Electronic Supplementary Material (ESI) for Green Chemistry

Supporting Information

Intramolecular etherification of five-membered cyclic carbonates bearing hydroxyalkyl groups

Karolina M. Tomczyk, Piotr A. Guńka, Paweł G. Parzuchowski, Janusz Zachara, Gabriel Rokicki*

Faculty of Chemistry, Warsaw University of Technology, Noakowskiego 3, 00-664 Warsaw, Poland

* Address correspondence to Gabriel Rokicki, e-mail: gabro@ch.pw.edu.pl

Table S1. Selected bond lengths and angles for compound **15**.

	Bond Lengths		
O1–C1	1.1969(16)	O4–C3	1.4323(16)
O2–C1	1.3379(16)	O4–C4	1.4376(16)
O2–C2	1.4483(15)	C2–C3	1.5145(18)
O3–C1	1.3417(16)	C2–C5	1.5391(17)
O3–C5	1.4504(15)	C4–C5	1.5146(18)
	Bond Angles		
C1–O2–C2	110.62(9)	O2–C2–C5	103.44(9)
C1–O3–C5	110.22(9)	C3–C2–C5	103.82(10)
C3–O4–C4	103.91(9)	O4–C3–C2	104.44(9)
O1–C1–O2	123.90(12)	O4–C4–C5	104.34(10)
O1–C1–O3	124.24(12)	O3–C5–C2	103.71(9)
O2–C1–O3	111.85(10)	O3–C5–C4	110.25(10)
O2–C2–C3	110.26(10)	C4–C5–C2	103.72(10)

Table S2. Selected bond lengths and angles for compound **20**.

	Bond Lengths		
O1–C1	1.1972(19)	O5–C6	1.4496(18)
O2–C1	1.3393(19)	O5–C8	1.335(2)
O2–C3	1.4470(18)	O6–C7	1.442(2)
O3–C1	1.3411(19)	O6–C8	1.333(2)
O3–C2	1.4531(18)	O7–C8	1.202(2)
O4–C4	1.4373(19)	C2–C3	1.540(2)
O4–C5	1.4337(18)	C6–C7	1.539(2)
	Bond Angles		
C1–O2–C3	110.44(12)	O3–C2–C4	110.33(13)
C1–O3–C2	110.35(12)	O2–C3–C5	112.55(12)
C5–O4–C4	104.98(11)	O4–C4–C2	105.04(12)
C8–O5–C6	109.99(13)	O4–C5–C3	105.72(12)
C8–O6–C7	110.13(13)	O5–C6–C5	110.42(12)
O1–C1–O2	124.00(14)	O6–C8–O5	112.60(14)
O1–C1–O3	124.11(15)	O7–C8–O5	123.67(16)
O2–C1–O3	111.89(13)	O7–C8–O6	123.74(16)

Table S3. Crystal data and structure refinement parameters for compounds **15** and **20**.

	15	**20**
Sum formula	$C_5H_6O_4$	$C_8H_8O_7$
Formula weight	130.10	216.14
Temperature /K	100(2)	100(2)
Crystal system	orthorhombic	orthorhombic
Space group	$P2_12_12_1$	$P2_12_12_1$
a, b, c /Å	4.25566(10), 10.2167(2), 12.2711(3)	7.9412(2), 10.0129(3), 10.8310(3)
α, β, γ /°	90.00, 90.00, 90.00	90.00, 90.00, 90.00
Volume /Å^3	533.53(2)	861.22(4)
Z	4	4
ρ_{calc} /g·cm^{-3}	1.620	1.667
λ /Å	1.5418	0.7107
μ /mm^{-1}	1.252	0.151
F(000)	272	448
Crystal size /mm^3	0.30 × 0.10 × 0.10	0.60 × 0.35 × 0.20
θ range for data collection	5.63 to 67.00°	6.54 to 55.00°
Index ranges hkl	−5:4, −12:12, −14:14	−10:10, −12:13, −13:14
Reflections collected	7552	11027
Independent reflections	945 [R_{int} = 0.0296]	1982 [R_{int} = 0.0333]
Data/restraints/parameters	945/0/89	1982/0/161
Goodness-of-fit on F^2	1.062	1.083
Final R indices [I >2σ(I)]	R1 = 0.0225, wR2 = 0.0574	R1 = 0.0255, wR2 = 0.0550
Final R indices [all data]	R1 = 0.0236, wR2 = 0.0587	R1 = 0.0295, wR2 = 0.0571
$\Delta\rho_{max}$/ $\Delta\rho_{min}$ /e·Å^{-3}	0.145/ −0.133	0.231/−0.173

