Pressure-Promoted Solvation of Resorcinol

*Fatemeh Safari, Anna Olejniczak, Andrzej Katrusiak**

Faculty of Chemistry, Adam Mickiewicz University, ul. Uniwersytetu Poznańskiego 8, 61-614 Poznań, Poland

Supporting Information

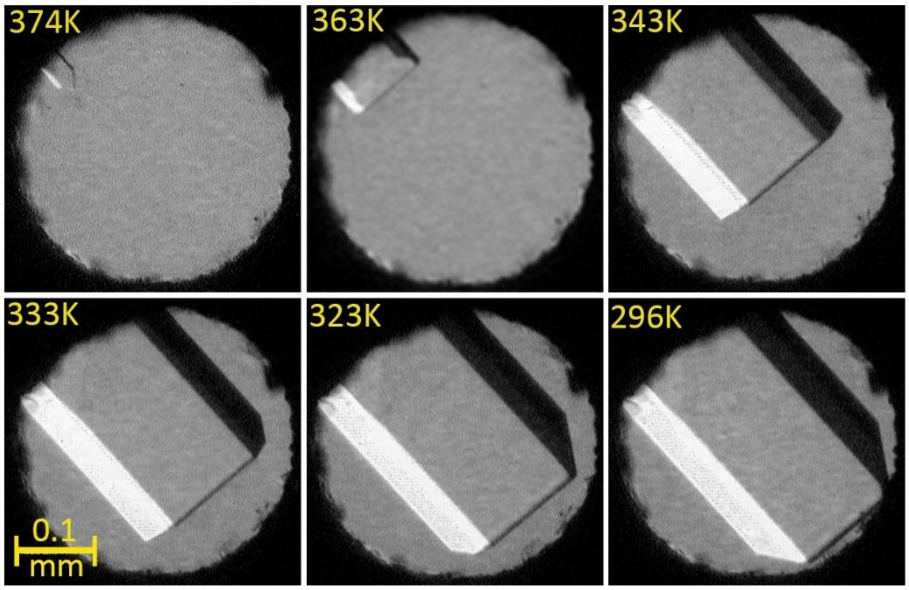

Figure S1. Single crystals of monohydrate of orcinol grown isochorically in the diamond-anvil cell at 0.2 GPa from its solution in the methanol.

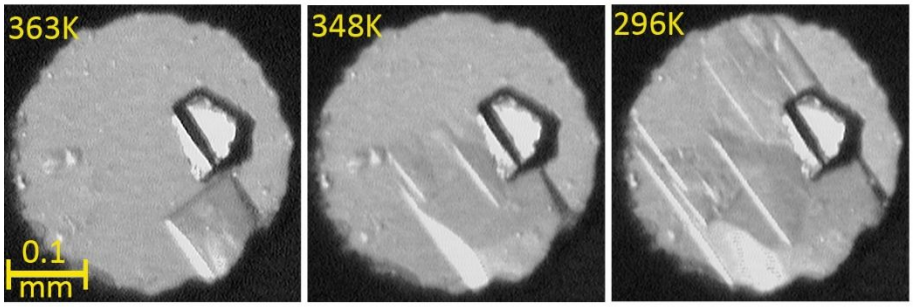

Figure S2. Isochoric growth of a single crystal of orcinol monohydrate 0.2 GPa from its water solution. The ruby chip for pressure calibration lies at the right side of the DAC chamber.

Table S1. Selected experimental data for co-crystals of resorcinol.

