C5—S4—C3—C2	−8.2 (4)	C3—S4—C5—N1	17.0 (4)
C7—N1—C2—C3	−96.9 (6)	C3—S4—C5—C6	114.4 (4)
C5—N1—C2—C3	18.1 (6)	N31—C32—C33—C38	−18.7 (7)
S4—C3—C2—N1	−3.5 (5)		

Despite the fairly large size of crystal (II), it showed weak diffraction power, *i.e.* only 4.2% of observed reflections satisfied the $I > 2\sigma(I)$ criterion in the $25 < \theta < 27°$ resolution shell. Therefore, the reflection to parameter ratio is rather small. The data were corrected for Lorentz and polarization effects and an absorption correction (North *et al.*, 1968) was applied to the data of crystal (I). Both structures were solved by direct methods and refined by full-matrix least-squares calculations based on F^2, with anisotropic displacement parameters for all non-H atoms. The amide-H atoms in (I) were located in difference Fourier maps and all other H atoms in (I), as well as all those in (II), were placed in geometrical positions.

For both compounds, data collection: *STADI*4 (Stoe & Cie, 1996*a*); cell refinement: *STADI*4; data reduction: *X-RED* (Stoe & Cie, 1996*b*); program(s) used to solve structures: *SHELXS*97 (Sheldrick, 1997*b*); program(s) used to refine structures: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *PLATON*93 (Spek, 1993*a*) and *PLUTON*93 (Spek, 1993*b*); software used to prepare material for publication: *SHELXL*97.

The authors would like to thank the Ministry of Science and Technology of the Republic of Croatia, for partially supporting this work (Grant No. 119420).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1291). Services for accessing these data are described at the back of the journal.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Herak, J. J. (1996). *Sulfur Rep.* **18**, 379–396.
Herak, J. J. & Vinković, M. (1996). *Croat. Chem. Acta*, **69**, 1367–1376.
Herak, J. J., Vinković, M. & Lukić, I. (1995). *Tetrahedron*, **51**, 5083–5092.
Herak, J. J., Vinković, M., Mandić, Z., Lukić, I., Tomić, M. & Kovačević, M. (1994). *Tetrahedron Asymmetry*, **5**, 1605–1612.
Nardelli, M. (1993). *PARST*93. University of Parma, Italy.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Page, M. I. (1987). *Advances in Physical Organic Chemistry*, Vol. 23, edited by D. Bethell, pp. 165–270. London: Academic Press.
Sheldrick, G. M. (1997*a*). *SHELXL97. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXS97. Program for the Solution of Crystal Structures.* University of Göttingen, Germany.
Spek, A. L. (1993*a*). *PLATON93. Molecular Geometry Program.* University of Utrecht, The Netherlands.
Spek, A. L. (1993*b*). *PLUTON93. Molecular Graphics Program.* University of Utrecht, The Netherlands.
Stoe & Cie (1996*a*). *STADI4. Diffractometer Control Program.* Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1996*b*). *X-RED. Data Reduction Program.* Stoe & Cie, Darmstadt, Germany.
Sweet R. M. (1972). *Cephalosporins and Penicillins*, edited by E. H. Flynn, pp. 281–309. London: Academic Press.

Acta Cryst. (1999). C**55**, 659–661

Acefluoranthylene†

Martin Lutz,[a] Anthony L. Spek,[a] Martin Sarobe[b] and Leonardus W. Jenneskens[b]

[a]*Bijvoet Center for Biomolecular Research, Department of Crystal and Structural Chemistry, Utrecht University, Padualaan 8, NL-3584 CH Utrecht, The Netherlands, and* [b]*Debye Institute, Department of Physical Organic Chemistry, Utrecht University, Padualaan 8, NL-3584 CH Utrecht, The Netherlands. E-mail: m.lutz@chem.uu.nl*

(*Received 8 September 1998; accepted 12 October 1998*)

Abstract

The title compound, $C_{18}H_{10}$, expresses a curvature in the solid state about the planar central pentagon and shows distinct bond length alternations. The single bonds in the fused cyclopenteno ring are the longest bonds in the molecule, in the range 1.487 (2)–1.491 (2) Å. The shortest bond, at 1.354 (2) Å, is a double bond between the two adjacent hexagons.

