

Acta Crystallographica Section C

**Crystal Structure
Communications**

ISSN 0108-2701

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Ziemer and Grubert

Electronic paper

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2,2',4,4',6,6'-Hexamethyl-4,4'-bi[4H-pyranyl]

Burkhard Ziemer* and Lutz Grubert

Institut für Chemie, Humboldt-Universität zu Berlin, Hessische Strasse 1-2, 10115 Berlin, Germany

Correspondence e-mail: burkhard=ziemer@chemie.hu-berlin.de

Received 12 May 2000

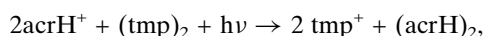
Accepted 30 May 2000

Data validation number: IUC0000155

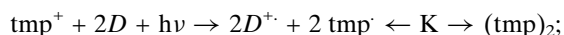
The title molecule, C₁₆H₂₂O₂, reveals C_i point symmetry in the crystal structure. The structure was disordered. The pyran ring is not planar; the O atom lies significantly out of the least-squares plane (ten times the r.m.s. deviation of all six atoms).

Comment

Bipyranyls and pyrylium salts are used for photochemical redox reactions. Bipyranyls may be applied as 2e donors in photoreductions, e.g. such as

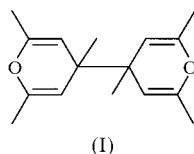


where acrH⁺ is acridinium and tmp⁺ is 2,4,6-trimethylpyrylium. For photo-oxidations with pyrylium salts as e acceptors, attention has to be paid to the equilibrium of bipyranyl and the pyranyl radical,



where *D* is an e donor.

The title molecule, (I), reveals C_i point symmetry in the crystal structure. The structure was disordered in such a way



that each of three C atoms of the pyran ring occupies statistically two sites with a ratio of 89/11. The sites of the other three ring atoms are not split. This means that the two ring positions are arranged like a roof with an interfacial angle of about 24°. A second crystal studied showed the same effect, with a similar occupation ratio of 81/19 and a corresponding

interfacial angle of about 27°. The pyran ring is not planar; the O atom lies significantly out of the least-squares plane (ten times the r.m.s. deviation of all six atoms).

Experimental

The title compound was synthesized according to Balaban *et al.* (1964) and crystallized from ethanol.

Crystal data

 C₁₆H₂₂O₂
M_r = 246.34

 Monoclinic, *P*2₁/*c*
a = 7.842 (2) Å

b = 11.458 (3) Å

c = 8.824 (2) Å

 β = 114.26 (3)°

V = 722.9 (3) Å³
Z = 2

D_x = 1.132 Mg m⁻³

 Mo *K*α radiation

Cell parameters from 5000 reflections

 θ = 2–24°

 μ = 0.073 mm⁻¹
T = 180 (2) K

Plate, colourless

0.52 × 0.40 × 0.22 mm

Data collection

Stoe IPDS diffractometer

 φ -rotation, φ -incr. = 1.5°, 160 exposure scans

5901 measured reflections

1716 independent reflections

 1393 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.033

 θ_{max} = 28.0°

h = −10 → 10

k = −15 → 15

l = −11 → 11

Intensity decay: none

Refinement

 Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.044

wR(*F*²) = 0.119

S = 1.039

1716 reflections

121 parameters

H atoms treated by a mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0650P)^2 + 0.1273P]$

 where *P* = (*F_o*² + 2*F_c*²)/3

 (Δ/σ)_{max} = 0.003

 Δρ_{max} = 0.22 e Å⁻³

 Δρ_{min} = −0.16 e Å⁻³

The structure is disordered so that split atomic positions of C3, C4 and C5 had to be introduced for the least squares refinement. Without such a 'split model', all parameters of quality for a structure determination become worse as follows: *R*₁(gt) increases from 0.0438 to 0.0751; *wR*₂(all) increases from 0.1185 to 0.2182; GoF changes from 1.039 to 1.066; the long axes of the displacement ellipsoids of C3, C4 and C5 increase by a factor of about 1.35; and last, but not least, the three highest peaks in the final difference map become significantly larger (1.02, 0.82 and 0.77 e Å⁻³) than its r.m.s. (0.07 e Å⁻³); the peaks lie nearest to C4 (1.09 Å), C5 (0.82 Å) and C3 (0.79 Å), respectively.

Data collection: *IPDS-2.87* (Stoe & Cie, 1997); cell refinement: *IPDS-2.87*; data reduction: *IPDS-2.87*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97*.

References

- Balaban, A. T., Bratu, C. & Rentea, C. N. (1964). *Tetrahedron*, **20**, 265–269.
 Sheldrick, G. M. (1990). *Acta Cryst. A* **46**, 467–473.
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
 Stoe & Cie (1997). *IPDS-2.87*. Stoe & Cie, Darmstadt, Germany.