Crystal structures of non-proteinogenic amino acid peroxosolvates: rare example of H-bonded hydrogen peroxide chains.

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Experimental part

Amino acids and 50% hydrogen peroxide were purchased from Aldrich. 96% hydrogen peroxide was prepared by an extraction method.[1] Handling procedures for concentrated hydrogen peroxide are described in detail (danger of explosion!).[2,3] Briefly, 0.42 g of amino acid was dissolved in 0.5 ml of hydrogen peroxide. Colourless crystals of **1** and **2** were obtained by cooling to -18° C saturated solutions (rt) of respective anhydrous amino acids in 96% hydrogen peroxide. Yield 43 and 28% for 1 and 2 respectively.

Peroxide content was estimated by permanganometry. Carbon, hydrogen and nitrogen content was determined using the Perkin-Elmer 2400 series II Analyzer (CHN).

Anal. Calc. for $H_9O_4C_3N$ (1): OO (peroxide), 25.99; N, 11.38; C, 29.27; H, 7.37. Found: OO (peroxide), 25.20; N, 11.14; C, 29.16; H, 7.35.

Anal. Calc. for $H_{13}O_5C_9N$ (1): OO (peroxide), 14.87; N, 6.51; C, 50.23; H, 6.09. Found: OO (peroxide), 14.34; N, 6.61; C, 50.23; H, 6.09.

1 Y. Wolanov, O. Lev, A. V. Churakov, A. G. Medvedev, V. M. Novotortsev and P. V. Prikhodchenko, *Tetrahedron*, 2010, **66**, 5130.
2 W. C. Schumb, C. N. Satterfield and R. P. Wentworth, *Hydrogen peroxide*, Reinhold publishing corp., New York, 1955.
3 O. Maass and W. H. Hatcher, *J. Amer. Chem. Soc.*, 1920, **42**, 2569.

Table S1. Crystal data, data collection and refinement parameters for **1** and **2.**

	1	**2**
formula	$C_3H_7NO_2 \cdot H_2O_2$	$C_9H_{11}NO_3 \cdot H_2O_2$
fw	123.11	215.20
colour, habit	colourless prism	colourless needle
cryst size, mm	0.30×0.25×0.25	0.38×0.05×0.05
crystal system	monoclinic	tetragonal
space group	*P*c	$P\text{-}42_1c$
a, Å	13.1669(7)	18.7167(5)
b, Å	6.3974(4)	18.7167(5)
c, Å	10.7817(6)	5.7741(3)
β, deg	90.605(1)	90
V, Å^3	908.13(9)	2022.75(13)
Z	6	8
ρ_{calc}, g/cm^3	1.351	1.413
μ, mm^{-1}	0.125	0.116
F(000)	396	912
θ range, deg	3.09 to 30.50	2.18 to 29.98

total no. of reflns	10591	23552
unique reflns, R_{int}	2766, 0.0172	1693, 0.0429
reflns with $I>2\sigma(I)$	2700	1518
no. of variables	325	188
R_1 ($I>2\sigma(I)$)	0.0243	0.0299
wR_2 (all data)	0.0669	0.0778
Goof on F^2	1.046	1.055
largest diff peak/hole, e/Å^3	0.263 / -0.130	0.327 / -0.148

Methods

Differential scanning calorimetry (DSC) was performed on differential scanning calorimeter, DSC-60 PLUS, Shimadzu. Experiments were carried out in the range starting from the +20˚C following heating to +150˚C under argon flow at a heating rate of 5°C/min. Noise level less than 0.5 μW.

X-ray powder diffraction measurements were performed on a D8 Advance diffractometer (Bruker AXS, Karlsruhe, Germany). XRD patterns in the range 5˚ to 75˚ 2θ were recorded at room temperature using CuKα radiation under the following measurement conditions: reflection geometry, tube voltage of 40 kV, tube current of 40 mA, Ni filter, LYNXEYE detector, step scan mode with a step size 0.02˚ 2θ, and counting time of 0.5 s/step. XRD patterns were processed by DIFFRAC.SUITE Eva (Bruker), DIFFRAC.SUITE TOPAS 4.2 (Bruker) software. Calculated powder patterns were obtained using Mercury (CCDC) software and CIF files.

X-ray powder diffraction

Sarcosine peroxosolvate $C_3H_7NO_2 \cdot H_2O_2$ (**1**) and phenylserine peroxosolvate $C_9H_{11}NO_3 \cdot H_2O_2$ (**2**) crystals were isolated from hydrogen peroxide solution and dried on filter paper. Obtained crystals were carefully ground in a mortar. Resulting powder and initial crystals were placed to low background Si sample holder. X-ray powder diffractograms of **1** and **2** are presented in the Figure S1. Sample displacement correction was taken into account in DIFFRAC.SUITE Eva (Bruker) software. The peak position mismatch caused by different temperatures of X-ray analysis (150 K) and powder diffraction (298 K) experiments.