Comment

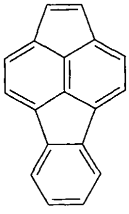
Polycyclic aromatic hydrocarbons are of great interest as components of the smoke produced during incomplete combustion of fossil fuels and tobacco. They are of particular interest because of the high toxicity of many representatives of this class of compounds (Jacob, 1996). In recent times, polycyclic aromatic compounds have also attracted interest as key planar intermediates in the formation of fullerenes.

Acefluoranthylene, (I), was prepared in the context of our investigations into the generation of polycyclic aromatic compounds using flash vacuum thermolysis (FVT; Sarobe *et al.*, 1995; Jenneskens *et al.*, 1996). Fig. 1 shows a molecular plot of the title compound with the atom-numbering scheme. The molecule is curved in the solid state. The maximum deviation from planarity is with atoms C17 and C18, which deviate by 0.347 (5) and 0.305 (5) Å, respectively, from the least-squares plane defined by the central five-membered ring. A rigid-body motion analysis (Schomaker & Trueblood, 1968) leads to $R = \Sigma(|U_{\rm obs} - U_{\rm calc}|)/\Sigma|U_{\rm obs}| = 0.065$. This low R value is a strong indication that the whole molecule behaves as a rigid body (Dunitz, 1979), which is to be expected for a polycyclic aromatic system. A comparison of the anisotropic vibration parameters in the covalent bond direction of atom pairs (Hirshfeld, 1976) shows only a variation of less than 2σ and confirms the consistency of the model.

---

† Cyclopenta[*cd*]fluoranthene.

© 1999 International Union of Crystallography
Printed in Great Britain – all rights reserved



(I)

Bond-length equalisation due to cyclic delocalization of π-electrons can be considered as a geometric criterion of aromaticity (Schleyer & Jiao, 1996). The title compound, however, shows strong variations in bond lengths, leading to the picture of alternating single and double bonds (see scheme above). The longest bond distances are found as single bonds in the five-membered rings, in the range 1.487 (2)–1.491 (3) Å. The shortest bond is the double bond between two six-membered rings, at 1.354 (2) Å (C10—C16). This variation in bond lengths closely resembles the situation found in the related polycyclic aromatic hydrocarbon, pyracylene ($C_{14}H_8$; Freiermuth *et al.*, 1990†) and is also confirmed by our semiempirical *AM*1 calculations (Dewar *et al.*, 1985) for (I). These *AM*1 calculations, as well as high level *ab-initio* studies (Sarobe, 1998), find (I) in a planar state, with molecular symmetry C_{2v}. The curvature found in the present study must therefore be a result of crystal-specific forces, such as the H···π interactions described below.

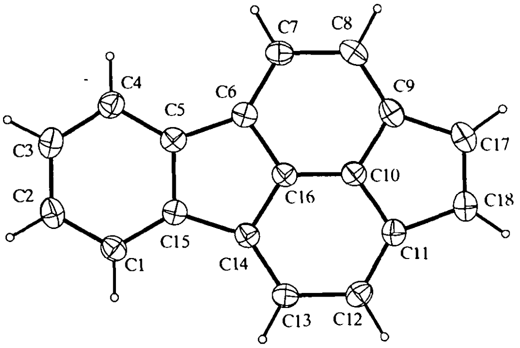


Fig. 1. *PLATON* (Spek, 1998) plot of (I), showing the atom-labelling scheme and with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

The variation in bond lengths can be considered as a preliminary stage to the fullerene molecule, C_{60}, where the bond lengths of adjacent hexagons are about 1.391 Å and those within the pentagon are about 1.455 Å (Vainshtein *et al.*, 1995). In contrast to C_{60}, in (I), the pentagons are not fully surrounded by hexagons. The symmetry is lower and the bond length variation is larger. The greatest deviation from the situation in C_{60} is found in the bond lengths of the pentagons not shared with hexagons; the bond lengths between adjacent pentagons and hexagons are indeed rather similar in (I) and C_{60}. It should be noted here that the 'isolated pentagon rule' (Schmalz *et al.*, 1988), which is formulated with relation to fullerenes, is also valid for (I).