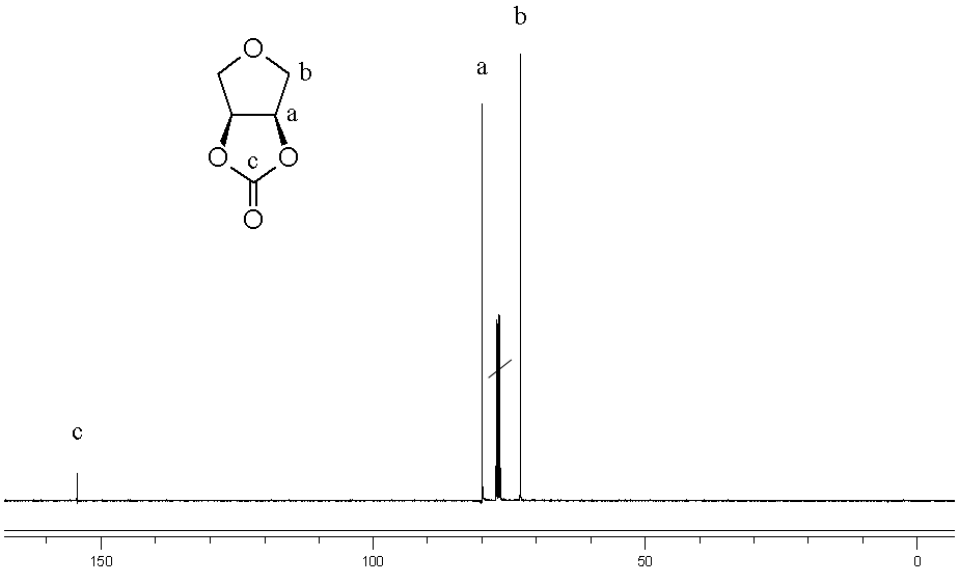

Figure S1. ^{13}C NMR (400 MHz, $CDCl_3$) spectrum of **15**.

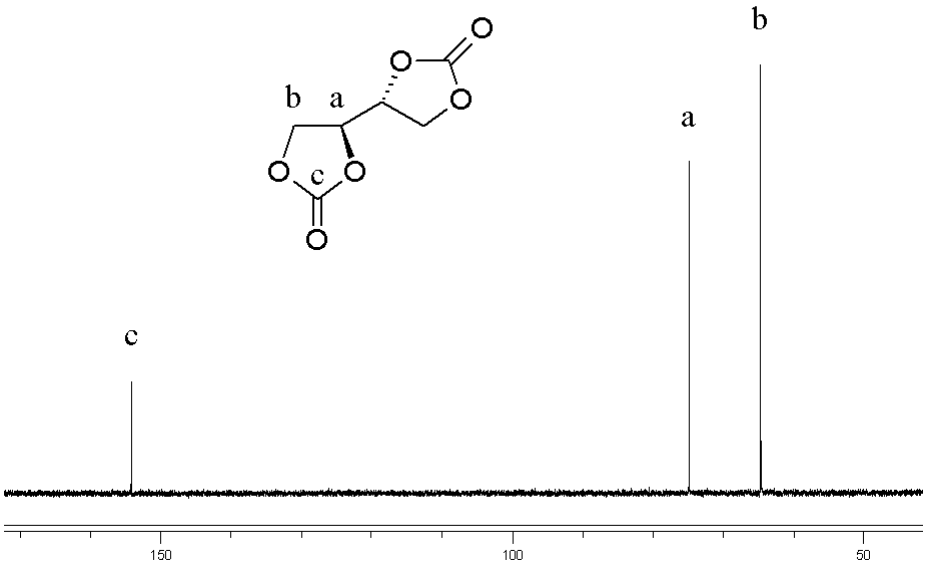

Figure S2. ^{13}C NMR (400 MHz, DMSO-d_6) spectrum of **16**.

