

SYNTHESIS OF γ -TRIFLUOROMETHYL TETRONATE DERIVATIVES FROM SQUARATES

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Single Crystal X-Ray Diffraction Study

Single crystals were mounted on a quartz fiber, and diffraction data were collected in θ ranges specified in Tables S1 at 103 K on a Bruker SMART APEX CCD diffractometer with graphite monochromatized Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$). The absorption correction was made using SADABS. The structure was solved by direct methods and refined by the full-matrix least-squares on F^2 by using SHELXTL. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in calculated positions. Final refinement details are compiled in Tables S1. The supplementary crystallographic data for this paper [CCDC] can also be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033; or deposit@ccdc.cam.ac.uk).

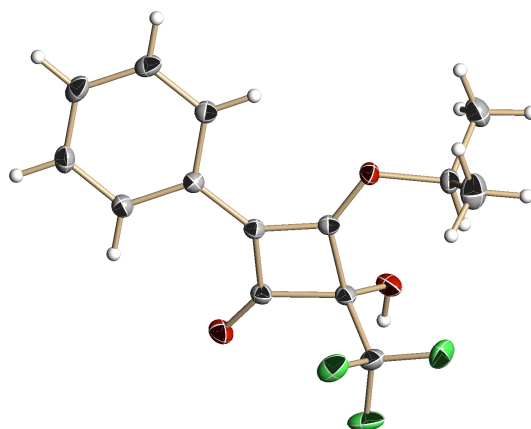


Figure S1. ORTEP drawing of **7a**.

Table S1. Selected Crystallographic data and collection parameters for **7a**.

formula	C ₁₄ H ₁₃ F ₃ O ₃
fw	286.24
crystal system	triclinic
space group	P-1
<i>a</i> , Å	8.9825(18)
<i>b</i> , Å	9.0227(18)
<i>c</i> , Å	9.5256(19)
α , deg	66.736(3)
β , deg	67.602(3)
γ , deg	75.132(4)
volume, Å ³	650.6(2)
<i>Z</i>	2
<i>D</i> (calcd), Mg m ⁻³	1.461
μ , mm ⁻¹	0.129
<i>F</i> (000)	296
crystal size, mm	0.5 x 0.5 x 0.1
θ range for data collection, deg	2.45 to 28.43
index ranges	-7 \leq h \leq 12, -6 \leq k \leq 12, -12 \leq l \leq 12
reflections collected	4049
independent reflections [<i>R</i> (int)]	3051 [<i>R</i> (int) = 0.0152]
coverage of independent reflections	93.1%
data / restraints / parameters	3051 / 0 / 184
goodness-of-fit on <i>F</i> ²	0.586
<i>R</i> ₁ [4730 data; <i>I</i> > 2s(<i>I</i>)]	0.0388
<i>wR</i> ₂ [4730 data; <i>I</i> > 2s(<i>I</i>)]	0.1152
<i>R</i> ₁ (all data)	0.0402
<i>wR</i> ₂ (all data)	0.1181
largest diff. peak and hole, e Å ⁻³	0.416 and -0.318

NMR Charts

