

Lars Kr. Hansen,^{a*} German L.
Perlovich^{b,c} and Annette
Bauer-Brandl^b^aDepartment of Chemistry, University of Tromsø, 9037 Tromsø, Norway, ^bDepartment of Pharmaceutics and Biopharmaceutics, University of Tromsø, 9037 Tromsø, Norway, and ^cInstitute of Solution Chemistry, Russian Academy of Sciences, 153045 Ivanovo, Russian Federation

Correspondence e-mail: larsk@chem.uit.no

Key indicators

Single-crystal X-ray study

T = 298 K

Mean $\sigma(C-C)$ = 0.002 Å

R factor = 0.039

wR factor = 0.111

Data-to-parameter ratio = 17.2

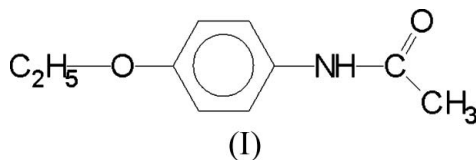
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Redetermination of *p*-ethoxyacetanilide (phenacetin)The crystal structure of the title compound, C₁₀H₁₃NO₂, has been redetermined in order to obtain more precise structure parameters.

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Comment

Phenacetin was used as an analgesic and antipyretic drug until 1983 when it was removed from the market because it can induce nephropathy and cancer. Analysis of a great number of experimental data shows that the thermodynamic parameters of the sublimation and fusion processes are very sensitive to the geometric and topological parameters of hydrogen bonds on the one hand, and to the conformational state of the molecule in the crystal structure on the other. Therefore, any improvement in these parameters gives better opportunities for understanding the thermodynamic properties of molecular crystals.



In the literature, there are only two papers devoted to a full determination of the crystal structure of phenacetin (*p*-ethoxyacetanilide), (I) (Patel *et al.*, 1983*a,b*). In this work, we present more precise crystal structure parameters, due to the higher quality of the single crystals used.

A view of the molecular structure of (I) with the atomic numbering is presented in Fig. 1. The bond lengths are within the normal range of such bonds (Allen *et al.*, 1987). The parameters of the hydrogen-bond geometry of (I) are shown in Table 1. There is one short intermolecular hydrogen bond and one intramolecular hydrogen bond (Taylor & Kennard, 1982; Bondi, 1964).

The data obtained by us are approximately five times more precise than the results given by Patel *et al.* (1983*a*). For example, the bond lengths for the non-H atoms found from our new study compared with the results from Patel *et al.* (in square brackets) are: C5–C6 1.374 (2) [1.35 (1)], C8–C9 1.509 (2) [1.50 (1)], O7–C8 1.422 (2) [1.44 (1)], O7–C1 1.374 (2) [1.35 (1)], C4–N10 1.421 (2) [1.40 (1)], N10–C11 1.444 (2) [1.34 (1)] and C11–C12 1.497 (2) Å [1.48 (1) Å]. The same comparison for the torsion angles is: C9–C8–O7–C1 171.6 (2) [172 (1)], C4–N10–C11–C12 178.7 (2) [178 (1)] and C4–N10–C11–O13 -1.2 (3)° [-1 (1)°]. Finally, the *R* factor obtained by us is also much lower: 0.0389 [0.088].

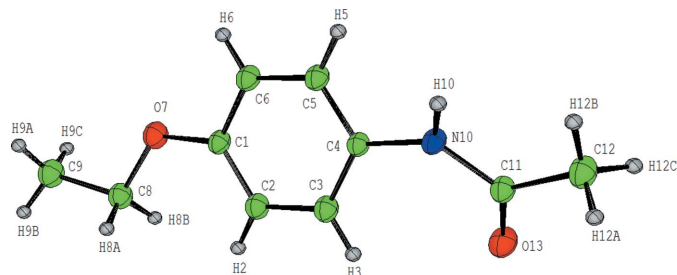


Figure 1
A view of the molecular structure of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Experimental

Crystals of (I) were grown by slow evaporation of an ethanol solution.

Crystal data

$C_{10}H_{13}NO_2$	$Z = 4$
$M_r = 179.21$	$D_x = 1.238 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 13.3236 (14) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 9.6159 (15) \text{ \AA}$	$T = 298 (2) \text{ K}$
$c = 7.7331 (15) \text{ \AA}$	Block, colourless
$\beta = 103.992 (14)^\circ$	$0.50 \times 0.40 \times 0.30 \text{ mm}$
$V = 961.4 (3) \text{ \AA}^3$	

Data collection

Enraf–Nonius CAD-4 diffractometer	2299 measured reflections
$\omega/2\theta$ scans	2083 independent reflections
Absorption correction: ψ scan	1056 reflections with $I > 2\sigma(I)$
[<i>ABSCALC</i> in <i>OSCAIL</i> (McArdle & Daly, 1999) and North <i>et al.</i> (1968)]	$R_{\text{int}} = 0.015$
$T_{\text{min}} = 0.958$, $T_{\text{max}} = 0.975$	$\theta_{\text{max}} = 27.0^\circ$
	3 standard reflections
	frequency: 120 min
	intensity decay: 1%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0541P)^2 + 0.0361P]$
$R[F^2 > 2\sigma(F^2)] = 0.039$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.111$	$(\Delta/\sigma)_{\text{max}} = 0.009$
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.16 \text{ e \AA}^{-3}$
2083 reflections	$\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$
121 parameters	Extinction correction: <i>SHELXL97</i> (Sheldrick, 1997)
H-atom parameters constrained	Extinction coefficient: 0.021 (3)

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C3-H3 \cdots O13$	0.93	2.42	2.915 (2)	114
$N10-H10 \cdots O13^i$	0.86	2.09	2.9426 (18)	175

Symmetry code: (i) $-x, y - \frac{1}{2}, -z - \frac{1}{2}$.

All H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C–H distances at 0.93 \AA , the N–H distance at 0.86 \AA and the CH_2 C–H distances at 0.97 \AA , with $U_{\text{iso}}(\text{H}) = 1.3U_{\text{eq}}(\text{C}, \text{N})$. The CH_3 C–H distances were fixed at 0.96 \AA , with $U_{\text{iso}}(\text{H}) = 1.4U_{\text{eq}}(\text{C})$. In addition, the methyl groups were allowed to rotate but not to tip.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992); cell refinement: *CELDIM* in *CAD-4-PC Software*; data reduction: *XCAD4* (McArdle & Higgins, 1995); program(s) used to solve structure: *OSCAIL-X for Windows* (McArdle, 2005) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL-X for Windows* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1993) and *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *OSCAIL-X for Windows*.

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