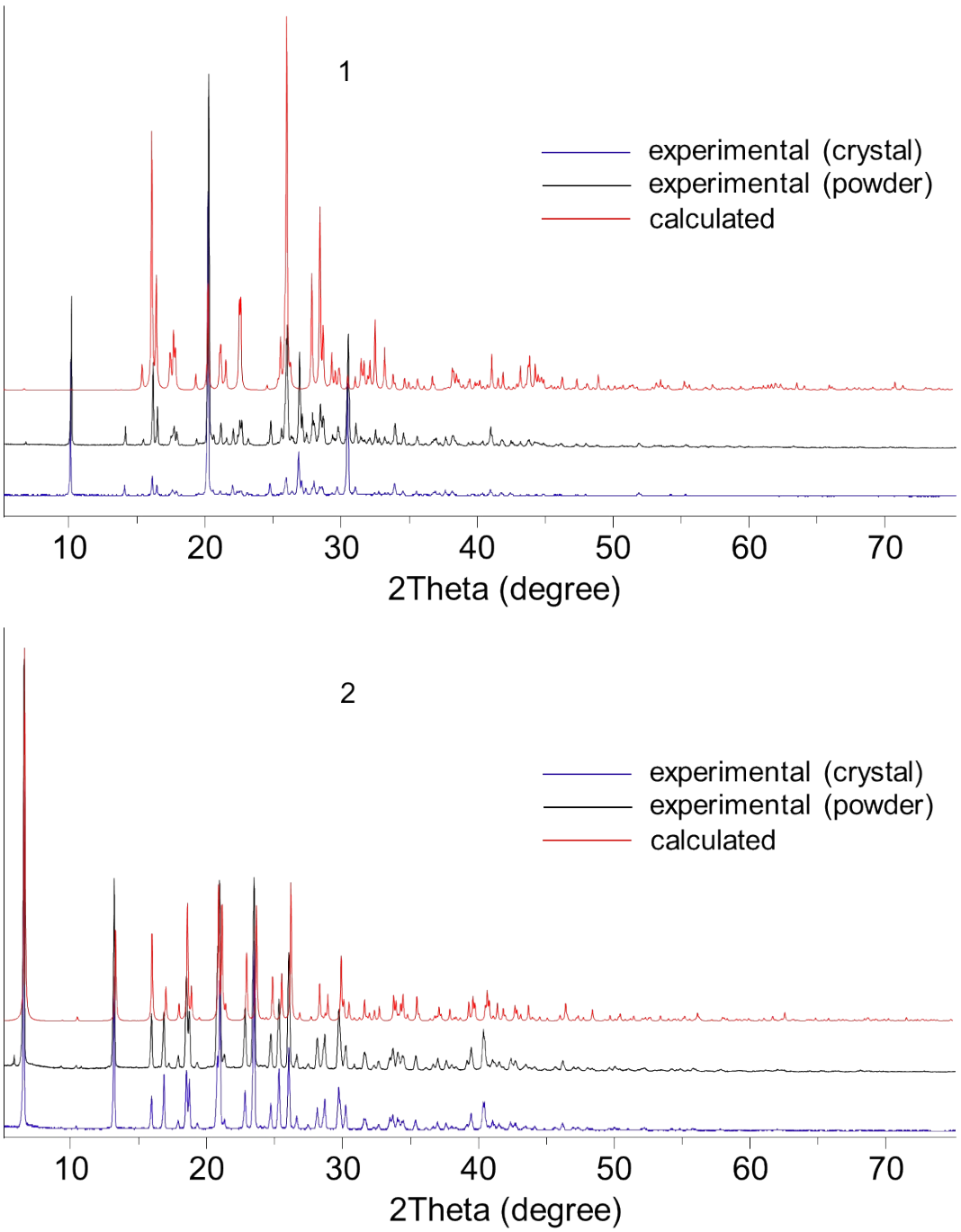

Figure S1. X-ray powder diffractograms of sarcosine $C_3H_7NO_2 \cdot H_2O_2$ (**1**) and phenylserine $C_9H_{11}NO_3 \cdot H_2O_2$ (**2**) peroxosolvates. Calculated powder diffractograms was obtained using Mercury (CCDC) software.

Graphical representations of the Rietweld refinement results obtained sarcosine $C_3H_7NO_2 \cdot H_2O_2$ (**1**) and phenylserine $C_9H_{11}NO_3 \cdot H_2O_2$ (**2**) peroxosolvates are presented in Figure S2. Sample displacement (mm) and unit cell parameters were refined, spherical harmonics was applied for preferred orientation correction. Sample **1** contains an unidentified phase before and after grinding which can be attributed to the possible phase transition or decomposition product. Unfortunately we cannot perform indexing of additional peaks due to their small amount. Sample 2 contains a small peaks at 5.7° after grinding. We used initial dried crystals powder diffractogram for Rietweld refinement with hkl phase obtained from crystallographic file. Rietweld refinement results presented in Table S2.

In order to shed light on the question of a possible phase transition when the crystals are cooled from room temperature to the low temperatures, we performed differential scanning calorimetry (DSC) studies for compound **1** starting from the room temperature and cooling down to -90°C and then heating to room temperature (see DSC results section).

