X-ray Structure Analysis Online

# Synthesis and Structural Studies of Ethylnaphtho[2,1-b]furan-2-carboxylate

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Ethyl naphtho[2,1-b]furan-2-carboxylate was synthesized and characterized by spectroscopic techniques, and the structure of the compound was confirmed by X-ray diffraction studies. The title compound crystallizes in the monoclinic crystal system and space group $P2_1/c$ with cell parameters: $a$ = 13.112(4)Å, $b$ = 5.910(1)Å, $c$ = 18.657(5)Å, $\beta$ = 123.785(6)°. The furan ring is planar. The molecule is stabilized by weak interactions.

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Many compounds involving a naphthofuran ring have attracted much attention in view of their diverse pharmacological properties, such as antibacterial, antitumor and antielmintic activities.[1] The derivatives of naphthofurans play vital roles as intermediates in the organic synthesis of number of heterocyclic pharmacological-active compounds.[2] The bioactivity of naphthofuran derivatives is desirable in the synthesis of novel condensed heterocyclic compounds.[3] They also possess a broad spectrum of biological activities that are constituents of important natural product.[4] With their varieties of pharmacological importance, a number of research activities are in focus. In view of these important properties, we report here on spectroscopic studies and the crystal structure of ethyl naphtho[2,1-b]furan-2-carboxylate using the X-ray diffraction method. The schematic diagram of the molecule is given in Fig. 1.

Spectral details: White crystalline solids. UV-Visible (SP-2102 UVPC Spectrum) $\lambda_{max}$ = 300.3; $^{13}$C-NMR ($CDCl_3$): 14.4 ($CH_3$); 61.4 ($CH_2$); 112.7 (CH); 122.7 (CH); 123.3 (CH); 125.3 (C); 127.2 (C); 128.01, (CH); 128.0, (CH); 128.9, (CH); 129.0, (CH); 130.5 (C); 145, (C); 153.9 (C); 159.5 (C=O). Synthesis, spectral data (FT-IR, and $^1$H NMR) and elemental analysis of the title compound was reported earlier.[5]

A single crystal of the title compound with dimensions of 0.30 × 0.27 × 0.25 mm was chosen for an X-ray diffraction study. The data were collected on a DIPLabo Image Plate system equipped with a normal focus, 3 kW sealed X-ray source (graphite monochromated). The crystal-detector distance was fixed at 120 mm with a detector area of 441 × 240 mm$^2$. Thirty six frames of data were collected at room temperature by an oscillation method. Each exposure of the image plate was set to a period of 400 s. Successive frames were scanned. Image processing and data reduction were conducted using Denzo. The reflections were merged with Scalepack. All of the frames could be indexed using a primitive monoclinic lattice. An absorption correction was not applied. The structure was solved by direct methods using SHELXS-97.[6] All of the non-hydrogen atoms were revealed in the first Fourier map, itself. After four

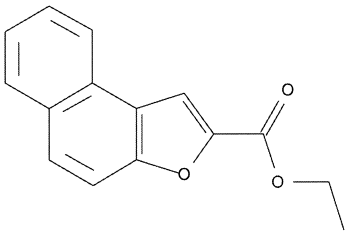

Fig. 1 Schematic diagram.

Table 1 Crystal data and structure refinement table

| | |
|---|---|
| Empirical formula | $C_{15}H_{12}O_3$ |
| Formula weight | 240.5 |
| Temperature | 293 K |
| Wavelength | 0.71073 Å |
| Crystal system | Monoclinic |
| Space group | $P2_1/c$ |
| Cell dimensions | $a$ = 13.112(4)Å |
| | $b$ = 5.910(1)Å |
| | $c$ = 18.657(5)Å |
| | $\beta$ = 123.785(6)° |
| Volume | 1201.6(5)Å$^3$ |
| $Z$ | 4 |
| Density (calculated) | 1.328 Mg/m$^3$ |
| Absorption coefficient | 0.092 mm$^{-1}$ |
| $F_{000}$ | 504 |
| Crystal size | 0.30 × 0.27 × 0.25 mm |
| Theta range for data collection | 3.7° to 23.3° |
| Index ranges | $-14 \le h \le 14$ |
| | $-5 \le k \le 6$ |
| | $-20 \le l \le 20$ |
| Reflections collected/Unique | 2612/1550 [$R_{int}$ = 0.0311] |
| Absorption correction | None |
| Refinement method | Full-Matrix least squares of $F^2$ |
| Data/restraints/parameters | 1550/0/165 |
| Goodness-of-fit on $F^2$ | 1.32 |
| Final $R$ indices | $R_1$ = 0.0929, $wR_2$ = 0.2803 |
| Extinction coefficient | 0.06(3) |
| Largest diff.peak and hole | 0.38 and –0.23 e.Å$^{-3}$ |
| CCDC Number | 851582 |

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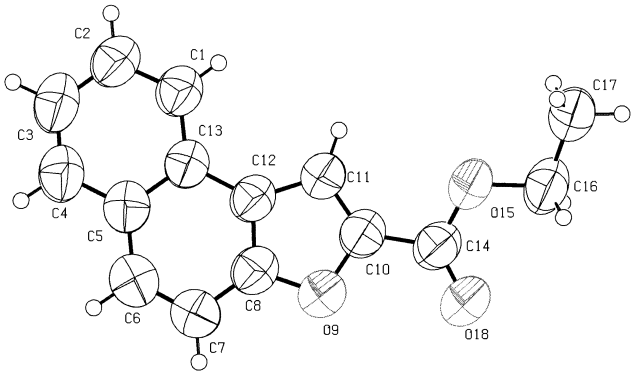


Fig. 2 ORTEP drawing of the molecule with thermal ellipsoids drawn at 50% probability.

refinements with anisotropic parameters for the non-hydrogen atoms, the residuals saturated to 0.09. The details of the crystal data and refinement are given in Table 1. Figure 2 represents the ORTEP drawing of the molecule with thermal ellipsoids drawn at 50% probability. The crystallographic data are deposited in Cambridge Crystallographic Data Center (Ref. No. CCDC 851582).

The naphthofuran moiety is essentially planar, with a mean deviation of 0.016 Å from the least-squares plane, defined by thirteen constituent atoms. This is in comparision with the value of 0.011 Å, defined for 7-bromo-1-(4-flurophenylsulfonyl)-2-methylnaphto[2,1-b]furan.[7] The torsion angle value of –173.1(4)˚ for C17–C16–O15–C14 indicates that the ethyl group is in an *antiperiplanar* orientation with the naphtho-furan moiety. In the absence of hydrogen bonds, the molecule is stabilized by intermolecular interactions of the type C–O···Centroid. C14–O18···Cg(1) [–*x*, 2–*y*, –*z*] has a length of 3.493(5)Å with a bond angle of 94.3(3)˚, and C14–O18···Cg(3) [–*x*, 2–*y*, –*z*] has a length of 3.726(4)Å with a bond angle value of 108.2(3)˚, where Cg(1) and Cg(3) are the centroids of rings defined by the atoms O9–C8–C12–C11–C10 and C5–C6–C7–C8–C12–C13, respectively. These interactions lead to the close packing of the neighboring molecules.

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