Electronic Supplementary Information

Tetrahydroxidohexaoxidopentaborate(1-) salts of C$_6$- linked substituted diimidazolium and dipyrrolidinium cations: synthesis, characterization and XRD studies.

Ahmad R. Al-Dulayymi [1], Michael A. Beckett [2],* Radek Braganca [1], Simon J. Coles [3], Peter N. Horton [3], and Thomas A. Rixon [2]

[1]   Bio-composites Centre, Bangor University, Bangor Gwynedd, LL57 2UW, UK.

[2]   School of Environmental and Natural Sciences, Bangor University, Bangor, LL57 2UW, UK.

[3]   School of Chemistry, University of Southampton, Southampton, SO17 1BJ, UK

* Correspondence: m.a.beckett@bangor.ac.uk

$[CH_3(C_3H_3N_2)(CH_2)_6(C_3H_3N_2)CH_3][B_5O_6(OH)_4]_2$ (**1**).

$[C_2H_5(C_3H_3N_2)(CH_2)_6(C_3H_3N_2)C_2H_5][B_5O_6(OH)_4]_2 \cdot 3H_2O$ (**2**).

$[CH_3(C_3H_3N_2)CH_2(C_6H_4)CH_2(C_3H_3N_2)CH_3][B_5O_6(OH)_4]_2$ (**3**).

$[CH_3(C_4H_8N)(CH_2)_6(C_4H_8N)CH_3][B_5O_6(OH)_4]_2$ (**4a** and **4b**).

$[C_2H_5(C_4H_8N)(CH_2)_6(C_4H_8N)C_2H_5][B_5O_6(OH)_4]_2$ (**5**).

$[C_4H_9(C_4H_8N)(CH_2)_6(C_4H_8N)C_4H_9] [B_5O_6(OH)_4]_2 \cdot 4B(OH)_3$ (**6**).

$[C_3H_5(C_4H_8N)(CH_2)_6(C_4H_8N)C_3H_5][B_5O_6(OH)_4]_2$ (**7**).

IR plots of compounds 1-7
– Figures S1-S7.

^1H, ^{13}C and ^{11}B NMR spectra for compounds 1-2, 4-7
(all in D$_2$O)
– Figures S8-S25.

TGA/DSC plots of compounds 1 and 7
– Figures S26, S27.

Selected X-ray data for 1-3, 4a, 4b, 5-7
– Figures S28-43, and Tables S1-S59.
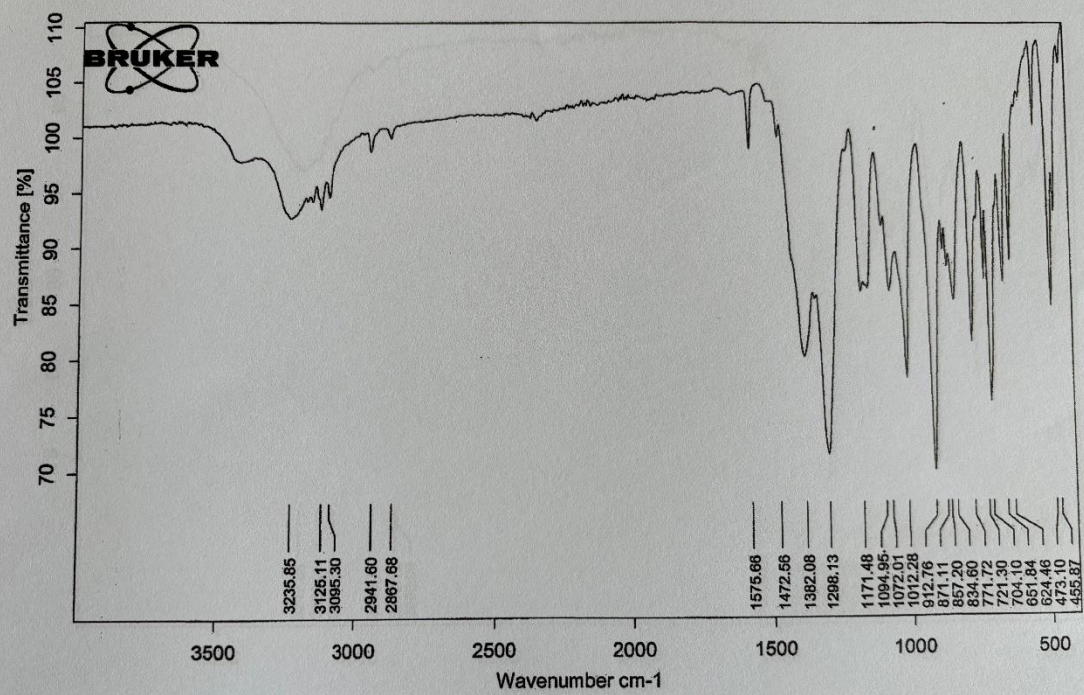
Figure S1: IR of **1**.

Figure S2: IR of **2**.

Analyst   FTIR User
Date   16 July 2018 20:25

Sample Name	Description	Quality Checks
AR 106 + B Crystals	Sample 011 date Thursday, July 05 2018	The Quality Checks give rise to multiple warnings for the sample.

3
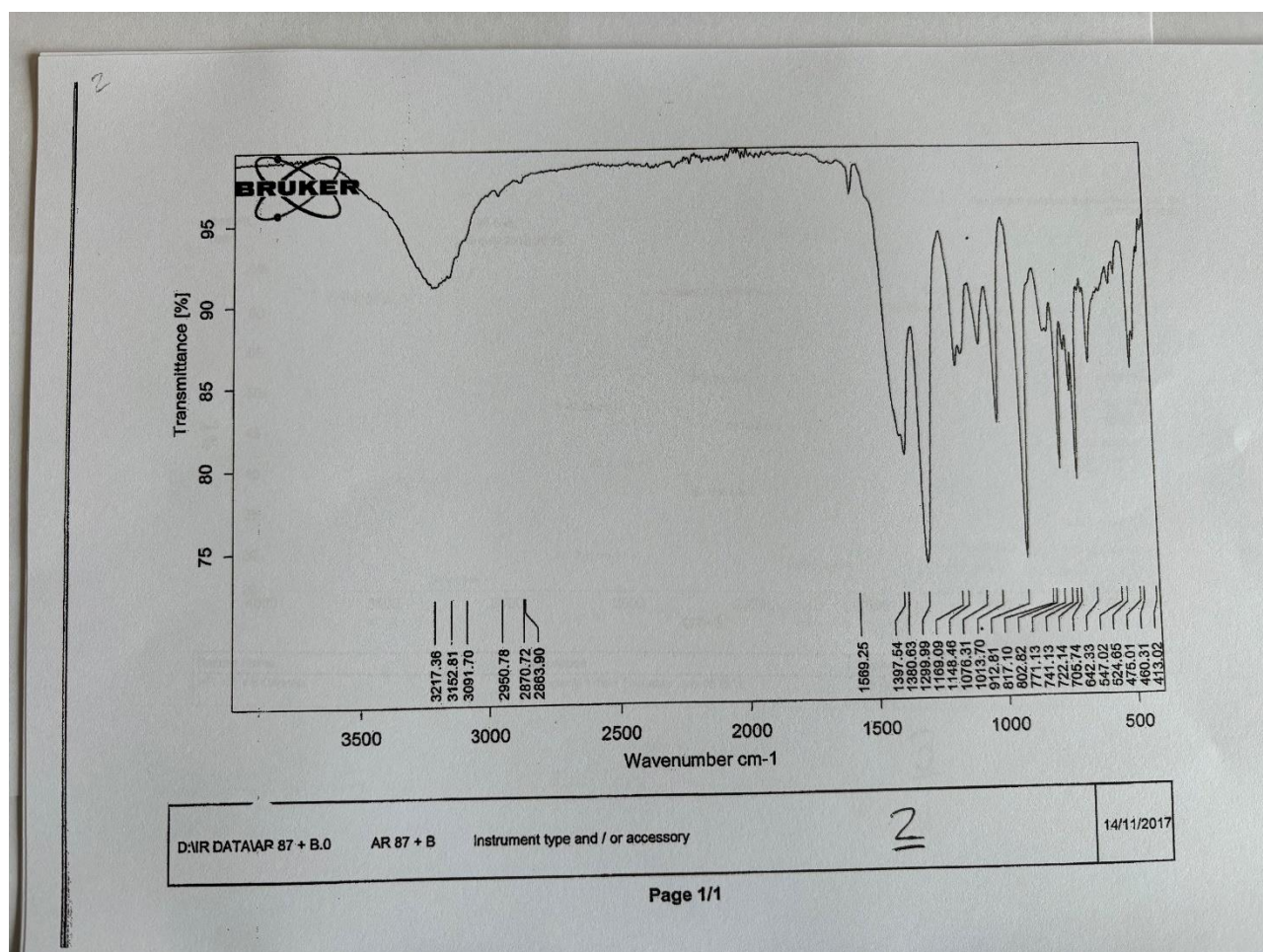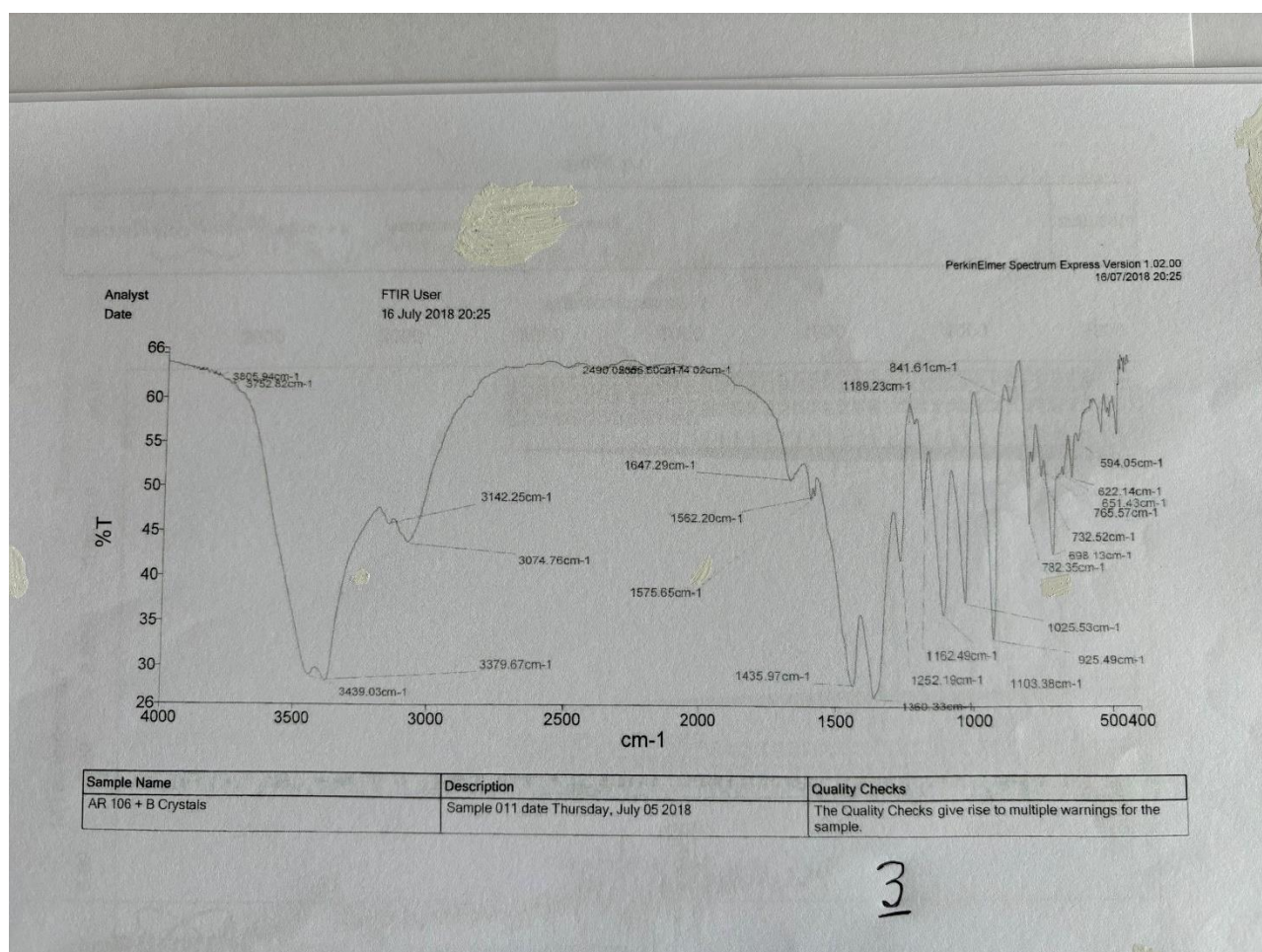
Figure S3: IR of **3**.
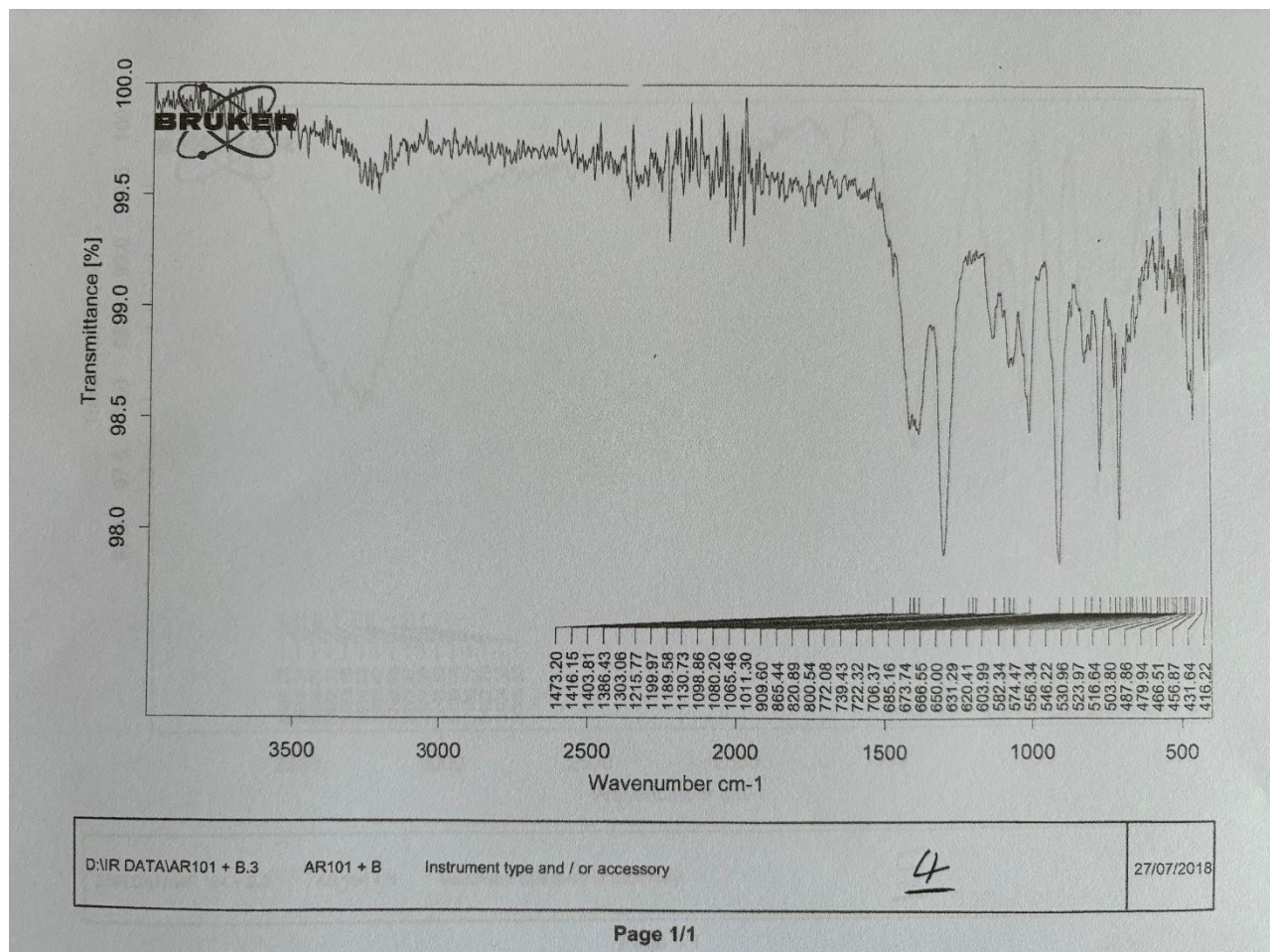
Figure S4: IR of **4**
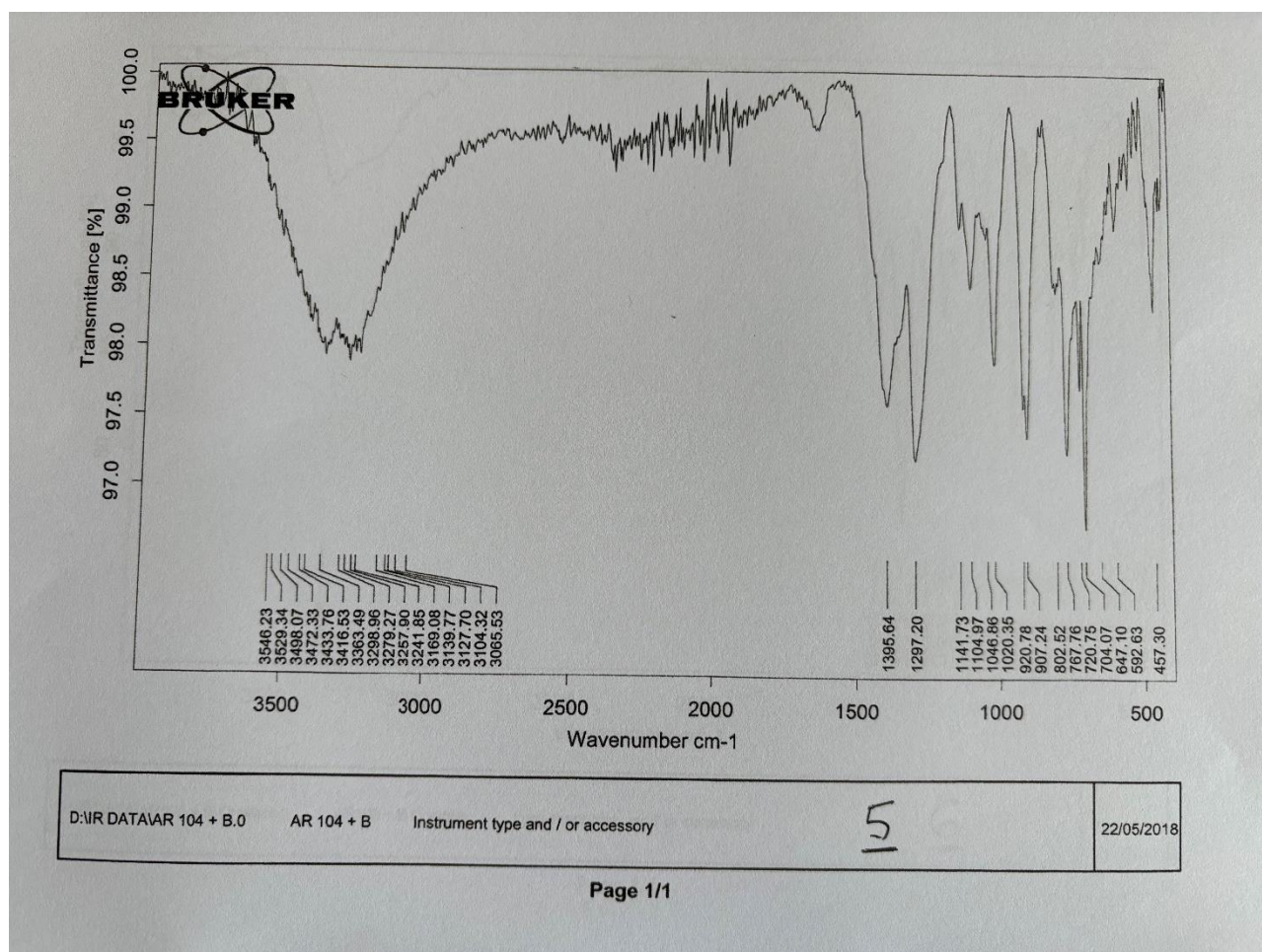
Figure S5: IR of **5**
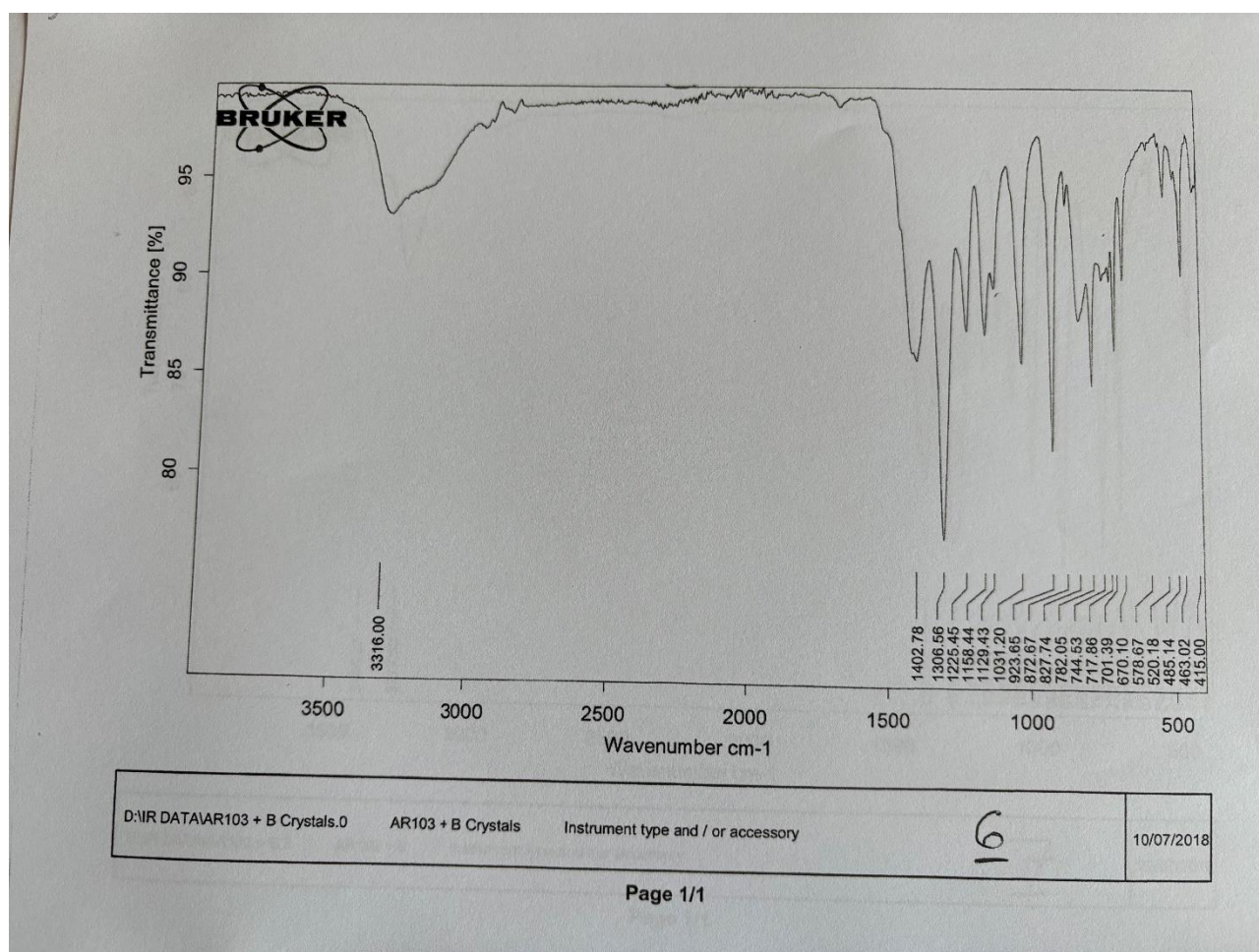
Figure S6: IR of **6**
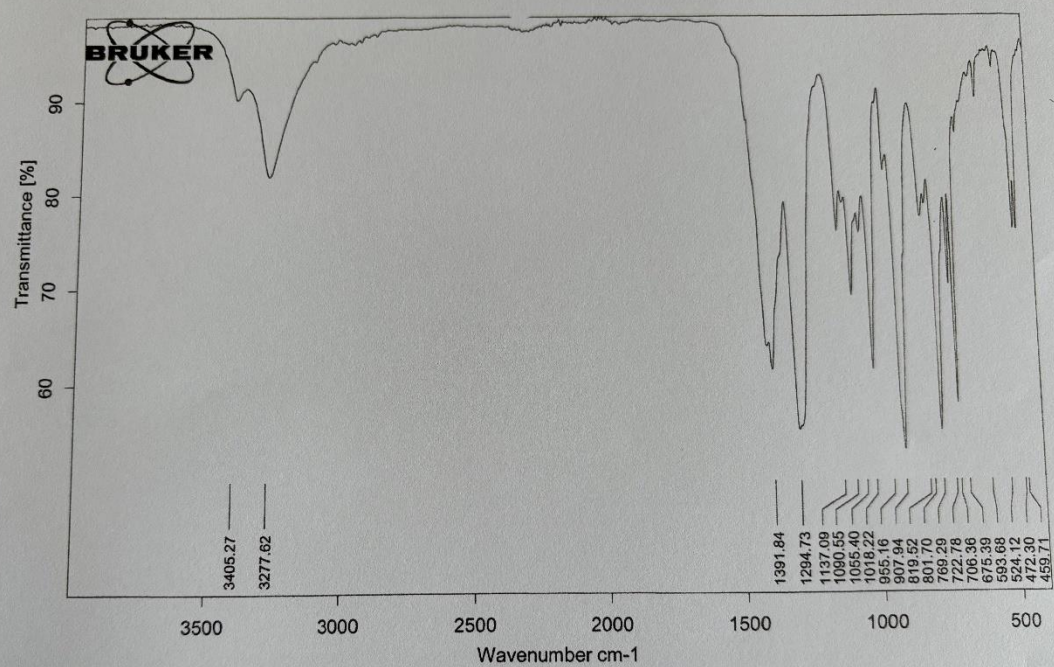
Figure S7: IR of **7**