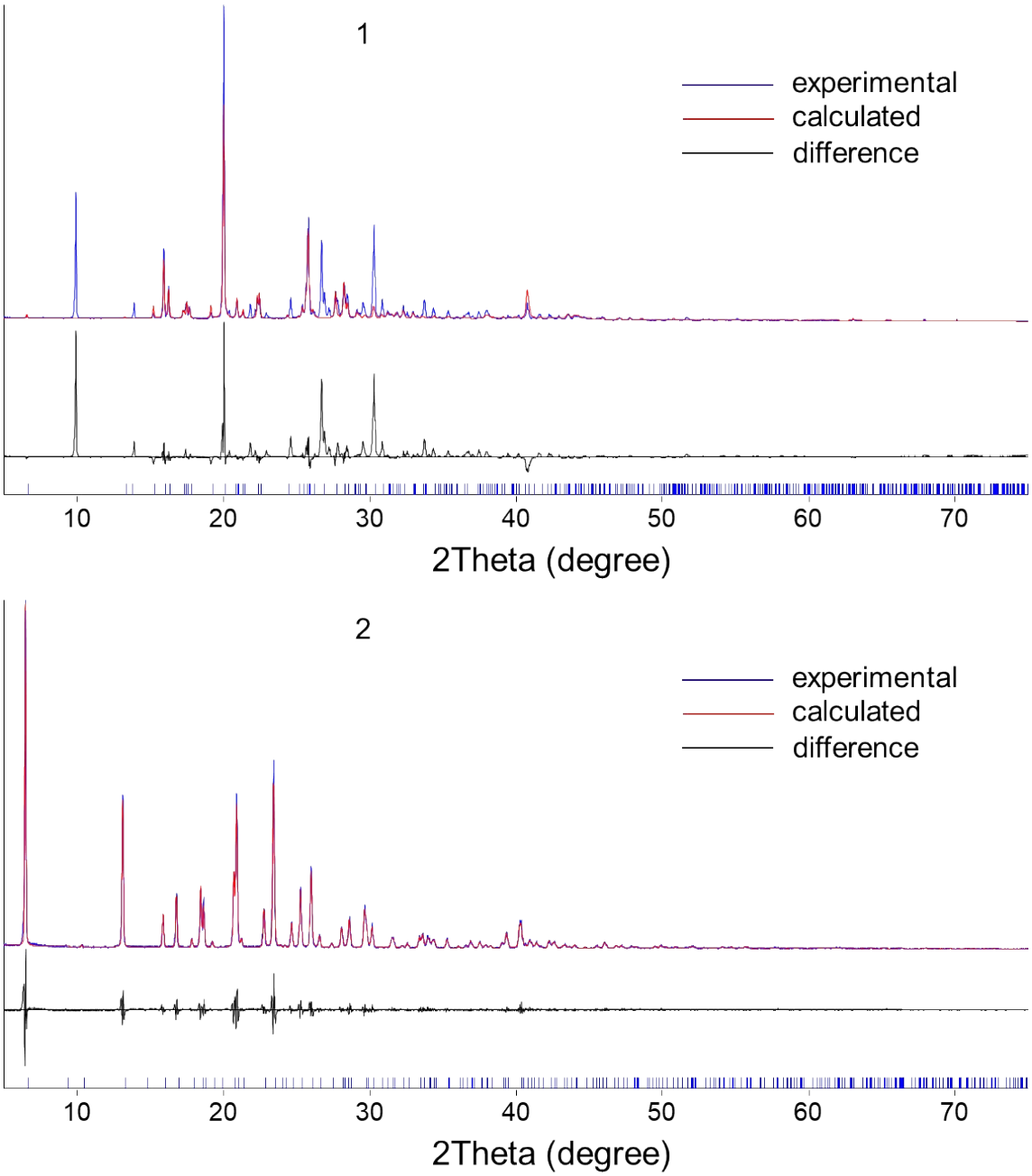

Figure S2. Rietweld refinement results for sarcosine $C_3H_7NO_2 \bullet H_2O_2$ (**1**) and phenylserine $C_9H_{11}NO_3 \bullet H_2O_2$ (**2**).

Table S2. Rietweld refinement results for **1** and **2.**

Compound	Results

Compound	Results
1	**R-Values** Rexp : 2.84 Rwp : 36.42 Rp : 25.56 GOF : 12.81 Rexp`: 4.97 Rwp`: 63.65 Rp` : 55.95 DW : 0.03 **Quantitative Analysis - Rietveld** Phase 1 : Structure 100.000 % **Background** Chebychev polynomial, Coefficient 0 676.7349 1 -335.5877 2 -49.99457 **Instrument** Primary radius (mm) 280 Secondary radius (mm) 280 **Corrections** Specimen displacement 0.3286099 LP Factor 0 **Miscellaneous** Start X 5 Finish X 75 **Structure 1** Phase name Structure R-Bragg 21.375 Spacegroup Pc Scale 0.00818649881 Cell Mass 738.653 Cell Volume (Å^3) 922.45889 Wt% - Rietveld 100.000 Crystal Linear Absorption Coeff. (1/cm) 10.874 Crystal Density (g/cm^3) 1.330 Preferred Orientation (Dir 1 : 1 0 0) 0.306198 Preferred Orientation Spherical Harmonics Order 4 y00 1 y20 -0.88272 y22m -1.276885 y22p 0.2058258 y40 -0.08925413 y42m -0.1280573 y42p 0.1137169 y44m -0.1786822 y44p -0.6127033 PV_TCHZ peak type U -0.03079052 V -0.03434624 W 0.008466503 Z 0 X 0.296459 Y 0 Lattice parameters a (Å) 13.2436052 b (Å) 6.4269771 c (Å) 10.8387319 beta (°) 90.81957 Site Np x y z Atom Occ Beq

