# Structure of Ethyl α-(*p*-Tolylhydrazono)-2-furanpropionate

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**Abstract.** $C_{16}H_{18}N_2O_3$, $M_r = 286.3$, monoclinic, $P2_1/c$, $a = 8.587$ (2), $b = 17.313$ (4), $c = 10.680$ (2) Å, $\beta = 110.44$ (1)°, $V = 1487.8$ Å$^3$, $Z = 4$, $D_x = 1.28$, $D_m = 1.26$ Mg m$^{-3}$, $\lambda$(Cu $K\alpha$) = 1.5418 Å, $\mu$(Cu $K\alpha$) = 0.69 mm$^{-1}$, $F(000) = 608$, $T = 300$ K, final $R = 0.063$ for 1892 observed reflections. The structure conforms to the non-hydrogen-bonded α form. The hydrazine moiety forms an extended planar zigzag chain with the neighboring atoms. Intermolecular N—H⋯O bonds link the molecules along **c**.

**Introduction.** The title compound, synthesized *via* Japp–Klingemann reaction (Holla, 1979), belongs to the general family of ethyl pyruvate phenylhydrazones and is an intermediate for the synthesis of indole and pyrazole derivatives which are of biological interest for their antitubercular activity (Ambekar & Siddappa, 1965). As a result of hydrogen bonding, it can have *anti* or *syn* conformations (Fig. 1*a,b*). The structures of two compounds belonging to this family, ethyl α-(4-chloro-2-methylphenylhydrazono)propionate (Ravindra Acharya & Sake Gowda, 1981) and ethyl α-phenylhydrazono-2-furanpropionate (Puttaraja, Sake Gowda, Gabe & Le Page, 1982), have been determined. Both compounds conform to the non-hydrogen-bonded α form. The present work is a continuation of an investigation of compounds from this family.

**Experimental.** Brownish crystals of dimensions 0.5 × 0.41 × 0.3 mm mounted on Syntex $P2_1$ diffractometer, $\theta$–$2\theta$ scan, variable scan rate, lattice parameters from least-squares refinement of 15 centered reflections, density measurements by flotation, Cu $K\alpha$ intensity data collected in the range $5.1 \le 2\theta \le 114.7°$,
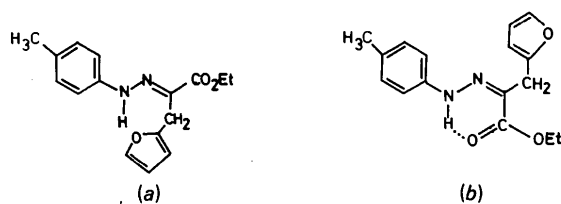
Table 1. *Positional parameters* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å$^2$) *for non-H atoms with e.s.d.'s in parentheses*

| | $x$ | $y$ | $z$ | $B_{eq}$* |
|---|---|---|---|---|
| O(1) | 798 (2) | 885 (1) | 8080 (1) | 3.72 (07) |
| O(2) | −878 (2) | 1480 (1) | 6242 (1) | 4.33 (08) |
| O(3) | 2299 (2) | 2393 (1) | 11820 (2) | 4.45 (08) |
| N(1) | −1793 (2) | 2464 (1) | 7806 (2) | 3.49 (08) |
| N(2) | −2298 (3) | 2965 (1) | 8531 (2) | 3.92 (09) |
| C(1) | −283 (2) | 1435 (1) | 7449 (2) | 3.27 (09) |
| C(2) | −670 (3) | 1958 (1) | 8389 (2) | 3.22 (09) |
| C(3) | 154 (3) | 1873 (1) | 9885 (2) | 3.67 (11) |
| C(4) | 1578 (3) | 2409 (1) | 10454 (2) | 3.56 (09) |
| C(5) | 2368 (3) | 2926 (1) | 9964 (3) | 4.33 (12) |
| C(6) | 3660 (4) | 3250 (2) | 11066 (3) | 5.19 (13) |
| C(7) | 3556 (3) | 2913 (2) | 12142 (3) | 5.11 (13) |
| C(8) | 1206 (3) | 316 (1) | 7213 (3) | 4.39 (11) |
| C(9) | −59 (4) | −313 (2) | 6817 (4) | 5.65 (14) |
| C(10) | −3518 (3) | 3513 (1) | 7889 (2) | 3.53 (10) |
| C(11) | −4227 (3) | 3565 (1) | 6514 (2) | 3.81 (10) |
| C(12) | −5420 (3) | 4122 (1) | 5935 (3) | 3.98 (10) |
| C(13) | −5943 (3) | 4636 (1) | 6710 (3) | 4.13 (11) |
| C(14) | −5232 (3) | 4570 (2) | 8090 (3) | 4.44 (11) |
| C(15) | −4043 (3) | 4022 (1) | 8684 (3) | 4.18 (11) |
| C(16) | −7213 (4) | 5258 (2) | 6085 (4) | 5.17 (13) |

\* $B_{eq}$ is the arithmetic mean of the principal axes of the thermal ellipsoids.