$C_6H_6O_2$	$C_6H_4(OH)_2.H_2O$	$3C_6H_4(OH)_2.2H_2O)$			$C_6H_4(OH)_2.CH_3OH$	
Pressure (GPa)	0.8 (2)	0.55 (2)	0.83 (2)	0.93 (2)	0.49 (2)	0.7 (2)
Temperature (K)	296(2)	296 (2)			296 (2)	
Formula weight	128.12	199			142.15	
Crystal color	colorless	Colorless			colorless	
Crystal size (mm)	0.25×0.2×0.15	0.25×0.2×0.12			0.25×0.2×0.1	
Crystal system	Orthorhombic	Monoclinic			Orthorhombic	
Space group	$P2_12_12_1$	$C2/c$			$P2_12_12_1$	
Unit cell (Å) a	5.6567(18)	8.1180(14)	8.041(3)	8.0312(8)	6.0242(12)	5.9240(3)
b	7.6544(5)	8.1403(18)	8.110(3)	8.1080(6)	8.1523(12)	8.1201(3)
c	13.226(3)	26.192(4)	26.029(8)	26.01(4)	14.32(3)	14.087(9)
β ( )	90	95.915 (17)	95.67 (3)	95.31 (3)	90	90
Volume ($Å^3$)	572.7(2)	1721.6(6)	1689.1(10)	1686(2)	703.4(14)	677.6(4)
Z	4	12	12	12	4	4
Density (g/cm^3)	1.486	1.4.6	1.433	1.435	1.342	1.393
Wavelength MoKα (Å)	0.71073	0.71073	0.71073		0.71073	0.71073
Absorption (mm^{-1})	0.120	0.111	0.114	0.114	0.105	0.109
F(000)	272	768	768	768	304	304
2θ max (deg.)	53.146	54.34	55.006	54.362	56.656	57.308
Index ranges h_{min}/h_{max}	-3/3	-8/8	-8/8	-10/9	-8/7	-7/7
k_{min}/k_{max}	-9/9	-7/7	-8/8	-9/10	-10/10	-10/10
l_{min}/l_{max}	-15/14	-27/27	-26/26	-8/8	-4/4	-4/4
Refl. Collected	2295	3075	2597	4286	3847	3806
Refl. observed ($I>4\sigma_I$)	430	541	522	435	536	523
R(int)	0.0349	0.1228	0.2309	0.0459	0.0548	0.0616
Data/restraints/parameters	430/0/87	541/25/133	522/6/134	435/30/135	536/0/95	523/0/95
Goodness-of-fit on F^2	1.150	1.017	0.988	1.129	1.217	1.100
Final R_1 ($I>2\sigma_I$)	0.0371/	0.0751/	0.1014/	0.0506/	0.0440/	0.0351/
	0.0871	0.1887	0.2441	0.1248	0.0800	0.0793
R_1/wR_2 (all data)	0.0571/	0.1346/	0.2222/	0.0808/	0.0909/	0.0408/
	0.1138	0.2432	0.3311	0.1488	0.0932	0.0819
Absorption corrections	DAC, gasket and sample crystal					

Table S2. Selected experimental data for monohydrate of orcinol.

$C_6H_6O_2$	$C_7H_6(OH)_2.H_2O$	
Pressure (GPa)	0.2(2)	
Temperature (K)	296(2)	
Formula weight	142.15	
Crystal color	Colorless	
Crystal size (mm)	0.25×0.2×0.15	0.25×0.2×0.12
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell (Å) a	9.74(4)	9.622.(10)
b	8.0023(16)	7.8957(9)
c	9.308(3)	9.184(5)
β ( )	96.35 (9)	97.59 (8)
Volume (Å^3)	721(3)	691.6(8)
Z	4	4
Density (g/cm^3)	1.309	1.365
Wavelength MoKα (Å)	0.71073	0.71073
Absorption (mm^{-1})	0.102	0.107
F(000)	304	304
2θ max (deg.)	54.618	52.896
Index ranges h_{min}/h_{max}	-2/2	-7/7
k_{min}/k_{max}	-10/10	-9/9
l_{min}/l_{max}	-11/11	-8/8
Refl. Collected	2939	2509
Refl. observed ($I>4\sigma_I$)	286	354
R(int)	0.1550	0.0459
Data/restraints/parameters	286/24/91	354/33/97
Goodness-of-fit on F^2	1.018	1.137
Final R_1 ($I>2\sigma_I$)	0.0574/	0.0371/
	0.1308	0.0423
R_1/wR_2 (all data)	0.1454/	0.0899/
	0.1927	0.0528
Absorption corrections	DAC, gasket and sample crystal	

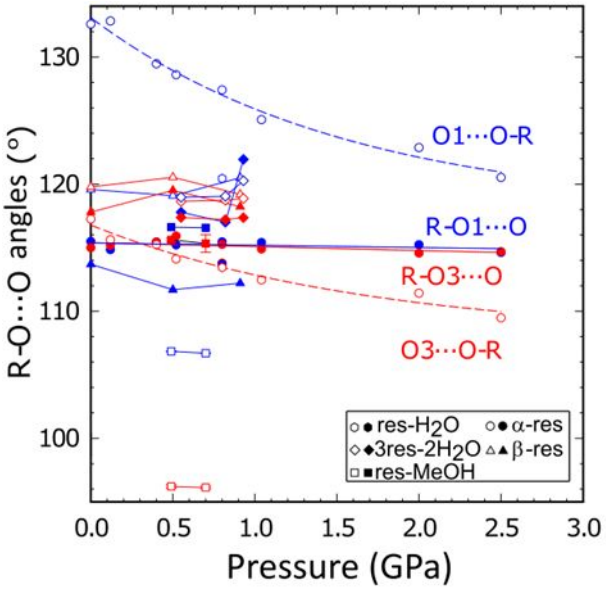

Figure S3. Pressure dependence of hydrogen-bond angles R-O···O on the H-donor side (full symbols and full lines) and on the H-acceptor side (open symbols and dashed lines).