Compound	Results							
	O11	2	0.91975	0.32810	0.55751	O	1	4
	O12	2	1.06642	0.43582	0.47590	O	1	4
	N1	2	0.91166	-0.02708	0.42951	N	1	4
	C11	2	0.99453	0.30714	0.48827	C	1	4
	C12	2	1.00037	0.11080	0.40891	C	1	4
	C13	2	0.90630	-0.20430	0.34027	C	1	4
	H1A	2	0.91500	-0.07900	0.50900	H	1	5
	H1B	2	0.85760	0.04200	0.42180	H	1	5
	H12A	2	1.05900	0.03400	0.42800	H	1	5
	H12B	2	1.00030	0.15500	0.32800	H	1	5
	H13A	2	0.96850	-0.27900	0.34900	H	1	5
	H13B	2	0.85030	-0.29000	0.35700	H	1	5
	H13C	2	0.89940	-0.15400	0.26700	H	1	5
	O21	2	0.57687	0.28724	0.37058	O	1	4
	O22	2	0.73447	0.16184	0.35526	O	1	4
	N2	2	0.57599	0.29469	0.61644	N	1	4
	C21	2	0.65874	0.22223	0.41551	C	1	4
	C22	2	0.66822	0.20994	0.55623	C	1	4
	C23	2	0.57411	0.25400	0.75253	C	1	4
	H2A	2	0.57430	0.43400	0.60370	H	1	5
	H2B	2	0.52300	0.23800	0.58340	H	1	5
	H22A	2	0.72580	0.29300	0.58600	H	1	5
	H22B	2	0.67350	0.06800	0.58000	H	1	5
	H23A	2	0.57250	0.11200	0.76100	H	1	5
	H23B	2	0.63040	0.31100	0.79200	H	1	5
	H23C	2	0.51580	0.32000	0.79000	H	1	5
	O31	2	0.39519	0.11307	0.55471	O	1	4
	O32	2	0.24511	0.06396	0.46043	O	1	4
	N3	2	0.24333	0.42174	0.33997	N	1	4
	C31	2	0.32356	0.16685	0.48315	C	1	4
	C32	2	0.33368	0.37795	0.41938	C	1	4
	C33	2	0.24607	0.63129	0.28049	C	1	4
	H3A	2	0.23990	0.32300	0.28270	H	1	5
	H3B	2	0.19200	0.40900	0.37850	H	1	5
	H32A	2	0.39090	0.37500	0.36610	H	1	5
	H32B	2	0.33930	0.47800	0.48200	H	1	5
	H33A	2	0.24970	0.73100	0.34100	H	1	5
	H33B	2	0.30620	0.63900	0.23300	H	1	5
	H33C	2	0.18730	0.64300	0.23200	H	1	5
	O1	2	0.88543	0.28896	0.14211	O	1	4
	O2	2	0.77486	0.28285	0.12250	O	1	4
	H1	2	0.90100	0.41800	0.11400	H	1	5
	H2	2	0.75780	0.25400	0.19500	H	1	5
	O3	2	0.54494	0.26326	0.12950	O	1	4
	O4	2	0.43358	0.28186	0.13102	O	1	4
	H3	2	0.56020	0.27200	0.21000	H	1	5
	H4	2	0.41990	0.16500	0.10400	H	1	5
	O5	2	0.21153	0.24876	0.10919	O	1	4
	O6	2	0.10071	0.23925	0.12388	O	1	4
	H5	2	0.21960	0.14100	0.06700	H	1	5
	H6	2	0.08570	0.34100	0.08370	H	1	5
2	**R-Values**							
	Rexp : 6.09 Rwp : 14.92 Rp : 11.03 GOF : 2.45 Rexp`: 7.86 Rwp`: 19.25 Rp` : 14.81 DW : 0.42							
	Quantitative Analysis - Rietveld							
	Phase 1 : hkl_Phase 0.000 %							
	Background							
	One on X 790(35)							
	Chebychev polynomial, Coefficient 0 64.8(17)							

Compound	Results

1	-44.7(18)
2	-11.90(99)

Instrument

Primary radius (mm)	280
Secondary radius (mm)	280

Corrections

Specimen displacement	0.40638(93)
LP Factor	0

Miscellaneous

Start X	5
Finish X	75

hkl Phase - 1 Lebail method

Phase name	hkl_Phase
R-Bragg	1.170
Spacegroup	"p -4 21c"
Cell Mass	0.000
Cell Volume (Å^3)	2065.553(87)
Wt% - Rietveld	0.000
PV_TCHZ peak type	
U	0.042(11)
V	-0.0015(34)
W	0.00632(23)
Z	0
X	0.1888(40)
Y	0
Lattice parameters	
a (Å)	18.89140(23)
c (Å)	5.78773(20)

h	k	l	m	d	Th2	I
1	1	0	4	13.35824	6.61155	2.45
0	2	0	4	9.44570	9.35534	0.0105
2	1	0	8	8.44849	10.46251	0.0317
2	2	0	4	6.67912	13.24524	5.11
3	1	0	8	5.97399	14.81692	0.0306
0	1	1	8	5.53384	16.00284	1.82
3	2	0	8	5.23953	16.90814	3.21
0	2	1	8	4.93499	17.95993	0.619
2	1	1	16	4.77476	18.56786	4.89
0	4	0	4	4.72285	18.77378	3.36
4	1	0	8	4.58184	19.35704	0.565
3	3	0	4	4.45275	19.92387	0.04
0	3	1	8	4.26127	20.82892	7.69
4	2	0	8	4.22425	21.01353	15.4
3	1	1	16	4.15683	21.35830	0.817
3	2	1	16	3.88429	22.87642	5.28
4	3	0	8	3.77828	23.52733	24.4
5	1	0	8	3.70491	24.00013	0.255
0	4	1	8	3.65917	24.30465	0.332
4	1	1	16	3.59240	24.76348	4.21
5	2	0	8	3.50805	25.36878	10.7
4	2	1	16	3.41209	26.09460	15.3
4	4	0	4	3.33956	26.67169	2.62
5	3	0	8	3.23985	27.50843	1.11
0	5	1	8	3.16381	28.18308	2.49
4	3	1	16	3.16381	28.18308	2.49
0	6	0	4	3.14857	28.32234	0.168
5	1	1	16	3.12035	28.58386	0.929

