Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Redetermination and H-atom refinement of (*S*)-(+)-ibuprofen. Corrigendum.

Lars Kr. Hansen,[a]* German L. Perlovich[b,c] and Annette Bauer-Brandl[b]

[a]Department of Chemistry, University of Tromsø, 9037 Tromsø, Norway, [b]Department of Pharmaceutics and Biopharmaceutics, University of Tromsø, 9037 Tromsø, Norway, and [c]Institute 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 σ(C–C) = 0.005 Å
R factor = 0.038
wR factor = 0.104
Data-to-parameter ratio = 7.0

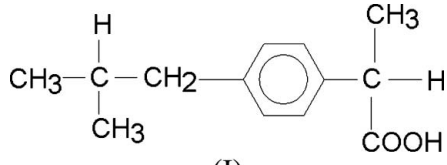
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 8 May 2006
Accepted 31 May 2006

In the paper by Hansen, Perlovich & Bauer-Brandl [*Acta Cryst.* (2003), E**59**, o1357–o1358], the coordinates of the *R* enantiomer of the title compound, $C_{13}H_{18}O_2$, were incorrectly given instead of those of the *S* enantiomer. The correct coordinates of the *S* enantiomer are given here.

Comment

In the paper by Hansen *et al.* (2003), the coordinates of the *R* enantiomer were incorrectly given instead of those of the *S* enantiomer, (I). The correct coordinates of the *S* enantiomer are given in the deposited replacement CIF. Molecular geometry parameters are not affected, except for the signs of torsion angles; the correct values are given in Table 1 below for the torsion angles in Table 2 of the previous report (where there was also an error in the atom numbering). Fig. 1 shows the correct structure of the two independent molecules, which form a hydrogen-bonded dimer without crystallographic symmetry.



(I)

Experimental

Table 1
Selected torsion angles (°).

C5*B*—C4*B*—C2*B*—C3*B*	29.1 (4)	O1*A*—C1*A*—C2*A*—C4*A*	−81.7 (4)
C7*B*—C10*B*—C11*B*—C12*B*	−68.0 (5)	C3*A*—C2*A*—C4*A*—C5*A*	−144.4 (4)
C4*B*—C2*B*—C1*B*—O1*B*	83.5 (3)	C7*A*—C10*A*—C11*A*—C13*A*	67.9 (5)

All H atoms were refined freely [C—H = 0.85 (3)–114 (5) Å].

Data collection: *CAD-4-PC* Software (Enraf–Nonius, 1992); cell refinement: *CELDIM* in *CAD-4-PC* Software; data reduction: *XCAD* (McArdle & Higgins, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *OSCAIL* (McArdle, 1993).

References

Enraf–Nonius (1992). *CAD-4-PC Software*. Enraf–Nonius, Delft, The Netherlands.

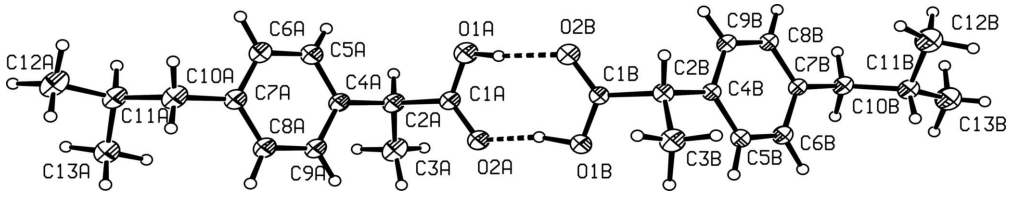


Figure 1
The structure of the two independent molecules of (I). Displacement ellipsoids are shown at the 30% probability level. Hydrogen bonds are shown as dashed lines.

