centered on the  $sp^2$  C(7) and C(8), are planar within  $\pm 0.006$  Å and

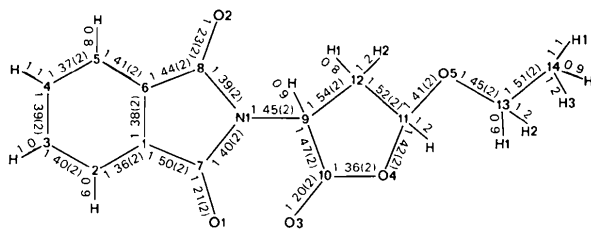


Fig. 1. The atom-labeling scheme and bond distances ( $\text{\AA}$ ) with e.s.d.'s in parentheses. For the bonds involving H atoms the e.s.d.'s are 0.1 Å.

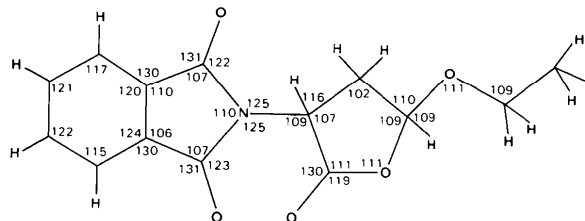


Fig. 2. Valence angles ( $^\circ$ ) involving the non-hydrogen atoms; the e.s.d.'s are 1°.

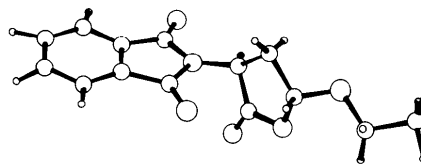


Fig. 3. A perspective view of the molecule.

$\pm 0.030$  Å respectively. These two planes make a dihedral angle of 2.5°. O(4) is significantly (0.070 Å) out of the least-squares plane through the  $\gamma$ -butyrolactone ring. The main  $\gamma$ -butyrolactone fragment defined by C(9)–C(10)=[O(13)]–O(4) is essentially planar [within  $\pm 0.002$  (1) Å].

The  $\gamma$ -butyrolactone ring is bent along the line connecting C(9) with the middle of the C(11)–O(4) bond. The dihedral angle between the plane of the above-mentioned  $\gamma$ -butyrolactone fragment and the plane defined by C(9)–C(12)–C(11) is 4.8°; this value does not agree with that found in the structure of 4-methylthio-2-phthalimido- $\gamma$ -butyrolactone (Sikirica & Vicković, 1977) in which a typical envelope conformation for the  $\gamma$ -butyrolactone ring, with a folding angle of 29.0°, was observed. The  $\gamma$ -butyrolactone substituents are in the 2 and 4 positions and, rather unexpectedly, are *trans* to each other. Although it has been generally accepted that the *trans* isomers are less stable than the *cis*, this structure analysis confirms a previous suggestion that the difference in free energies between the *cis* and *trans* isomers of the  $\gamma$ -butyrolactone envelope conformers is small (Hussain, Ollis, Smith & Stoddart, 1975).