Compound	Results						
	6	1	0	8	3.10573	28.72134	7.11
	5	2	1	16	3.00000	29.75654	10.5
	6	2	0	8	2.98699	29.88910	4.32
	5	4	0	8	2.95034	30.26923	6.21
	0	0	2	2	2.89386	30.87455	0.362
	0	1	2	8	2.86050	31.24383	0.273
	1	1	2	8	2.82826	31.60923	1.38
	5	3	1	16	2.82705	31.62305	1.52
	6	3	0	8	2.81616	31.74855	2.44
	0	2	2	8	2.76692	32.32891	0.448
	0	6	1	8	2.76579	32.34245	0.435
	2	1	2	16	2.73771	32.68344	0.885
	6	1	1	16	2.73662	32.69685	0.941
	7	1	0	8	2.67165	33.51519	2.03
	5	5	0	4	2.67165	33.51519	2.03
	2	2	2	8	2.65534	33.72713	2.38
	6	2	1	16	2.65435	33.74018	2.86
	0	3	2	8	2.62949	34.06874	1.54
	5	4	1	16	2.62853	34.08167	1.94
	6	4	0	8	2.61977	34.19913	2.25
	3	1	2	16	2.60439	34.40737	2.25
	7	2	0	8	2.59493	34.53664	2.76
	3	2	2	16	2.53317	35.40619	2.14
	6	3	1	16	2.53231	35.41869	2.15
	7	3	0	8	2.48056	36.18278	1
	0	4	2	8	2.46749	36.38109	0.0594
	4	1	2	16	2.44671	36.70108	0.752
	0	7	1	8	2.44593	36.71320	0.807
	3	3	2	8	2.42645	37.01869	1.97
	7	1	1	16	2.42569	37.03072	1.85
	6	5	0	8	2.41880	37.14004	0.37
	4	2	2	16	2.38738	37.64701	1.64
	6	4	1	16	2.38666	37.65887	2
	7	2	1	16	2.36783	37.96962	0.454
	0	8	0	4	2.36143	38.07664	1.78
	7	4	0	8	2.34319	38.38451	0.715
	8	1	0	8	2.34319	38.38451	0.715
	4	3	2	16	2.29742	39.18022	1.54
	0	5	2	8	2.29742	39.18022	1.54
	8	2	0	8	2.29092	39.29589	0.639
	5	1	2	16	2.28061	39.48082	4.37
	7	3	1	16	2.27998	39.49221	5.4
	5	2	2	16	2.23233	40.37139	5.18
	6	5	1	16	2.23174	40.38257	6.37
	6	6	0	4	2.22637	40.48421	11.4
	8	3	0	8	2.21107	40.77679	1.58
	7	5	0	8	2.19608	41.06765	5.32
	4	4	2	8	2.18700	41.24597	1.1
	0	8	1	8	2.18644	41.25695	0.935
	8	1	1	16	2.17194	41.54504	2.19
	7	4	1	16	2.17194	41.54504	2.19
	5	3	2	16	2.15826	41.82064	1.31
	0	6	2	8	2.13063	42.38895	2.84
	8	2	1	16	2.13012	42.39970	3.17
	6	1	2	16	2.11721	42.67080	2.22
	8	4	0	8	2.11212	42.77864	3.9
	9	1	0	8	2.08621	43.33675	1.52
	6	2	2	16	2.07841	43.50748	2.95
	5	4	2	16	2.06595	43.78352	0.692
	8	3	1	16	2.06548	43.79399	0.711
	7	5	1	16	2.05324	44.06859	0.284
	9	2	0	8	2.04906	44.16329	1.55
	7	6	0	8	2.04906	44.16329	1.55