1 *[CH₃(C₃H₃N₂)(CH₂)₆(C₃H₃N₂)CH₃][B₅O₆(OH)₄]₂*
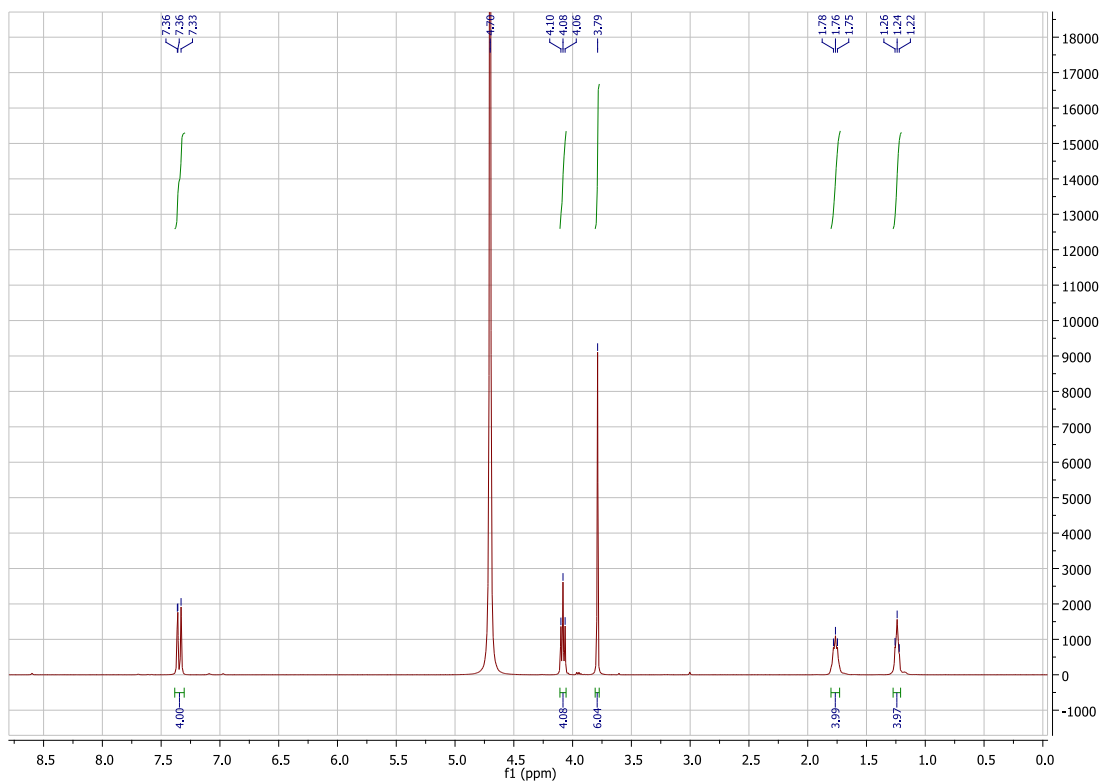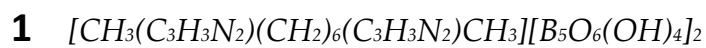
Figure S8: ¹H NMR (400 MHz) spectrum of **1**
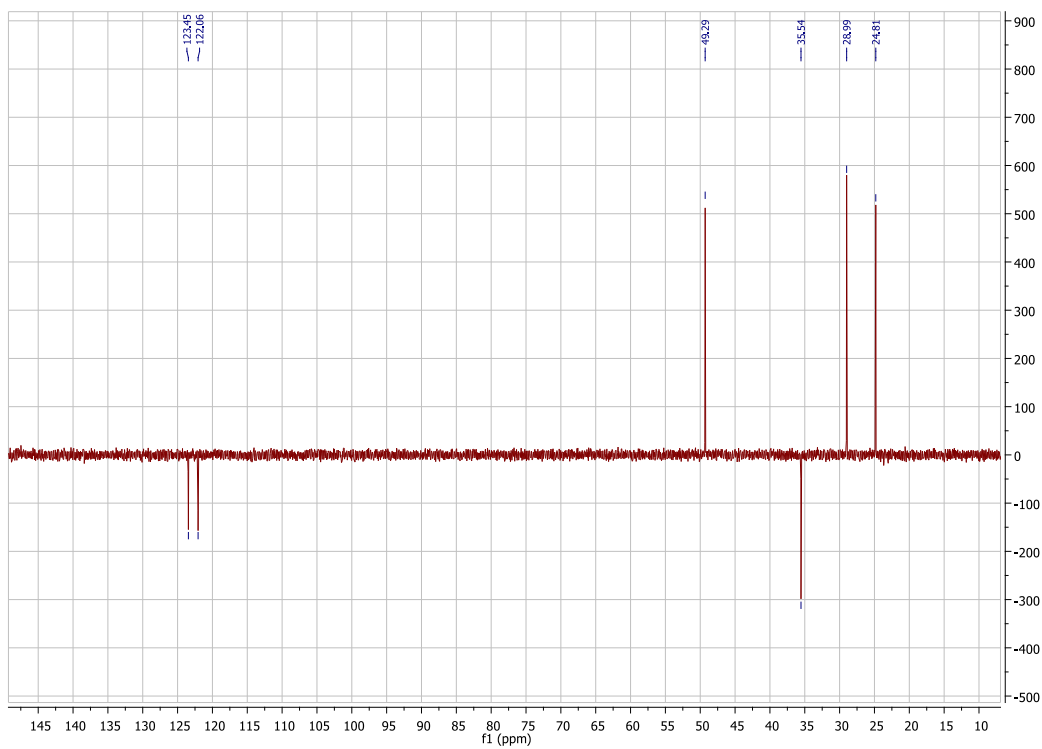
Figure S9: ¹³C DEPT NMR (100 MHz) spectrum of **1**
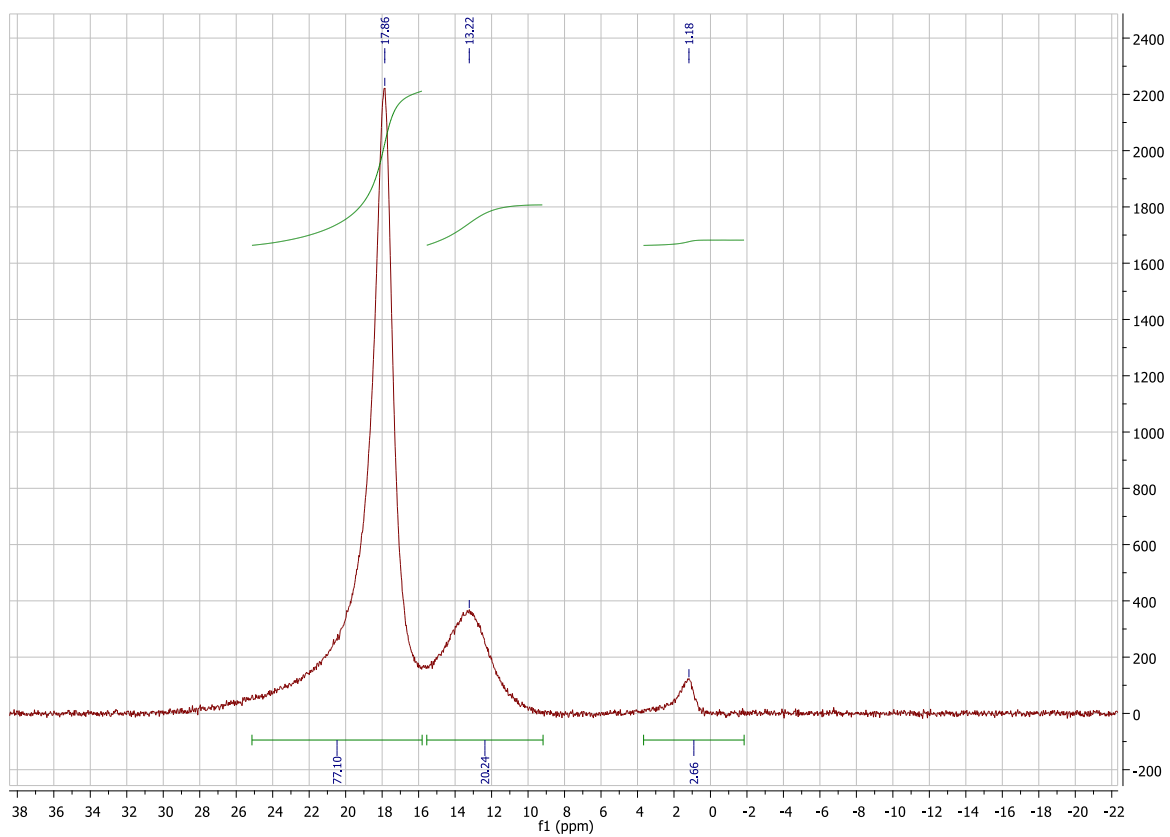
Figure S10: ^{11}B NMR (128 MHz) spectrum of **1**

2 $[C_2H_5(C_3H_3N_2)(CH_2)_6(C_3H_3N_2)C_2H_5][B_5O_6(OH)_4]_2 \cdot 3H_2O$
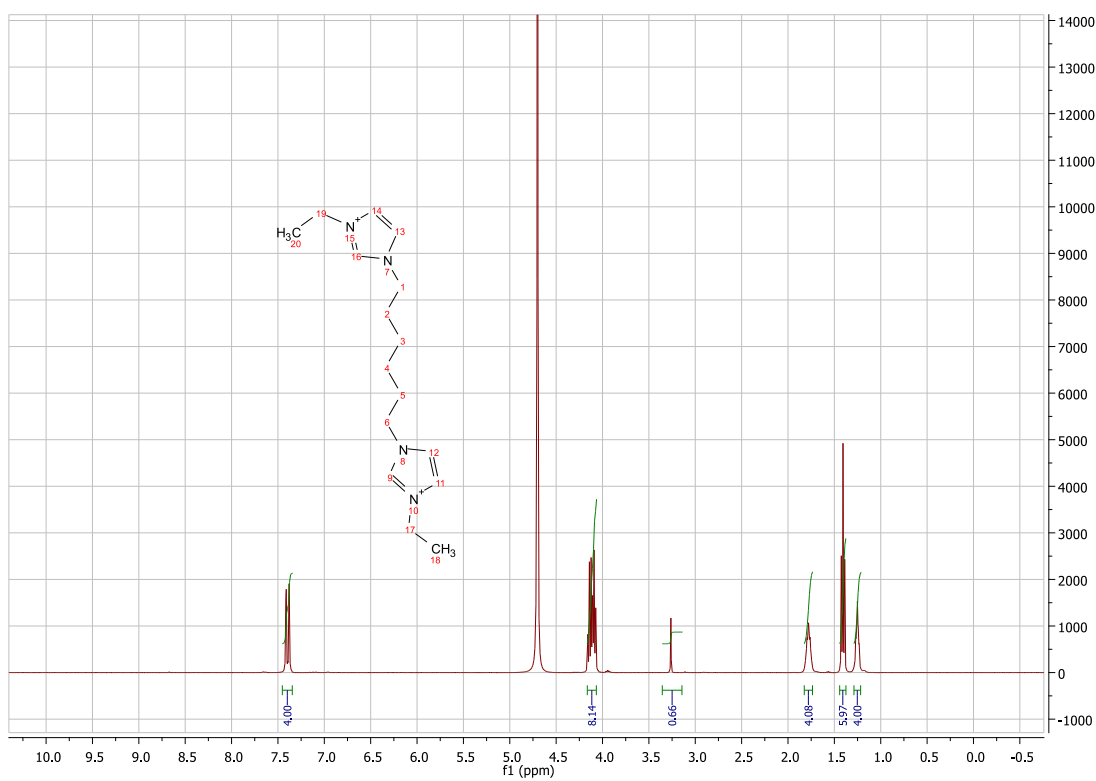
Figure S11: ^{1}H NMR (400 MHz) spectrum of **2**
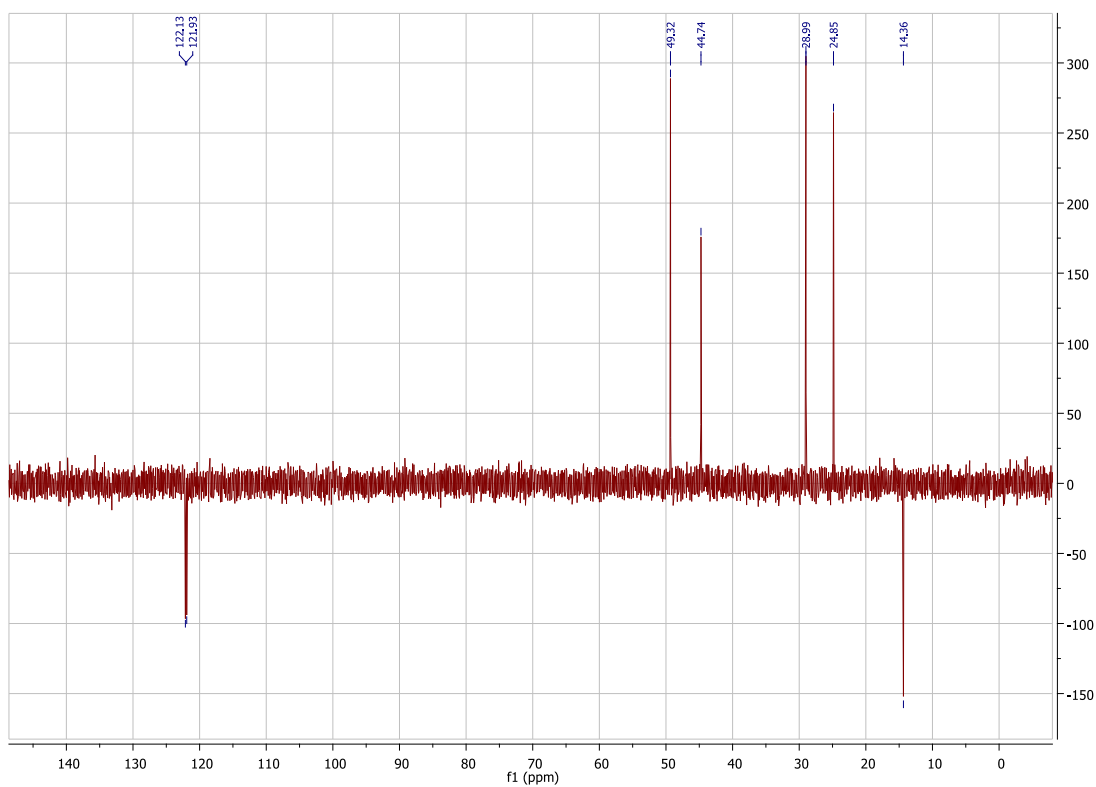
Figure S12: ^{13}C DEPT NMR (100 MHz) spectrum of **2**
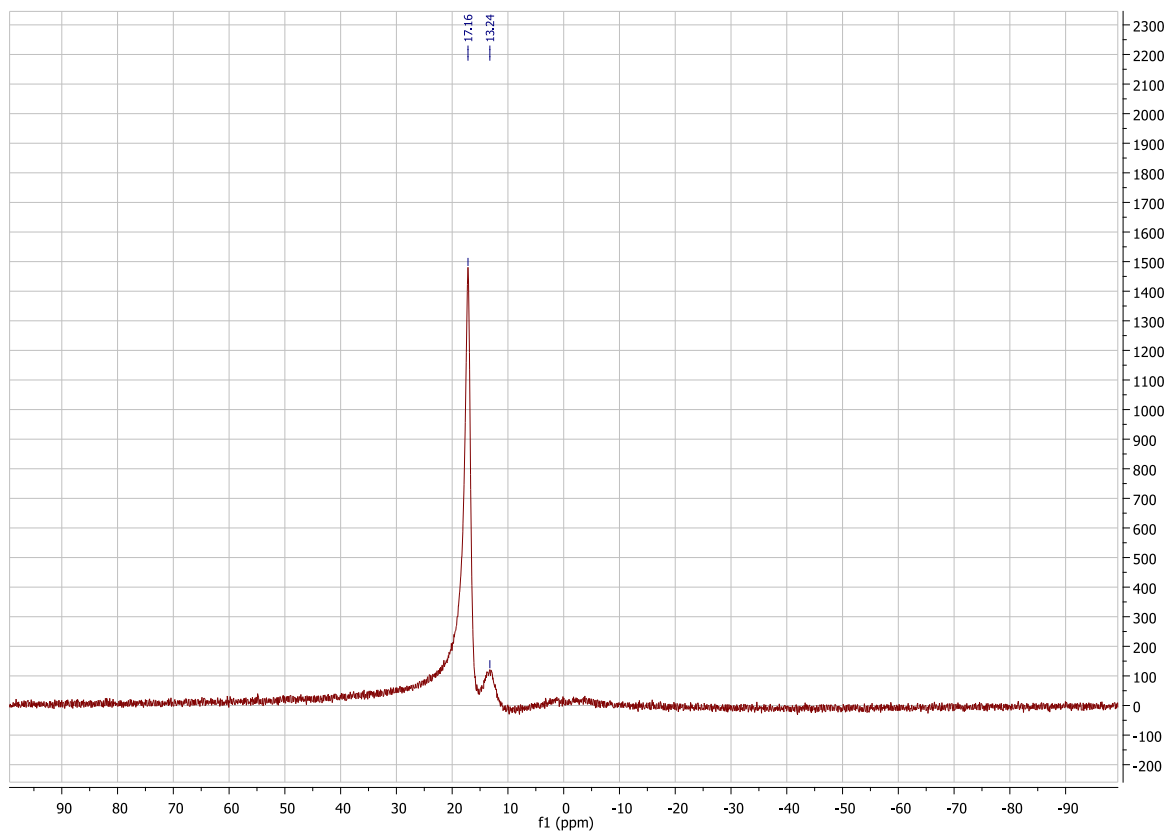
Figure S13: ^{11}B NMR (128 MHz) spectrum of **2**

4 $[CH_3(C_4H_8N)(CH_2)_6(C_4H_8N)CH_3][B_5O_6(OH)_4]_2$
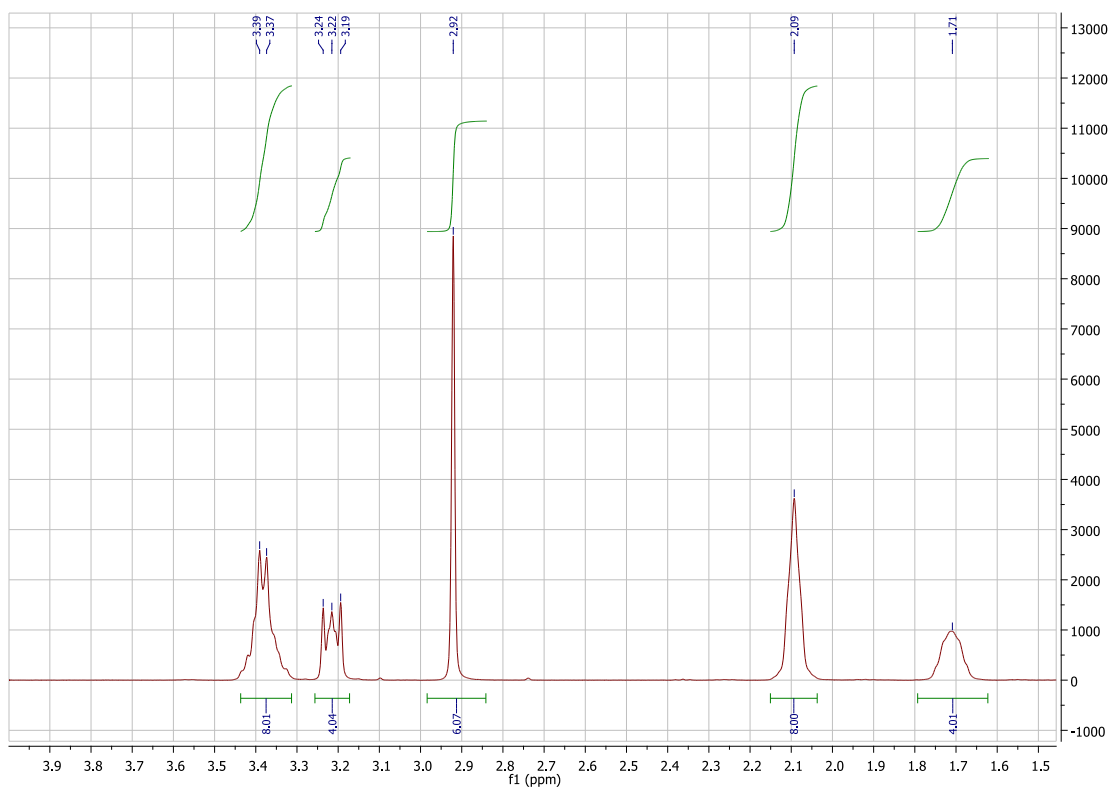
Figure S14: ^{1}H NMR (400 MHz) spectrum of **4**
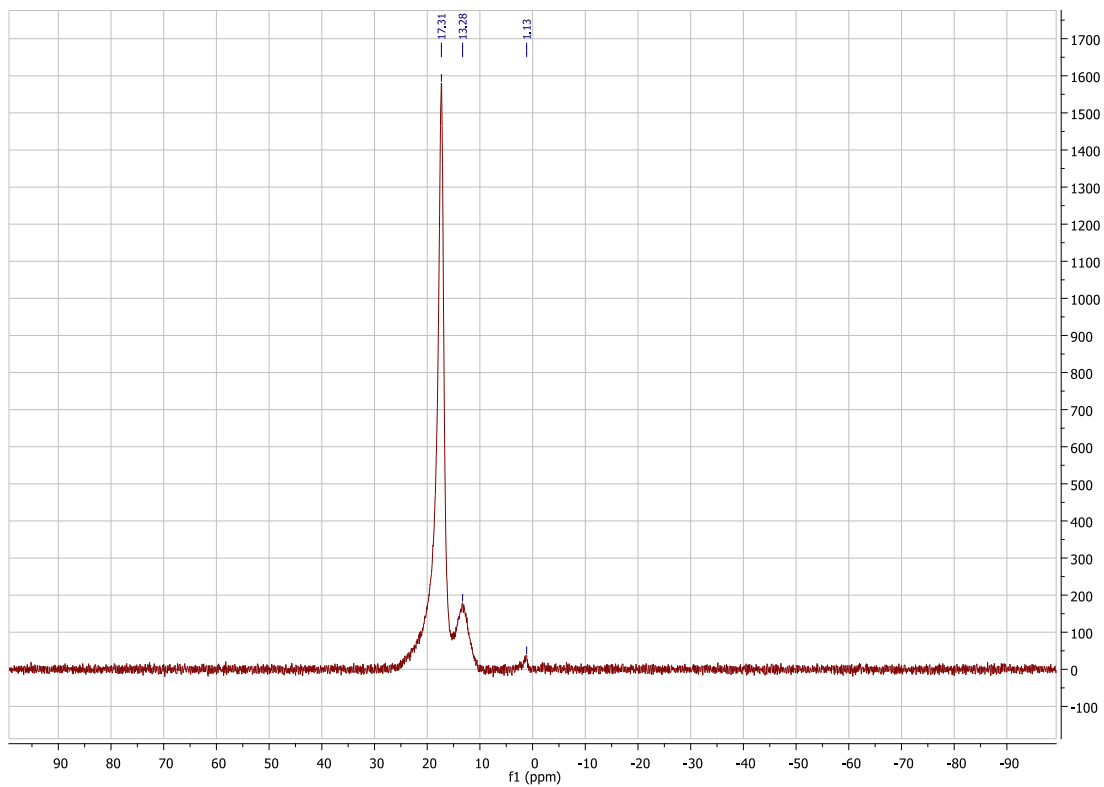
Figure S15: ^{11}B NMR (128 MHz) spectrum of **4**
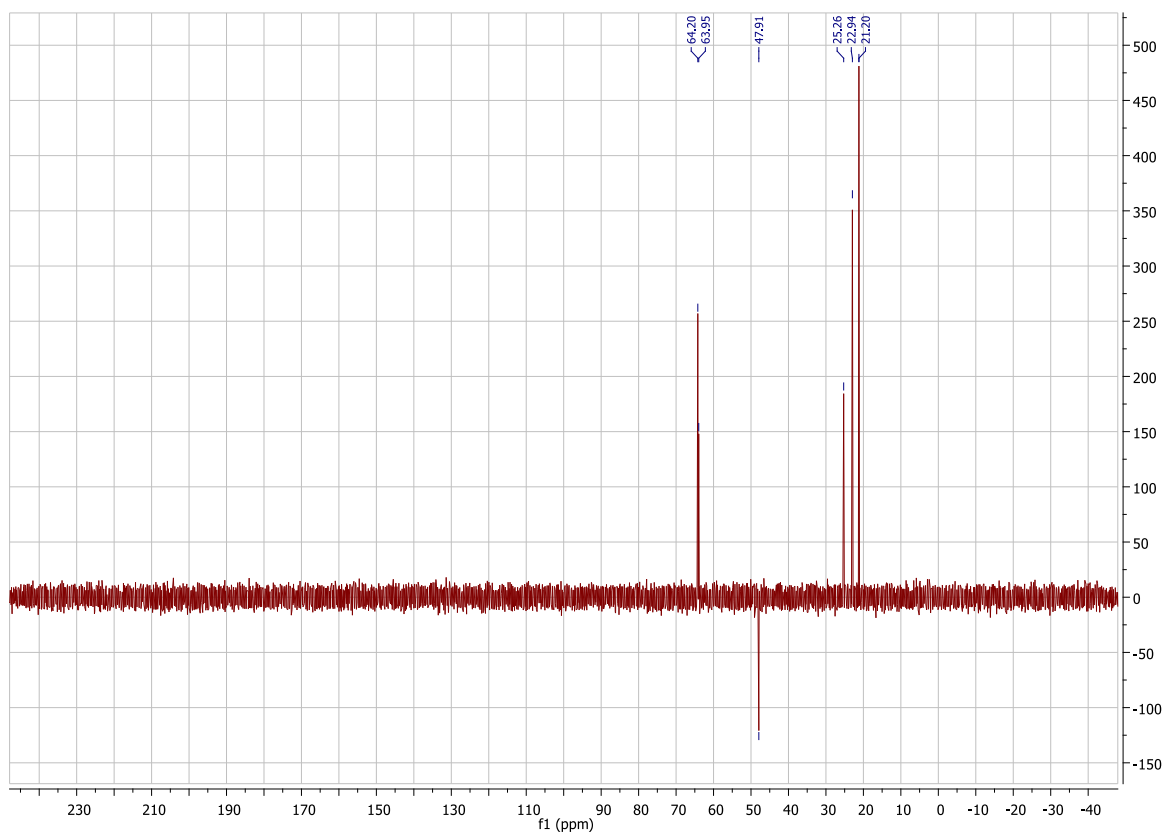
Figure S16: ^{13}C DEPT NMR (100 MHz) spectrum of **4**

