Ethyl 5,5''-dimethyl-2,2';6',2''-terpyridine-4'-carboxylate

Kickelbick, G.; Hoogenboom, R.; Schubert, U.S.

Published in:
Acta Crystallographica, Section E: Structure Reports

DOI:
10.1107/S1600536805038547

Published: 01/01/2005

Document Version
Publisher's PDF, also known as Version of Record (includes final page, issue and volume numbers)

Please check the document version of this publication:
• A submitted manuscript is the author's version of the article upon submission and before peer-review. There can be important differences between the submitted version and the official published version of record. People interested in the research are advised to contact the author for the final version of the publication, or visit the DOI to the publisher's website.
• The final author version and the galley proof are versions of the publication after peer review.
• The final published version features the final layout of the paper including the volume, issue and page numbers.

Link to publication

Citation for published version (APA):

General rights
Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

• Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
• You may not further distribute the material or use it for any profit-making activity or commercial gain
• You may freely distribute the URL identifying the publication in the public portal ?

Take down policy
If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

Download date: 11. Dec. 2018
Guido Kickelbick,a Richard Hoogenboomb and Ulrich S. Schubertb**

*a Vienna University of Technology, Institute of Materials Chemistry, Getreidemarkt 9/165, A-1060 Wien, Austria, and b Laboratory of Macromolecular Chemistry and Nanoscience, Eindhoven University of Technology, PO Box 513, 5600 MB Eindhoven, The Netherlands

Correspondence e-mail: u.s.schubert@tue.nl

Key indicators

Single-crystal X-ray study

T = 173 K

Mean |C–C| = 0.002 Å

R factor = 0.049

wR factor = 0.149

Data-to-parameter ratio = 18.2

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

We report here the crystal structure of another 2,2′:6′,2″-terpyridine with three substituents, namely the title compound.
trimethylstannyl-5-methylpyridine, (6), by the addition of trimethylstannylechloride (in the literature method 2-tributylstannyl-5-methylpyridine was used). Compounds (3) and (6) were coupled via a Pd(PPH3)4-catalysed Stille coupling, resulting in the title compound (7), which crystalized as single crystals by slow evaporation of a CDCl3 solution. The reaction scheme of the synthesis is shown above.

Crystal data

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
</table>
| C20H19N3O2 | |}

Data collection

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
</table>
| Bruker SMART CCD area-detector diffractometer | |}

Refinement

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
</table>
| R[F2 > 2σ(F2)] = 0.049 | |}


table 1

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
</table>
| Selected geometric parameters (Å, °) | |}

H atoms were located in difference Fourier maps and refined with a riding model, with C–H distances of 0.95 (aromatic H), 0.98 (methyl H) and 0.99 Å (methylene H), and with Uiso(H) = 1.2Ueq(C) for aromatic and methylene H, or 1.5Ueq(C) for methyl H.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1998); software used to prepare material for publication: SHELXTL.

Guido Kickelbick thanks the Fonds zur Förderung der wissenschaftlichen Forschung (FWF), Austria, for financial support. Richard Hoogenboom and Ulrich S. Schubert thank the Dutch Scientific Organization (NWO) and the Fonds der Chemischen Industrie for financial support.

Experimental

Compound (7) was synthesized starting from 2,6-dihydroxyisonicotinic acid, (1), and 2-bromo-5-methylpyridine, (4), following a modified literature procedure (Fallahpour, 2000; Heller & Schubert, 2002). Compound (1) was converted into 2,6-dibromoisonicotinic acid ethyl ester, (3), via 2,6-dibromoisonicotinic acid, (2). 2-Bromo-5-methylpyridine was lithiated, resulting in (5), and converted into 2-

"Figure 1" The structure of (7), with displacement ellipsoids shown at the 50% probability level. For clarity, H atoms have been omitted.

"Figure 2" Projection of the structure along [001]. For clarity, H atoms have been omitted.

The molecular structure of (7) is shown in Fig. 1. The three rings of the terpyridine unit, N1/C2–C6, C7/N8/C9–C12 and C14/N15/C16–C19, are coplanar. The plane through the terpyridine unit makes an angle to the carboyxylic ester group (C21/O22/O23) of 10.99 (4)°. All bond lengths and angles can be regarded as normal. The crystal packing reveals π–π stacking interactions between the conjugated aromatic rings in the structure, with a distance between the mean planes of 3.494 Å (Fig. 2).
References