Compound	Results					
	6	3	2	16	2.01824	44.87413	1.28
	8	5	0	8	2.00248	45.24665	0.454
	9	3	0	8	1.99133	45.51431	0.645
	8	4	1	16	1.98413	45.68870	2.78
	0	7	2	8	1.97369	45.94428	0.102
	0	9	1	8	1.97328	45.95436	0.093
	7	1	2	16	1.96300	46.20879	2.21
	5	5	2	8	1.96300	46.20879	2.21
	9	1	1	16	1.96260	46.21883	2.58
	6	4	2	16	1.94215	46.73434	0.195
	7	2	2	16	1.93196	46.99542	1.02
	9	2	1	16	1.93158	47.00533	1.07
	7	6	1	16	1.93158	47.00533	1.07
	0	1	3	8	1.91926	47.32539	1.24
	9	4	0	8	1.91813	47.35495	1.98
	7	7	0	4	1.90832	47.61342	0.392
	8	5	1	16	1.89242	48.03862	2.09
	0	2	3	8	1.89022	48.09802	0.522
	0	10	0	4	1.88914	48.12721	0.25
	8	6	0	8	1.88914	48.12721	0.25
	7	3	2	16	1.88335	48.28462	0.121
	9	3	1	16	1.88299	48.29433	0.143
	2	1	3	16	1.88083	48.35349	0.571
	10	1	0	8	1.87976	48.38256	1.21
	6	5	2	16	1.85588	49.04583	0.611
	10	2	0	8	1.85245	49.14264	0.275
	0	3	3	8	1.84461	49.36543	0.147
	3	1	3	16	1.83588	49.61602	1.19
	9	5	0	8	1.83490	49.64454	1.65
	0	8	2	8	1.82959	49.79837	0.423
	8	1	2	16	1.82107	50.04736	1.28
	7	4	2	16	1.82107	50.04736	1.28
	9	4	1	16	1.82074	50.05680	1.42
	3	2	3	16	1.81042	50.36226	0.867
	10	3	0	8	1.80947	50.39048	1.2
	8	2	2	16	1.79620	50.78896	0.445
	0	10	1	8	1.79589	50.79831	0.462
	8	6	1	16	1.79589	50.79831	0.462
	10	1	1	16	1.78783	51.04374	1.44
	0	4	3	8	1.78598	51.10054	1.03
	4	1	3	16	1.77805	51.34492	0.221
	8	7	0	8	1.77715	51.37275	0.273
	6	6	2	8	1.76458	51.76577	0.833
	10	2	1	16	1.76429	51.77499	0.838
	8	3	2	16	1.75693	52.00792	0.867
	4	2	3	16	1.75489	52.07315	0.824
	10	4	0	8	1.75402	52.10070	0.839
	7	5	2	16	1.74938	52.24927	1.41
	9	5	1	16	1.74910	52.25843	1.39
	9	6	0	8	1.74651	52.34175	0.754
	10	3	1	16	1.72703	52.97774	1.71
	0	5	3	8	1.71821	53.27114	0.486
	4	3	3	16	1.71821	53.27114	0.486
	5	1	3	16	1.71115	53.50849	0.571
	11	1	0	8	1.71035	53.53552	0.637
	8	4	2	16	1.70605	53.68130	0.632
	0	9	2	8	1.69913	53.91740	0.133
	8	7	1	16	1.69887	53.92637	0.124
	9	1	2	16	1.69230	54.15280	0.406
	5	2	3	16	1.69047	54.21622	0.831
	11	2	0	8	1.68970	54.24301	1.15
	10	5	0	8	1.68970	54.24301	1.15
	10	4	1	16	1.67863	54.63038	1.86

Compound	Results					
	9	2	2	16	1.67229	54.85483	0.835
	7	6	2	16	1.67229	54.85483	0.835
	9	6	1	16	1.67204	54.86369	0.821
	8	8	0	4	1.66978	54.94426	0.521
	5	3	3	16	1.65761	55.38203	0.656
	11	3	0	8	1.65689	55.40843	0.705
	9	7	0	8	1.65689	55.40843	0.705
	8	5	2	16	1.64668	55.78158	1.41
	0	11	1	8	1.64644	55.79034	1.42
	0	6	3	8	1.64500	55.84376	1.18
	9	3	2	16	1.64046	56.01167	0.614
	11	1	1	16	1.64023	56.02041	0.619
	6	1	3	16	1.63880	56.07368	0.684
	11	2	1	16	1.62199	56.70692	0.408
	10	5	1	16	1.62199	56.70692	0.408
	6	2	3	16	1.62060	56.75977	0.361
	10	6	0	8	1.61992	56.78574	0.359
	5	4	3	16	1.61467	56.98726	0.453
	11	4	0	8	1.61400	57.01318	0.599
	9	4	2	16	1.59881	57.60538	2.09
	7	7	2	8	1.59311	57.83073	0.691
	11	3	1	16	1.59290	57.83929	0.743
	9	7	1	16	1.59290	57.83929	0.743
	6	3	3	16	1.59159	57.89147	1.19
	0	10	2	8	1.58190	58.27978	0.541
	8	6	2	16	1.58190	58.27978	0.541
	10	1	2	16	1.57639	58.50349	0.554
	0	12	0	4	1.57428	58.58926	0.271
	0	7	3	8	1.56946	58.78682	0.271
	12	1	0	8	1.56885	58.81223	0.304
	9	8	0	8	1.56885	58.81223	0.304
	7	1	3	16	1.56407	59.00932	0.369
	11	5	0	8	1.56346	59.03467	0.387
	10	2	2	16	1.56018	59.17143	0.977
	10	6	1	16	1.55997	59.17987	1.04
	11	4	1	16	1.55468	59.40147	0.65
	6	4	3	16	1.55346	59.45279	0.43
	12	2	0	8	1.55286	59.47802	0.346
	9	5	2	16	1.54964	59.61416	0.348
	7	2	3	16	1.54824	59.67376	0.663
	10	7	0	8	1.54764	59.69894	0.857
	10	3	2	16	1.53423	60.27451	1.97
	12	3	0	8	1.52728	60.57770	1.7
	7	3	3	16	1.52288	60.77130	1.5
	0	12	1	8	1.51909	60.93884	0.709
	8	7	2	16	1.51439	61.14830	0.921
	12	1	1	16	1.51420	61.15657	0.937
	9	8	1	16	1.51420	61.15657	0.937
	11	5	1	16	1.50936	61.37385	0.782
	6	5	3	16	1.50825	61.42418	0.905
	11	6	0	8	1.50770	61.44893	1.01
	10	4	2	16	1.50000	61.79884	1.29
	12	2	1	16	1.49982	61.80706	1.27
	9	6	2	16	1.49529	62.01481	1.03
	10	7	1	16	1.49511	62.02301	1.04
	0	8	3	8	1.49403	62.07304	0.928
	12	4	0	8	1.49350	62.09763	0.807
	8	1	3	16	1.48938	62.28846	1.05
	7	4	3	16	1.48938	62.28846	1.05
	9	9	0	4	1.48425	62.52795	2.82
	0	11	2	8	1.47690	62.87441	0.485
	12	3	1	16	1.47673	62.88255	0.458
	8	2	3	16	1.47568	62.93218	0.388

