Supporting information for:

Synthesis and Solid State Dynamics of a Crystalline Steroid Molecular Rotor Without the Alkyne Axle: Steroid Dimers Based on a 1,4-Di(1,3-dioxan-2-yl)benzene Moiety

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(20*S*)-5 α -pregnan-3 β ,16 β ,20-triol 3-monoacetate (17)



¹H NMR (400 MHz, CDCl₃) spectrum of compound **17**





					~73.65 ~73.07 —66.53	63.00 54.17 53.87 44.59
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41 40 39	38 37 36 35 34 33	32 31 30 29 28 ¹³ C NMR (100.53 M	8 27 26 25 24 f1 (ppm) //Hz, CDCl ₃) spectru	23 22 21 20 m of compound 17	19 18 17	16 15 14 13 12 S-6



Dimer SR-18



¹H NMR (400 MHz, CDCl₃) spectrum of compound *SR*-18





 13 C NMR (100.53 MHz, CDCl₃) spectrum of compound *SR*-18



¹³C NMR (100.53 MHz, CDCl₃) spectrum of compound *SR*-18



HSQC (CDCl₃) spectrum pf compoudn SR-18



NOESY(CDCl₃) spectrum pf compoudn SR-18



HMBC (CDCl₃) spectrum pf compoudn **SR-18**



Dimer RR-18







¹³C NMR (100.53 MHz, CDCl₃) spectrum of compound **RR-18**



 ^{13}C NMR (100.53 MHz, CDCl₃) spectrum of compound **RR-18**



HSQC (CDCl₃) spectrum of compound **RR-18**



HMBC (CDCl₃) spectrum of compound **RR-18**



NOESY(CDCl₃) spectrum of compound **RR-18**



Figure S-1 Crystal structures of steroid dimers *SR*-18 (A) and *RR*-18 (B) with the thermal ellipsoids drawn at 30% probability.

Parameter	SR-18	<i>RR</i> -18
Empirical formula	C ₅₄ H ₇₈ O ₈	C ₅₄ H ₇₈ O ₈
Formula weight	855.16	855.16
Temperature	130(2) K	130(2) K
Wavelength	1.54184 Å	1.54184 Å
Crystal system	Monoclinic	Triclinic
Space group	<i>P</i> 2 ₁	P1
	a = 13.7565(7) Å	a = 13.4848(8) Å
	b = 10.3255(6) Å	b = 13.8175(9) Å
Unit cell dimensions	c = 16.8608(7) Å	c = 14.4535(10) Å
	$\alpha = 90^{\circ}$	$\alpha = 74.959(6)^{\circ}$
	$\beta = 100.716(4)^{\circ}$	$\beta = 88.351(5)^{\circ}$
	$\gamma = 90^{\circ}$	$\gamma = 65.812(6)^{\circ}$
Volume	2353.2(2) Å ³	2363.3(3) Å ³
Z	2	2
Density (calculated)	1.207 Mg/m ³	1.202 Mg/m ³
Absorption coefficient	0.624 mm^{-1}	0.621 mm^{-1}
F(000)	932	932
Crystal size	0.370 x 0.140 x 0.060 mm ³	0.410 x 0.290 x 0.150 mm ³
Theta range for data collection	3.817 to 77.853°.	3.607 to 78.091°.
Index ranges	-17<=h<=17, -12<=k<=10, -	-16<=h<=16, -17<=k<=17, -
	21<=l<=21	18<=l<=18
Reflections collected	45982	45764
Independent reflections	9330 [R(int) = 0.1087]	18197 [R(int) = 0.0766]
Completeness to theta = 67.684°	100.0 %	100.0 %
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	9330 / 1 / 567	18197 / 3 / 1133
Goodness-of-fit on F ²	1.040	1.065
Final R indices [I>2sigma(I)]	R1 = 0.0563, WR2 = 0.1199	R1 = 0.0595, wR2 = 0.1210
R indices (all data)	R1 = 0.0869, WR2 = 0.1397	R1 = 0.0920, wR2 = 0.1445
Absolute structure parameter	0.13(18)	-0.02(14)
Largest diff. peak and hole	$0.200 \text{ and } -0.263 \text{ e.Å}^{-3}$	$0.234 \text{ and } -0.343 \text{ e.\AA}^{-3}$

Table S-1. Crystal data and structure refinement for compounds SR-18 and RR-18.



Figure S2. Overlay of the two symmetry independent molecules in the asymmetric unit of *RR*-18 highlighting the differences in the conformation of the molecules.



Figure S3. Alternative dynamic model for the motion of *RR*-18. The simulated line shapes (dotted red line) do not match the evolution of the experimental ¹³C CPMAS (75.47 MHz) peaks at higher temperatures (solid black line).