5 *[C₂H₅(C₄H₈N)(CH₂)₆(C₄H₈N)C₂H₅][B₅O₆(OH)₄]₂*
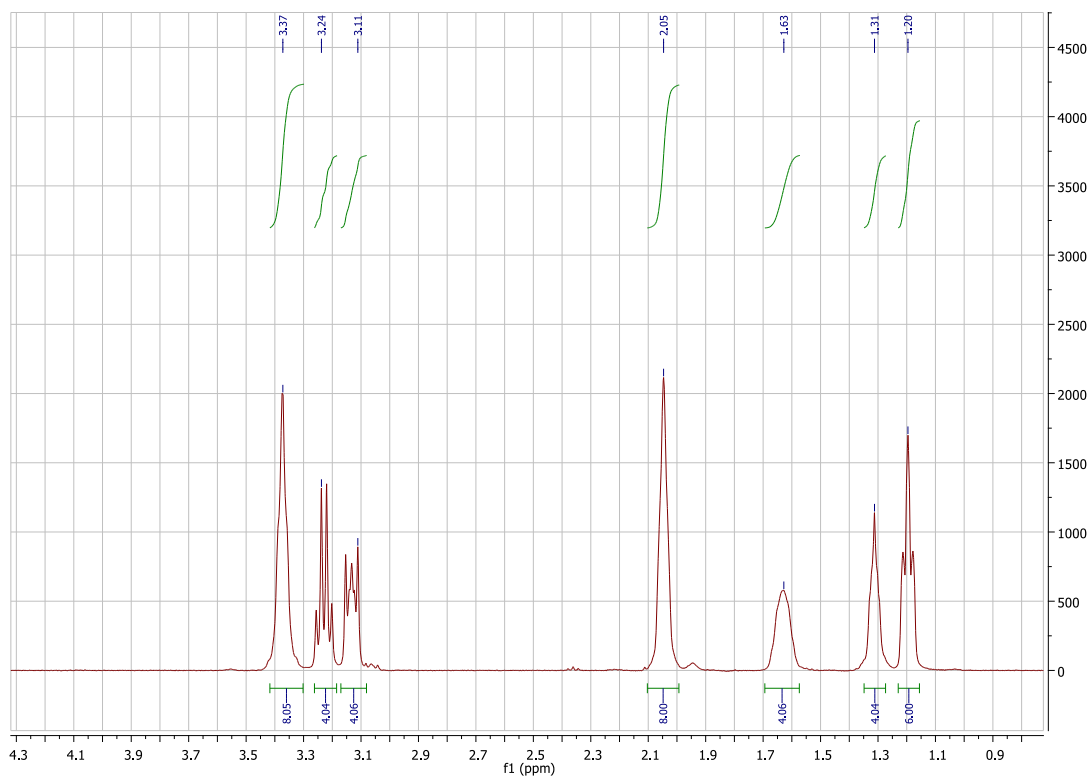
Figure S17: ¹H NMR (400 MHz) spectrum of **5**
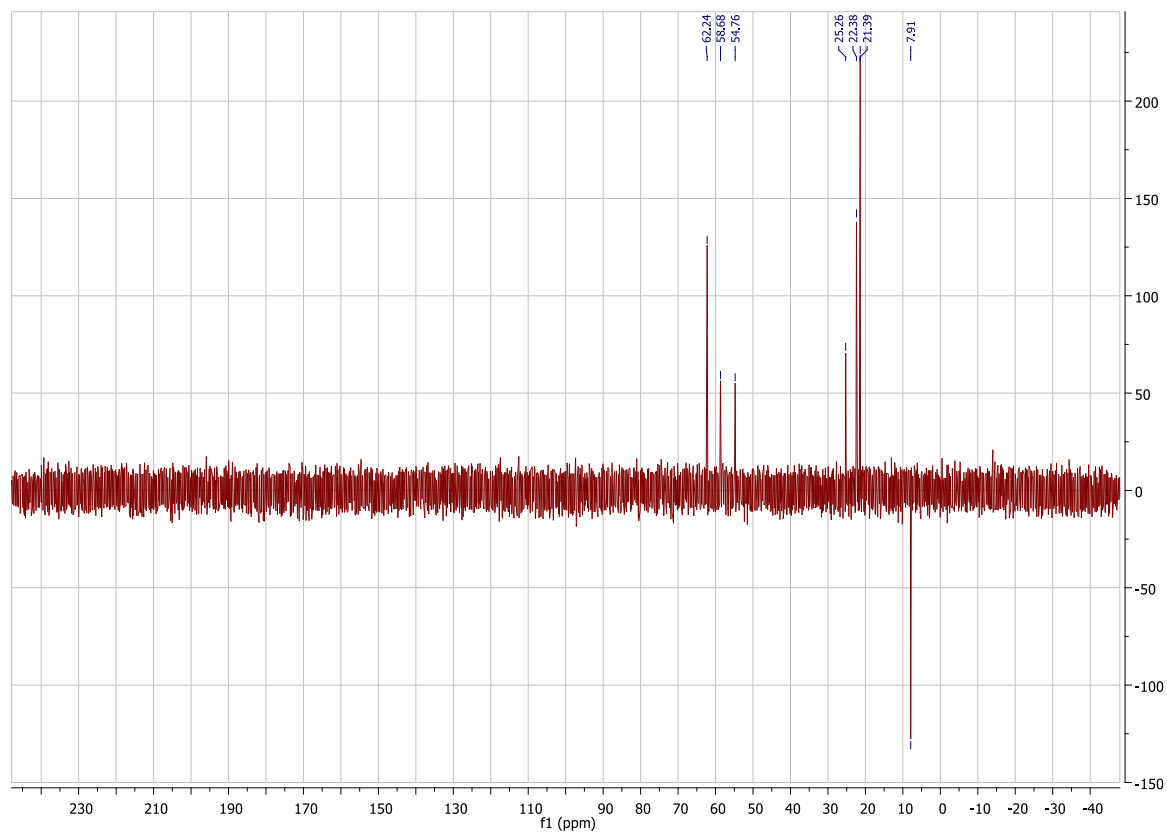
Figure S18: ¹³C DEPT NMR (100 MHz) spectrum of **5**
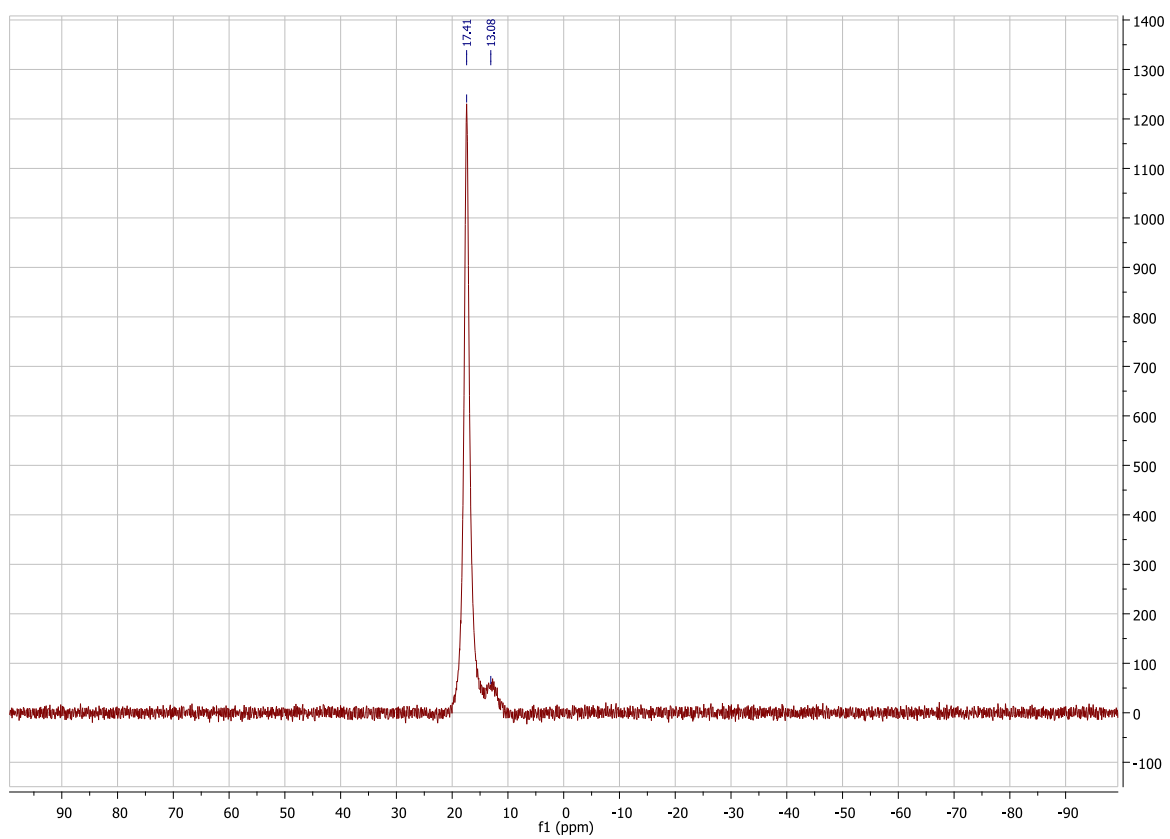
Figure S19: ^{11}B NMR (128 MHz) spectrum of **5**

6   *[C₄H₉(C₄H₈N)(CH₂)₆(C₄H₈N)C₄H₉] [B₅O₆(OH)₄]₂·4B(OH)₃*
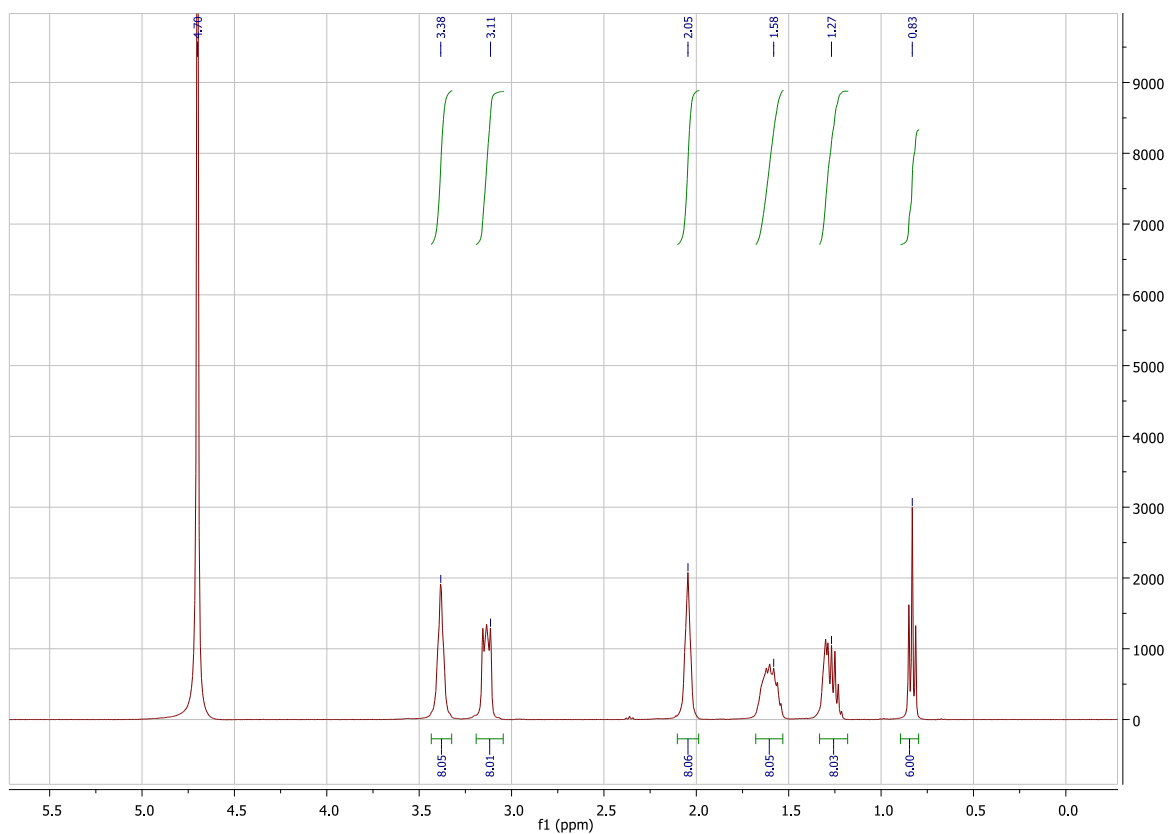
Figure S20: ¹H NMR (400 MHz) spectrum of **6**
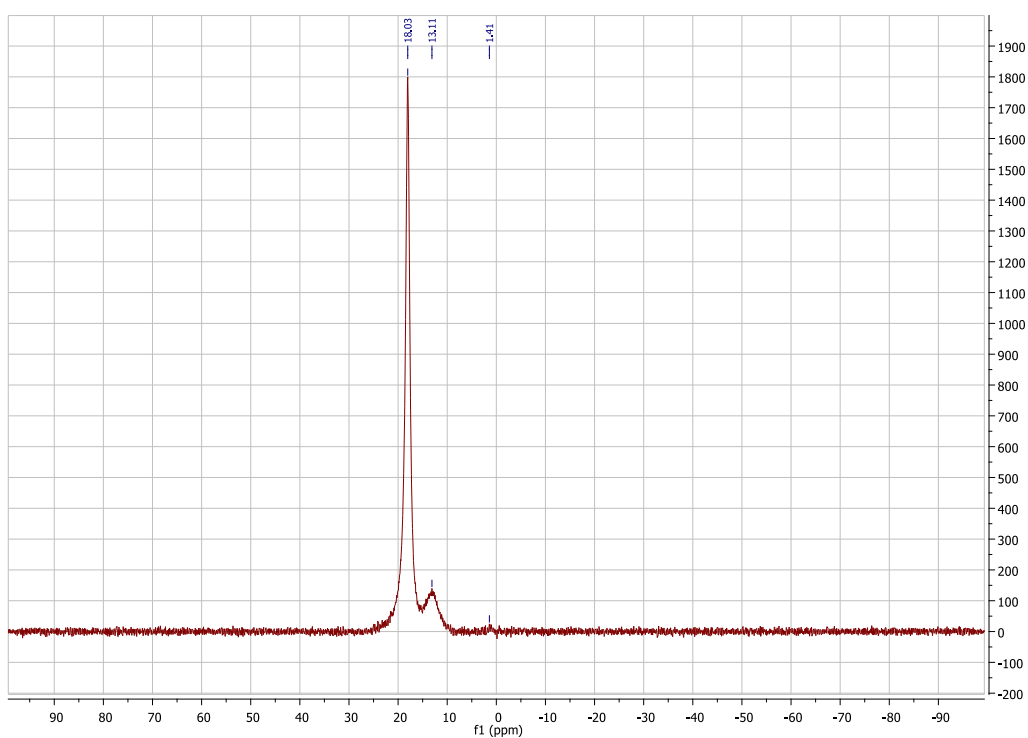
Figure S21: ¹¹B NMR (128 MHz) spectrum of **6**
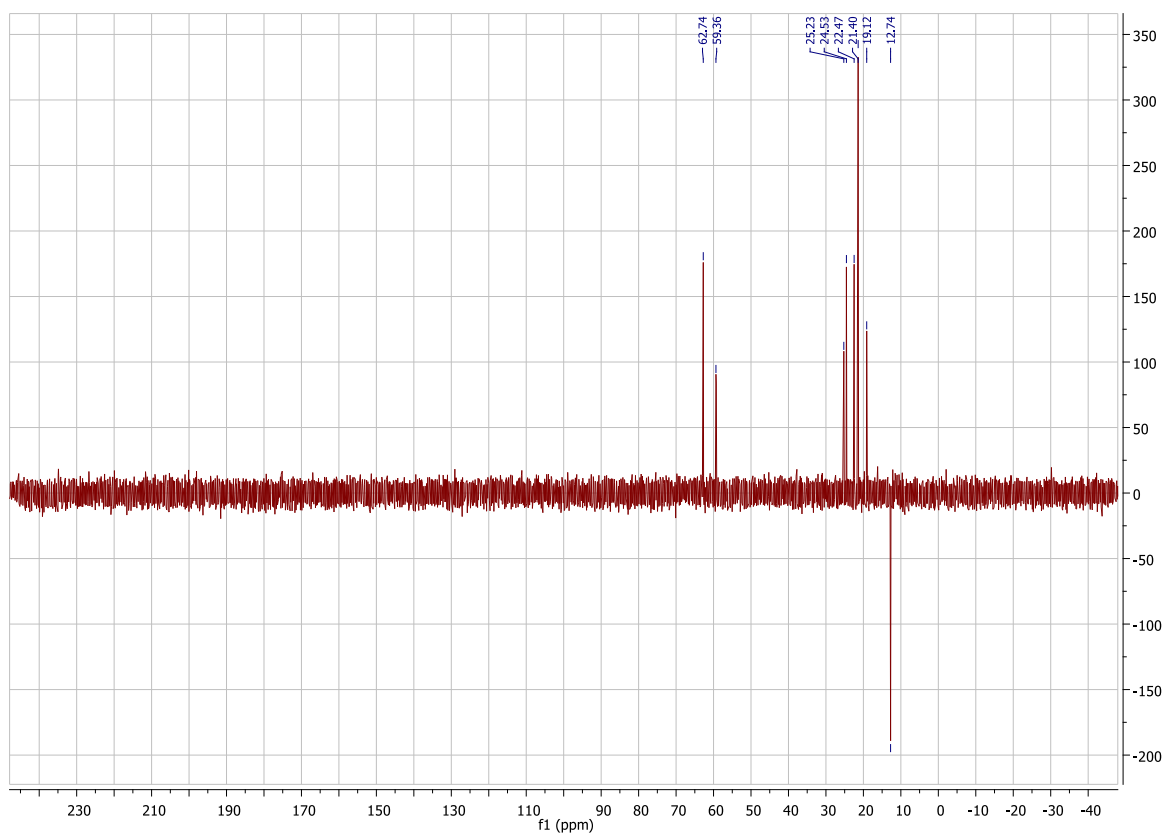
Figure S22: ^{13}C DEPT NMR (100 MHz) spectrum of **6**

7   [C₃H₅(C₄H₈N)(CH₂)₆(C₄H₈N)C₃H₅][B₅O₆(OH)₄]₂.
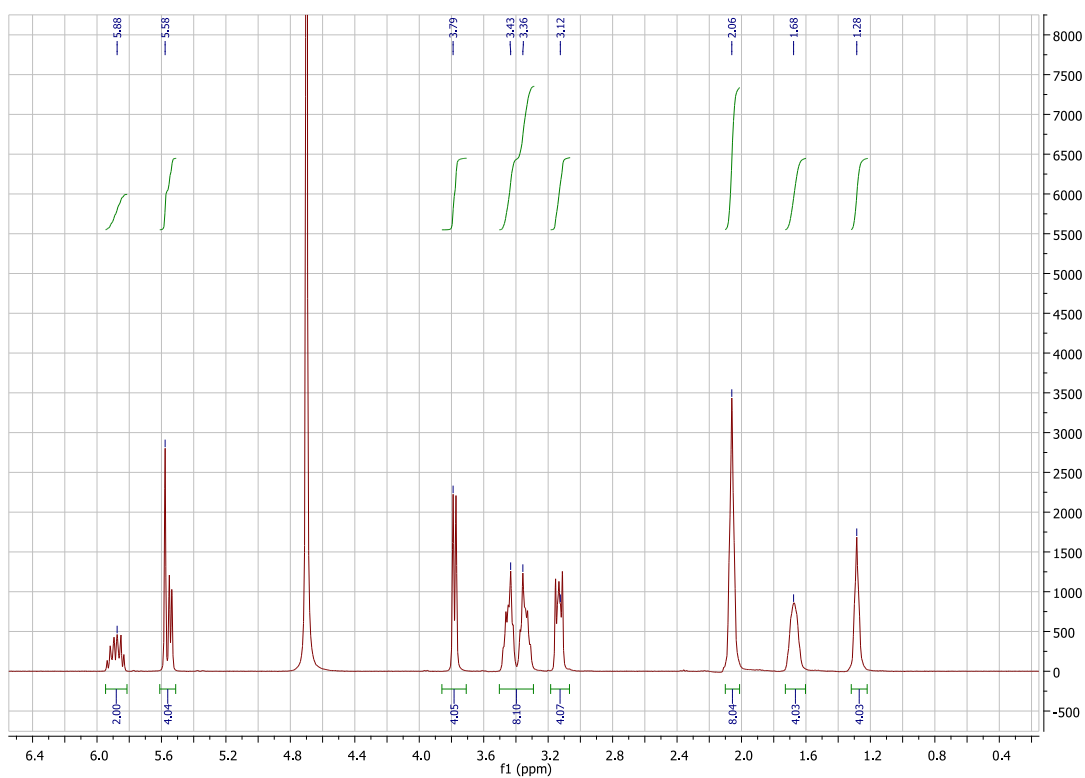
Figure S23: ¹H NMR (400 MHz) spectrum of **7**
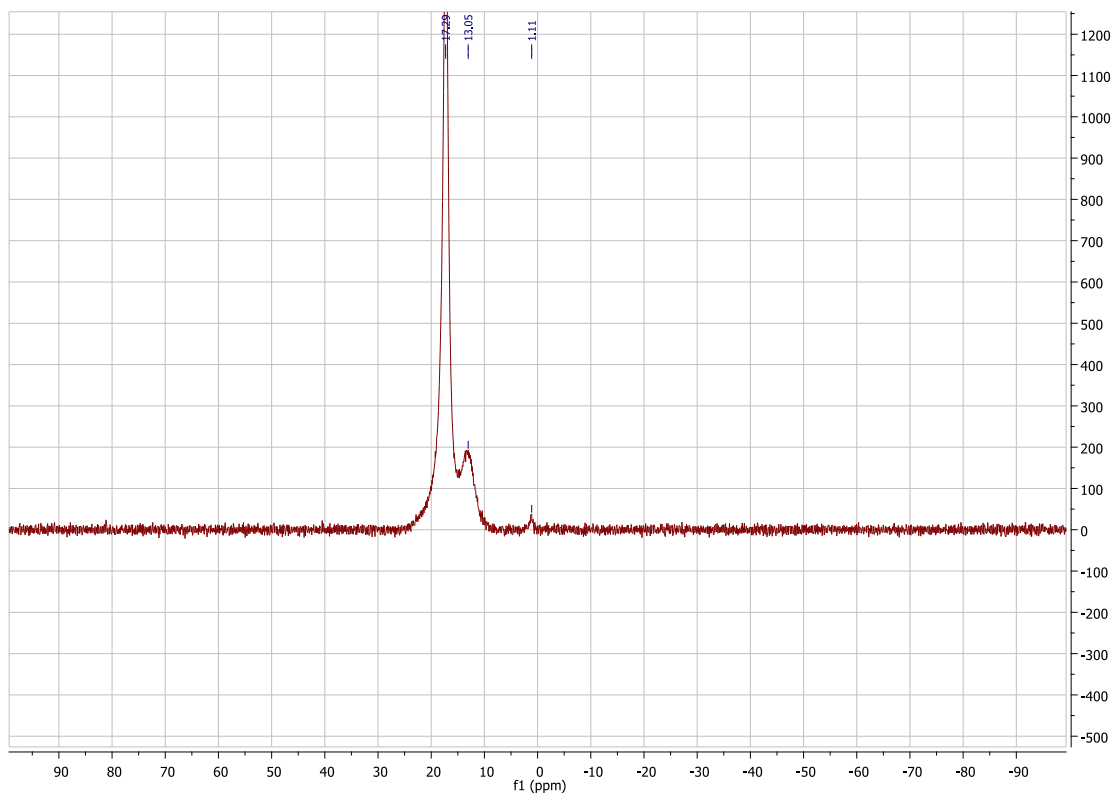
Figure S24: ¹¹B NMR (125 MHz) spectrum of **7**
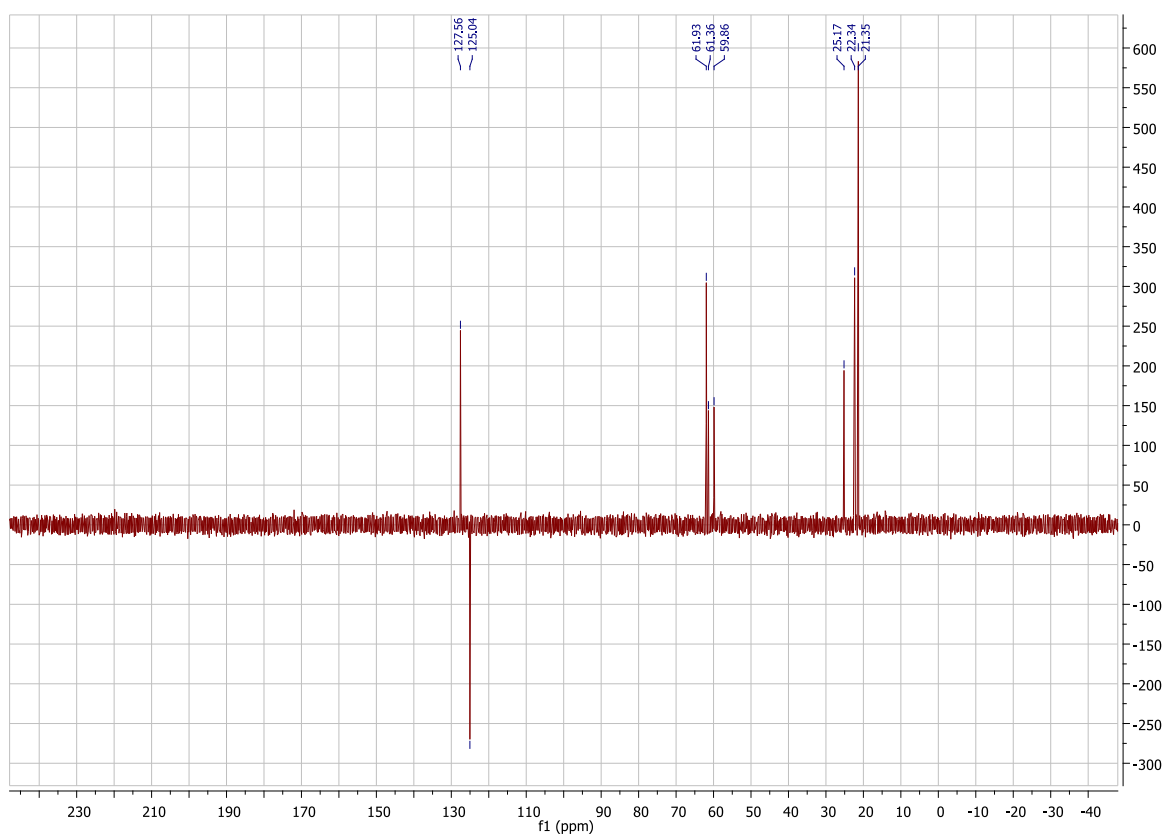
Figure S25: ^{13}C DEPT NMR (100 MHz) spectrum of **7**
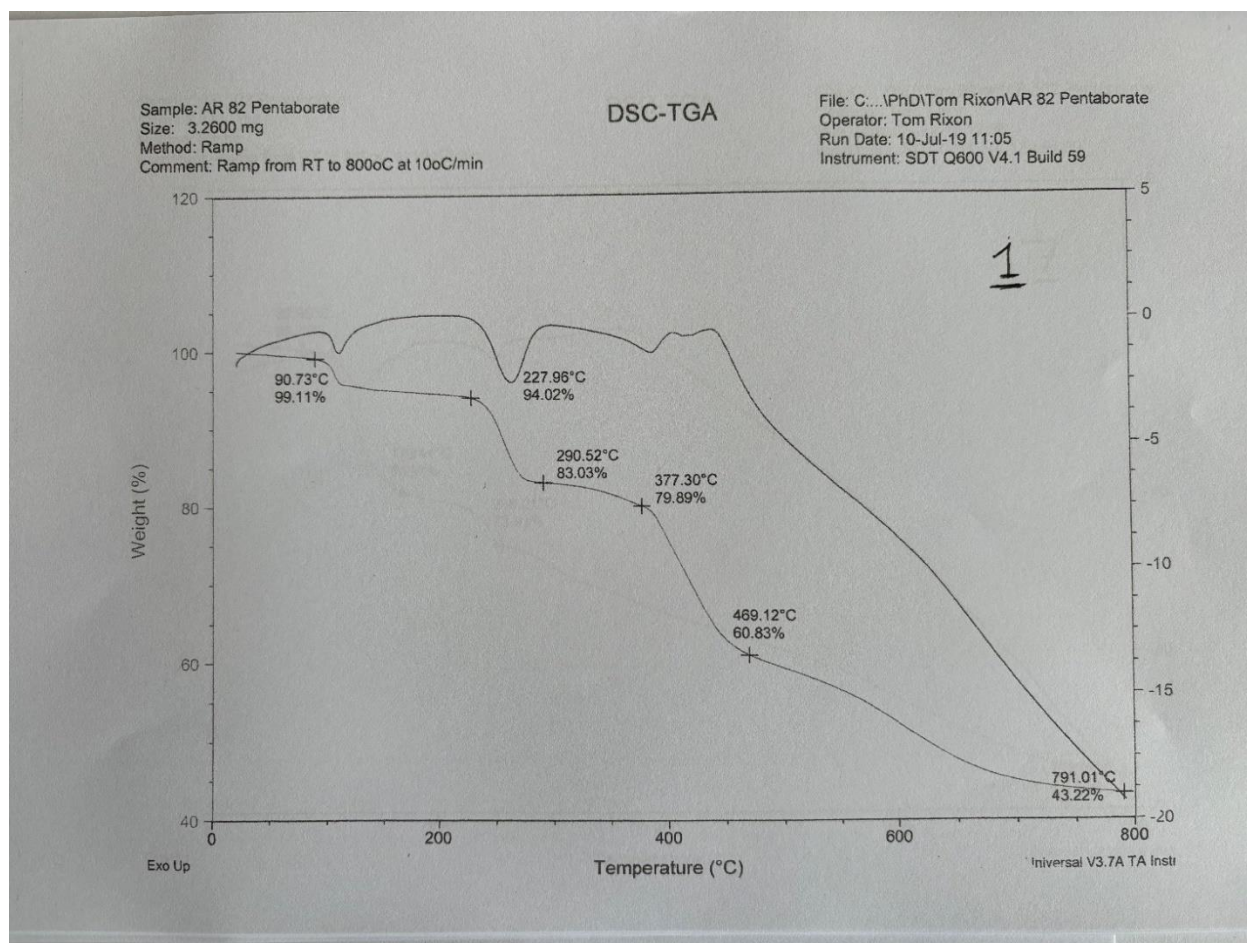
Figure S26: DSC/TGA trace of **1**.
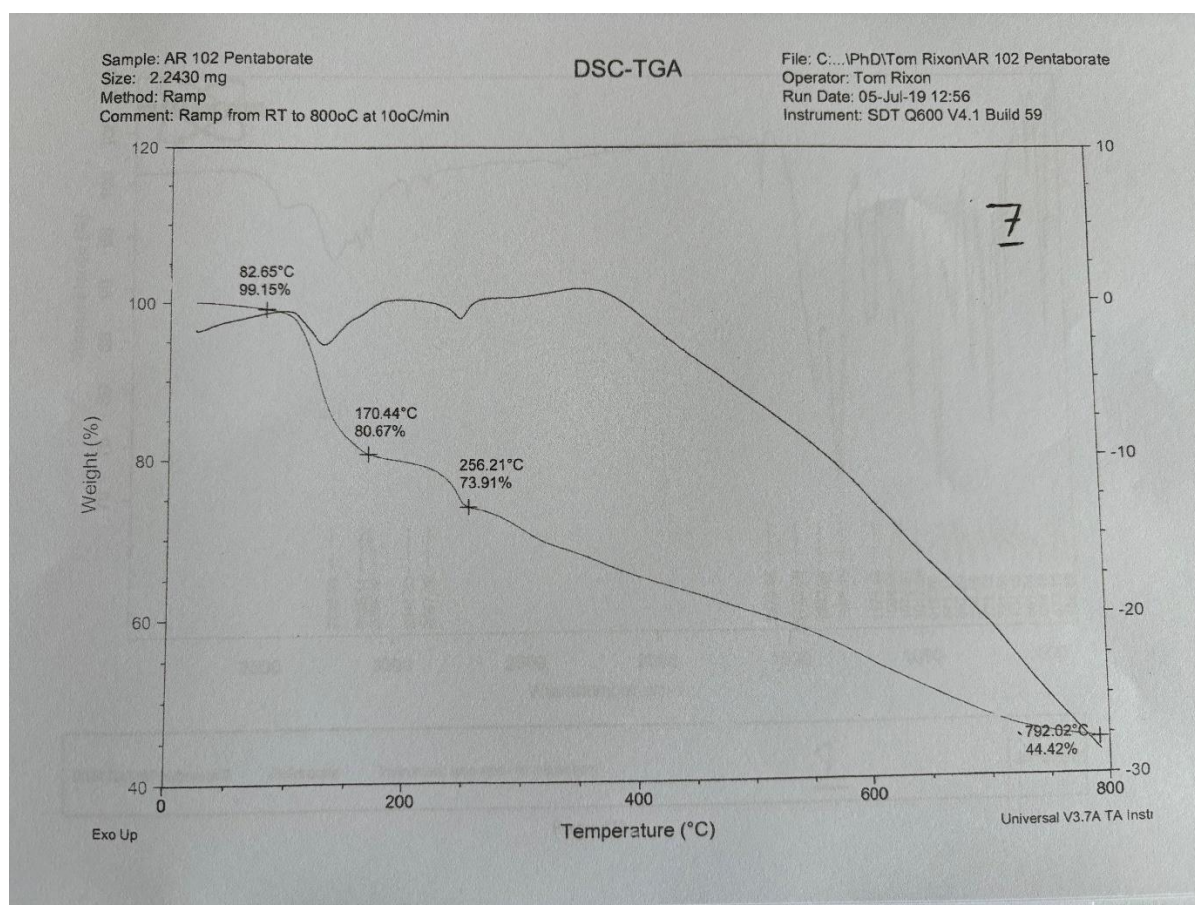
Figure S27: DSC/TGA trace of **7**.