Table S3. Geometry of O···O contacts in β- resorcinol.

Pressure (GPa) / Distances[Å]	0.0001(2)	0.50(2)	0.91(2)
O-H···O^{3655}	2.709 (2)	2.6620(14)	2.640 (4)
O-H···O^{2665}	2.780 (3)	2.749 (13)	2.721 (4)

Table S4. Geometry of O···O contacts in α- resorcinol at 0.0001-2.5(2) GPa range.

Pressure (GPa) / Distances[Å]	0.0001(2)	0.12(2)	0.40(2)	0.52(2)	0.80(2)	1.04(2)
O-H···O^{4465}	2.718(2)	2.719(14)	2.687(4)	2.671(8)	2.662(4)	2.637(5)
O-H···O^{3655}	2.720(3)	2.703(13)	2.692(4)	2.706(8)	2.664(4)	2.639(4)

Table S5. Geometry of O···O contacts in Res·CH_3OH.

Pressure (GPa) / Distances[Å]	0.49(2)	0.70(2)
O-H···O^{1555}	2.770 (2)	2.744(4)
O-H···O^{1545}	2.723 (3)	2.706 (10)
O-H···O^{4745}	2.708 (2)	2.686 (5)

Table S6. Geometry of O···O contacts in 3Res·$2H_2O$.

Pressure (GPa) / Distances[Å]	0.55(2)	0.83(2)	0.93(2)
O-H···O^{1555}	2.695(1)	2.668 (4)	2.660 (4)
O-H···O^{1565}	2.761 (4)	2.746 (10)	2.733 (5)
O-H···O^{3455}	2.824 (2)	2.791 (15)	2.826 (4)
O-H···O^{4656}	2.761 (6)	2.746 (12)	2.733 (3)

Table S7. Geometry of O···O contacts in Res·H_2O.

Pressure (GPa) / Distances[Å]	0.80(2)
O-H···O^{1555}	2.713(2)
O-H···O^{2664}	2.993 (10)
O-H···O^{4746}	2.763 (2)
O-H···O^{4752}	2.742 (6)

Table S8. Hydrogen-bond angles C-O···O in Res·CH_3OH, Res·H_2O and 3Res·$2H_2O$.

Pressure (GPa) / Angle [°]	Res·CH_3OH		Res·H_2O	3Res·$2H_2O$		
	0.49(2)	0.70(2)	0.80(2)	0.55(2)	0.83(2)	0.93(2)
R-O3···O	115.61 (2)	115.33 (4)	113.77 (1)	117.37 (4)	117.21 (4)	117.36 (3)
O3···O-R	96.21 (3)	96.12 (5)	113.44 (4)	118.65 (3)	118.74 (3)	118.87 (6)
R-O1···O	116.61 (1)	116.56 (2)	113.70 (5)	117.78 (5)	117.20 (2)	121.95 (5)
O1···O-R	106.84 (3)	106.69 (4)	120.44 (6)	118.97 (1)	119.05 (2)	120.27 (4)

Table S9. ORTEP symmetry code.[1]

Compound	ORTEP code	Symmetry code
α- resorcinol	3655	$^{3}/_{2}$-x, $^{1}/_{2}$+ y, $^{1}/_{2}$ +z
	4465	- $^{1}/_{2}$+x, $^{3}/_{2}$-y, $^{1}/_{2}$+z
β- resorcinol	2665	1-x, 1-y, $^{1}/_{2}$+z
	3655	$^{3}/_{2}$-x, $^{1}/_{2}$+y, $^{1}/_{2}$+z
$C_6H_4(OH)_2 \cdot H_2O$	2664	$^{3}/_{2}$-x, 1-y, - $^{1}/_{2}$+z
	4746	2-x, - $^{1}/_{2}$+y, $^{3}/_{2}$-z
	4756	2-x, $^{1}/_{2}$+y, $^{3}/_{2}$-z
$3C_6H_4(OH)_2 \cdot 2H_2O$	1565	+x, 1+y, z

Reference

(1) Johnson, C. K. ORTEPII. report ORNL-5138, TN: Oak Ridge National Laboratory, Memphis, **1976**,