The packing of the molecules in the crystal of (I) is influenced by two C—H···π interactions, which are shown in Fig. 2. The distance of H2 to the centre of gravity of hexagon 5^{i} (C10–C14 and C16) is 2.689 Å, with an angle of 156° at H2, and the distance of H7 to the centre of gravity of hexagon 3^{ii} (C1–C5 and C15) is 2.687 Å, with an angle of 145° at H7 [symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z$; (ii) $1-x, -y, z+\frac{1}{2}$]. This influence of C—H···π interactions on the packing is well known in crystal engineering of aromatic compounds (Hunter, 1994). It should be noted that both aromatic rings involved in these interactions in (I) are hexagons, which have better acceptor capabilities.

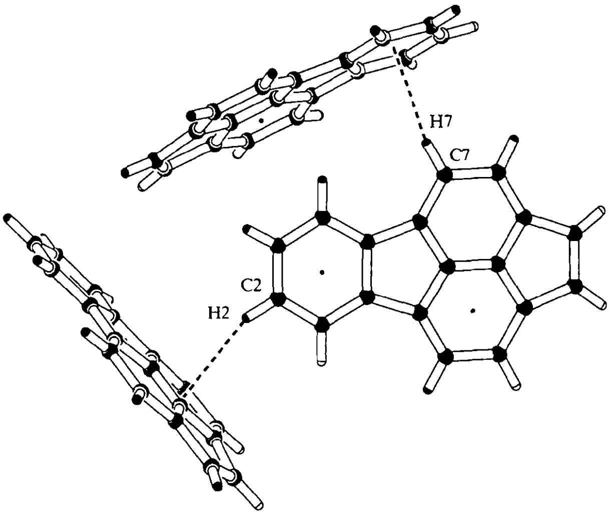


Fig. 2. *PLUTON* (Spek, 1991) plot, showing the C—H···π interactions.

Experimental

Acefluoranthylene was prepared according to the procedures described previously by Sarobe *et al.* (1995).

Crystal data

$C_{18}H_{10}$
$M_r = 226.26$
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

---

† The structure of pyracylene is published in the monoclinic space group *P*2₁/*n* (No. 14), but closely fits into the orthorhombic space group *Cmca* (No. 64), and it should therefore be carefully re-checked.

Orthorhombic
$Pna2_1$
a = 12.5684 (9) Å
b = 14.4723 (10) Å
c = 6.1016 (9) Å
V = 1109.8 (2) Å^3
Z = 4
D_x = 1.354 Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
θ = 10.93–17.45°
μ = 0.077 mm^{-1}
T = 150(2) K
Block
0.50 × 0.25 × 0.25 mm
Red

Data collection

Enraf–Nonius CAD-4T diffractometer
ω scans
Absorption correction: none
2782 measured reflections
1391 independent reflections
1260 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.036
$\theta_{\rm max}$ = 27.44°
$h = -16 \rightarrow 16$
$k = -18 \rightarrow 0$
$l = -7 \rightarrow 0$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.089
S = 1.024
1391 reflections
163 parameters
H-atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.0850P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 0.183 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.189 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected bond lengths (Å)*

C1—C15	1.383 (2)	C9—C10	1.406 (3)
C1—C2	1.394 (3)	C9—C17	1.488 (3)
C2—C3	1.383 (3)	C10—C16	1.354 (2)
C3—C4	1.406 (3)	C10—C11	1.396 (3)
C4—C5	1.383 (3)	C11—C12	1.380 (3)
C5—C15	1.431 (3)	C11—C18	1.491 (2)
C5—C6	1.487 (2)	C12—C13	1.444 (3)
C6—C7	1.387 (3)	C13—C14	1.383 (3)
C6—C16	1.406 (3)	C14—C16	1.404 (3)
C7—C8	1.448 (3)	C14—C15	1.491 (3)
C8—C9	1.385 (3)	C17—C18	1.362 (3)

The absolute structure was assigned arbitrarily. A check for additional higher symmetry with *ADDSYM* in *PLATON* (Spek, 1998) did not indicate any missed symmetry.