Submitted by: **Mike A Beckett / Thomas A. Rixon**
                Bangor University

Solved by: **Peter N Horton**

Sample ID: **MAB/TR/AR82 B5**
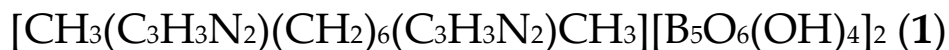
$[CH_3(C_3H_3N_2)(CH_2)_6(C_3H_3N_2)CH_3][B_5O_6(OH)_4]_2$ (**1**)

Crystal Data and Experimental
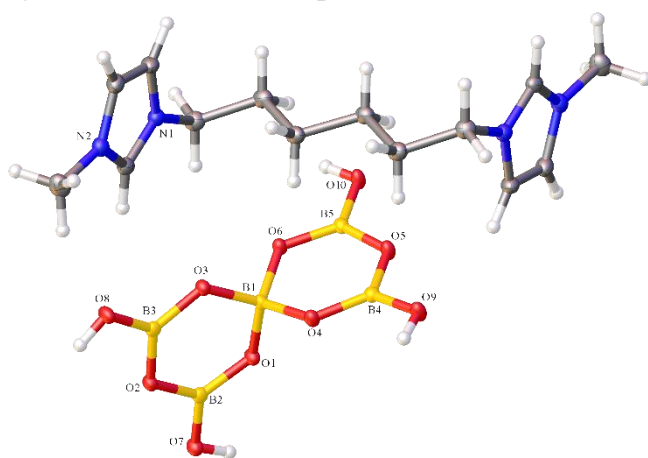
Experimental. Single colourless plate-shaped crystals of (**2017ncs0795**) were recrystallised from water. A suitable crystal (0.290×0.140×0.015) mm³ was selected and mounted on a MITIGEN holder in perfluoroether oil on a Rigaku FRE+ diffractometer equipped with HF Varimax confocal mirrors and an AFC12 goniometer and HG Saturn 724+ detector. The crystal was kept at T = 100(2) K during data collection. Using **Olex2** (Dolomanov et al., 2009), the structure was solved with the **ShelXT** (Sheldrick, 2015) structure solution program, using the Intrinsic Phasing solution method. The model was refined with version 2014/7 of **ShelXL** (Sheldrick, 2015) using Least Squares minimisation.

Crystal Data. $C_{14}H_{32}B_{10}N_4O_{20}$, M_r = 684.53, monoclinic, $P2_1/c$ (No. 14), a = 9.6754(2) Å, b = 16.7781(3) Å, c = 9.1677(2) Å, β = 92.680(2)°, α = γ = 90°, V = 1486.61(5) Å³, T = 100(2) K, Z = 2, Z' = 0.5, μ(MoK$_\alpha$) = 0.131 mm⁻¹, 20877 reflections measured, 3403 unique (R_{int} = 0.0339) which were used in all calculations. The final wR_2 was 0.0851 (all data) and R_1 was 0.0335 (I > 2(I)).

Compound	2017ncs0795
Formula	$C_{14}H_{32}B_{10}N_4O_{20}$
$D_{calc.}$/ g cm⁻³	1.529
μ/mm⁻¹	0.131
Formula Weight	684.53
Colour	colourless
Shape	plate
Size/mm³	0.290×0.140×0.015
T/K	100(2)
Crystal System	monoclinic
Space Group	$P2_1/c$
a/Å	9.6754(2)
b/Å	16.7781(3)
c/Å	9.1677(2)
α/°	90
β/°	92.680(2)
γ/°	90
V/Å³	1486.61(5)
Z	2
Z'	0.5
Wavelength/Å	0.71075
Radiation type	MoK$_\alpha$
Θ_{min}/°	2.107
Θ_{max}/°	27.482
Measured Refl.	20877
Independent Refl.	3403
Reflections Used	2908
R_{int}	0.0339
Parameters	234
Restraints	0
Largest Peak	0.265
Deepest Hole	-0.208
GooF	1.031
wR_2 (all data)	0.0851
wR_2	0.0809
R_1 (all data)	0.0414
R_1	0.0335

Structure Quality Indicators

Reflections:

d min (Mo)	I/σ	Rint	complete at 2θ=55°
0.77	26.1	3.39%	100%

Refinement:

Shift	Max Peak	Min Peak	GooF
0.001	0.3	-0.2	1.031

A colourless plate-shaped crystal with dimensions $0.290 \times 0.140 \times 0.015$ mm^3 was mounted on a MITIGEN holder in perfluoroether oil. X-ray diffraction data were collected using a Rigaku FRE+ diffractometer equipped with HF Varimax confocal mirrors and an AFC12 goniometer and HG Saturn 724+ detector equipped with an Oxford Cryosystems low-temperature device, operating at T = 100(2) K.

Data were measured using profile data from ω-scans of 1.0 ° per frame for 4.0 s using MoK$_\alpha$ radiation (Rotating Anode, 45.0 kV, 55.0 mA). The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.39.34b, 2017). The maximum resolution achieved was Θ = 27.482°.

Cell parameters were retrieved using the **CrysAlisPro** (Rigaku, V1.171.39.34b, 2017) software and refined using **CrysAlisPro** (Rigaku, V1.171.39.34b, 2017) on 9503 reflections, 46 % of the observed reflections. Data reduction was performed using the **CrysAlisPro** (Rigaku, V1.171.39.34b, 2017) software which corrects for Lorentz polarisation. The final completeness is 100.00 % out to 27.482° in Θ.

A multi-scan absorption correction was performed using CrysAlisPro 1.171.39.34b (Rigaku Oxford Diffraction, 2017) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm. The absorption coefficient μ of this material is 0.131 mm^{-1} at this wavelength (λ = 0.71075Å) and the minimum and maximum transmissions are 0.57426 and 1.00000.

The structure was solved in the space group $P2_1/c$ (# 14) by Intrinsic Phasing using the **ShelXT** (Sheldrick, 2015) structure solution program and refined by Least Squares using version 2014/7 of **ShelXL** (Sheldrick, 2015). All non-hydrogen atoms were refined anisotropically. Most hydrogen atom positions were calculated geometrically and refined using the riding model, but some hydrogen atoms were refined freely.

_exptl_absorpt_process_details_: CrysAlisPro 1.171.39.34b (Rigaku Oxford Diffraction, 2017) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

The value of Z' is 0.5. This means that only half of the formula unit is present in the asymmetric unit, with the other half consisting of symmetry equivalent atoms.
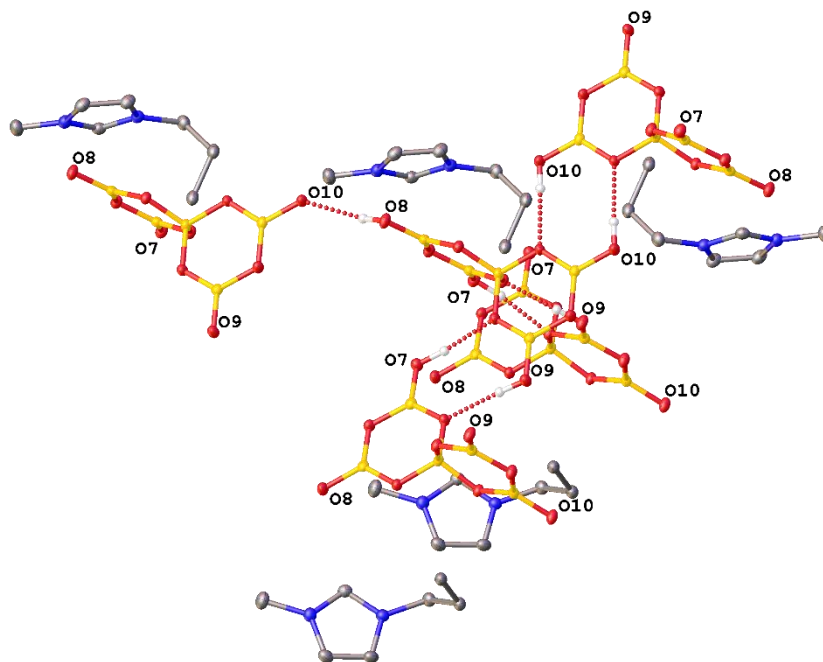
Figure S28: The following hydrogen bonding interactions with a maximum D-D distance of 2.9 Å and a minimum angle of 120 ° are present in **2017ncs0795**: O7–O4_*1: 2.754 Å, O8–O10_*2: 2.798 Å, O9–O1_*3: 2.694 Å, O10–O6_*4: 2.721 Å.
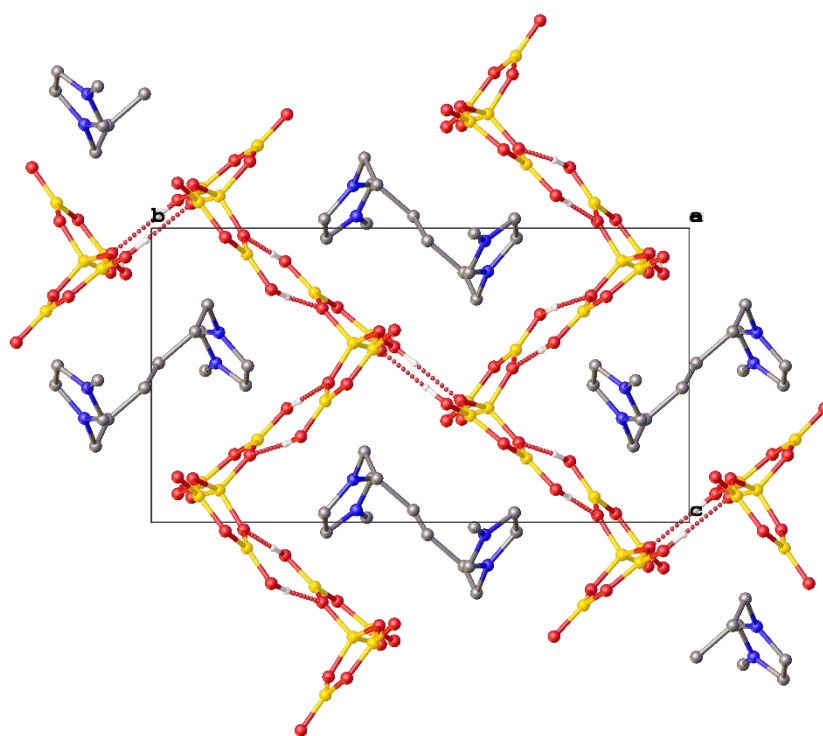
Figure S19: Packing diagram of 2017ncs0795.

Reflection Statistics

Total reflections (after filtering)	21377	Unique reflections	3403
Completeness	1.0	Mean I/σ	26.07
hkl_{max} collected	(12, 21, 11)	hkl_{min} collected	(-12, -21, -11)
hkl_{max} used	(12, 21, 11)	hkl_{min} used	(-12, 0, 0)
Lim d_{max} collected	100.0	Lim d_{min} collected	0.36
d_{max} used	9.66	d_{min} used	0.77
Friedel pairs	5332	Friedel pairs merged	1
Inconsistent equivalents	0	R_{int}	0.0339
R_{sigma}	0.0228	Intensity transformed	0
Omitted reflections	0	Omitted by user (OMIT hkl)	0
Multiplicity	(5944, 3130, 1704, 842, 135, 3)	Maximum multiplicity	19
Removed systematic absences	500	Filtered off (Shel/OMIT)	0

Table S1: Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **2017ncs0795**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
N1	7154.8(10)	6249.8(6)	8539.8(11)	15.6(2)
N2	5201.6(10)	6194.2(6)	9576.5(11)	16.9(2)
C1	5919.6(12)	5903.2(7)	8500.6(13)	16.2(2)
C2	7222.7(12)	6786.8(7)	9679.0(14)	19.1(3)
C3	6005.0(12)	6749.5(7)	10331.3(14)	19.7(3)
C4	8289.5(12)	6041.8(8)	7595.7(14)	18.9(3)
C5	9584.8(12)	5809.2(7)	8509.7(14)	19.2(3)
C6	9339.4(11)	5131.4(7)	9579.5(14)	18.6(3)
C7	3758.7(12)	6000.8(8)	9832.1(15)	23.9(3)
O1	7622.2(8)	3290.1(5)	4754.6(9)	14.34(18)
O2	5223.3(8)	3630.8(5)	4706.0(9)	14.45(18)
O3	6777.2(8)	4232.8(5)	6491.1(9)	13.17(18)
O4	8280.1(8)	3171.8(5)	7288.4(9)	14.12(18)
O5	10702.8(8)	3454.8(5)	7315.8(9)	15.40(18)
O6	9149.7(8)	4258.8(4)	5849.6(9)	12.63(17)
O7	6062.9(8)	2775.6(5)	2923.8(9)	16.22(19)
O8	4435.9(8)	4517.5(5)	6544.4(10)	17.16(19)
O9	9877.7(8)	2483.0(5)	8877.6(9)	16.93(19)
O10	11569.8(8)	4513.4(5)	5970.0(9)	15.77(19)
B1	7943.2(12)	3744.9(8)	6102.8(14)	12.5(3)
B2	6324.8(13)	3231.8(7)	4129.4(14)	13.2(3)
B3	5476.4(12)	4123.0(7)	5901.8(15)	13.1(3)
B4	9590.9(13)	3031.7(8)	7818.5(14)	13.0(3)
B5	10458.6(12)	4076.6(8)	6362.6(14)	12.8(3)

Table S2: Anisotropic Displacement Parameters (×10⁴) **2017ncs0795**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11}+ ... +2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	15.7(5)	14.6(5)	16.3(5)	-0.6(4)	-0.6(4)	0.8(4)
N2	15.5(5)	18.1(5)	17.1(5)	1.1(4)	-0.3(4)	1.1(4)
C1	18.2(5)	14.8(6)	15.5(6)	1.0(5)	-1.8(4)	0.4(4)
C2	17.9(5)	16.2(6)	22.9(7)	-5.1(5)	-3.0(5)	1.3(4)
C3	19.9(6)	18.9(6)	20.0(6)	-3.7(5)	-2.4(5)	4.0(5)
C4	19.3(6)	20.0(6)	17.7(6)	-1.5(5)	4.3(5)	-0.7(5)
C5	15.6(5)	18.3(6)	23.9(7)	-3.1(5)	4.2(5)	-0.1(5)
C6	14.6(5)	17.9(6)	23.4(7)	-3.7(5)	3.0(5)	-0.3(4)
C7	18.3(6)	28.5(7)	24.9(7)	4.0(6)	3.4(5)	-2.7(5)
O1	10.8(4)	16.0(4)	16.1(4)	-3.3(3)	-0.1(3)	1.3(3)
O2	10.6(4)	15.2(4)	17.3(4)	-2.1(3)	-1.6(3)	0.4(3)
O3	10.1(4)	13.7(4)	15.6(4)	-1.6(3)	0.1(3)	0.4(3)
O4	10.6(4)	14.4(4)	17.2(4)	3.0(3)	-0.2(3)	-1.4(3)
O5	10.3(4)	17.5(4)	18.3(4)	5.1(3)	-0.5(3)	0.1(3)
O6	9.9(3)	13.1(4)	14.8(4)	2.6(3)	0.1(3)	-1.0(3)
O7	13.5(4)	17.9(4)	17.1(4)	-3.0(3)	-0.6(3)	1.2(3)
O8	9.8(4)	19.3(4)	22.3(5)	-4.9(4)	-0.3(3)	0.7(3)
O9	11.6(4)	18.4(4)	20.7(5)	6.7(4)	-0.6(3)	-1.9(3)
O10	10.0(4)	18.0(4)	19.2(5)	6.2(4)	-0.3(3)	0.0(3)
B1	9.4(5)	13.6(6)	14.4(6)	0.2(5)	-0.1(5)	-0.7(4)
B2	12.9(5)	11.9(6)	14.9(6)	2.5(5)	0.7(5)	-0.8(5)
B3	12.3(5)	11.1(6)	15.8(6)	2.3(5)	0.8(5)	-0.7(4)
B4	12.8(5)	13.1(6)	13.2(6)	-1.2(5)	0.5(5)	0.7(5)
B5	11.2(5)	14.4(6)	12.9(6)	-1.9(5)	0.4(4)	0.6(5)

Table S3: Bond Lengths in Å for **2017ncs0795**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N1	C1	1.3281(15)	O3	B1	1.4515(14)
N1	C2	1.3786(16)	O3	B3	1.3588(14)
N1	C4	1.4714(15)	O4	B1	1.4762(15)
N2	C1	1.3259(15)	O4	B4	1.3573(14)
N2	C3	1.3787(16)	O5	B4	1.3857(14)
N2	C7	1.4628(15)	O5	B5	1.3743(15)
C2	C3	1.3474(17)	O6	B1	1.4783(14)
C4	C5	1.5254(17)	O6	B5	1.3648(14)
C5	C6	1.5275(17)	O7	B2	1.3585(16)
C6	C6¹	1.527(2)	O8	B3	1.3616(15)
O1	B1	1.4733(15)	O9	B4	1.3573(15)
O1	B2	1.3589(14)	O10	B5	1.3633(15)
O2	B2	1.3842(14)	----		
O2	B3	1.3851(15)	¹2-x,1-y,2-z		

Table S4: Bond Angles in ° for **2017ncs0795**.

Atom	Atom	Atom	Angle/°
C1	N1	C2	108.52(10)
C1	N1	C4	125.13(10)
C2	N1	C4	126.17(10)
C1	N2	C3	108.64(10)
C1	N2	C7	125.07(11)
C3	N2	C7	126.11(10)
N2	C1	N1	108.71(10)
C3	C2	N1	107.11(11)
C2	C3	N2	107.02(11)
N1	C4	C5	110.73(10)
C4	C5	C6	113.25(10)
C6[1]	C6	C5	112.81(12)
B2	O1	B1	123.06(9)
B2	O2	B3	118.67(9)
B3	O3	B1	122.83(9)
B4	O4	B1	123.16(9)
B5	O5	B4	119.16(9)
B5	O6	B1	122.83(9)
O1	B1	O4	108.11(9)

Atom	Atom	Atom	Angle/°
O1	B1	O6	107.90(9)
O3	B1	O1	111.23(9)
O3	B1	O4	109.53(9)
O3	B1	O6	109.76(9)
O4	B1	O6	110.29(9)
O1	B2	O2	121.11(11)
O7	B2	O1	121.15(10)
O7	B2	O2	117.74(10)
O3	B3	O2	121.45(10)
O3	B3	O8	116.86(11)
O8	B3	O2	121.69(10)
O4	B4	O5	121.28(11)
O4	B4	O9	122.00(10)
O9	B4	O5	116.71(10)
O6	B5	O5	121.22(10)
O10	B5	O5	117.49(10)
O10	B5	O6	121.28(11)

----
[1]2-x,1-y,2-z

Table S5: Torsion Angles in ° for **2017ncs0795**.