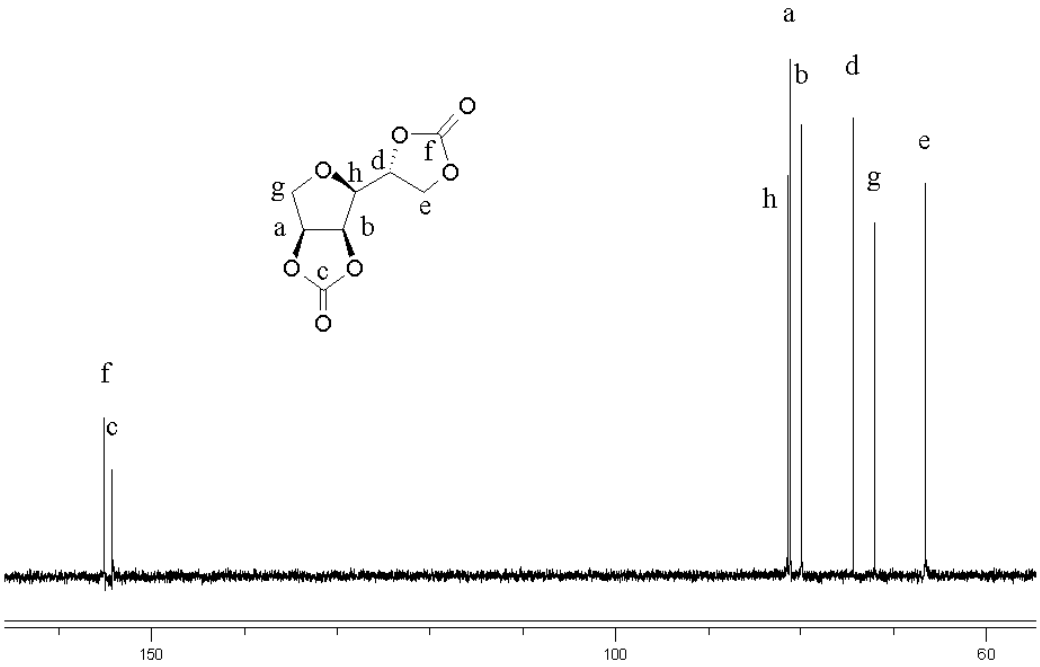

Figure S3. ^{13}C NMR (400 MHz, DMSO-d_6) spectrum of **20**.