Compound	Results						
	10	8	0	8	1.47517	62.95658	0.411
	11	1	2	16	1.47241	63.08827	1.93
	11	2	2	16	1.45917	63.72745	0.854
	10	5	2	16	1.45917	63.72745	0.854
	11	6	1	16	1.45901	63.73553	0.783
	8	3	3	16	1.45368	63.99697	0.616
	12	5	0	8	1.45318	64.02114	0.608
	7	5	3	16	1.44939	64.20876	0.391
	13	1	0	8	1.44890	64.23289	0.407
	11	7	0	8	1.44890	64.23289	0.407
	0	0	4	2	1.44693	64.33099	0.632
	8	8	2	8	1.44629	64.36313	0.747
	12	4	1	16	1.44613	64.37116	0.776
	0	1	4	8	1.44271	64.54218	0.721
	1	1	4	8	1.43852	64.75301	0.48
	11	3	2	16	1.43788	64.78503	0.481
	9	7	2	16	1.43788	64.78503	0.481
	13	2	0	8	1.43629	64.86588	0.504
	0	2	4	8	1.43025	65.17356	1.74
	10	8	1	16	1.42947	65.21346	2.03
	2	1	4	16	1.42617	65.38331	1.21
	8	4	3	16	1.42445	65.47173	1.03
	0	9	3	8	1.42042	65.68097	0.826
	9	1	3	16	1.41642	65.88987	0.799
	13	3	0	8	1.41597	65.91367	0.729
	2	2	4	8	1.41413	66.01044	0.631
	10	6	2	16	1.41353	66.04216	0.684
	0	3	4	8	1.41018	66.21881	0.481
	11	4	2	16	1.40959	66.25047	0.455
	0	13	1	8	1.40944	66.25839	0.45
	12	5	1	16	1.40944	66.25839	0.45
	12	6	0	8	1.40808	66.33038	0.38
	3	1	4	16	1.40627	66.42684	0.375
	13	1	1	16	1.40553	66.46635	0.494
	11	7	1	16	1.40553	66.46635	0.494
	9	2	3	16	1.40463	66.51455	0.881
	7	6	3	16	1.40463	66.51455	0.881
	10	9	0	8	1.40419	66.53824	1.2
	3	2	4	16	1.39473	67.04898	1.74
	13	2	1	16	1.39400	67.08831	0.952
	8	5	3	16	1.38935	67.34289	0.739
	13	4	0	8	1.38892	67.36644	0.811
	11	8	0	8	1.38892	67.36644	0.811
	9	3	3	16	1.38561	67.54919	0.93
	0	4	4	8	1.38346	67.66827	0.447
	0	12	2	8	1.38290	67.69960	0.417
	4	1	4	16	1.37977	67.87409	0.749
	12	1	2	16	1.37921	67.90537	0.801
	9	8	2	16	1.37921	67.90537	0.801
	3	3	4	8	1.37610	68.07961	1.5
	11	5	2	16	1.37555	68.11084	1.98
	13	3	1	16	1.37541	68.11864	2.14
	4	2	4	16	1.36886	68.48976	0.986
	12	2	2	16	1.36831	68.52090	0.855
	12	6	1	16	1.36817	68.52869	0.831
	10	7	2	16	1.36474	68.72549	1.78
	10	9	1	16	1.36460	68.73327	1.89
	9	4	3	16	1.36023	68.98492	1.22
	12	7	0	8	1.35983	69.00819	1.16
	13	5	0	8	1.35632	69.21212	2.54
	0	5	4	8	1.35124	69.51015	0.624
	4	3	4	16	1.35124	69.51015	0.624
	12	3	2	16	1.35071	69.54108	0.511