Atom	Atom	Atom	Atom	Angle/°
N1	C2	C3	N2	-0.46(14)
N1	C4	C5	C6	55.76(13)
C1	N1	C2	C3	0.56(14)
C1	N1	C4	C5	-122.80(12)
C1	N2	C3	C2	0.20(13)
C2	N1	C1	N2	-0.44(13)
C2	N1	C4	C5	51.82(15)
C3	N2	C1	N1	0.15(13)
C4	N1	C1	N2	174.98(10)
C4	N1	C2	C3	-174.80(11)
C4	C5	C6	C6[1]	176.42(12)
C7	N2	C1	N1	175.57(10)
C7	N2	C3	C2	-175.16(11)
B1	O1	B2	O2	2.65(17)
B1	O1	B2	O7	-176.88(10)
B1	O3	B3	O2	-11.53(17)
B1	O3	B3	O8	168.30(10)
B1	O4	B4	O5	3.21(17)
B1	O4	B4	O9	-177.86(10)
B1	O6	B5	O5	-8.87(17)
B1	O6	B5	O10	172.35(10)
B2	O1	B1	O3	-11.10(15)
B2	O1	B1	O4	109.19(11)
B2	O1	B1	O6	-131.53(10)
B2	O2	B3	O3	1.46(16)
B2	O2	B3	O8	-178.37(10)
B3	O2	B2	O1	2.86(16)
B3	O2	B2	O7	-177.59(10)
B3	O3	B1	O1	15.44(15)

Atom	Atom	Atom	Atom	Angle/°
B3	O3	B1	O4	-104.01(12)
B3	O3	B1	O6	134.77(10)
B4	O4	B1	O1	104.37(11)
B4	O4	B1	O3	-134.29(10)
B4	O4	B1	O6	-13.38(15)
B4	O5	B5	O6	-3.22(17)
B4	O5	B5	O10	175.61(10)
B5	O5	B4	O4	6.05(17)
B5	O5	B4	O9	-172.94(10)
B5	O6	B1	O1	-101.71(12)
B5	O6	B1	O3	136.94(10)
B5	O6	B1	O4	16.18(15)

----

[1]2-x,1-y,2-z

Table S6: Hydrogen Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **2017ncs0795**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H1	5603	5512	7815	19
H2	7985	7120	9953	23
H3	5749	7049	11155	24
H4A	8498	6503	6970	23
H4B	8001	5592	6952	23
H5A	9928	6281	9064	23
H5B	10314	5646	7848	23
H6A	8656	5307	10283	22
H6B	8942	4670	9033	22
H7A	3167	6452	9532	36
H7B	3661	5893	10873	36
H7C	3482	5528	9262	36
H7	6837(19)	2492(11)	2678(19)	42(5)
H8	3630(20)	4426(11)	6250(20)	48(5)
H9	9140(18)	2248(10)	9141(18)	37(5)
H10	11331(16)	4892(10)	5412(18)	29(4)

Table S7: Hydrogen Bond information for **2017ncs0795**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
O7	H7	O4[1]	0.924(19)	1.834(19)	2.7541(11)	173.3(16)
O8	H8	O10[2]	0.83(2)	2.00(2)	2.7984(11)	161.0(18)
O9	H9	O1[3]	0.860(18)	1.834(18)	2.6936(11)	177.1(17)
O10	H10	O6[4]	0.842(17)	1.879(17)	2.7208(12)	178.4(15)

––––

[1]+x,1/2-y,-1/2+z; [2]-1+x,+y,+z; [3]+x,1/2-y,1/2+z; [4]2-x,1-y,1-z
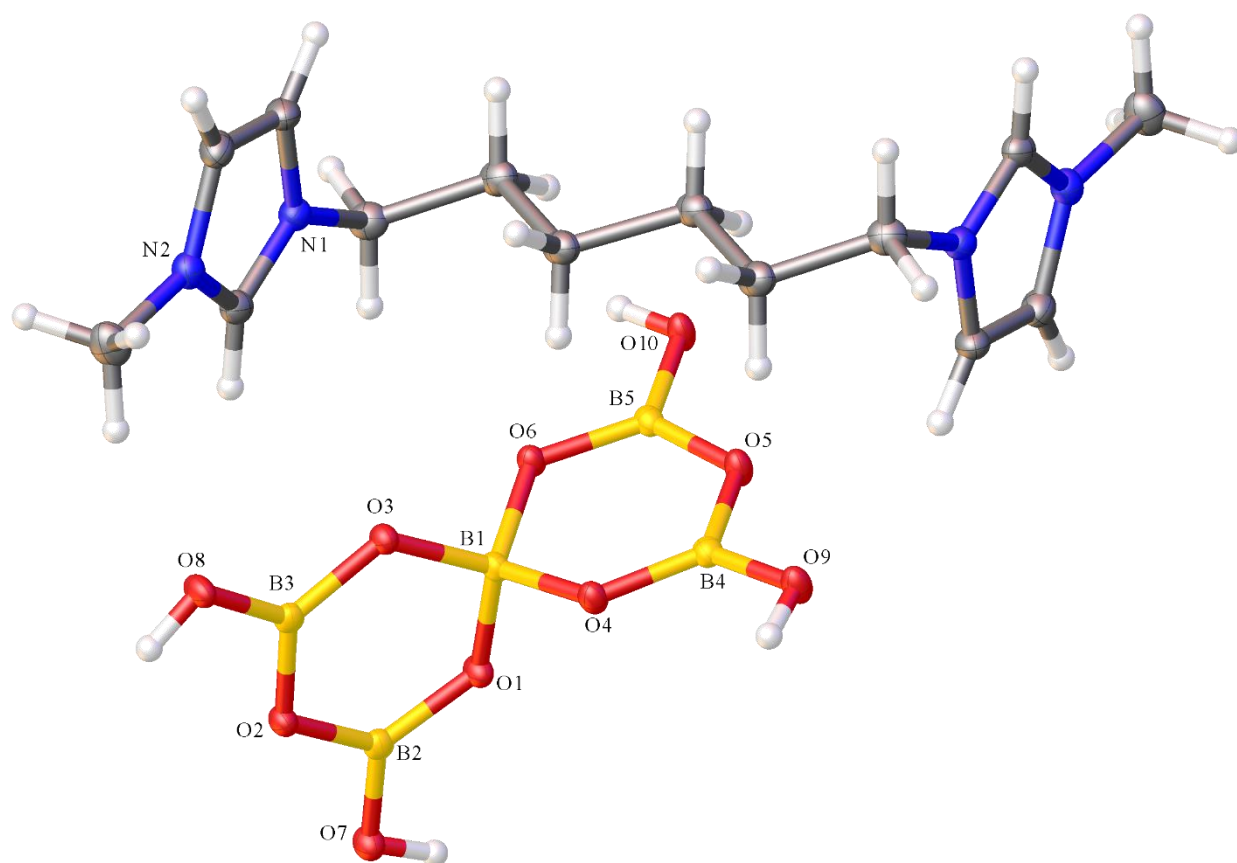
Citations for 1

CrysAlisPro Software System, Rigaku Oxford Diffraction, (2017).

O.V. Dolomanov and L.J. Bourhis and R.J. Gildea and J.A.K. Howard and H. Puschmann, Olex2: A complete structure solution, refinement and analysis program, *J. Appl. Cryst.*, (2009), **42**, 339-341.

Sheldrick, G.M., Crystal structure refinement with ShelXL, *Acta Cryst.*, (2015), **C27**, 3-8.

Sheldrick, G.M., ShelXT-Integrated space-group and crystal-structure determination, *Acta Cryst.*, (2015), **A71**, 3-8.

Submitted by: **Mike A. Beckett / Thomas A. Rixon**
Bangor University

Solved by: **Peter N. Horton**

Sample ID: **MAB/TR/AR87/**
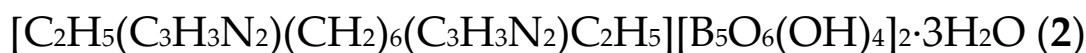
[C₂H₅(C₃H₃N₂)(CH₂)₆(C₃H₃N₂)C₂H₅][B₅O₆(OH)₄]₂·3H₂O (2)

$[C_2H_5(C_3H_3N_2)(CH_2)_6(C_3H_3N_2)C_2H_5][B_5O_6(OH)_4]_2 \cdot 3H_2O$ (**2**)

Crystal Data and Experimental
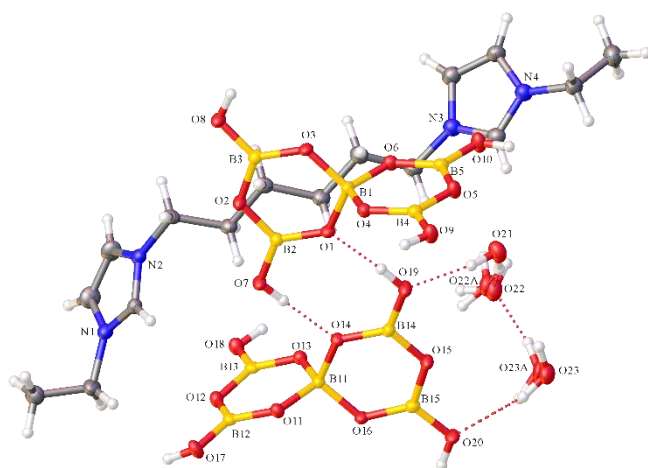
Experimental. Single colourless plate-shaped crystals of (**2017ncs0859**) were recrystallised from water. A suitable crystal (0.280×0.100×0.010) mm³ was selected and mounted on a MITIGEN holder in perfluoroether oil on a Rigaku FRE+ diffractometer equipped with HF Varimax confocal mirrors and an AFC12 goniometer and HG Saturn 724+ detector. The crystal was kept at T = 100(2) K during data collection. Using **Olex2** (Dolomanov et al., 2009), the structure was solved with the **ShelXT** (Sheldrick, 2015) structure solution program, using the Intrinsic Phasing solution method. The model was refined with version 2014/7 of **ShelXL** (Sheldrick, 2015) using Least Squares minimisation.

Crystal Data. $C_{16}H_{42}B_{10}N_4O_{23}$, M_r = 766.63, triclinic, P-1 (No. 2), a = 9.4616(4) Å, b = 9.4780(3) Å, c = 19.8856(8) Å, α = 86.483(3)°, β = 81.244(3)°, γ = 75.624(3)°, V = 1706.79(12) Å³, T = 100(2) K, Z = 2, Z' = 1, μ(MoK$_\alpha$) = 0.129 mm⁻¹, 38202 reflections measured, 7820 unique (R_{int} = 0.0424) which were used in all calculations. The final wR_2 was 0.1431 (all data) and R_1 was 0.0457 (I > 2(I)).

Compound	2017ncs0859
Formula	$C_{16}H_{42}B_{10}N_4O_{23}$
$D_{calc.}$/ g cm⁻³	1.492
μ/mm⁻¹	0.129
Formula Weight	766.63
Colour	colourless
Shape	plate
Size/mm³	0.280×0.100×0.010
T/K	100(2)
Crystal System	triclinic
Space Group	P-1
a/Å	9.4616(4)
b/Å	9.4780(3)
c/Å	19.8856(8)
α/°	86.483(3)
β/°	81.244(3)
γ/°	75.624(3)
V/Å³	1706.79(12)
Z	2
Z'	1
Wavelength/Å	0.71075
Radiation type	MoK$_\alpha$
Θ_{min}/°	2.073
Θ_{max}/°	27.484
Measured Refl.	38202
Independent Refl.	7820
Reflections Used	5988
R_{int}	0.0424
Parameters	537
Restraints	21
Largest Peak	0.337
Deepest Hole	-0.322
GooF	0.970
wR_2 (all data)	0.1431
wR_2	0.1276
R_1 (all data)	0.0664
R_1	0.0457

Structure Quality Indicators

Reflections:

d min (Mo)	I/σ	Rint	complete at 2θ=55°
0.77	17.1	4.24%	100%

Refinement:

Shift	Max Peak	Min Peak	GooF
0.001	0.3	-0.3	0.970

A colourless plate-shaped crystal with dimensions 0.280×0.100×0.010 mm³ was mounted on a MITIGEN holder in perfluoroether oil. X-ray diffraction data were collected using a Rigaku FRE+ diffractometer equipped with HF Varimax confocal mirrors and an AFC12 goniometer and HG Saturn 724+ detector equipped with an Oxford Cryosystems low-temperature device, operating at T = 100(2) K.

Data were measured using profile data from ω-scans of 1.0 ° per frame for 10.0 s using MoK$_\alpha$ radiation (Rotating Anode, 45.0 kV, 55.0 mA). The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.39.34b, 2017). The maximum resolution achieved was Θ = 27.484°.

Cell parameters were retrieved using the **CrysAlisPro** (Rigaku, V1.171.39.34b, 2017) software and refined using **CrysAlisPro** (Rigaku, V1.171.39.34b, 2017) on 11311 reflections, 30 % of the observed reflections. Data reduction was performed using the **CrysAlisPro** (Rigaku, V1.171.39.34b, 2017) software which corrects for Lorentz polarisation. The final completeness is 100.00 % out to 27.484° in Θ.

A multi-scan absorption correction was performed using CrysAlisPro 1.171.39.34b (Rigaku Oxford Diffraction, 2017) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm. The absorption coefficient μ of this material is 0.129 mm^{-1} at this wavelength (λ = 0.71075Å) and the minimum and maximum transmissions are 0.60935 and 1.00000.

The structure was solved in the space group P-1 (# 2) by Intrinsic Phasing using the **ShelXT** (Sheldrick, 2015) structure solution program and refined by Least Squares using version 2014/7 of **ShelXL** (Sheldrick, 2015). All non-hydrogen atoms were refined anisotropically. Most hydrogen atom positions were calculated geometrically and refined using the riding model, but some hydrogen atoms were refined freely.

_exptl_absorpt_process_details: CrysAlisPro 1.171.39.34b (Rigaku Oxford Diffraction, 2017) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

There is a single molecule in the asymmetric unit, which is represented by the reported sum formula. In other words: Z is 2 and Z' is 1.
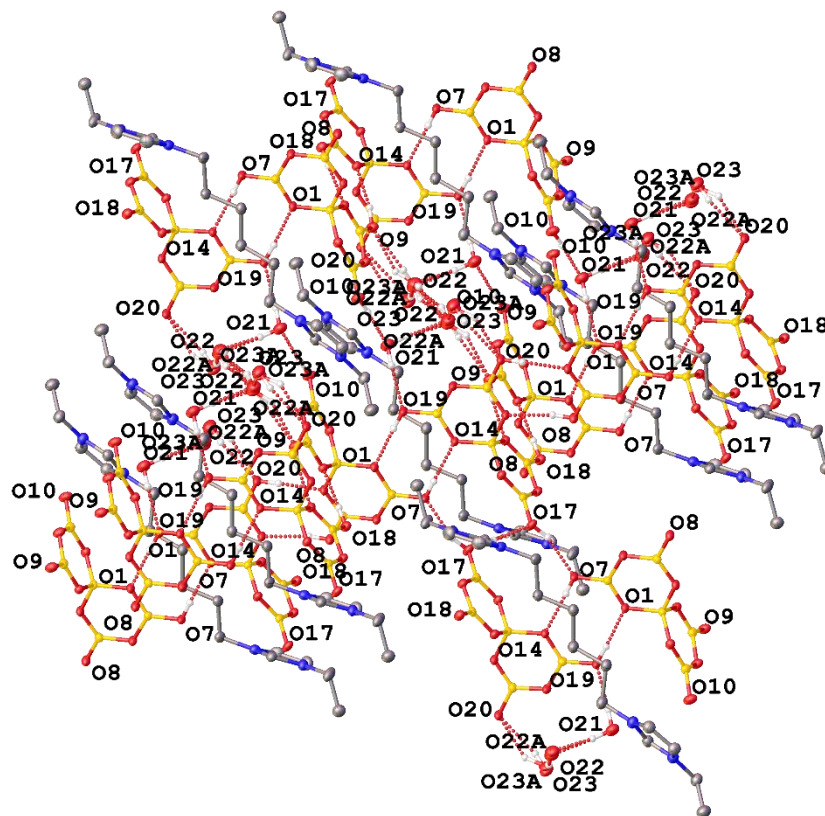
Figure S30: The following hydrogen bonding interactions with a maximum D-D distance of 2.9 Å and a minimum angle of 120 ° are present in **2017ncs0859**: O7–O14: 2.713 Å, O8–O16_*5: 2.718 Å, O9–O13*_6: 2.74 Å, O10–O21_*7: 2.685 Å, O17–O7*_3: 2.7 Å, O18–O4_*4: 2.786 Å, O19–O1: 2.683 Å, O20–O3*_1: 2.759 Å, O22–O9_*4: 3.041 Å, O22–O22*_2: 2.927 Å, O21–O19: 2.781 Å, O21–O22: 2.78 Å, O21–O22A: 2.768 Å, O23–O20: 2.911 Å, O22A–O22A_*2: 2.85 Å, O22A–O9*_4: 2.9 Å, O23A–O22: 2.7 Å, O23A–O20: 2.795 Å.
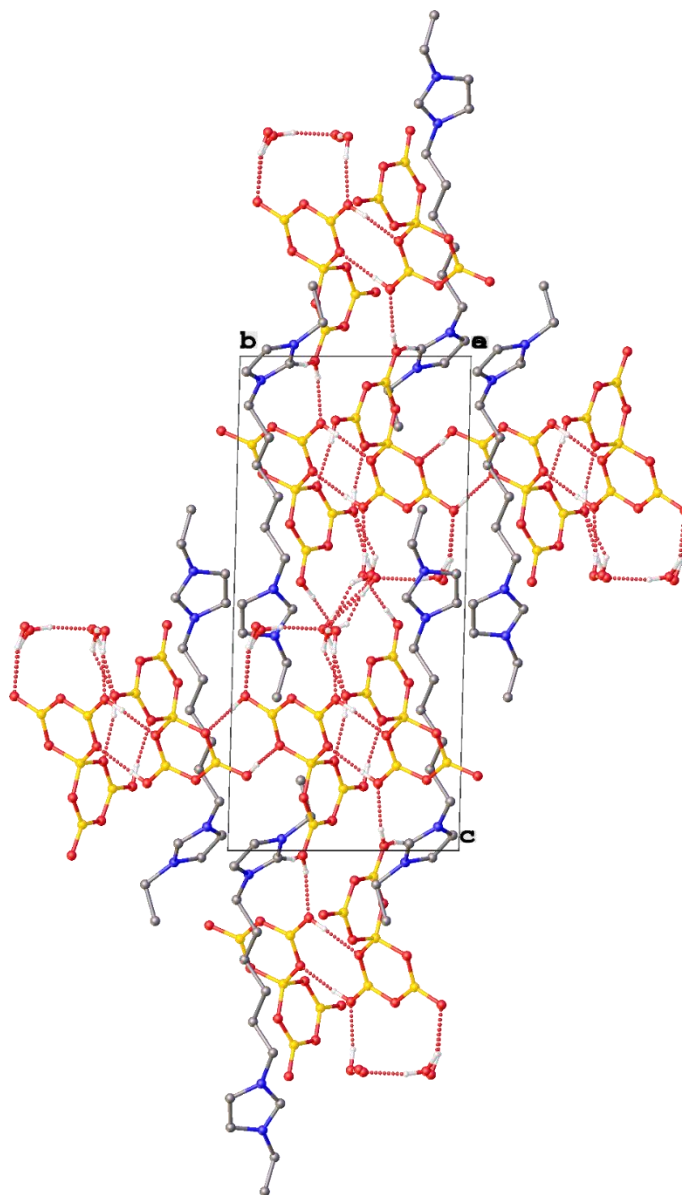
Figure S31: Packing diagram of 2017ncs0859.

Reflection Statistics

Total reflections (after filtering)	38202	Unique reflections	7820
Completeness	0.998	Mean I/σ	17.09
hkl$_{max}$ collected	(12, 12, 25)	hkl$_{min}$ collected	(-12, -11, -25)
hkl$_{max}$ used	(12, 12, 25)	hkl$_{min}$ used	(-12, -12, 0)
Lim d$_{max}$ collected	100.0	Lim d$_{min}$ collected	0.36
d$_{max}$ used	9.82	d$_{min}$ used	0.77
Friedel pairs	7114	Friedel pairs merged	1
Inconsistent equivalents	5	R$_{int}$	0.0424
R$_{sigma}$	0.0387	Intensity transformed	0
Omitted reflections	0	Omitted by user (OMIT hkl)	0
Multiplicity	(3743, 4464, 3167, 2220, 934, 367, 34, 5)	Maximum multiplicity	12
Removed systematic absences	0	Filtered off (Shel/OMIT)	0

Table S8: Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{Å}^2 \times 10^3$) for **2017ncs0859**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
N1	12929.9(15)	7685.5(15)	-236.2(7)	20.9(3)
N2	11322.8(15)	9071.8(15)	482.9(7)	19.2(3)
N3	7351.1(16)	8306.2(16)	4712.2(8)	23.7(3)
N4	5913.0(16)	8202.7(16)	5654.9(8)	22.9(3)
C1	13741(2)	6924(2)	-1420.9(9)	28.3(4)
C2	13647(2)	6451(2)	-683.6(10)	33.0(5)
C3	11624.3(18)	7840.9(17)	144.9(8)	18.4(3)
C4	13487(2)	8852(2)	-139(1)	29.0(4)
C5	12489(2)	9709(2)	311.9(10)	28.1(4)
C6	10015.8(19)	9574.7(19)	994.7(9)	23.7(4)
C7	10129.8(19)	8662.9(19)	1645.4(9)	22.2(4)
C8	8841.6(18)	9169.9(19)	2203.1(9)	22.6(4)
C9	9049.9(19)	8206.4(19)	2842.3(9)	24.7(4)
C10	7935.4(19)	8737(2)	3462.5(9)	25.3(4)
C11	8370(2)	7834(2)	4088.1(10)	28.1(4)
C12	7061(2)	9645(2)	5009.0(9)	25.7(4)
C13	6156(2)	9590.2(19)	5597.5(9)	24.4(4)
C14	6634.6(19)	7463.5(19)	5113.9(9)	23.3(4)
C15	4954(2)	7655(2)	6202.6(9)	25.7(4)
C16	5178(2)	8046(2)	6897.8(10)	33.5(4)
O1	5644.4(11)	6745.3(12)	2328.4(6)	16.3(2)
O2	5565.8(12)	8528.9(12)	1429.7(6)	18.7(3)
O3	3624.9(11)	8900.5(11)	2374.3(6)	15.8(2)
O4	3162.8(12)	6533.8(12)	2637.1(5)	15.2(2)
O5	2840.4(13)	6002.1(13)	3835.0(6)	20.1(3)
O6	4319.4(12)	7656.3(12)	3396.5(6)	16.5(2)
O7	7492.1(12)	6425.8(13)	1363.3(6)	19.6(3)
O8	3781.6(12)	10729.3(12)	1511.3(6)	19.8(3)
O9	1681.1(13)	4928.5(13)	3130.2(6)	21.2(3)
O10	3977.8(14)	7123.9(13)	4564.5(6)	23.6(3)
B1	4185.9(19)	7468.1(19)	2696.1(9)	14.1(4)
B2	6226.9(19)	7213.4(19)	1715.3(9)	15.2(4)
B3	4301.2(19)	9386(2)	1783.0(9)	16.0(4)
B4	2569(2)	5835.3(19)	3184.4(9)	16.7(4)
B5	3721(2)	6934(2)	3928.9(10)	16.8(4)
O11	10387.4(12)	3217.1(12)	1069.6(5)	15.6(2)
O12	12222.4(12)	4483.3(12)	604.7(6)	17.3(2)
O13	11433.6(12)	4508.6(12)	1806.4(5)	15.7(2)
O14	8977.7(11)	4210.3(11)	2107.2(5)	15.2(2)
O15	9162.2(12)	2600.1(12)	3075.7(6)	20.4(3)
O16	11075.5(11)	2139.5(11)	2127.1(6)	16.1(2)
O17	11108.9(13)	3239.2(13)	-96.8(6)	20.9(3)
O18	13200.8(12)	5797.9(13)	1298.3(6)	19.2(3)
O19	7077.3(13)	4512.5(13)	3054.6(6)	22.0(3)
O20	11150.1(13)	589.7(13)	3119.3(6)	20.7(3)
B11	10465.1(19)	3517.0(19)	1767.5(9)	14.1(4)
B12	11237.4(19)	3662.2(19)	527.9(9)	15.3(4)
B13	12280.0(19)	4926.9(19)	1251.4(9)	15.4(4)
B14	8408(2)	3793.0(19)	2733.6(9)	16.1(4)
B15	10490(2)	1777(2)	2761.3(9)	16.3(4)
O22	10240(40)	3900(30)	4480(20)	33(2)
O21	7223.7(15)	4449.0(15)	4442.9(6)	29.8(3)
O23	9790(20)	970(20)	4532(11)	31(2)
O22A	10220(40)	4090(30)	4429(19)	29(2)
O23A	9840(20)	1180(20)	4462(10)	26.1(16)

Table S9: Anisotropic Displacement Parameters (×10^4) **2017ncs0859**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11}+ ... +2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	20.4(7)	17.9(7)	21.6(7)	1.9(6)	0.1(6)	-2.1(6)
N2	23.0(7)	16.1(7)	18.0(7)	0.5(6)	-3.8(6)	-3.2(6)
N3	24.4(8)	23.3(8)	23.0(8)	-2.3(6)	-1.0(6)	-6.1(6)
N4	24.1(8)	21.9(8)	22.7(8)	-1.6(6)	-1.3(6)	-6.6(6)
C1	31.6(10)	28.6(10)	25.4(10)	-1.8(8)	-4.0(8)	-8.5(8)
C2	38.3(11)	23.7(10)	27.4(10)	-0.3(8)	11.0(8)	0.7(8)
C3	21.3(8)	15.9(8)	17.5(8)	0.7(7)	-1.1(6)	-4.8(7)
C4	23.2(9)	31.5(10)	35.1(11)	1.4(9)	-3.3(8)	-13.2(8)
C5	33.7(10)	25.5(10)	31(1)	-1.1(8)	-7.8(8)	-15.8(8)
C6	25.6(9)	21.2(9)	19.7(9)	-4.1(7)	-0.5(7)	2.0(7)
C7	22.5(9)	19.5(9)	21.8(9)	-1.6(7)	-1.7(7)	-0.3(7)
C8	21.0(9)	21.6(9)	24.1(9)	-4.1(7)	-2.1(7)	-3.0(7)
C9	23.3(9)	23.0(9)	25(1)	-1.4(8)	0.5(7)	-2.4(7)
C10	23.5(9)	26.2(10)	24.1(9)	-2.4(8)	0.2(7)	-3.6(7)
C11	26.9(9)	26.4(10)	26.9(10)	-2.3(8)	2.7(8)	-2.7(8)
C12	28.1(9)	22.2(9)	28.2(10)	0.1(8)	-4.9(8)	-8.2(8)
C13	29.3(9)	19.7(9)	25.2(9)	-3.1(7)	-3.2(7)	-7.6(7)
C14	23.4(9)	20.9(9)	24.5(9)	-2.9(7)	-3.1(7)	-3.1(7)
C15	27.2(9)	23.5(9)	25.7(10)	-0.3(8)	1.8(7)	-7.9(7)
C16	39.3(11)	34.1(11)	25.4(10)	1.1(8)	-2.0(8)	-7.9(9)
O1	14.7(5)	13.5(5)	17.1(6)	0.5(4)	0.8(4)	1.2(4)
O2	16.5(6)	17.0(6)	17.7(6)	4.0(5)	2.4(4)	0.7(5)
O3	13.9(5)	12.2(5)	18.2(6)	0.6(4)	1.0(4)	0.6(4)
O4	16.6(5)	14.9(6)	14.2(5)	-0.7(4)	-1.4(4)	-4.4(4)
O5	26.0(6)	21.8(6)	15.0(6)	0.4(5)	-1.1(5)	-11.9(5)
O6	16.3(5)	16.8(6)	17.1(6)	-1.5(5)	-1.4(4)	-5.7(4)
O7	18.0(6)	18.2(6)	16.3(6)	3.4(5)	1.7(5)	4.0(5)
O8	17.6(6)	16.5(6)	20.6(6)	3.2(5)	2.4(5)	0.5(5)
O9	27.4(6)	23.5(6)	16.4(6)	0.7(5)	-3.7(5)	-13.3(5)
O10	32.4(7)	27.1(7)	15.4(6)	0.1(5)	-3.7(5)	-15.1(6)
B1	13.6(8)	12.6(8)	15.5(9)	0.6(7)	-0.1(7)	-3.3(7)
B2	15.3(8)	15.0(9)	14.4(9)	-0.3(7)	-1.7(7)	-1.9(7)
B3	13.8(8)	15.4(9)	18.4(9)	1.6(7)	-3.1(7)	-3.1(7)
B4	16.4(9)	14.5(9)	18.1(9)	-1.4(7)	-0.7(7)	-2.4(7)
B5	17.8(9)	14.3(9)	17.4(9)	-2.2(7)	-1.1(7)	-3.0(7)
O11	16.3(5)	17.0(6)	13.6(6)	-1.3(5)	0.3(4)	-5.3(4)
O12	18.5(6)	19.2(6)	14.2(6)	0.4(5)	0.7(4)	-6.8(5)
O13	16.7(5)	15.8(6)	14.4(6)	-1.0(4)	-0.3(4)	-4.6(4)
O14	13.2(5)	14.8(6)	15.0(6)	1.1(4)	0.2(4)	-0.2(4)
O15	19.5(6)	19.2(6)	16.0(6)	4.1(5)	2.3(4)	3.5(5)
O16	14.9(5)	12.9(5)	16.8(6)	0.9(4)	1.8(4)	0.9(4)
O17	22.6(6)	27.6(7)	13.7(6)	-4.2(5)	3.1(5)	-10.8(5)
O18	19.9(6)	22.1(6)	17.1(6)	-0.8(5)	-0.7(5)	-9.1(5)
O19	18.8(6)	22.0(6)	17.6(6)	5.4(5)	2.8(5)	4.6(5)
O20	18.5(6)	18.1(6)	19.0(6)	3.4(5)	1.7(5)	4.5(5)
B11	14.4(8)	13.3(8)	13.1(8)	0.1(7)	-0.1(6)	-2.1(7)
B12	14.6(8)	13.5(8)	15.3(9)	-0.7(7)	0.0(7)	0.1(7)
B13	13.1(8)	13.2(9)	17.1(9)	-1.2(7)	-0.5(7)	1.0(7)
B14	17.8(9)	14.0(9)	15.0(9)	1.5(7)	-1.4(7)	-2.0(7)
B15	15.5(8)	14.4(9)	18.1(9)	-0.7(7)	-1.1(7)	-2.4(7)
O22	36.5(15)	36(5)	25(4)	7(5)	-2(2)	-10(4)
O21	35.1(7)	35.3(8)	20.3(7)	-2.6(6)	-0.9(5)	-12.4(6)
O23	35.7(18)	30(5)	25(4)	6(3)	4(2)	-7(3)
O22A	34.6(16)	28(5)	24(4)	7(4)	-2(2)	-11(4)
O23A	31.6(18)	26(4)	19(3)	13(2)	-2(2)	-7(2)

Table S10: Bond Lengths in Å for **2017ncs0859**.

