

1-methyl-4-phenylpiperidine hydrochloride (Tillack, Seccombe, Kennard & Oh, 1974) exhibit the normal chair conformation for the piperidine ring, as does the title compound. The bond distances C6—C7 and C8—C9 in the title compound [1.321 (10) and 1.336 (10) Å] are similar to those in 9-(2,4-cyclopentadienylidene)bicyclo[3.3.1]nonane (Fronczek, Garcia & McLaughlin, 1990) at 1.340 (1) and 1.339 (2) Å, respectively.

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Structure of 3-Ethyl-4-oxa-1,5,6-trihydrophthalimide

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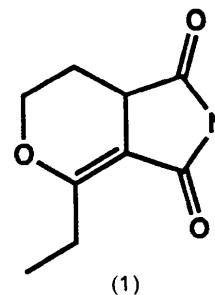
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Abstract. 4-Ethyl-1,3,6,7-tetrahydro-5H-pyrano[3,4-c]pyrrole-1,3-dione, C₉H₁₁NO₃, *M_r* = 181.19, monoclinic, *P*2₁/*c*, *a* = 12.262 (3), *b* = 8.027 (2), *c* = 8.596 (1) Å, β = 92.36 (2)°, *V* = 845.4 (3) Å³, *Z* = 4, *D_x* = 1.42 g cm⁻³, λ(Mo *K*α) = 0.7107 Å, μ = 1.2 cm⁻¹, *F*(000) = 384, *T* = 165 K, *R* = 0.050 (*wR* = 0.075) for 1365 unique, observed reflections. The compound is a derivative of phthalimide substituted in the six-membered ring by an ether oxygen and an ethyl group.

Experimental. Crystals (colorless prisms) of C₉H₁₁NO₃ [hereafter (1)] obtained from an ethyl acetate/hexane solution by Sean Esslinger and Professor Robert M. Williams (Colorado State University). Crystal dimensions 0.47 × 0.60 × 0.44 mm. Nicolet *R3m* diffractometer, unit-cell constants from least-squares fit of setting angles for 25 reflections (2θ_{av} = 31.40°). Data collected (θ/2θ scans) to (sinθ)/λ = 0.5947 Å⁻¹, -11 ≤ *h* ≤ 11, 0 ≤ *k* ≤ 10, 0 ≤ *l* ≤ 15. Three standard reflections (400, 020, 005) measured every 97 reflections, no trend in intensity observed; Lorentz-polarization corrections;

no absorption correction applied due to low absorption coefficient; 1623 unique reflections, 1365 reflections with *F_o* > 4.0σ(*F_o*) observed.



Structure solved by direct methods (*SOLV*; Sheldrick, 1983); block-diagonal (max. 103 parameters/block, 125 parameters total, data/parameters = 10.9) weighted {*w* = [σ²(*F*) + *gF*²]⁻¹, *g* = 1.37 × 10⁻³} least-squares refinement on *F*. H atoms in idealized positions [C—H = 0.96 Å, *U*(H) = 1.2 × *U*_{iso}(C)] with the exception of the H atom on N(1) (located in difference map and refined with isotropic thermal parameters). Non-H atoms refined with anisotropic thermal parameters. At convergence [(Δ/σ)_{max} =

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Table 1. Atomic coordinates and isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) for (1)

| | x | y | z | U |
|-------|-------------|-------------|--------------|---------|
| O(1) | 0.8387 (1) | 0.0476 (2) | 0.1339 (2) | 25 (1)* |
| O(2) | 0.4994 (1) | 0.1669 (1) | -0.2315 (1) | 20 (1)* |
| O(3) | 0.7062 (1) | 0.5532 (1) | 0.0324 (2) | 28 (1)* |
| N(1) | 0.5860 (1) | 0.3846 (2) | -0.1072 (2) | 18 (1)* |
| HN(1) | 0.5482 (18) | 0.4697 (27) | -0.1692 (26) | 37 (6) |
| C(1) | 0.7610 (1) | -0.0827 (2) | 0.0872 (2) | 22 (1)* |
| C(2) | 0.6892 (1) | -0.0350 (2) | -0.0535 (2) | 20 (1)* |
| C(3) | 0.6292 (1) | 0.1226 (2) | -0.0094 (2) | 17 (1)* |
| C(4) | 0.5644 (1) | 0.2199 (2) | -0.1312 (2) | 16 (1)* |
| C(5) | 0.6734 (1) | 0.4145 (2) | 0.0012 (2) | 17 (1)* |
| C(6) | 0.7096 (1) | 0.2496 (2) | 0.0534 (2) | 17 (1)* |
| C(7) | 0.8085 (1) | 0.2071 (2) | 0.1150 (2) | 19 (1)* |
| C(8) | 0.8971 (1) | 0.3259 (2) | 0.1676 (2) | 24 (1)* |
| C(9) | 1.0078 (2) | 0.2849 (3) | 0.1055 (3) | 37 (1)* |

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_i tensor.