Compound	Results						
	13	4	1	16	1.35058	69.54881	0.49
	11	8	1	16	1.35058	69.54881	0.49
	0	10	3	8	1.34978	69.59596	0.406
	8	6	3	16	1.34978	69.59596	0.406
	0	14	0	4	1.34939	69.61915	0.389
	5	1	4	16	1.34779	69.71340	0.538
	10	1	3	16	1.34635	69.79910	1.54
	14	1	0	8	1.34596	69.82226	2.24
	5	2	4	16	1.33762	70.32158	1.39
	11	6	2	16	1.33711	70.35235	1.01
	10	2	3	16	1.33621	70.40695	0.842
	14	2	0	8	1.33582	70.43002	0.889
	10	10	0	4	1.33582	70.43002	0.889
	9	5	3	16	1.32957	70.81091	1.74
	11	9	0	8	1.32919	70.83392	1.67
	4	4	4	8	1.32767	70.92746	1.53
	12	4	2	16	1.32717	70.95811	1.55
	12	7	1	16	1.32379	71.16720	1.31
	5	3	4	16	1.32116	71.33015	0.559
	9	9	2	8	1.32067	71.36073	0.6
	13	5	1	16	1.32055	71.36837	0.62
	10	3	3	16	1.31980	71.41500	0.859
	14	3	0	8	1.31943	71.43793	1.05
	13	6	0	8	1.31943	71.43793	1.05
	0	6	4	8	1.31475	71.73188	1.35
	10	8	2	16	1.31426	71.76239	1.1
	0	14	1	8	1.31414	71.77002	1.05
	6	1	4	16	1.31157	71.93240	0.758
	14	1	1	16	1.31097	71.97049	0.913
	12	8	0	8	1.30988	72.03980	1.32
	8	7	3	16	1.30710	72.21715	3.65
	6	2	4	16	1.30219	72.53259	2.1
	14	2	1	16	1.30161	72.57056	1.62
	5	4	4	16	1.29911	72.73221	0.694
	0	13	2	8	1.29864	72.76255	0.641
	12	5	2	16	1.29864	72.76255	0.641
	10	4	3	16	1.29782	72.81639	0.536
	14	4	0	8	1.29747	72.83914	0.498
	13	1	2	16	1.29559	72.96192	0.947
	11	7	2	16	1.29559	72.96192	0.947
	11	9	1	16	1.29547	72.96950	1.07
	9	6	3	16	1.29476	73.01571	2.89
	6	3	4	16	1.28700	73.52856	2.12
	13	2	2	16	1.28654	73.55878	1.23
	14	3	1	16	1.28643	73.56633	1.1
	13	6	1	16	1.28643	73.56633	1.1
	0	11	3	8	1.28277	73.81088	1.52
	11	1	3	16	1.27982	74.00917	1.36
	13	7	0	8	1.27949	74.03178	1.39
	12	8	1	16	1.27757	74.16134	2.16
	0	7	4	8	1.27521	74.32166	1.82
	7	1	4	16	1.27232	74.51945	0.633
	5	5	4	8	1.27232	74.51945	0.633
	13	3	2	16	1.27188	74.54951	0.659
	11	2	3	16	1.27110	74.60287	0.815
	10	5	3	16	1.27110	74.60287	0.815
	14	5	0	8	1.27077	74.62541	0.917
	11	10	0	8	1.27077	74.62541	0.917
	6	4	4	16	1.26659	74.91446	1.5
	12	6	2	16	1.26615	74.94447	1.48
	14	4	1	16	1.26604	74.95197	1.49
	7	2	4	16	1.26375	75.11169	2.41
	10	9	2	16	1.26332	75.14167	2.58

Compound	Results						
	12	9	0	8	1.25943	75.41431	2.11

DSC analysis

DSC experiment was performed for compound **1**. The crystals were isolated from hydrogen peroxide solution, quickly dried on filter paper and closed in aluminum crucible to prevent exposure to air. The results of DSC studies are depicted on the Figure S3. We observed reversible pair of peaks: exothermal effect on cooling with maximum at –58°C and endothermal effect on heating with minimum at –31°C. These thermal effects can attribute to phase transition and explain additional phase in crystalline powder of **1**.

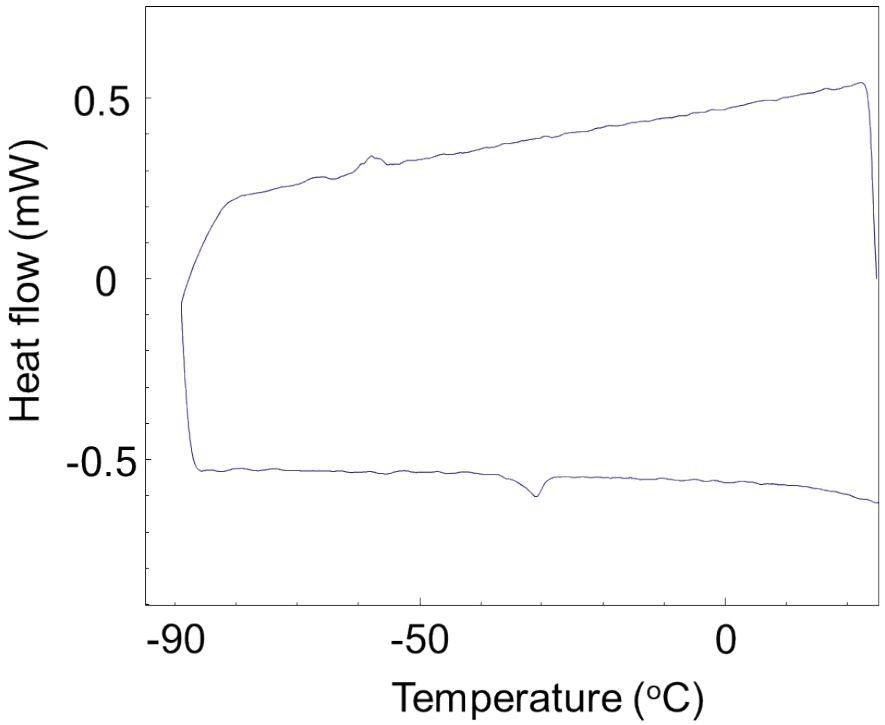

Figure S3. Differential scanning calorimetry of sarcosine $C_3H_7NO_2 \bullet H_2O_2$ peroxosolvate (**1**).