Atom	Atom	Length/Å
N1	C2	1.473(2)
N1	C3	1.327(2)
N1	C4	1.373(2)
N2	C3	1.329(2)
N2	C5	1.375(2)
N2	C6	1.475(2)
N3	C11	1.470(2)
N3	C12	1.380(2)
N3	C14	1.327(2)
N4	C13	1.385(2)
N4	C14	1.321(2)
N4	C15	1.464(2)
C1	C2	1.503(3)
C4	C5	1.344(3)
C6	C7	1.514(2)
C7	C8	1.520(2)
C8	C9	1.527(3)
C9	C10	1.517(2)
C10	C11	1.516(3)
C12	C13	1.347(3)
C15	C16	1.512(3)
O1	B1	1.481(2)
O1	B2	1.355(2)
O2	B2	1.379(2)
O2	B3	1.384(2)
O3	B1	1.474(2)
O3	B3	1.362(2)

Atom	Atom	Length/Å
O4	B1	1.486(2)
O4	B4	1.360(2)
O5	B4	1.382(2)
O5	B5	1.394(2)
O6	B1	1.443(2)
O6	B5	1.353(2)
O7	B2	1.359(2)
O8	B3	1.357(2)
O9	B4	1.362(2)
O10	B5	1.353(2)
O11	B11	1.450(2)
O11	B12	1.354(2)
O12	B12	1.384(2)
O12	B13	1.390(2)
O13	B11	1.479(2)
O13	B13	1.361(2)
O14	B11	1.475(2)
O14	B14	1.356(2)
O15	B14	1.379(2)
O15	B15	1.383(2)
O16	B11	1.479(2)
O16	B15	1.358(2)
O17	B12	1.360(2)
O18	B13	1.357(2)
O19	B14	1.357(2)
O20	B15	1.360(2)

Table S11: Bond Angles in ° for **2017ncs0859**.

Atom	Atom	Atom	Angle/°
C3	N1	C2	124.90(14)
C3	N1	C4	108.70(15)
C4	N1	C2	126.40(15)
C3	N2	C5	108.06(15)
C3	N2	C6	124.93(14)
C5	N2	C6	126.81(15)
C12	N3	C11	126.46(15)
C14	N3	C11	125.01(15)
C14	N3	C12	108.35(15)
C13	N4	C15	126.15(15)
C14	N4	C13	108.80(15)
C14	N4	C15	124.97(15)
N1	C2	C1	111.39(15)
N1	C3	N2	108.74(14)
C5	C4	N1	106.88(16)
C4	C5	N2	107.62(16)
N2	C6	C7	111.17(14)
C6	C7	C8	113.58(14)
C7	C8	C9	110.25(14)
C10	C9	C8	114.67(15)
C11	C10	C9	110.27(15)
N3	C11	C10	113.24(15)
C13	C12	N3	107.40(16)
C12	C13	N4	106.55(16)
N4	C14	N3	108.89(16)
N4	C15	C16	112.15(15)

Atom	Atom	Atom	Angle/°
B2	O1	B1	123.92(13)
B2	O2	B3	119.05(13)
B3	O3	B1	123.53(13)
B4	O4	B1	122.31(13)
B4	O5	B5	118.76(13)
B5	O6	B1	123.86(13)
O1	B1	O4	107.30(12)
O3	B1	O1	110.05(12)
O3	B1	O4	108.47(12)
O6	B1	O1	109.54(13)
O6	B1	O3	109.57(13)
O6	B1	O4	111.87(13)
O1	B2	O2	120.98(14)
O1	B2	O7	121.70(15)
O7	B2	O2	117.31(14)
O3	B3	O2	121.41(15)
O8	B3	O2	116.53(15)
O8	B3	O3	122.06(15)
O4	B4	O5	121.62(14)
O4	B4	O9	122.64(15)
O9	B4	O5	115.74(15)
O6	B5	O5	121.18(15)
O6	B5	O10	119.45(15)
O10	B5	O5	119.37(15)
B12	O11	B11	123.24(12)
B12	O12	B13	118.94(13)

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
B13	O13	B11	122.85(13)	O11	B12	O17	117.15(14)
B14	O14	B11	123.42(13)	O17	B12	O12	121.13(15)
B14	O15	B15	119.36(14)	O13	B13	O12	120.84(14)
B15	O16	B11	123.07(13)	O18	B13	O12	116.97(14)
O11	B11	O13	111.90(13)	O18	B13	O13	122.18(15)
O11	B11	O14	109.44(13)	O14	B14	O15	121.17(15)
O11	B11	O16	109.40(13)	O14	B14	O19	122.24(15)
O14	B11	O13	108.14(12)	O19	B14	O15	116.59(14)
O14	B11	O16	110.29(12)	O16	B15	O15	121.08(15)
O16	B11	O13	107.65(12)	O16	B15	O20	122.70(15)
O11	B12	O12	121.70(15)	O20	B15	O15	116.22(15)

Table S12: Torsion Angles in ° for **2017ncs0859**.

Atom	Atom	Atom	Atom	Angle/°
N1	C4	C5	N2	-0.6(2)
N2	C6	C7	C8	177.62(14)
N3	C12	C13	N4	0.5(2)
C2	N1	C3	N2	179.82(16)
C2	N1	C4	C5	-179.39(17)
C3	N1	C2	C1	112.36(19)
C3	N1	C4	C5	0.3(2)
C3	N2	C5	C4	0.7(2)
C3	N2	C6	C7	71.8(2)
C4	N1	C2	C1	-68.0(2)
C4	N1	C3	N2	0.14(19)
C5	N2	C3	N1	-0.50(19)
C6	N2	C3	N1	-175.65(14)
C6	N2	C5	C4	175.71(16)
C6	C7	C8	C9	-179.37(15)
C7	C8	C9	C10	172.07(15)
C8	C9	C10	C11	-171.83(15)
C9	C10	C11	N3	178.91(15)
C11	N3	C12	C13	-175.37(16)
C11	N3	C14	N4	174.95(16)
C12	N3	C11	C10	-62.4(2)
C12	N3	C14	N4	-0.5(2)
C13	N4	C14	N3	0.8(2)
C13	N4	C15	C16	-46.0(2)
C14	N3	C11	C10	122.98(19)
C14	N3	C12	C13	0.0(2)
C14	N4	C13	C12	-0.8(2)
C14	N4	C15	C16	137.60(18)
C15	N4	C13	C12	-177.69(17)
C15	N4	C14	N3	177.76(15)
B1	O1	B2	O2	8.6(2)
B1	O1	B2	O7	-172.58(14)
B1	O3	B3	O2	1.7(2)
B1	O3	B3	O8	-177.87(14)
B1	O4	B4	O5	2.6(2)
B1	O4	B4	O9	-177.13(14)
B1	O6	B5	O5	-4.0(2)
B1	O6	B5	O10	176.28(14)
B2	O1	B1	O3	-11.7(2)
B2	O1	B1	O4	106.16(16)
B2	O1	B1	O6	-132.21(15)
B2	O2	B3	O3	-6.0(2)

Atom	Atom	Atom	Atom	Angle/°
B2	O2	B3	O8	173.59(14)
B3	O2	B2	O1	1.0(2)
B3	O2	B2	O7	-177.92(14)
B3	O3	B1	O1	6.5(2)
B3	O3	B1	O4	-110.61(16)
B3	O3	B1	O6	127.01(15)
B4	O4	B1	O1	113.71(15)
B4	O4	B1	O3	-127.43(15)
B4	O4	B1	O6	-6.5(2)
B4	O5	B5	O6	-0.7(2)
B4	O5	B5	O10	179.03(15)
B5	O5	B4	O4	1.3(2)
B5	O5	B4	O9	-178.95(14)
B5	O6	B1	O1	-111.68(16)
B5	O6	B1	O3	127.50(15)
B5	O6	B1	O4	7.2(2)
B11	O11	B12	O12	-2.0(2)
B11	O11	B12	O17	176.54(14)
B11	O13	B13	O12	2.9(2)
B11	O13	B13	O18	-177.72(14)
B11	O14	B14	O15	-4.9(2)
B11	O14	B14	O19	175.39(14)
B11	O16	B15	O15	8.5(2)
B11	O16	B15	O20	-172.43(14)
B12	O11	B11	O13	7.1(2)
B12	O11	B11	O14	126.95(15)
B12	O11	B11	O16	-112.11(15)
B12	O12	B13	O13	3.0(2)
B12	O12	B13	O18	-176.39(14)
B13	O12	B12	O11	-3.5(2)
B13	O12	B12	O17	178.02(14)
B13	O13	B11	O11	-7.6(2)
B13	O13	B11	O14	-128.19(14)
B13	O13	B11	O16	112.65(15)
B14	O14	B11	O11	133.19(15)
B14	O14	B11	O13	-104.68(16)
B14	O14	B11	O16	12.8(2)
B14	O15	B15	O16	1.2(2)
B14	O15	B15	O20	-177.95(14)
B15	O15	B14	O14	-3.0(2)
B15	O15	B14	O19	176.73(14)
B15	O16	B11	O11	-134.95(14)
B15	O16	B11	O13	103.24(16)
B15	O16	B11	O14	-14.5(2)

Table S13: Hydrogen Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **2017ncs0859**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H1A	14169	6072	-1706	42
H1B	14364	7619	-1511	42
H1C	12750	7388	-1527	42
H2A	13081	5693	-602	40
H2B	14652	6019	-572	40
H3	11007	7181	172	22
H4	14403	9021	-350	35
H5	12575	10595	482	34
H6A	9120	9514	809	28
H6B	9921	10607	1095	28
H7A	11054	8689	1815	27

Atom	x	y	z	U_{eq}
H7B	10190	7639	1543	27
H8A	7913	9126	2043	27
H8B	8771	10193	2311	27
H9A	8993	7213	2742	30
H9B	10050	8137	2954	30
H10A	7886	9773	3535	30
H10B	6947	8661	3386	30
H11A	8408	6802	4012	34
H11B	9371	7893	4151	34
H12	7432	10457	4831	31
H13	5762	10353	5911	29
H14	6640	6488	5027	28
H15A	5155	6582	6177	31
H15B	3913	8068	6139	31
H16A	4942	9109	6932	50
H16B	6208	7639	6964	50
H16C	4531	7644	7249	50
H7	7910(20)	5743(19)	1584(10)	29
H8	2980(18)	11170(20)	1744(10)	30
H9	1530(20)	4920(20)	2732(8)	32
H10	3570(20)	6610(20)	4864(10)	35
H17	11750(20)	3390(20)	-427(9)	31
H18	13180(20)	5960(20)	1720(8)	29
H19	6610(20)	5259(19)	2841(11)	33
H20	11957(19)	80(20)	2895(10)	31
H22A	10617	4309	4117	49
H22B	10268	4453	4810	49
H21A	7115	4375	4021	45
H21B	8163	4099	4453	45
H23A	8997	1679	4526	47
H23B	10002	666	4117	47
H22C	10314	4900	4583	43
H22D	10882	3924	4068	43
H23C	9943	2051	4522	39
H23D	10554	741	4123	39

Table S14: Hydrogen Bond information for **2017ncs0859**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
O7	H7	O14	0.811(15)	1.903(16)	2.7130(16)	176(2)
O8	H8	O16[1]	0.849(15)	1.877(15)	2.7182(15)	171(2)
O9	H9	O13[2]	0.828(16)	1.923(16)	2.7395(16)	169(2)
O10	H10	O21[3]	0.856(15)	1.831(16)	2.6848(18)	175(2)
O17	H17	O7[4]	0.856(15)	1.903(17)	2.7002(16)	154(2)
O18	H18	O4[5]	0.860(15)	1.930(16)	2.7860(16)	174(2)
O19	H19	O1	0.861(16)	1.824(16)	2.6830(16)	175(2)
O20	H20	O3[6]	0.869(15)	1.892(16)	2.7593(16)	176(2)
O22	H22A	O9[5]	0.87	2.18	3.04(4)	169.6
O22	H22B	O22[7]	0.87	2.08	2.93(6)	163.5
O21	H21A	O19	0.87	1.92	2.7814(18)	168.6
O21	H21B	O22	0.87	1.93	2.78(4)	163.7
O21	H21B	O22A	0.87	1.94	2.77(4)	158.4
O23	H23B	O20	0.87	2.11	2.91(2)	153.3
O22A	H22C	O22A[7]	0.87	2.17	2.85(6)	134.5
O22A	H22D	O9[5]	0.87	2.16	2.90(4)	142.4
O23A	H23C	O22	0.87	1.84	2.70(3)	169.7
O23A	H23D	O20	0.92	1.99	2.80(2)	145.0

----

[1]-1+x,1+y,+z; [2]-1+x,+y,+z; [3]1-x,1-y,1-z; [4]2-x,1-y,-z; [5]1+x,+y,+z; [6]1+x,-1+y,+z; [7]2-x,1-y,1-z

Table S15: Atomic Occupancies for all atoms that are not fully occupied in **2017ncs0859**.

Atom	Occupancy
O22	0.5
H22A	0.5
H22B	0.5
O23	0.5
H23A	0.5
H23B	0.5
O22A	0.5
H22C	0.5
H22D	0.5
O23A	0.5
H23C	0.5
H23D	0.5
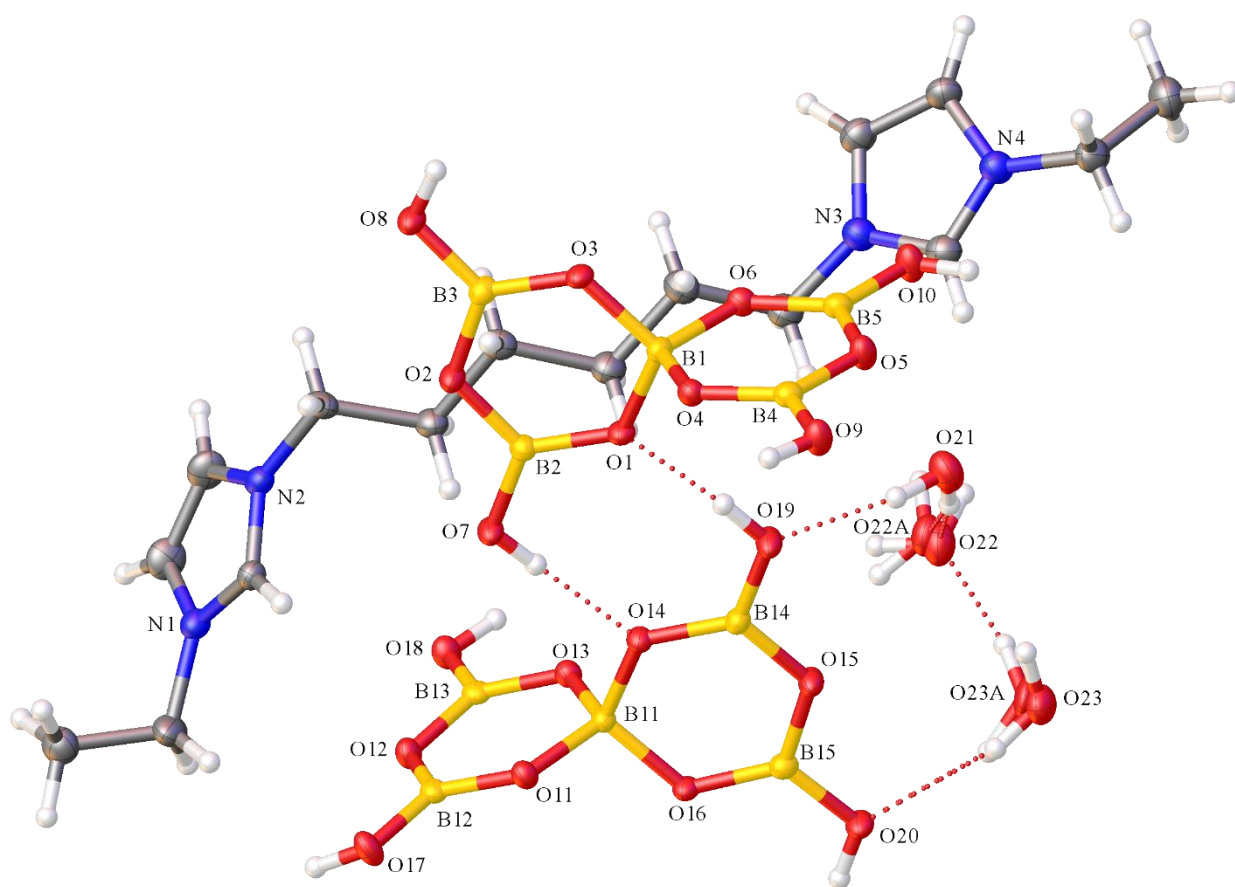
Citations for 2

CrysAlisPro Software System, Rigaku Oxford Diffraction, (2017).

O.V. Dolomanov and L.J. Bourhis and R.J. Gildea and J.A.K. Howard and H. Puschmann, Olex2: A complete structure solution, refinement and analysis program, *J. Appl. Cryst.*, (2009), **42**, 339-341.

Sheldrick, G.M., Crystal structure refinement with ShelXL, *Acta Cryst.*, (2015), **C27**, 3-8.

Sheldrick, G.M., ShelXT-Integrated space-group and crystal-structure determination, *Acta Cryst.*, (2015), **A71**, 3-8.

Submitted by: **Mike A Beckett / Thomas A. Rixon**
                Bangor University

Solved by: **Peter N Horton**

Sample ID: **MAB/TR/AR106B5**
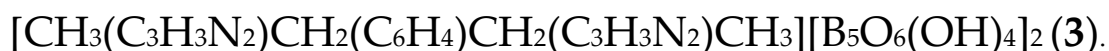
[CH$_3$(C$_3$H$_3$N$_2$)CH$_2$(C$_6$H$_4$)CH$_2$(C$_3$H$_3$N$_2$)CH$_3$][B$_5$O$_6$(OH)$_4$]$_2$ (**3**).

Crystal Data and Experimental
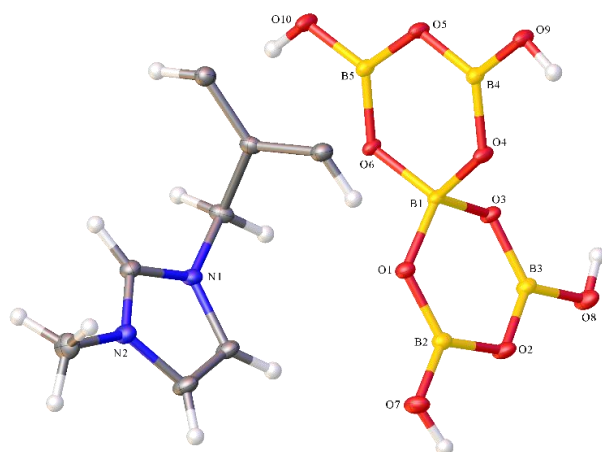
Experimental. Single colourless block-shaped crystals of **2018ncs0811** were recrystallised from water. A suitable crystal 0.130×0.100×0.060 mm^3 was selected and mounted on a MITIGEN holder in perfluoroether oil on a Rigaku FRE+ diffractometer equipped with VHF Varimax confocal mirrors and an AFC12 goniometer and HyPix 6000 detector. The crystal was kept at a steady T = 100(2) K during data collection. The structure was solved with the **ShelXT** (Sheldrick, 2015) structure solution program using the Intrinsic Phasing solution method and by using **Olex2** (Dolomanov et al., 2009) as the graphical interface. The model was refined with version 2018/3 of **ShelXL** (Sheldrick, 2015) using Least Squares minimisation.

Crystal Data. C$_{16}$H$_{28}$B$_{10}$N$_4$O$_{20}$, M_r = 704.52, triclinic, P-1 (No. 2), a = 9.0501(2) Å, b = 9.1652(2) Å, c = 9.5476(2) Å, α = 104.254(2)°, β = 94.968(2)°, γ = 103.538(2)°, V = 737.43(3) Å^3, T = 100(2) K, Z = 1, Z' = 0.5, μ(MoK$_\alpha$) = 0.135 mm^{-1}, 44644 reflections measured, 4490 unique (R_{int} = 0.0242) which were used in all calculations. The final wR_2 was 0.0890 (all data) and R_1 was 0.0312 (I > 2(I)).

Compound	2018ncs0811
Formula	C$_{16}$H$_{28}$B$_{10}$N$_4$O$_{20}$
$D_{calc.}$/ g cm^{-3}	1.586
μ/mm^{-1}	0.135
Formula Weight	704.52
Colour	colourless
Shape	block
Size/mm^3	0.130×0.100×0.060
T/K	100(2)
Crystal System	triclinic
Space Group	P-1
a/Å	9.0501(2)
b/Å	9.1652(2)
c/Å	9.5476(2)
α/°	104.254(2)
β/°	94.968(2)
γ/°	103.538(2)
V/Å^3	737.43(3)
Z	1
Z'	0.5
Wavelength/Å	0.71075
Radiation type	MoK$_\alpha$
Θ_{min}/°	2.227
Θ_{max}/°	30.507
Measured Refl.	44644
Independent Refl.	4490
Reflections with I > 2(I)	4220
R_{int}	0.0242
Parameters	243
Restraints	0
Largest Peak	0.454
Deepest Hole	-0.216
GooF	1.061
wR_2 (all data)	0.0890
wR_2	0.0880
R_1 (all data)	0.0326
R_1	0.0312

Structure Quality Indicators

Reflections:

d min (Mo)	I/σ	Rint	complete 100% (IUCr)
0.70	90.3	2.42%	100%

Refinement:

Shift	Max Peak	Min Peak	GooF
0.001	0.5	-0.2	1.061

A colourless block-shaped crystal with dimensions 0.130×0.100×0.060 mm^3 was mounted on a MITIGEN holder in perfluoroether oil. Data were collected using an Rigaku FRE+ diffractometer equipped with VHF Varimax confocal mirrors and an AFC12 goniometer and HyPix 6000 detector equipped with an Oxford Cryosystems low-temperature device operating at T = 100(2) K.

Data were measured using profile data from ω-scans 0.5 ° per frame for 1.0 s using MoK$_\alpha$ radiation (Rotating anode, 45.0 kV, 55.0 mA). The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.40.18b, 2018) The maximum resolution that was achieved was Θ = 30.507° (0.70 Å).

The diffraction pattern was indexed. The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.40.18b, 2018) and the unit cell was refined using **CrysAlisPro** (Rigaku, V1.171.40.18b, 2018) on 36394 reflections, 82% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using **CrysAlisPro** (Rigaku, V1.171.40.18b, 2018). The final completeness is 99.90 % out to 30.507° in Θ. A multi-scan absorption correction was performed using CrysAlisPro 1.171.40.18b (Rigaku Oxford Diffraction, 2018) using spherical harmonics as implemented in SCALE3 ABSPACK. The absorption coefficient μ of this material is 0.135 mm^{-1} at this wavelength (λ = 0.71075 Å) and the minimum and maximum transmissions are 0.815 and 1.000.

The structure was solved and the space group P-1 (# 2) determined by the **ShelXT** (Sheldrick, 2015) structure solution program using Intrinsic Phasing and refined by Least Squares using version 2018/3 of **ShelXL** (Sheldrick, 2015). All non-hydrogen atoms were refined anisotropically. Most hydrogen atom positions were calculated geometrically and refined using the riding model, but some hydrogen atoms were refined freely.

_exptl_absorpt_process_details: CrysAlisPro 1.171.40.18b (Rigaku Oxford Diffraction, 2018) using spherical harmonics as implemented in SCALE3 ABSPACK.

The value of Z' is 0.5. This means that only half of the formula unit is present in the asymmetric unit, with the other half consisting of symmetry equivalent atoms.