Table 2. Bond lengths (\AA) and angles ($^\circ$) for (1) with *e.s.d.*'s in parentheses

| | | | |
|-----------------|------------|-----------------|------------|
| O(1)—C(1) | 1.459 (2) | O(1)—C(7) | 1.340 (2) |
| O(2)—C(4) | 1.227 (2) | O(3)—C(5) | 1.210 (2) |
| N(1)—HN(1) | 0.972 (22) | N(1)—C(4) | 1.363 (2) |
| N(1)—C(5) | 1.412 (2) | C(1)—C(2) | 1.515 (2) |
| C(2)—C(3) | 1.519 (2) | C(3)—C(4) | 1.506 (2) |
| C(3)—C(6) | 1.503 (2) | C(5)—C(6) | 1.460 (2) |
| C(6)—C(7) | 1.348 (2) | C(7)—C(8) | 1.501 (2) |
| C(8)—C(9) | 1.515 (3) | | |
| C(1)—O(1)—C(7) | 118.5 (1) | HN(1)—N(1)—C(4) | 120.9 (13) |
| HN(1)—N(1)—C(5) | 125.1 (13) | C(4)—N(1)—C(5) | 113.7 (1) |
| O(1)—C(1)—C(2) | 112.9 (1) | C(1)—C(2)—C(3) | 106.4 (1) |
| C(2)—C(3)—C(4) | 120.3 (1) | C(2)—C(3)—C(6) | 109.7 (1) |
| C(4)—C(3)—C(6) | 102.6 (1) | O(2)—C(4)—N(1) | 124.0 (1) |
| O(2)—C(4)—C(3) | 128.2 (1) | N(1)—C(4)—C(3) | 107.7 (1) |
| O(3)—C(5)—N(1) | 122.6 (1) | O(3)—C(5)—C(6) | 132.2 (2) |
| N(1)—C(5)—C(6) | 105.2 (1) | C(3)—C(6)—C(5) | 108.6 (1) |
| C(3)—C(6)—C(7) | 122.4 (1) | C(5)—C(6)—C(7) | 127.4 (1) |
| O(1)—C(7)—C(6) | 121.9 (1) | O(1)—C(7)—C(8) | 112.2 (1) |
| C(6)—C(7)—C(8) | 125.9 (1) | C(7)—C(8)—C(9) | 113.8 (2) |

0.036, $(\Delta/\sigma)_{\text{mean}} = 0.011$ for last two cycles] $R = 0.050$, $wR = 0.075$, $S = 1.81$, slope of normal probability plot = 1.56, $(\Delta\rho)_{\text{max}} = 0.40$, $(\Delta\rho)_{\text{min}} = -0.32 \text{ e \AA}^{-3}$. Neutral-atom scattering factors and anomalous-dispersion corrections used (*International Tables for X-ray Crystallography*, 1974, Vol. IV); all calculations performed using the *SHELXTL* program library (Sheldrick, 1983). Table 1 gives atomic coordinates and Table 2 gives bond lengths and angles.* Fig. 1 shows the structure of (1) and the atom-numbering scheme used.

Related literature. Phthalimide and seven derivatives of phthalimide have been studied previously: phthalimide (Matzat, 1972), 5-methyl-1,3,4,6-tetra-

* Lists of anisotropic thermal parameters, H-atom coordinates and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53989 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

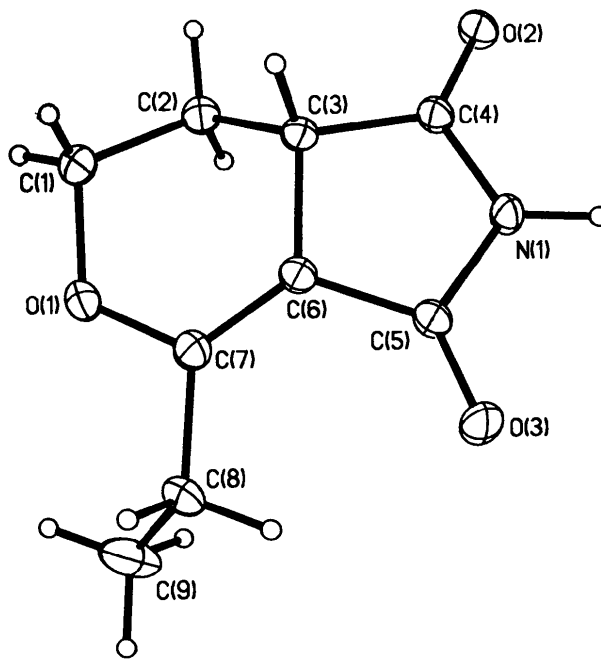


Fig. 1. The structure of (1) (50% probability thermal ellipsoids).

oxaperhydropyrrolo[3,4-*c*]pyridine (Amorese, Gavuzzo, Mazza, Casini & Ferappi, 1982), 4-(4'-*N*-diethylaminophenylazo)phthalimide (Golinski, 1985), 3-(4-diethylaminophenylazo)phthalimide (Golinski, 1988), 1,2,3,6-tetrahydrophthalimide (Kirfel, 1976), pyromellitic di-imide (Bulgarovskaya, Novakovskaya, Federov & Zvonkova, 1976), 3,4,5,6-tetrahydrophthalimide (Kirfel, 1975), and 3,6-dithia-3,4,5,6-tetrahydrophthalimide (Kirfel, Will & Fickentscher, 1975). In the structure of (1), hydrogen bonds from the amide atom N(1) to the amide atom O(2) more distant from the ethyl group link molecules of (1) into chains [N(1)—O(2)ⁱ, 2.834 (2), O(2)—HN(1)ⁱ, 1.88 (2) \AA , N(1)—HN(1)—O(2)ⁱ, 167 (2)^o].

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