2071 independent reflections, $h$ 0 to 8, $k$ 0 to 16, $l$ −10 to 10, 1892 reflections with $I \ge 2\sigma(I)$ considered observed, data corrected for Lorentz and polarization effects, no absorption correction. Standard reflections showed no systematic variation. Structure solved by direct methods using *SHELX*76 (Sheldrick, 1976). All the non-H atoms located from the *E* map and subsequent difference Fourier maps. All the H atoms except the methyl H atoms located from the difference map, the methyl H fixed from geometrical considerations. Full-matrix least-squares refinement (on *F*) with anisotropic thermal parameters for non-H atoms and isotropic for H atoms. Final $R = 0.063$ and $wR = 0.082$, $w = [\sigma^2(F) + 0.0264F^2]^{-1}$, $\sigma$'s based on counting statistics, refinement was terminated when the maximum shift/e.s.d. for non-H atoms was 0.2, the minimum and maximum residual electron densities in the final difference Fourier map were −0.05 and 0.19 e Å$^{-3}$. Scattering factors for C, N and O are from Cromer & Mann (1968) and for H from Stewart, Davidson & Simpson (1965).



Fig. 1. (*a*) Non-hydrogen-bonded (α form) and (*b*) hydrogen-bonded (β form) molecules.
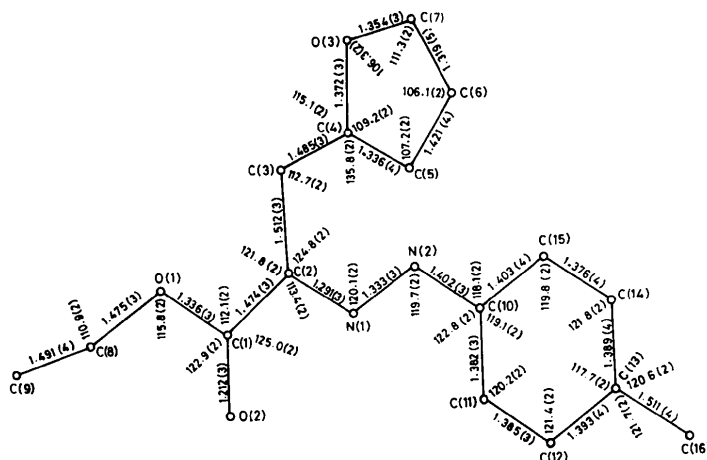
Fig. 2. Molecule viewed along a with bond lengths (Å) and angles (°). The e.s.d.'s are in parentheses.

**Discussion.** The final positional parameters are given in Table 1.* The molecule viewed along a with bond lengths and angles is shown in Fig. 2.

The structure obtained confirms the presence of the non-hydrogen-bonded α form of the molecule in the solid state. The hydrazine moiety forms an extended planar zigzag chain with neighbors C(2), C(1) and O(1).

The geometry of the phenyl ring is normal with bond lengths and angles having average values 1·388 (4) Å and 120·0 (2)° respectively. The methyl C atom attached to the ring has a deviation of 0·053 (4) Å from the mean plane of the ring. In the furan ring the C=C and C—O bonds have average values of 1·328 (5) and 1·363 (3) Å respectively, while the C—C bond length is 1·421 (4) Å. The ring is essentially planar and makes a dihedral angle of 85·9° with the mean plane of the phenyl ring.

The bond lengths in the propionate group are all normal and agree with the standard values associated with $C(sp^2)$—$C(sp^3)$, $C(sp^2)$—$C(sp^2)$ and $C(sp^2)$—O single-bond distances. The alcohol α-C atom, C(8), is syn to C=O about the C—O bond and the phenyl-hydrazono group is trans to ethoxycarbonyl group about the N(1)—C(2) bond; the C(1)—C(2)—N(1)—N(2) torsion angle is 178·5 (2)°.

The molecules are held together by hydrogen bonding along c between N(2) and O(2) of the molecule at x, ½−y, ½+z [N(2)···O(2) = 2·88 Å and N(2)—HN(2)···O(2) = 162·5°]. All other contacts are of van der Waals type. The molecular packing viewed along c is shown in Fig. 3.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44834 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
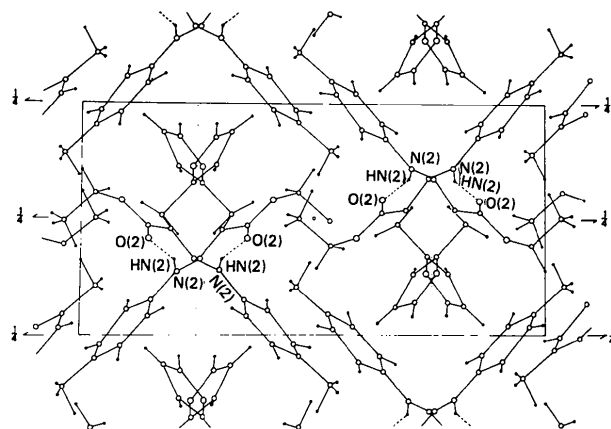


Fig. 3. Molecular packing viewed down c.

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