Figure S32: The following hydrogen bonding interactions with a maximum D-D distance of 3.2 Å and a minimum angle of 120 ° are present in **2018ncs0811**: C1–O9_*4: 3.047 Å, O7–O8_*5: 3.127 Å, O8–O3_*1: 2.748 Å, O9–O4_*2: 2.661 Å, O10–O6_*3: 2.705 Å.
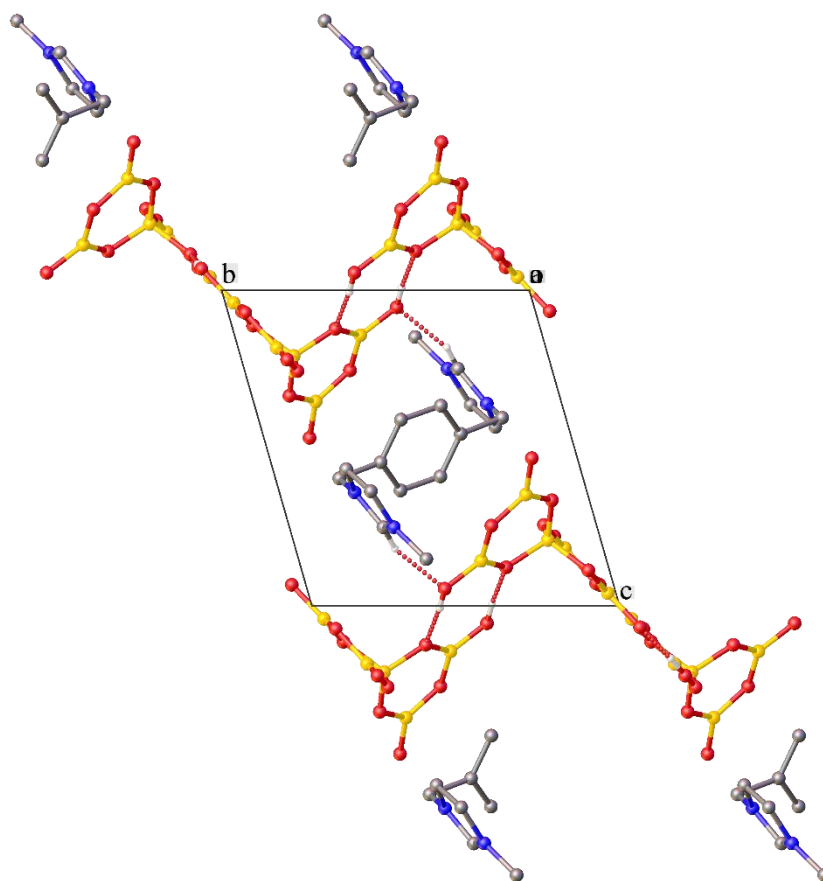
Figure S33: Packing diagram of 2018ncs0811.

Reflection Statistics

Total reflections (after filtering)	44644	Unique reflections	4490
Completeness	0.999	Mean I/σ	55.22
hkl_{max} collected	(12, 13, 13)	hkl_{min} collected	(-12, -13, -13)
hkl_{max} used	(12, 12, 13)	hkl_{min} used	(-12, -13, 0)
Lim d_{max} collected	100.0	Lim d_{min} collected	0.36
d_{max} used	9.14	d_{min} used	0.7
Friedel pairs	4465	Friedel pairs merged	1
Inconsistent equivalents	0	R_{int}	0.0242
R_{sigma}	0.0111	Intensity transformed	0
Omitted reflections	0	Omitted by user (OMIT hkl)	0
Multiplicity	(237, 1121, 1503, 1451, 1444, 1082, 719, 518, 358, 243, 137, 74, 41, 23, 3, 1)	Maximum multiplicity	23
Removed systematic absences	0	Filtered off (Shel/OMIT)	0

Table S16: Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **2018ncs0811**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
N(1)	1159.2(7)	2439.9(7)	3597.9(7)	12.09(12)
N(2)	-410.9(8)	3391.9(8)	2494.6(7)	14.08(13)
C(1)	938.7(9)	3053.5(9)	2491.6(8)	13.41(14)
C(2)	-92.6(9)	2380.4(9)	4330.3(9)	15.20(14)
C(3)	-1080.1(9)	2976.1(10)	3631.5(9)	17.02(15)
C(4)	2601.5(9)	2072.5(9)	4040.6(9)	14.36(14)
C(5)	3877.0(8)	3567.4(9)	4551.5(8)	12.41(14)
C(6)	3925.7(9)	4622.1(9)	5896.1(9)	14.09(14)
C(7)	4960.4(9)	3951.0(9)	3658.7(8)	14.07(14)
C(8)	-1031.6(10)	4150.2(11)	1487.5(10)	20.41(16)
O(1)	2406.8(6)	1709.0(6)	7433.0(6)	11.81(11)
O(2)	1457.1(6)	555.8(7)	9278.4(6)	14.75(12)
O(3)	4045.6(6)	687.5(6)	8870.1(6)	11.91(11)
O(4)	4820.1(6)	3331.9(6)	8774.0(6)	12.24(11)
O(5)	6948.9(6)	3424.4(6)	7458.7(6)	12.65(11)
O(6)	4737.3(6)	1221.6(6)	6653.0(6)	11.03(11)
O(7)	-154.9(7)	1481.3(7)	7798.0(7)	16.85(12)
O(8)	2986.3(7)	-460.7(7)	10671.1(7)	17.24(12)
O(9)	6997.8(6)	5525.3(6)	9453.7(6)	13.37(11)
O(10)	6840.0(7)	1483.6(7)	5321.9(6)	14.82(12)
B(1)	3984.3(9)	1726.3(9)	7910.9(9)	10.02(14)
B(2)	1248.9(9)	1251.2(9)	8168.5(9)	11.86(15)
B(3)	2858.1(9)	265.8(10)	9592.5(9)	12.13(15)
B(4)	6232.0(9)	4083.1(9)	8566.9(9)	10.74(14)
B(5)	6154.5(9)	2016.5(9)	6470.7(9)	10.62(14)

Table S17: Anisotropic Displacement Parameters (×10^4) **2018ncs0811**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + \ldots + 2hka^* \times b^* \times U_{12}]\$$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	9.4(3)	12.7(3)	14.1(3)	5.3(2)	2.6(2)	1.0(2)
N(2)	12.7(3)	15.6(3)	15.8(3)	8.0(2)	3.7(2)	3.0(2)
C(1)	12.0(3)	14.1(3)	14.2(3)	5.5(3)	3.5(2)	1.3(2)
C(2)	13.0(3)	18.0(3)	16.9(3)	8.4(3)	6.2(3)	3.2(3)
C(3)	13.9(3)	21.3(4)	20.1(4)	11.1(3)	7.8(3)	5.2(3)
C(4)	10.5(3)	12.7(3)	20.0(3)	5.9(3)	0.9(3)	2.3(2)
C(5)	9.3(3)	12.6(3)	15.8(3)	5.3(3)	0.9(2)	2.8(2)
C(6)	11.4(3)	15.6(3)	15.9(3)	5.7(3)	3.5(2)	2.9(3)
C(7)	12.3(3)	14.8(3)	14.8(3)	3.7(3)	2.3(2)	3.4(3)
C(8)	21.5(4)	23.5(4)	21.1(4)	13.1(3)	2.6(3)	8.1(3)
O(1)	9.9(2)	13.5(2)	12.6(2)	4.83(19)	3.42(18)	2.19(18)
O(2)	10.8(2)	19.3(3)	17.7(3)	10.2(2)	6.32(19)	4.0(2)
O(3)	10.3(2)	14.2(2)	13.3(2)	6.80(19)	5.03(18)	2.84(18)
O(4)	11.5(2)	10.5(2)	12.3(2)	0.41(18)	5.67(18)	-0.16(18)
O(5)	10.2(2)	12.0(2)	13.4(2)	0.17(19)	5.43(18)	0.61(18)
O(6)	10.9(2)	10.6(2)	10.6(2)	1.76(18)	5.18(18)	0.84(18)
O(7)	10.2(2)	21.5(3)	21.1(3)	9.3(2)	3.9(2)	4.2(2)
O(8)	14.0(3)	23.9(3)	19.6(3)	13.9(2)	7.0(2)	6.0(2)
O(9)	11.9(2)	11.9(2)	13.1(2)	-0.21(19)	5.39(19)	-0.35(19)
O(10)	13.2(2)	13.5(3)	14.5(3)	-0.6(2)	7.4(2)	0.17(19)
B(1)	9.8(3)	9.8(3)	10.0(3)	2.6(2)	4.0(2)	0.9(2)
B(2)	10.4(3)	11.2(3)	13.2(3)	2.9(3)	2.8(3)	1.4(3)
B(3)	11.7(3)	12.1(3)	13.0(3)	4.5(3)	4.5(3)	1.9(3)
B(4)	10.5(3)	10.8(3)	10.5(3)	2.6(3)	3.0(3)	1.7(3)
B(5)	10.5(3)	10.4(3)	11.1(3)	3.0(3)	3.7(3)	2.4(3)

Table S18: Bond Lengths in Å for **2018ncs0811**.

Atom	Atom	Length/Å
N(1)	C(1)	1.3343(10)
N(1)	C(2)	1.3793(9)
N(1)	C(4)	1.4751(9)
N(2)	C(1)	1.3287(10)
N(2)	C(3)	1.3783(10)
N(2)	C(8)	1.4634(10)
C(2)	C(3)	1.3588(11)
C(4)	C(5)	1.5101(10)
C(5)	C(6)	1.3942(11)
C(5)	C(7)	1.3937(10)
C(6)	C(7)[1]	1.3935(10)
O(1)	B(1)	1.4559(9)
O(1)	B(2)	1.3543(9)
O(2)	B(2)	1.3853(10)
O(2)	B(3)	1.3782(10)
O(3)	B(1)	1.4811(9)
O(3)	B(3)	1.3584(9)
O(4)	B(1)	1.4800(9)
O(4)	B(4)	1.3582(9)
O(5)	B(4)	1.3758(9)
O(5)	B(5)	1.3859(9)
O(6)	B(1)	1.4618(9)
O(6)	B(5)	1.3674(9)
O(7)	B(2)	1.3668(10)
O(8)	B(3)	1.3677(10)
O(9)	B(4)	1.3648(9)
O(10)	B(5)	1.3499(9)

––––

[1]1-x,1-y,1-z

Table S19: Bond Angles in ° for **2018ncs0811**.

Atom	Atom	Atom	Angle/°
C(1)	N(1)	C(2)	109.00(6)
C(1)	N(1)	C(4)	124.09(6)
C(2)	N(1)	C(4)	126.54(6)
C(1)	N(2)	C(3)	108.86(6)
C(1)	N(2)	C(8)	124.80(7)
C(3)	N(2)	C(8)	126.28(7)
N(2)	C(1)	N(1)	108.38(6)
C(3)	C(2)	N(1)	106.59(7)
C(2)	C(3)	N(2)	107.17(7)
N(1)	C(4)	C(5)	108.98(6)
C(6)	C(5)	C(4)	119.81(7)
C(7)	C(5)	C(4)	120.79(7)
C(7)	C(5)	C(6)	119.29(7)
C(7)[1]	C(6)	C(5)	120.44(7)
C(6)[1]	C(7)	C(5)	120.26(7)
B(2)	O(1)	B(1)	121.80(6)
B(3)	O(2)	B(2)	118.88(6)
B(3)	O(3)	B(1)	121.00(6)
B(4)	O(4)	B(1)	123.46(6)
B(4)	O(5)	B(5)	118.71(6)
B(5)	O(6)	B(1)	123.67(6)
O(1)	B(1)	O(3)	111.43(6)
O(1)	B(1)	O(4)	108.52(6)
O(1)	B(1)	O(6)	110.57(6)
O(4)	B(1)	O(3)	107.46(6)
O(6)	B(1)	O(3)	108.56(6)
O(6)	B(1)	O(4)	110.25(6)
O(1)	B(2)	O(2)	121.52(7)
O(1)	B(2)	O(7)	118.86(7)
O(7)	B(2)	O(2)	119.62(7)
O(3)	B(3)	O(2)	121.54(7)
O(3)	B(3)	O(8)	122.05(7)
O(8)	B(3)	O(2)	116.41(7)
O(4)	B(4)	O(5)	121.52(7)
O(4)	B(4)	O(9)	121.20(7)
O(9)	B(4)	O(5)	117.29(6)
O(6)	B(5)	O(5)	121.14(6)
O(10)	B(5)	O(5)	116.84(6)
O(10)	B(5)	O(6)	122.01(7)

----
[1]1-x,1-y,1-z

Table S20: Torsion Angles in ° for **2018ncs0811**.

Atom	Atom	Atom	Atom	Angle/°
N(1)	C(2)	C(3)	N(2)	-0.25(9)
N(1)	C(4)	C(5)	C(6)	73.09(9)
N(1)	C(4)	C(5)	C(7)	-103.22(8)
C(1)	N(1)	C(2)	C(3)	0.07(9)
C(1)	N(1)	C(4)	C(5)	63.38(9)
C(1)	N(2)	C(3)	C(2)	0.35(9)
C(2)	N(1)	C(1)	N(2)	0.15(9)
C(2)	N(1)	C(4)	C(5)	-108.85(8)
C(3)	N(2)	C(1)	N(1)	-0.31(9)
C(4)	N(1)	C(1)	N(2)	-173.25(6)
C(4)	N(1)	C(2)	C(3)	173.27(7)
C(4)	C(5)	C(6)	C(7)[1]	-176.02(7)
C(4)	C(5)	C(7)	C(6)[1]	175.99(7)
C(6)	C(5)	C(7)	C(6)[1]	-0.35(12)
C(7)	C(5)	C(6)	C(7)[1]	0.35(12)
C(8)	N(2)	C(1)	N(1)	177.07(7)
C(8)	N(2)	C(3)	C(2)	-176.98(8)
B(1)	O(1)	B(2)	O(2)	-9.89(11)
B(1)	O(1)	B(2)	O(7)	170.28(7)
B(1)	O(3)	B(3)	O(2)	11.28(11)
B(1)	O(3)	B(3)	O(8)	-168.44(7)
B(1)	O(4)	B(4)	O(5)	4.23(11)
B(1)	O(4)	B(4)	O(9)	-175.73(7)
B(1)	O(6)	B(5)	O(5)	-3.24(11)
B(1)	O(6)	B(5)	O(10)	177.43(7)
B(2)	O(1)	B(1)	O(3)	20.97(9)
B(2)	O(1)	B(1)	O(4)	-97.15(8)
B(2)	O(1)	B(1)	O(6)	141.81(7)
B(2)	O(2)	B(3)	O(3)	1.98(11)
B(2)	O(2)	B(3)	O(8)	-178.28(7)
B(3)	O(2)	B(2)	O(1)	-2.83(11)
B(3)	O(2)	B(2)	O(7)	177.00(7)
B(3)	O(3)	B(1)	O(1)	-21.63(9)
B(3)	O(3)	B(1)	O(4)	97.13(7)
B(3)	O(3)	B(1)	O(6)	-143.64(7)
B(4)	O(4)	B(1)	O(1)	-132.72(7)
B(4)	O(4)	B(1)	O(3)	106.67(7)
B(4)	O(4)	B(1)	O(6)	-11.48(10)
B(4)	O(5)	B(5)	O(6)	-5.48(11)
B(4)	O(5)	B(5)	O(10)	173.89(7)
B(5)	O(5)	B(4)	O(4)	4.95(11)
B(5)	O(5)	B(4)	O(9)	-175.09(6)
B(5)	O(6)	B(1)	O(1)	130.96(7)
B(5)	O(6)	B(1)	O(3)	-106.51(7)
B(5)	O(6)	B(1)	O(4)	10.96(10)

––––

[1]1-x,1-y,1-z

Table S21: Hydrogen Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **2018ncs0811**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H(1)	1629.13	3220.04	1816.69	16
H(2)	-236.29	1997.77	5161.09	18
H(3)	-2050.04	3085.42	3880.03	20
H(4A)	2858.73	1345.9	3202.78	17
H(4B)	2477.03	1562.5	4840.24	17
H(6)	3194.58	4366.42	6513.04	17
H(7)	4939.48	3236.97	2743.06	17
H(8A)	-710.61	5284.37	1913.54	31
H(8B)	-2157.1	3790.04	1317.58	31
H(8C)	-642.78	3883.85	556.88	31
H(7A)	-819(19)	1164(19)	8338(18)	41(4)
H(8)	3909(19)	-535(18)	10811(17)	38(4)
H(9)	6417(17)	5864(17)	10059(17)	33(4)
H(10)	6290(19)	600(20)	4714(19)	47(4)

Table S22: Hydrogen Bond information for **2018ncs0811**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
C(1)	H(1)	O(9)[1]	0.95	2.15	3.0475(9)	157.4
O(7)	H(7A)	O(8)[2]	0.870(17)	2.278(17)	3.1269(8)	165.2(14)
O(8)	H(8)	O(3)[3]	0.855(16)	1.893(16)	2.7483(8)	179.6(18)
O(9)	H(9)	O(4)[4]	0.864(16)	1.799(16)	2.6614(8)	175.4(15)
O(10)	H(10)	O(6)[5]	0.877(18)	1.830(18)	2.7046(8)	175.3(17)

––––

[1]1-x,1-y,1-z; [2]-x,-y,2-z; [3]1-x,-y,2-z; [4]1-x,1-y,2-z; [5]1-x,-y,1-z
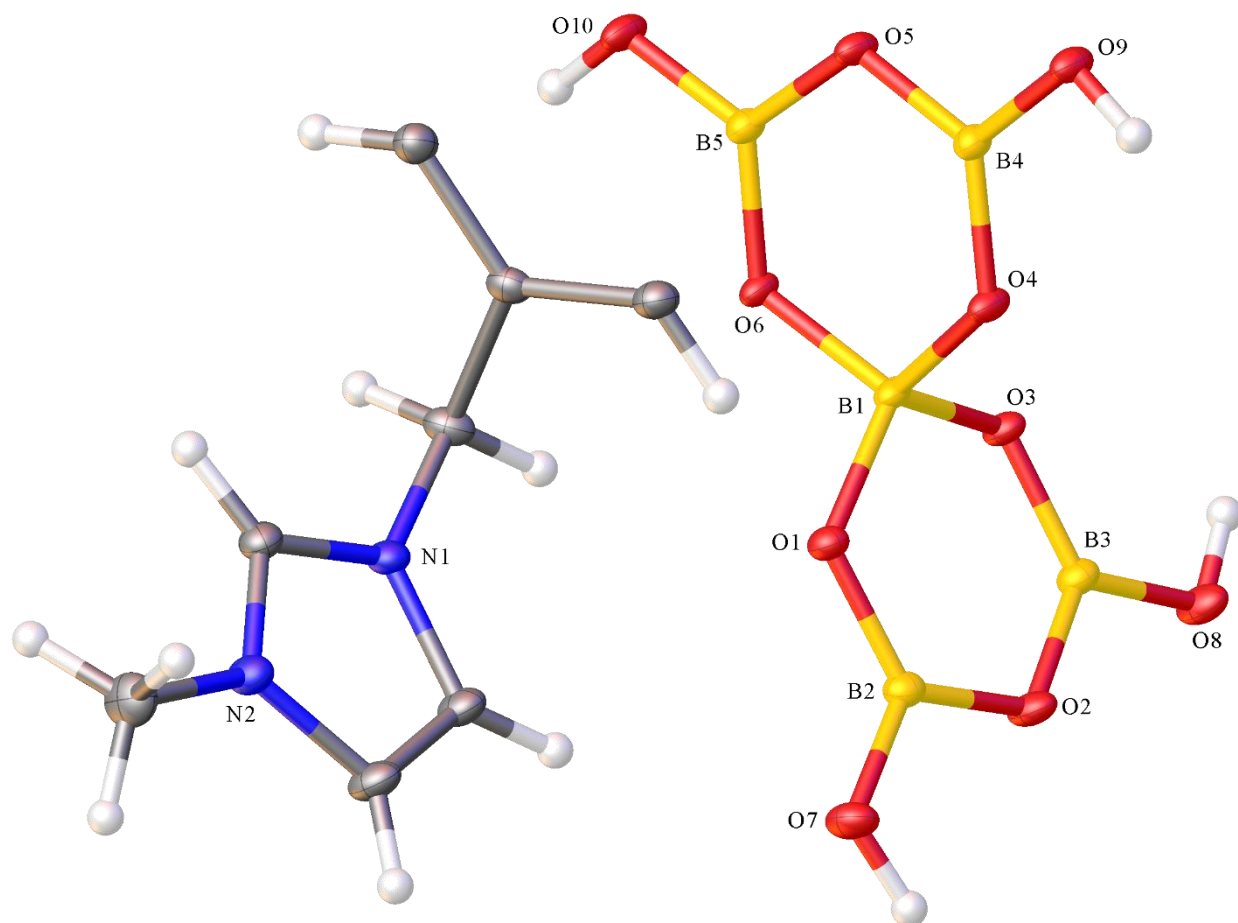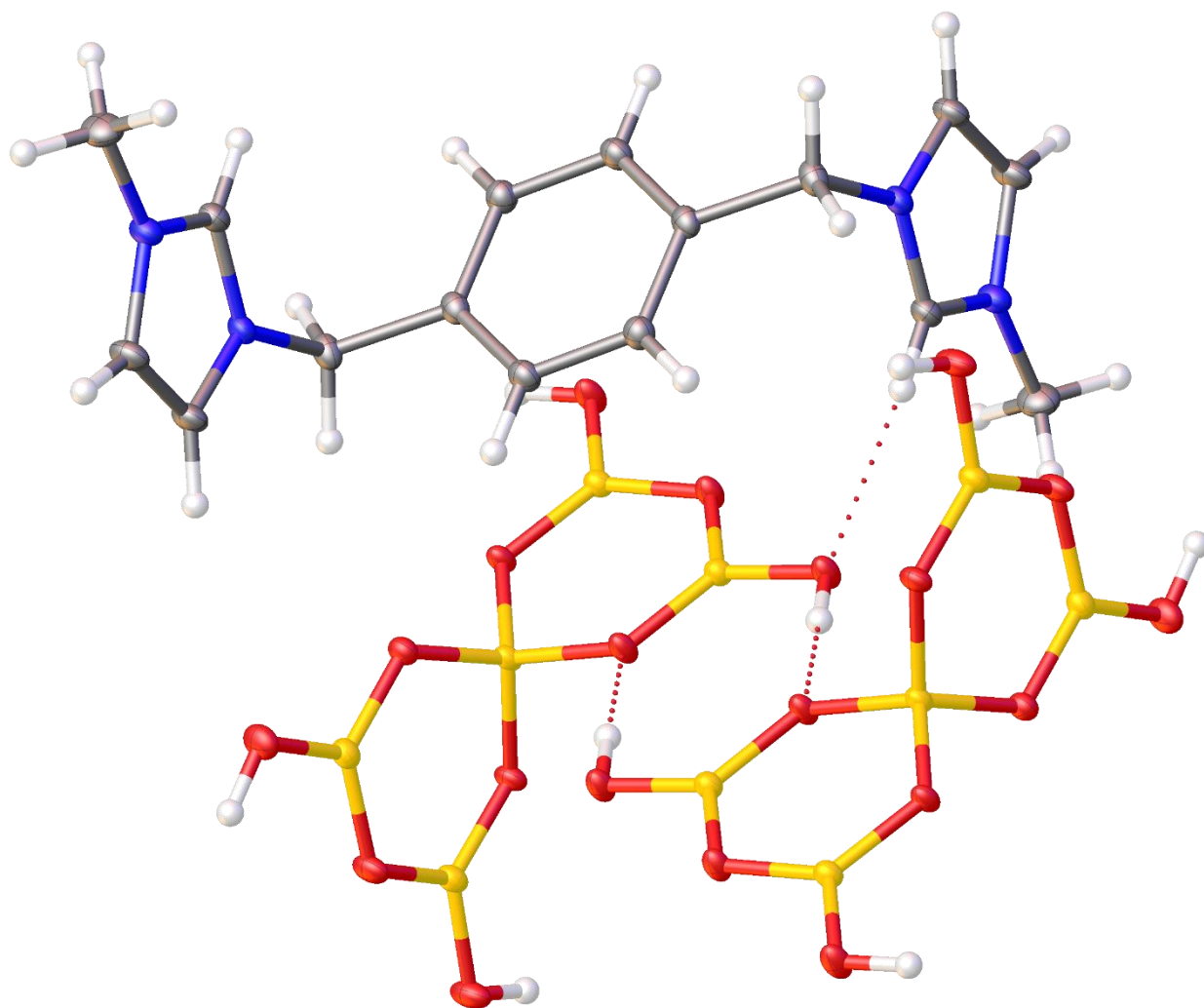
Citations for 3

CrysAlisPro Software System, Rigaku Oxford Diffraction, (2018).

O.V. Dolomanov and L.J. Bourhis and R.J. Gildea and J.A.K. Howard and H. Puschmann, Olex2: A complete structure solution, refinement and analysis program, *J. Appl. Cryst.*, (2009), **42**, 339-341.

Sheldrick, G.M., Crystal structure refinement with ShelXL, *Acta Cryst.*, (2015), **C27**, 3-8.

Sheldrick, G.M., ShelXT-Integrated space-group and crystal-structure determination, *Acta Cryst.*, (2015), **A71**, 3-8.

Submitted by: **Mike A Beckett / Thomas A. Rixon**

Bangor University

Solved by: **Peter N Horton**

Sample ID: **MAB/TR/AR101 B5**

$[CH_3(C_4H_8N)(CH_2)_6(C_4H_8N)CH_3][B_5O_6(OH)_4]_2$ (**4a**)

Crystal Data and Experimental
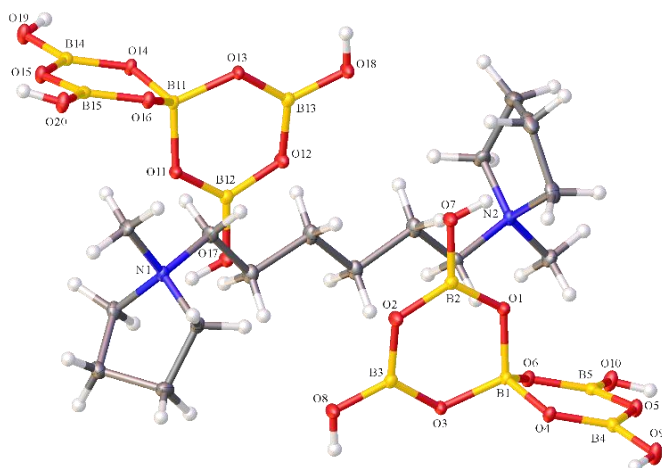
Experimental. Single colourless block-shaped crystals of **2018ncs0571** were recrystallised from water. A suitable crystal 0.220×0.200×0.120 mm³ was selected and mounted on a MITIGEN holder in perfluoroether oil on a Rigaku FRE+ diffractometer equipped with VHF Varimax confocal mirrors and an AFC12 goniometer and HyPix 6000 detector. The crystal was kept at a steady $T = 100(2)$ K during data collection. The structure was solved with the **ShelXT** (Sheldrick, 2015) structure solution program using the Intrinsic Phasing solution method and by using **Olex2** (Dolomanov et al., 2009) as the graphical interface. The model was refined with version 2018/3 of **ShelXL** (Sheldrick, 2015) using Least Squares minimisation.