Data collection: locally modified *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *SET*4 (Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1997). Program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1997). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *PLATON*. Software used to prepare material for publication: *PLATON*.

This work was supported by the Council for Chemical Sciences of the Netherlands Organization for Scientific Research (CW-NWO).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1006). Services for accessing these data are described at the back of the journal.

References

Altomare, A., Cascarano, C., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Burla, M. C., Polidori, G., Camalli, M. & Spagna, R. (1997). *SIR97. A Package for Crystal Structure Solution by Direct Methods and Refinement.* University of Bari, Italy.

Boer, J. L. de & Duisenberg, A. J. M. (1984). *Acta Cryst.* A**40**, C-410.

Dewar, M. J. S., Zoebisch, E. G., Healy, E. F. & Stewart, J. J. P. (1985). *J. Am. Chem. Soc.* **107**, 3902–3909.

Dunitz, J. D. (1979). *X-ray Analysis and the Structure of Organic Molecules*, pp. 244–261. London: Cornell University Press.

Enraf–Nonius (1989). *CAD-*4 *Software.* Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Freiermuth, B., Gerber, S., Riesen, A., Wirz, J. & Zehnder, M. (1990). *J. Am. Chem. Soc.* **112**, 738–744.

Hirshfeld, F. L. (1976). *Acta Cryst.* A**32**, 239–244.

Hunter, C. A. (1994). *Chem. Soc. Rev.* pp. 101–109.

Jacob, J. (1996). *Pure Appl. Chem.* **68**, 301–308.

Jenneskens, L. W., Sarobe, M. & Zwikker, J. W. (1996). *Pure Appl. Chem.* **68**, 219–224.

Sarobe, M. (1998). PhD thesis, University of Utrecht, The Netherlands.

Sarobe, M., Snoeijer, J. D., Jenneskens, L. W., Slagt, M. Q. & Zwikker, J. W. (1995). *Tetrahedron Lett.* **36**, 8489–8492.

Schleyer, P. v. R. & Jiao, H. (1996). *Pure Appl. Chem.* **68**, 209–218.

Schmalz, T. G., Seitz, W. A., Klein, D. J. & Hite, G. E. (1988). *J. Am. Chem. Soc.* **110**, 1113–1127.

Schomaker, V. & Trueblood, K. N. (1968). *Acta Cryst.* B**24**, 63–76.

Sheldrick, G. M. (1997). *SHELXL*97. *Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Spek, A. L. (1991). *PLUTON. Molecular Graphics Program.* University of Utrecht, The Netherlands.

Spek, A. L. (1997). *HELENA. Program for Data Reduction.* University of Utrecht, The Netherlands.

Spek, A. L. (1998). *PLATON. Program for the Analysis of Molecular Geometry.* University of Utrecht, The Netherlands.

Vainshtein, B. K., Fridkin, V. M. & Indenbom, V. L. (1995). *Modern Crystallography*, Vol. 2 (*Structure of Crystals*), 2nd ed., pp. 402–408. Berlin: Springer-Verlag.

Acta Cryst. (1999). C**55**, 661–663

Methyl 1,4,5,6-tetrahydro-2-methyl-4-(2-nitrophenyl)-6-oxopyridine-3-carboxylate

Viktor Kettmann and Ján Svetlík

Faculty of Pharmacy, Comenius University, Odbojárov 10, 832 32 Bratislava, Slovak Republic. E-mail: kettmann@fpharm.uniba.sk

(*Received 21 July 1998; accepted 16 November 1998*)

Abstract

The title compound, $C_{14}H_{14}N_2O_5$, represents a tetrahydropyridin-2-one analogue of 1,4-dihydropyridine-type calcium antagonists, and was selected for a crystal structure determination in order to clarify some aspects

© 1999 International Union of Crystallography
Printed in Great Britain – all rights reserved

Acta Crystallographica Section C
ISSN 0108-2701 © 1999