One-Pot Synthesis of Novel Highly Functionalized Furan-Based Polyphenolics

Joana L. C. Sousa, a Oualid Talhi, a,c,* Djenisa H. A. Rocha, a Diana C. G. A. Pinto, a Filipe A. Almeida Paz, b Khaldoun Bachari, c Gilbert Kirsch, d Artur M. S. Silva a,*

a QOPNA, Department of Chemistry, University of Aveiro, 3810-193 Aveiro, Portugal
b CICECO – Aveiro Institute of Materials, Department of Chemistry, University of Aveiro, 3810-193 Aveiro, Portugal
c Centre de Recherche Scientifique et Technique en Analyses Physico-Chimiques CRAPC, BP384, Bou-Ismail, 42004, Tipaza, Algeria
d Laboratoire Structure et Réactivité des Systèmes Moléculaires Complexes, UMR 7565, Université de Lorraine, Avenue du Général Delestreint, 57070 Metz, France
Fax: +351 234370084 (Artur M. S. Silva and Oualid Talhi).
E-mail: artur.silva@ua.pt, oualid.talhi@ua.pt.

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**Figure S18** – $^{13}$C NMR spectrum of furan 3i (125.77 MHz, CDCl$_3$).
Single-Crystal X-ray Diffraction Studies of compound 3a

Single crystals of compound 3a were manually harvested from the crystallization vial and immersed in highly viscous FOMBLIN Y perfluoropolyether vacuum oil (LVAC 140/13, Sigma-Aldrich) to avoid degradation caused by the evaporation of the solvent. Crystals were mounted on Hampton Research CryoLoops with the help of a Stemi 2000 stereomicroscope equipped with Carl Zeiss lenses. X-ray diffraction data were collected at 150(2) K on a Bruker D8 QUEST equipped with Mo Kα sealed tube (λ = 0.71073 Å), a multilayer TRIUMPH X-ray mirror, a PHOTON 100 CMOS detector, controlled by the APEX2 software package, and an Oxford Instruments Cryostrem 700+ Series low temperature device.

Diffraction images were processed using the software package SAINT+, and data were corrected for absorption by the multiscan semi-empirical method implemented in SADABS. The structure was solved using the algorithm implemented in SHELXT-2014, which allowed the immediate location of almost all of the heaviest atoms composing the molecular unit of the three compounds. The remaining missing and misplaced non-hydrogen atoms were located from difference Fourier maps calculated from successive full-matrix least-squares refinement cycles on \( F^2 \) using the latest SHELXL from the 2014 release. All structural refinements were performed using the graphical interface ShelXle.

Hydrogen atoms bound to carbon were placed at their idealized positions using appropriate HFIX instructions in SHELXL: 43 (aromatic carbon atoms) or 137 (for the terminal methyl group). These hydrogen atoms were included in subsequent refinement cycles with isotropic thermal displacements parameters (\( U_{iso} \)) fixed at 1.2 (for the former family of hydrogen atoms) or 1.5×\( U_{eq} \) (solely for those associated with the methyl group) of the parent carbon atoms. The hydrogen atom associated with the hydroxyl group was directly found from difference Fourier maps and it was included in the final structural model with the O–H distance restrained to 0.95(1) Å and the isotropic thermal displacement parameter (\( U_{iso} \)) fixed at 1.5×\( U_{eq} \) of the parent oxygen atom.

The last difference Fourier map synthesis showed the highest peak (0.243 eÅ\(^{-3}\)) and the deepest hole (-0.239 eÅ\(^{-3}\)) located at 0.83 and 0.49 Å from H7A and H7B, respectively. Structural drawings have been created using the software package Crystal Impact Diamond.
Crystallographic data (including structure factors) for the crystal structure of compound 3a have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication No. CCDC-1409624. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 2EZ, U.K. FAX: (+44) 1223 336033. E-mail: deposit@ccdc.cam.ac.uk.
References


(3) SAINT+ Data Integration Engine v. 8.27b© 1997-2012, Bruker AXS, Madison, Wisconsin, USA.

(4) G. M. Sheldrick SADABS 2012/1, Bruker AXS Area Detector Scaling and Absorption Correction 2012, Bruker AXS, Madison, Wisconsin, USA.


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Structure factor report

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e.g. ‘Acta Crystallographica Section C’
PUBL008_ALERT_1_A _publ_section_title is missing. Title of paper.
PUBL012_ALERT_1_A _publ_section_abstract is missing.
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PUBL024_ALERT_1_A The number of authors is greater than 5.
Please specify the role of each of the co-authors
for your paper.

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