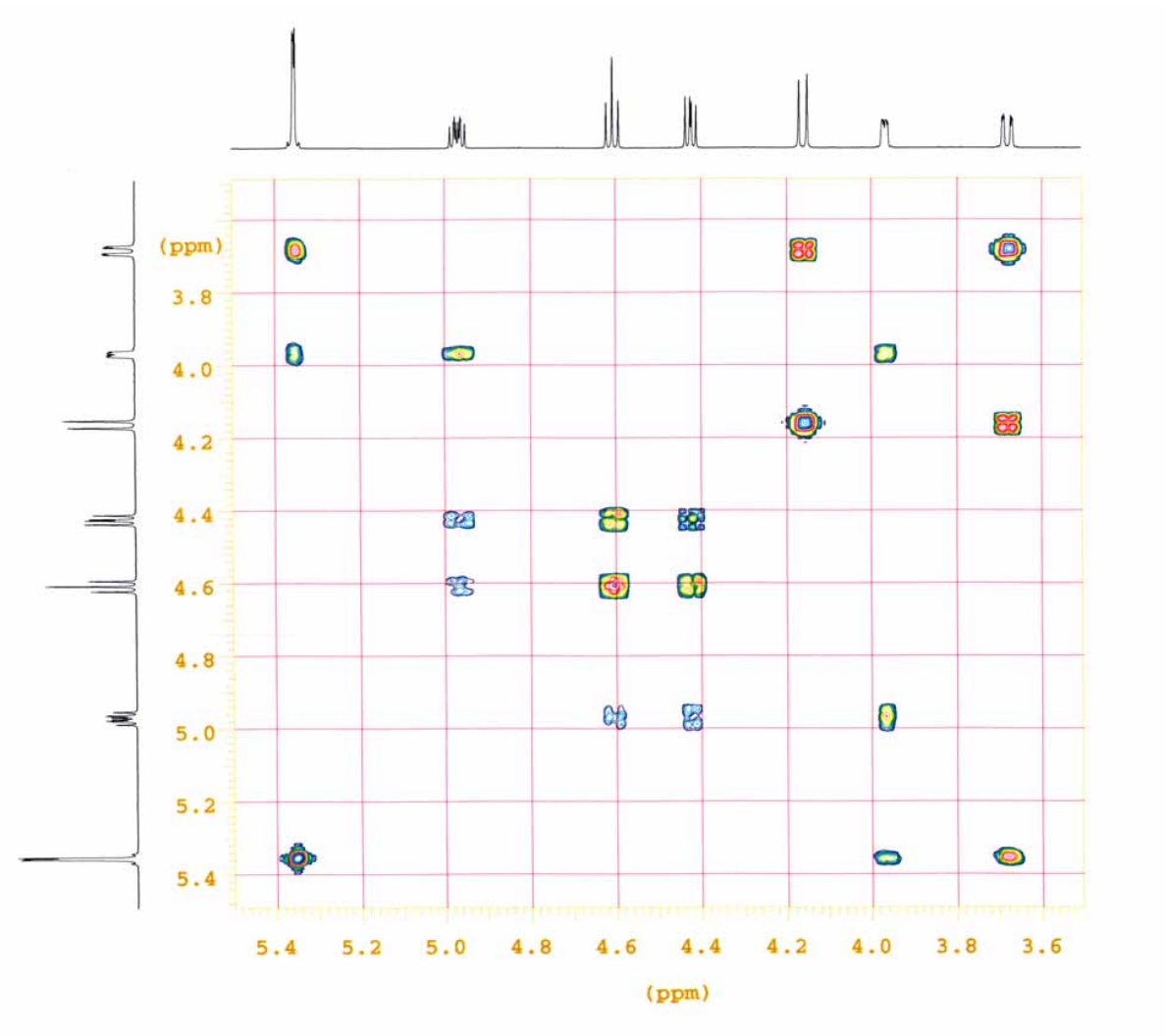

Figure S4. ^{1}H-^{1}H COSY NMR (600 MHz, DMSO-d_6) spectrum of **20**.