Table 2. Bond angles involving the H atoms with *e.s.d.*'s in parentheses

|                     |                      |                     |                      |
|---------------------|----------------------|---------------------|----------------------|
| C(1)–C(2)–H(C2)     | 126 (6) <sup>o</sup> | C(3)–C(2)–H(C2)     | 119 (6) <sup>o</sup> |
| C(2)–C(3)–H(C3)     | 115 (6)              | C(4)–C(3)–H(C3)     | 122 (6)              |
| C(3)–C(4)–H(C4)     | 110 (5)              | C(5)–C(4)–H(C4)     | 128 (5)              |
| C(4)–C(5)–H(C5)     | 133 (8)              | C(6)–C(5)–H(C5)     | 110 (8)              |
| N(1)–C(9)–H(C9)     | 103 (6)              | C(10)–C(9)–H(C9)    | 97 (7)               |
| C(12)–C(9)–H(C9)    | 123 (7)              | O(4)–C(11)–H(C11)   | 90 (4)               |
| O(5)–C(11)–H(C11)   | 107 (5)              | C(12)–C(11)–H(C11)  | 105 (5)              |
| C(9)–C(12)–H1(C12)  | 122 (5)              | C(11)–C(12)–H1(C12) | 105 (5)              |
| C(9)–C(12)–H2(C12)  | 93 (9)               | C(11)–C(12)–H2(C12) | 105 (9)              |
| O(5)–C(13)–H1(C13)  | 110 (5)              | C(14)–C(13)–H1(C13) | 99 (5)               |
| O(5)–C(13)–H2(C13)  | 99 (8)               | C(14)–C(13)–H2(C13) | 101 (8)              |
| C(13)–C(14)–H1(C14) | 89 (6)               | C(13)–C(14)–H2(C14) | 109 (8)              |
| C(13)–C(14)–H3(C14) | 111 (6)              |                     |                      |

The bond lengths and angles are within the expected values and agree well with those found in the above-mentioned 4-methylthio-2-phthalimido- $\gamma$ -butyrolactone and in  $\alpha$ -(3,5-dibromo-2-hydroxybenzylidene)- $\gamma$ -butyrolactone (Koenig, Chiu, Krebs & Walter, 1969). Because of the limited number of observed data, owing to the poor quality of the crystals, the estimated standard deviations of the interatomic distances and

angles are rather large and detailed discussion of the individual bonds and angles would be meaningless.

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## *N*-(*tert*-Butylphenylfluorosilyl)-*N*-(phenyldifluorosilyl)-2,4,6-trimethylaniline

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**Abstract.**  $C_{25}H_{30}F_3NSi_2$ ,  $M_r = 457.69$ , orthorhombic, *Pbcn*,  $a = 26.999$  (7),  $b = 9.209$  (3),  $c = 19.896$  (5) Å,  $U = 4947$  Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.229$  Mg m<sup>-3</sup>,  $\mu(Cu K\alpha) = 1.5$  mm<sup>-1</sup>; final  $R = 0.063$  for 2739 unique reflexions. The bonding geometry at the N atom is approximately planar.

**Introduction.** The title compound is one of a series of bis(fluorosilyl)anilines synthesized by Klingebiel, Neemann & Meller (1978). Its <sup>19</sup>F NMR spectrum shows an *AB* pattern for the SiF<sub>2</sub> group. The structure determination was undertaken to determine the bonding geometry at the N atom.

Crystals in the form of white blocks were obtained from petroleum spirit. A crystal 0.5 × 0.3 × 0.2 mm was used to collect data on a Syntex P2<sub>1</sub> diffractometer with monochromated Cu  $K\alpha$  radiation. Cell dimensions were obtained by least squares from 15 strong high-angle reflexions. 3958 reflexions were

collected in the range  $3^\circ < 2\theta < 116^\circ$ . After application of Lp corrections, averaging equivalent reflexions gave 2743 unique reflexions with  $F > 4\sigma(F)$ . Systematic absences  $0kl$ ,  $k$  odd;  $h0l$ ,  $l$  odd; and  $hk0$ ,  $h + k$  odd indicated space group *Pbcn*.

The structure was solved by multiresolution  $\sum_2$  sign expansion with  $323 E > 1.6$ . The best *E* map showed all but four non-hydrogen atoms. The missing atoms were located by a difference synthesis. Least-squares refinement gave  $R = 0.17$  (isotropic) and 0.10 (anisotropic). H atoms were then included at calculated positions and refined with C–H 1.08 Å, H–C–H 109.5°. The methyl H atoms of the mesitylene moiety could not be refined successfully, probably because of rotational disorder, and so were omitted. Refinement terminated at  $R = 0.063$ ,  $R' = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o| = 0.070$ . The weighting scheme was  $w = 1/[\sigma^2(F) + 0.001F^2]$ . Overall temperature factors for methyl and non-methyl H refined to 0.13 (1) and 0.10 (1) Å<sup>2</sup> respectively. F