

N,N'-Di-*n*-tetradecylpyromellitic diimideDaniel E. Lynch^{a*} and
Darren G. Hamilton^b^aSchool of Science and the Environment,
Coventry University, Coventry CV1 5FB,
England, and ^bDepartment of Chemistry, Mount
Holyoke College, South Hadley, Massachusetts
01075, USACorrespondence e-mail:
apx106@coventry.ac.uk

Key indicators

Single-crystal X-ray study
T = 150 K
Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
R factor = 0.059
wR factor = 0.203
Data-to-parameter ratio = 14.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The structure of the title compound, $\text{C}_{38}\text{H}_{60}\text{N}_2\text{O}_4$, has been determined and is similar to other compounds of this type, being essentially rod-shaped with the packing dominated by the lamellar arrangement of the molecules. The molecule lies on an inversion centre; thus only one alkyl chain, one imide ring and one of the non-bridgehead C atoms in the benzene ring are unique. The diimide moieties are arranged in a classic herring-bone structure, with two close non-hydrogen-atom contacts of 2.874 (5) and 2.946 (5) Å.

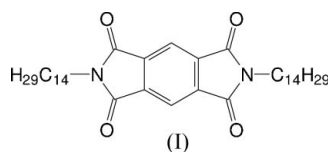
Received 9 February 2004

Accepted 15 March 2004

Online 24 March 2004

Comment

In a previous publication, we investigated the thin-film characteristics of neutral pseudorotaxanes consisting of 1:1 and 1:2 mixtures of bis(1,5-naphtho)-38-crown-10 with *N*-alkyl derivatives of both pyromellitic diimide and 1,4,5,8-naphthalene-tetracarboxylic diimide (Lynch *et al.*, 1999). The Langmuir spreading solutions used in this study were subsequently refrigerated for storage while the paper was being refereed and published. These solutions, over a period of several months, eventually evaporated to dryness, yielding crystals of varying quality. From the solution containing a 1:1 molar mixture of the crown and *N,N'*-di-*n*-tetradecylpyromellitic diimide, two distinct crystal forms were identified, separated and characterized using single-crystal X-ray techniques. One of those structures was that of (I), reported here, while the other form was that of a second polymorph of the crown (Lynch & Hamilton, 2004*a*). From the solution containing a 1:1 molar mixture of the crown and the naphthalene diimide analogue, crystals were obtained which gave the structure of the diimide; however, the poor quality of the crystals led to poor data and hence a poor structure refinement (Lynch & Hamilton, 2004*b*).



The structure of (I) is similar to other compounds of this type, being essentially rod-shaped (Fig. 1) with the packing dominated by the lamellar arrangement of the molecules. The diimide moieties arrange in a classic herring-bone structure with two close non-hydrogen-atom contacts: $\text{C6} \cdots \text{O21}(-x, -\frac{1}{2} + y, \frac{1}{2} - z) = 2.874 (5) \text{ \AA}$ and $\text{C2} \cdots \text{O61}(x, \frac{1}{2} - y, -\frac{1}{2} + z) = 2.946 (5) \text{ \AA}$. The chains are inclined at an angle of *ca* 40° to the plane of the diimide ring. The naphthalene diimide analogue similarly resides on an inversion centre, the molecules also pack in layers and the chains in this compound are also

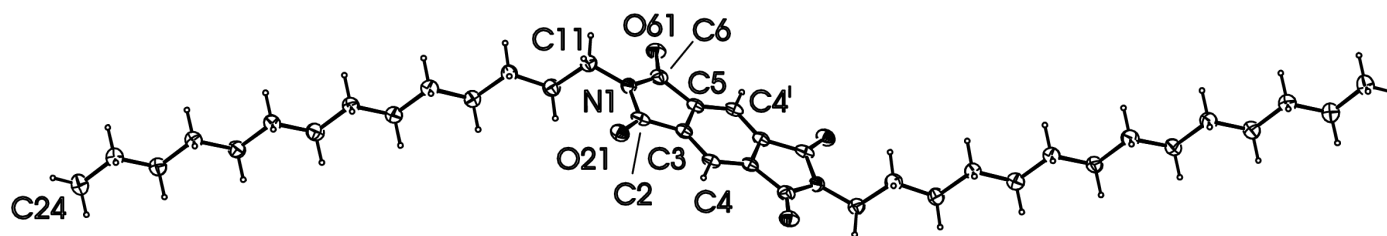


Figure 1

The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level. For clarity, only the first and last C atoms of the alkyl chain have been labelled. [Symmetry code (i): $-x, 1 - y, 1 - z$.]

inclined at an angle of *ca* 40° to the plane of the ring system, but this structure differs from (I) in that the naphthalene moieties are parallel to each other, displaying interplanar distances of *ca* 3.3–3.4 Å in the overlap regions.

Experimental

Crystals of the title compound were obtained following the total evaporation of an equimolar mixture of bis(1,5-naphtho)-38-crown-10 and (I) in 10 ml chloroform (0.1 mg cm⁻³) at 277 K.

Crystal data

C₃₈H₆₀N₂O₄

M_r = 608.88

Monoclinic, *P*2₁/*c*

a = 38.939 (3) Å

b = 4.9902 (3) Å

c = 8.9040 (5) Å

β = 95.896 (2)°

V = 1721.01 (19) Å³

Z = 2

D_x = 1.175 Mg m⁻³

Mo *K*α radiation

Cell parameters from 6262 reflections

θ = 1.0–27.5°

μ = 0.08 mm⁻¹

T = 150 (2) K

Plate, colourless

0.30 × 0.25 × 0.01 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer

φ and ω scans

Absorption correction: multi-scan (SORTAV; Blessing, 1995)

T_{min} = 0.992, *T_{max}* = 0.999

6743 measured reflections

2896 independent reflections

1366 reflections with *I* > 2σ(*I*)

R_{int} = 0.093

θ_{max} = 25.0°

h = -45 → 46

k = -5 → 5

l = -10 → 10

Refinement

Refinement on *F*²

R [*F*² > 2σ(*F*²)] = 0.059

wR (*F*²) = 0.203

S = 0.97

2896 reflections

200 parameters

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0882*P*)²]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.27 e Å⁻³

Δρ_{min} = -0.27 e Å⁻³

All H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C–H distances of 0.95 (aromatic H atoms), 0.99 (CH₂ H atoms) and 0.98 Å (CH₃ H atoms). The isotropic displacement parameters were set equal to 1.25*U_{eq}* of the carrier atom. The high *R_{int}* value was the result of weak high-angle data.

Data collection: DENZO (Otwinowski & Minor, 1997) and COLLECT (Hooft, 1998); cell refinement: DENZO and COLLECT; data reduction: DENZO, SCALEPACK (Otwinowski & Minor, 1997) and COLLECT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON97 (Spek, 1997); software used to prepare material for publication: SHELXL97.

The authors thank the EPSRC National Crystallography Service (Southampton, England).

References

- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–37.
- Hooft, R. (1998). COLLECT. Nonius BV, Delft, The Netherlands.
- Lynch, D. E., Hamilton, D. G., Calos, N. J., Wood, B. & Sanders, J. K. M. (1999). *Langmuir*, **15**, 5600–5605.
- Lynch, D. E. & Hamilton, D. G. (2004a). Unpublished results.
- Lynch, D. E. & Hamilton, D. G. (2004b). Private communication to the Cambridge Structural Database. Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, England.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
- Spek, A. L. (1997). PLATON97. University of Utrecht, The Netherlands.