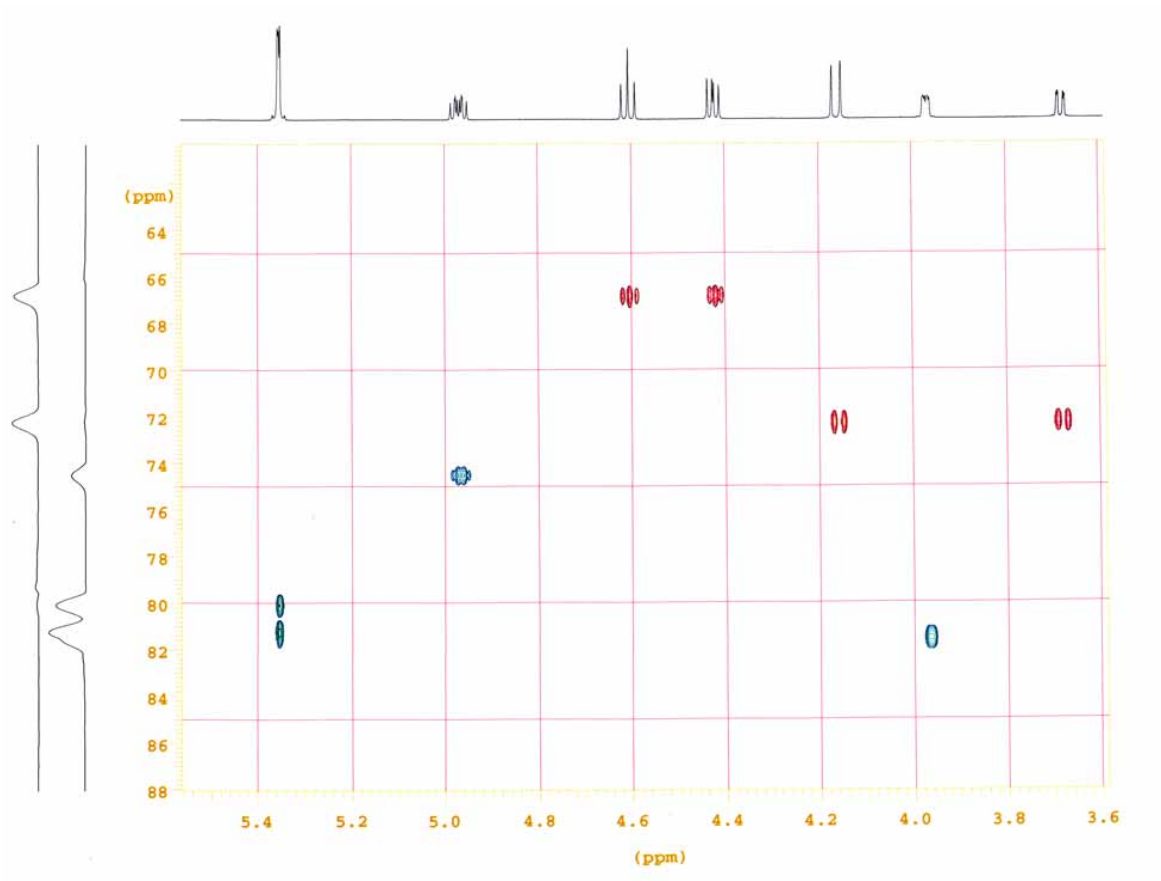

Figure S5. ^{1}H-^{13}C HSQC NMR (600 MHz, DMSO-d_6) spectrum of **20**.