Hansen, L. K., Perlovich, G. L. & Bauer-Brandl, A. (2003). *Acta Cryst.* E**59**, o1357–o1358.
McArdle, P. (1993). *J. Appl. Cryst.* **26**, 752.
McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65.
McArdle, P. & Higgins, T. (1995). *XCAD*. NUI Galway, Ireland.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Redetermination and H-atom refinement of (*S*)-(+)-ibuprofen

Lars Kr. Hansen,[a]* German L. Perlovich[b] and Annette Bauer-Brandl[c]

[a]Department of Chemistry, University of Tromsø, 9037 Tromsø, Norway, [b]Department of Pharmaceutics and Biopharmaceutics, University of Tromsø, 9037 Tromsø, Norway, and Institute of Solution Chemistry, Russian Academy of Sciences, 153045 Ivanovo, Russia, and [c]Department of Pharmaceutics and Biopharmaceutics, University of Tromsø, 9037 Tromsø, Norway

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

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.038
wR factor = 0.105
Data-to-parameter ratio = 7.0

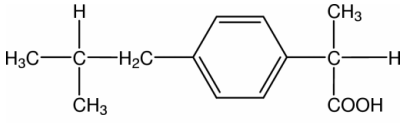
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 20 June 2003
Accepted 30 June 2003
Online 23 August 2003

The crystal structure of (*S*)-(+)-ibuprofen, $C_{13}H_{18}O_2$, has been redetermined. It crystallizes in the monoclinic space group $P2_1$ with two molecules in the asymmetric unit, giving a cyclic hydrogen-bonded dimer. All the H atoms were located from difference maps and refined isotropically.

Comment

The structure of racemic ibuprofen (IBP), (I), has been well described both by X-ray diffraction at 298 K (McConnell, 1974) and single-crystal pulsed neutron diffraction at 100 K (Shankland *et al.*, 1997). The structure of (*S*)-ibuprofen has also been solved by X-ray diffraction (Freer *et al.*, 1993). However, in contrast to the racemate, the quality and paucity of data did not allow the determination of the positions of the H atoms. It should be noted that the lack of this information is a serious obstacle for a logical classification of the hydrogen-bond topology of the compound, and for the calculation of crystal-lattice energy by methods which are sensitive to the geometry of hydrogen bonding (*e.g.* Gavezzotti & Filippini, 1997). With this background, the main aim of this work has been to find and refine the positions of the H atoms.



(I)

A view of the two crystallographically independent (*S*)-IBP molecules, with the atomic numbering, is presented in Fig. 1. The parameters of the hydrogen-bond geometry for both the racemic and *S*-enantiomer IBP are shown in Table 1 (Taylor & Kennard, 1982). As there are two independent molecules in the asymmetric unit, the two hydrogen bonds are not geometrically equivalent; one of them is shorter than the other. Comparison of these data gives the following conclusions: (*a*) the hydrogen bond in the racemate is more linear than in the *S*-enantiomer; (*b*) one of the O···O distances in (*S*)-IBP is essentially the same as that in the racemate, but the other is longer; (*c*) the H···O distance in the racemate is an average of the analogous parameters of the *S*-enantiomer. The conformations of the *S*-enantiomer molecules are different; the main parameters characterizing these differences are presented in Table 2, together with corresponding ones for the racemate. As can be seen, molecule *A* in the *S*-enantiomer structure has approximately the same conformation as the IBP molecule in the racemate.

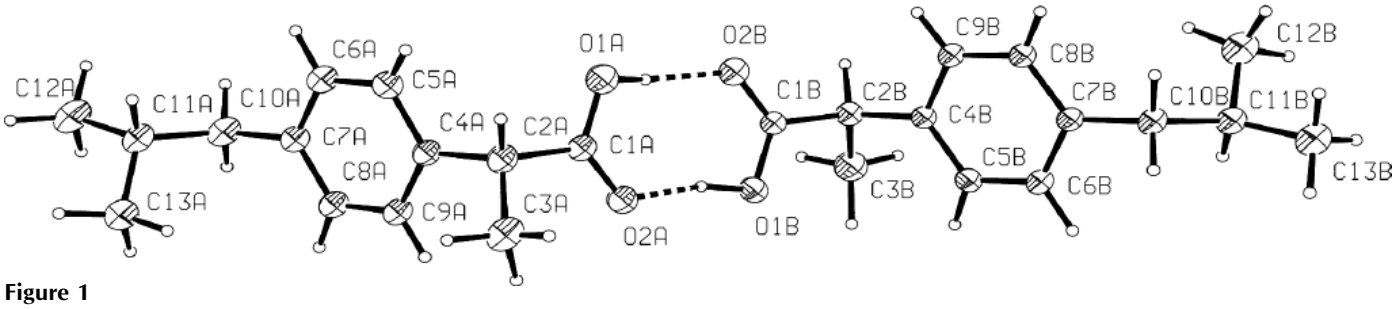


Figure 1
A view of the (*S*)-(+)-ibuprofen dimer, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 20% probability level.