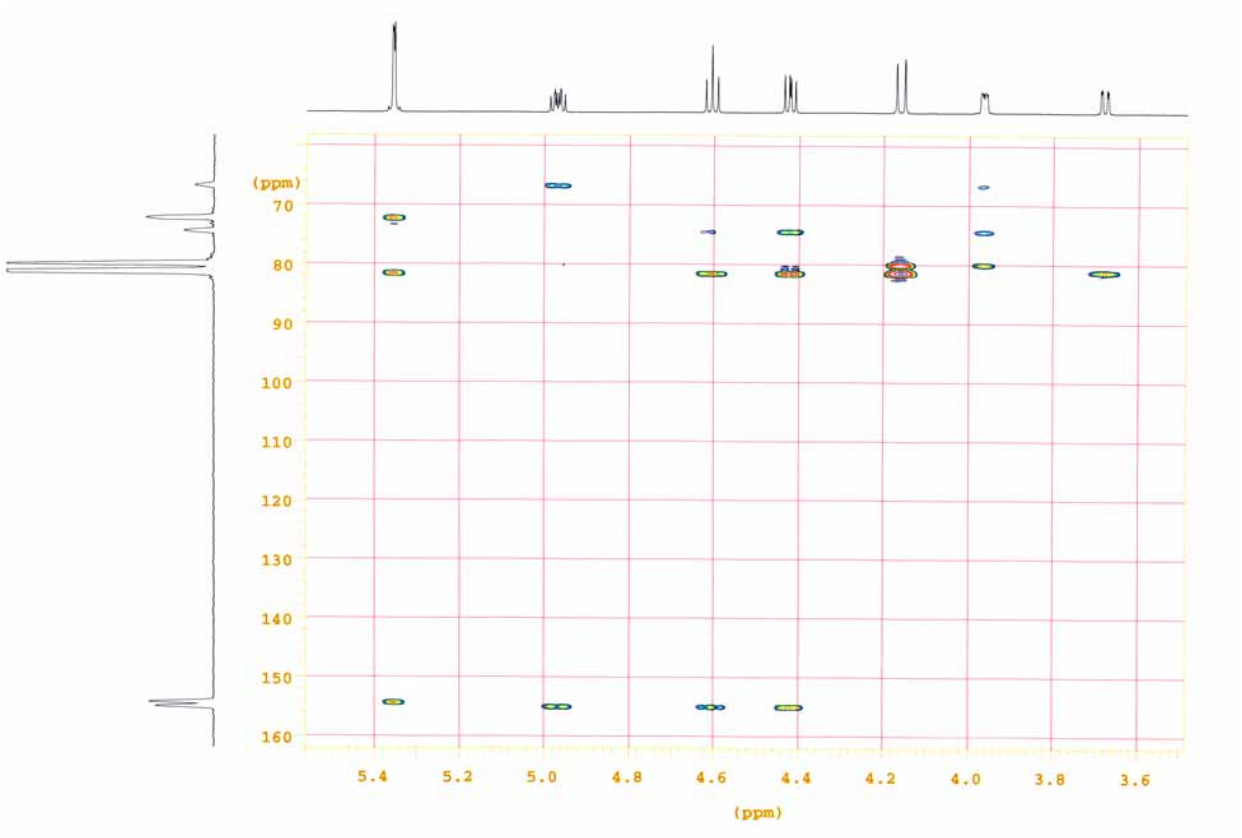

Figure S6. ^{1}H-^{13}C HMBC NMR (600 MHz, DMSO-d_6) spectrum of **20**.

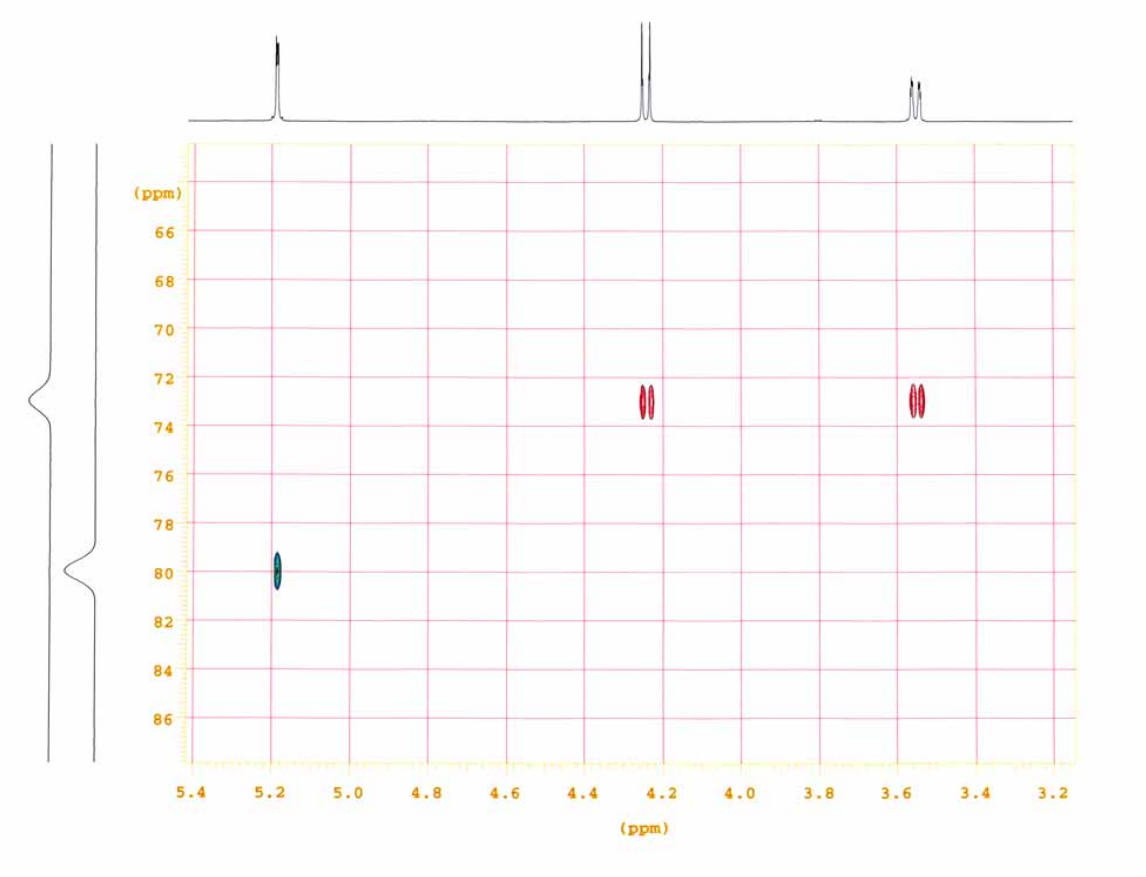

Figure S7. ^{1}H-^{13}C HSQC NMR (600 MHz, $CDCl_3$) spectrum of **15**.