Experimental

(*S*)–IBP single crystals were grown by slow evaporation from an *n*-heptanol solution.

Crystal data

$C_{13}H_{18}O_2$	D_x = 1.098 Mg m^{-3}
M_r = 206.27	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 25 reflections
a = 12.456 (4) Å	θ = 14–20°
b = 8.0362 (11) Å	μ = 0.07 mm^{-1}
c = 13.533 (3) Å	T = 298 (2) K
β = 112.86 (2)°	Block, colourless
V = 1248.2 (5) Å^3	0.40 × 0.40 × 0.30 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 27.0°
ω–2θ scans	$h = 0 \rightarrow 15$
3086 measured reflections	$k = -1 \rightarrow 10$
2910 independent reflections	$l = -17 \rightarrow 15$
1683 reflections with $I > 2\sigma(I)$	3 standard reflections
R_{int} = 0.019	frequency: 120 min
	intensity decay: 2%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.039	$(\Delta/\sigma)_{max}$ = 0.002
$wR(F^2)$ = 0.105	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
S = 0.98	$\Delta\rho_{min}$ = −0.11 e Å^{-3}
2910 reflections	Extinction correction: *SHELXL*97
416 parameters	Extinction coefficient: 0.016 (3)
All H-atom parameters refined	

Table 1
Hydrogen-bond geometry (Å, °) in the crystal structures of the title compound and of its racemate.

D—H···A	D—H	H···A	D···A	D—H···A
O1*A*—H1*AO*···O2*B*	0.94 (5)	1.73 (6)	2.651 (4)	169 (5)
O1*B*—H1*BO*···O2*A*	1.07 (5)	1.58 (5)	2.634 (4)	168 (4)
O2—H2O···O1^{i,a}	0.963 (13)	1.664 (10)	2.627 (7)	179.5 (7)

Symmetry code: (i) $1-x, 1-y, 1-z$. Reference: (*a*) Freer *et al.* (1993).

Table 2
Geometrical and conformational parameters (Å, °) of (*S*)- and racemic IBP.

	(*S*)-IBP *A*	(*S*)-IBP *B*	racemic IBP
C5—C4—C2—C3	144.4 (4)	−29.1 (4)	140.9 (4)
C7—C10—C11—C12	−67.9 (5)	68.0 (5)	−67.3 (4)
C4—C2—C1—O2	81.7 (4)	−83.5 (3)	88.7 (3)
O1—C1	1.219 (3)	1.226 (3)	1.222 (3)
O2—C1	1.302 (4)	1.302 (4)	1.305 (3)
C1—C2	1.496 (5)	1.518 (4)	1.509 (3)

Data collection: *CAD*-4-*PC Software* (Enraf–Nonius, 1992); cell refinement: *CELDIM* in *CAD*-4-*PC Software*; data reduction: *XCAD* (McArdle & Higgins, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: ORTEX (McArdle, 1995); software used to prepare material for publication: *OSCAIL* (Version 9; McArdle, 1993).

This work was generously supported by the Norwegian Research Council (project No. HS 58101).

References

Enraf–Nonius. (1992). *CAD*-4-*PC Software*. Version 1.1. Enraf–Nonius, Delft, The Netherlands.
Freer, A. A., Bunyan, J. M., Shankland, N. & Sheen, D. B. (1993). *Acta Cryst.* C**49**, 1378–1380.
Gavezzotti, A. & Filippini, G. (1997). *Energetic Aspects of Crystal Packing: Experiment and Computer Simulations*, ch. 3, pp. 61–97, in *Theoretical Aspects and Computer Modeling of the Molecular Solid State*, edited by A. Gavezzotti, p. 237. Chichester: John Wiley and Sons.
McArdle, P. (1993). *J. Appl. Cryst.* **26**, 752.
McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65.
McArdle, P. & Higgins, T. (1995). *XCAD*. National University of Ireland, Galway, Ireland.
McConnell, J. F. (1974). *Cryst. Struct. Commun.* **3**, 73–75.
Shankland, N., Wilson, C. C., Florence, A. J. & Cox, P. J. (1997). *Acta Cryst.* C**53**, 951–954.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.