Crystal Data. $C_{16}H_{42}B_{10}N_2O_{20}$, $M_r = 690.61$, triclinic, P-1 (No. 2), $a = 9.05340(10)$ Å, $b = 11.9367(2)$ Å, $c = 14.5824(2)$ Å, $\alpha = 94.1950(10)°$, $\beta = 104.1560(10)°$, $\gamma = 94.8550(10)°$, $V = 1515.35(4)$ Å³, $T = 100(2)$ K, $Z = 2$, $Z' = 1$, $\mu(MoK_\alpha) = 0.127$ mm⁻¹, 69149 reflections measured, 6954 unique ($R_{int} = 0.0243$) which were used in all calculations. The final wR_2 was 0.0906 (all data) and R_1 was 0.0314 (I > 2(I)).

Compound	2018ncs0571
Formula	$C_{16}H_{42}B_{10}N_2O_{20}$
$D_{calc.}$/ g cm⁻³	1.514
μ/mm⁻¹	0.127
Formula Weight	690.61
Colour	colourless
Shape	block
Size/mm³	0.220×0.200×0.120
T/K	100(2)
Crystal System	triclinic
Space Group	P-1
a/Å	9.05340(10)
b/Å	11.9367(2)
c/Å	14.5824(2)
α/°	94.1950(10)
β/°	104.1560(10)
γ/°	94.8550(10)
V/Å³	1515.35(4)
Z	2
Z′	1
Wavelength/Å	0.71075
Radiation type	MoK$_\alpha$
Θ_{min}/°	1.720
Θ_{max}/°	27.484
Measured Refl.	69149
Independent Refl.	6954
Reflections with I > 2(I)	6407
R_{int}	0.0243
Parameters	443
Restraints	0
Largest Peak	0.339
Deepest Hole	-0.298
GooF	1.044
wR_2 (all data)	0.0906
wR_2	0.0889
R_1 (all data)	0.0337
R_1	0.0314

Structure Quality Indicators

Reflections:

d min (Mo)	I/σ	Rint	complete 100% (IUCr)
0.77	85.7	2.43%	100%

Refinement:

Shift	Max Peak	Min Peak	GooF
0.001	0.3	-0.3	1.044

A colourless block-shaped crystal with dimensions 0.220×0.200×0.120 mm^3 was mounted on a MITIGEN holder in perfluoroether oil. Data were collected using an Rigaku FRE+ diffractometer equipped with VHF Varimax confocal mirrors and an AFC12 goniometer and HyPix 6000 detector equipped with an Oxford Cryosystems low-temperature device operating at T = 100(2) K.

Data were measured using profile data from ω-scans 0.5° per frame for 0.5 s using MoK$_\alpha$ radiation. The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.39.46, 2018). The maximum resolution that was achieved was Θ = 27.484° (0.77 Å).

The diffraction pattern was indexed. The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.39.46, 2018) and the unit cell was refined using **CrysAlisPro** (Rigaku, V1.171.39.46, 2018) on 42854 reflections, 62% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using **CrysAlisPro** (Rigaku, V1.171.39.46, 2018). The final completeness is 99.90 % out to 27.484° in Θ. A multi-scan absorption correction was performed using CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018) using spherical harmonics as implemented in SCALE3 ABSPACK. The absorption coefficient μ of this material is 0.127 mm^{-1} at this wavelength (λ = 0.71075 Å) and the minimum and maximum transmissions are 0.757 and 1.000.

The structure was solved and the space group P-1 (# 2) determined by the **ShelXT** (Sheldrick, 2015) structure solution program using Intrinsic Phasing and refined by Least Squares using version 2018/3 of **ShelXL** (Sheldrick, 2015). All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model. Hydrogen atom positions were calculated geometrically and refined using the riding model.

_exptl_absorpt_process_details: CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018) using spherical harmonics as implemented in SCALE3 ABSPACK.

There is a single molecule in the asymmetric unit, which is represented by the reported sum formula. In other words: Z is 2 and Z' is 1.
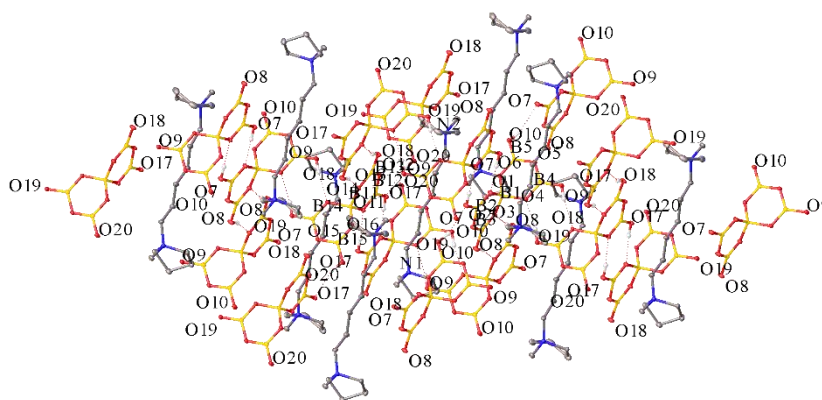
Figure S34: The following hydrogen bonding interactions with a maximum D-D distance of 3.02 Å and a minimum angle of 120 ° are present in **2018ncs0571**: O17–O11_*5: 2.784 Å, O7–O1*_1: 2.779 Å, O18–O13_*6: 2.804 Å, O10–O7*_8: 2.88 Å, O8–O3_*2: 2.768 Å, O20–O17*_7: 2.854 Å, O9–O14_*3: 2.769 Å, O19–O4*_4: 2.748 Å.
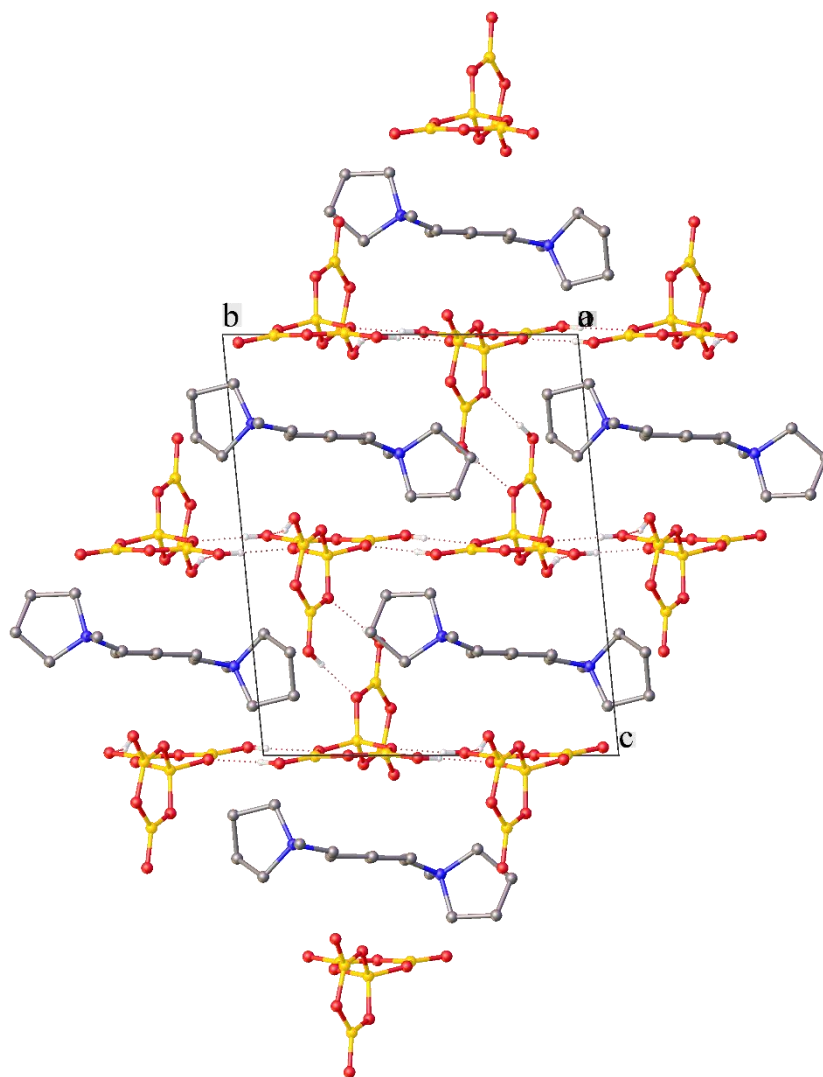
Figure S35: Packing diagram of 2018ncs0571.

Reflection Statistics

Total reflections (after filtering)	69149	Unique reflections	6954
Completeness	1.0	Mean I/σ	46.48
hkl_{max} collected	(11, 15, 18)	hkl_{min} collected	(-11, -15, -18)
hkl_{max} used	(11, 15, 18)	hkl_{min} used	(-11, -15, 0)
Lim d_{max} collected	100.0	Lim d_{min} collected	0.36
d_{max} used	11.84	d_{min} used	0.77
Friedel pairs	6880	Friedel pairs merged	1
Inconsistent equivalents	0	R_{int}	0.0243
R_{sigma}	0.0117	Intensity transformed	0
Omitted reflections	0	Omitted by user (OMIT hkl)	0
Multiplicity	(450, 1379, 2116, 2448, 2127, 1768, 1539, 1052, 477, 252, 174, 37, 12, 3)	Maximum multiplicity	19
Removed systematic absences	0	Filtered off (Shel/OMIT)	0

Table S23: Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **2018ncs0571**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
N(1)	3388.7(8)	5349.8(6)	2827.2(5)	10.03(15)
N(2)	11606.8(8)	9414.3(6)	2115.8(5)	10.11(15)
C(1)	3273.5(10)	4236.7(8)	2228.0(6)	13.93(18)
C(2)	3231.5(11)	3360.6(8)	2923.3(7)	16.35(19)
C(3)	4445.6(12)	3892.6(8)	3800.9(7)	18.1(2)
C(4)	4200.2(10)	5138.9(7)	3842.5(6)	11.58(17)
C(5)	4224.3(10)	6309.5(8)	2479.8(7)	13.81(18)
C(6)	5856.2(10)	6135.1(8)	2442.6(7)	14.73(18)
C(7)	6728.6(10)	7283.9(8)	2427.3(7)	15.15(19)
C(8)	8389.6(10)	7208.1(8)	2405.8(7)	14.52(18)
C(9)	9187.3(10)	8384.2(8)	2387.8(7)	14.29(18)
C(10)	10811.5(10)	8315.1(7)	2292.8(6)	12.28(17)
C(11)	11616.3(11)	10343.1(8)	2886.1(7)	16.81(19)
C(12)	10333.6(12)	11026.9(8)	2450.1(8)	23.8(2)
C(13)	10437.3(12)	11017.5(8)	1418.9(8)	23.9(2)
C(14)	10777.5(10)	9822.0(8)	1173.9(6)	13.95(18)
C(15)	1804.9(10)	5645.9(8)	2810.4(7)	13.96(18)
C(16)	13219.6(10)	9225.8(8)	2105.4(7)	14.32(18)
O(1)	10409.8(7)	8594.8(5)	5083.1(5)	11.05(13)
O(2)	8109.9(7)	7358.3(5)	4867.2(5)	12.67(14)
O(3)	10474.8(7)	6551.5(5)	5017.1(4)	9.96(13)
O(4)	12212.9(7)	7751.0(5)	6262.6(4)	10.35(13)
O(5)	14489.2(7)	8632.9(5)	5956.4(5)	11.74(13)
O(6)	12471.2(7)	7757.3(5)	4652.8(4)	10.52(13)
O(7)	7963.7(7)	9292.5(5)	4779.9(5)	12.37(14)
O(8)	8087.8(7)	5404.3(5)	4773.8(5)	13.51(14)
O(9)	14370.0(7)	8451.1(6)	7527.1(5)	15.40(14)
O(10)	14760.8(7)	8479.0(6)	4361.4(5)	14.30(14)
B(1)	11402.3(11)	7657.7(8)	5247.5(7)	9.10(18)
B(2)	8863.8(11)	8424.4(8)	4915.8(7)	9.73(18)
B(3)	8929.9(11)	6426.9(8)	4895.2(7)	9.94(18)
B(4)	13658.1(11)	8278.6(8)	6585.3(7)	10.74(19)
B(5)	13901.9(11)	8295.9(8)	4993.0(7)	10.26(18)
O(11)	4595.0(7)	6387.6(5)	-159.4(5)	10.75(13)
O(12)	6881.2(7)	7618.6(5)	4.7(5)	12.45(14)
O(13)	4523.4(7)	8429.3(5)	-98.4(4)	10.03(13)
O(14)	2853.3(7)	7223.3(5)	-1376.2(4)	10.57(13)
O(15)	569.8(7)	6289.0(5)	-1119.9(4)	11.49(13)
O(16)	2487.9(7)	7214.1(5)	211.2(4)	10.46(13)
O(17)	7019.9(7)	5672.4(5)	60.5(5)	12.04(13)
O(18)	6913.9(7)	9576.7(5)	191.7(5)	13.99(14)
O(19)	806.2(7)	6415.2(6)	-2674.4(5)	14.14(14)
O(20)	221.6(7)	6382.7(6)	457.3(5)	13.75(14)
B(11)	3604.1(11)	7323.1(8)	-349.7(7)	9.29(18)
B(12)	6132.2(11)	6550.5(8)	-36.2(7)	9.68(18)
B(13)	6067.7(11)	8551.1(8)	23.1(7)	10.17(18)
B(14)	1441.2(11)	6646.7(8)	-1727.1(7)	10.46(18)
B(15)	1093.7(11)	6625.1(8)	-154.4(7)	9.94(18)

Table S24: Anisotropic Displacement Parameters (×10⁴) **2018ncs0571**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11}+ ... +2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	8.6(3)	10.9(3)	9.8(3)	0.9(3)	1.3(3)	-0.8(3)
N(2)	9.4(3)	10.4(3)	9.8(3)	0.9(3)	2.0(3)	-1.1(3)
C(1)	13.4(4)	13.4(4)	13.2(4)	-3.3(3)	2.1(3)	-1.0(3)
C(2)	14.7(4)	11.3(4)	21.3(5)	0.9(3)	2.3(4)	-1.1(3)
C(3)	19.6(5)	13.9(4)	17.9(5)	3.4(3)	-1.5(4)	2.3(4)
C(4)	11.6(4)	13.6(4)	8.5(4)	1.7(3)	0.9(3)	-0.3(3)
C(5)	12.4(4)	13.5(4)	15.9(4)	6.3(3)	3.7(3)	-1.8(3)
C(6)	12.3(4)	16.4(4)	15.8(4)	2.7(3)	4.8(3)	-1.4(3)
C(7)	13.2(4)	16.9(4)	14.8(4)	3.1(3)	3.8(3)	-3.5(3)
C(8)	13.7(4)	15.7(4)	13.7(4)	2.3(3)	3.9(3)	-3.1(3)
C(9)	12.8(4)	15.4(4)	14.8(4)	2.1(3)	4.9(3)	-2.7(3)
C(10)	12.8(4)	10.4(4)	13.1(4)	2.4(3)	3.0(3)	-2.2(3)
C(11)	19.2(5)	13.7(4)	16.0(4)	-5.5(3)	5.1(4)	-2.8(3)
C(12)	17.7(5)	12.4(4)	41.0(6)	-4.3(4)	9.2(4)	-0.1(4)
C(13)	19.4(5)	13.3(5)	34.2(6)	6.8(4)	-3.4(4)	0.8(4)
C(14)	12.1(4)	16.7(4)	12.3(4)	6.1(3)	0.7(3)	0.2(3)
C(15)	8.1(4)	14.2(4)	19.2(4)	2.4(3)	2.4(3)	1.1(3)
C(16)	8.9(4)	16.0(4)	17.6(4)	2.4(3)	2.6(3)	0.4(3)
O(1)	7.7(3)	8.5(3)	16.1(3)	2.1(2)	1.4(2)	0.5(2)
O(2)	7.9(3)	10.2(3)	20.2(3)	2.4(2)	3.9(2)	0.4(2)
O(3)	7.4(3)	8.2(3)	13.8(3)	1.2(2)	1.8(2)	0.0(2)
O(4)	7.7(3)	12.8(3)	9.9(3)	2.2(2)	1.3(2)	-1.0(2)
O(5)	9.0(3)	13.7(3)	11.4(3)	1.4(2)	1.6(2)	-2.5(2)
O(6)	8.3(3)	12.9(3)	9.9(3)	1.0(2)	2.1(2)	-0.7(2)
O(7)	8.6(3)	9.5(3)	18.9(3)	1.9(2)	3.1(2)	1.1(2)
O(8)	8.1(3)	9.4(3)	22.5(3)	2.2(2)	3.0(3)	0.3(2)
O(9)	12.0(3)	20.9(3)	11.3(3)	1.6(3)	1.7(2)	-6.0(3)
O(10)	9.1(3)	20.5(3)	12.6(3)	1.6(3)	2.8(2)	-2.6(2)
B(1)	6.3(4)	9.1(4)	11.4(4)	1.4(3)	1.6(3)	-0.1(3)
B(2)	8.8(4)	11.1(4)	9.2(4)	1.4(3)	2.0(3)	0.7(3)
B(3)	8.6(4)	11.2(4)	9.6(4)	1.4(3)	1.7(3)	0.3(3)
B(4)	9.6(4)	9.3(4)	13.0(5)	1.2(3)	2.6(4)	-0.2(3)
B(5)	8.7(4)	9.1(4)	13.1(5)	2.8(3)	2.2(3)	1.6(3)
O(11)	7.5(3)	8.4(3)	15.5(3)	1.8(2)	1.4(2)	0.2(2)
O(12)	8.2(3)	10.2(3)	19.5(3)	2.9(2)	4.2(2)	0.1(2)
O(13)	7.0(3)	8.3(3)	14.0(3)	1.2(2)	1.6(2)	-0.3(2)
O(14)	7.8(3)	13.3(3)	9.8(3)	2.0(2)	1.4(2)	-1.8(2)
O(15)	8.8(3)	13.4(3)	11.1(3)	1.2(2)	1.6(2)	-2.8(2)
O(16)	8.2(3)	12.6(3)	10.0(3)	0.3(2)	2.2(2)	-1.4(2)
O(17)	8.6(3)	9.7(3)	18.0(3)	2.3(2)	3.4(2)	0.5(2)
O(18)	8.3(3)	9.7(3)	23.4(4)	1.8(3)	3.2(3)	-0.2(2)
O(19)	12.0(3)	17.8(3)	10.9(3)	1.8(2)	1.7(2)	-5.0(2)
O(20)	9.2(3)	18.5(3)	12.5(3)	-0.3(2)	3.1(2)	-3.5(2)
B(11)	6.4(4)	9.2(4)	11.3(4)	1.0(3)	0.9(3)	-0.3(3)
B(12)	8.8(4)	11.3(4)	8.8(4)	1.3(3)	2.1(3)	0.5(3)
B(13)	8.6(4)	11.3(4)	10.3(4)	2.2(3)	1.8(3)	-0.2(3)
B(14)	8.8(4)	9.3(4)	12.8(5)	1.5(3)	1.8(3)	0.5(3)
B(15)	9.0(4)	8.5(4)	12.0(4)	1.5(3)	1.7(3)	1.6(3)

Table S25: Bond Lengths in Å for **2018ncs0571**.

Atom	Atom	Length/Å		Atom	Atom	Length/Å
N(1)	C(1)	1.5172(11)		O(4)	B(4)	1.3587(11)
N(1)	C(4)	1.5326(11)		O(5)	B(4)	1.3879(11)
N(1)	C(5)	1.5056(11)		O(5)	B(5)	1.3907(12)
N(1)	C(15)	1.5004(11)		O(6)	B(1)	1.4521(11)
N(2)	C(10)	1.5124(11)		O(6)	B(5)	1.3547(11)
N(2)	C(11)	1.5174(11)		O(7)	B(2)	1.3679(11)
N(2)	C(14)	1.5304(11)		O(8)	B(3)	1.3591(11)
N(2)	C(16)	1.4995(11)		O(9)	B(4)	1.3591(12)
C(1)	C(2)	1.5134(13)		O(10)	B(5)	1.3608(12)
C(2)	C(3)	1.5245(13)		O(11)	B(11)	1.4907(11)
C(3)	C(4)	1.5218(13)		O(11)	B(12)	1.3541(11)
C(5)	C(6)	1.5227(12)		O(12)	B(12)	1.3827(11)
C(6)	C(7)	1.5279(12)		O(12)	B(13)	1.3879(12)
C(7)	C(8)	1.5218(13)		O(13)	B(11)	1.4712(11)
C(8)	C(9)	1.5285(12)		O(13)	B(13)	1.3594(11)
C(9)	C(10)	1.5188(12)		O(14)	B(11)	1.4762(11)
C(11)	C(12)	1.5147(14)		O(14)	B(14)	1.3600(11)
C(12)	C(13)	1.5287(17)		O(15)	B(14)	1.3888(11)
C(13)	C(14)	1.5206(14)		O(15)	B(15)	1.3880(11)
O(1)	B(1)	1.4900(11)		O(16)	B(11)	1.4508(11)
O(1)	B(2)	1.3555(11)		O(16)	B(15)	1.3556(11)
O(2)	B(2)	1.3808(11)		O(17)	B(12)	1.3687(12)
O(2)	B(3)	1.3858(11)		O(18)	B(13)	1.3606(11)
O(3)	B(1)	1.4734(11)		O(19)	B(14)	1.3579(12)
O(3)	B(3)	1.3599(11)		O(20)	B(15)	1.3588(12)
O(4)	B(1)	1.4741(11)				

Table S26: Bond Angles in ° for **2018ncs0571**.

Atom	Atom	Atom	Angle/°		Atom	Atom	Atom	Angle/°
C(1)	N(1)	C(4)	105.79(7)		B(2)	O(2)	B(3)	119.38(7)
C(5)	N(1)	C(1)	112.94(7)		B(3)	O(3)	B(1)	123.01(7)
C(5)	N(1)	C(4)	111.72(7)		B(4)	O(4)	B(1)	121.46(7)
C(15)	N(1)	C(1)	109.06(7)		B(4)	O(5)	B(5)	118.42(7)
C(15)	N(1)	C(4)	108.94(7)		B(5)	O(6)	B(1)	121.50(7)
C(15)	N(1)	C(5)	108.30(7)		O(3)	B(1)	O(1)	110.86(7)
C(10)	N(2)	C(11)	110.89(7)		O(3)	B(1)	O(4)	107.70(7)
C(10)	N(2)	C(14)	111.32(7)		O(4)	B(1)	O(1)	108.63(7)
C(11)	N(2)	C(14)	106.49(7)		O(6)	B(1)	O(1)	108.10(7)
C(16)	N(2)	C(10)	108.07(7)		O(6)	B(1)	O(3)	110.27(7)
C(16)	N(2)	C(11)	109.84(7)		O(6)	B(1)	O(4)	111.29(7)
C(16)	N(2)	C(14)	110.24(7)		O(1)	B(2)	O(2)	121.58(8)
C(2)	C(1)	N(1)	103.69(7)		O(1)	B(2)	O(7)	122.12(8)
C(1)	C(2)	C(3)	102.04(7)		O(7)	B(2)	O(2)	116.29(8)
C(4)	C(3)	C(2)	104.04(7)		O(3)	B(3)	O(2)	120.99(8)
C(3)	C(4)	N(1)	105.82(7)		O(8)	B(3)	O(2)	115.74(8)
N(1)	C(5)	C(6)	114.94(7)		O(8)	B(3)	O(3)	123.26(8)
C(5)	C(6)	C(7)	108.77(8)		O(4)	B(4)	O(5)	120.85(8)
C(8)	C(7)	C(6)	113.12(8)		O(4)	B(4)	O(9)	121.99(8)
C(7)	C(8)	C(9)	110.25(7)		O(9)	B(4)	O(5)	117.13(8)
C(10)	C(9)	C(8)	110.90(8)		O(6)	B(5)	O(5)	121.38(8)
N(2)	C(10)	C(9)	114.62(7)		O(6)	B(5)	O(10)	117.71(8)
C(12)	C(11)	N(2)	104.98(8)		O(10)	B(5)	O(5)	120.89(8)
C(11)	C(12)	C(13)	102.65(8)		B(12)	O(11)	B(11)	122.18(7)
C(14)	C(13)	C(12)	103.89(8)		B(12)	O(12)	B(13)	119.19(7)
C(13)	C(14)	N(2)	105.74(8)		B(13)	O(13)	B(11)	122.41(7)
B(2)	O(1)	B(1)	122.54(7)		B(14)	O(14)	B(11)	120.92(7)

Atom	Atom	Atom	Angle/°
B(15)	O(15)	B(14)	118.91(7)
B(15)	O(16)	B(11)	121.06(7)
O(13)	B(11)	O(11)	110.87(7)
O(13)	B(11)	O(14)	108.08(7)
O(14)	B(11)	O(11)	108.34(7)
O(16)	B(11)	O(11)	107.80(7)
O(16)	B(11)	O(13)	110.36(7)
O(16)	B(11)	O(14)	111.38(7)
O(11)	B(12)	O(12)	121.32(8)
O(11)	B(12)	O(17)	121.88(8)

Atom	Atom	Atom	Angle/°
O(17)	B(12)	O(12)	116.80(8)
O(13)	B(13)	O(12)	121.21(8)
O(13)	B(13)	O(18)	122.83(8)
O(18)	B(13)	O(12)	115.94(8)
O(14)	B(14)	O(15)	120.69(8)
O(19)	B(14)	O(14)	122.23(8)
O(19)	B(14)	O(15)	117.07(8)
O(16)	B(15)	O(15)	121.24(8)
O(16)	B(15)	O(20)	117.60(8)
O(20)	B(15)	O(15)	121.16(8)

Table S27: Torsion Angles in ° for **2018ncs0571**.

Atom	Atom	Atom	Atom	Angle/°
N(1)	C(1)	C(2)	C(3)	-42.17(9)
N(1)	C(5)	C(6)	C(7)	-160.17(7)
N(2)	C(11)	C(12)	C(13)	-37.23(9)
C(1)	N(1)	C(4)	C(3)	-3.01(9)
C(1)	N(1)	C(5)	C(6)	-58.44(10)
C(1)	C(2)	C(3)	C(4)	40.26(9)
C(2)	C(3)	C(4)	N(1)	-22.98(9)
C(4)	N(1)	C(1)	C(2)	28.06(8)
C(4)	N(1)	C(5)	C(6)	60.68(10)
C(5)	N(1)	C(1)	C(2)	150.56(7)
C(5)	N(1)	C(4)	C(3)	-126.28(8)
C(5)	C(6)	C(7)	C(8)	179.08(8)
C(6)	C(7)	C(8)	C(9)	179.80(7)
C(7)	C(8)	C(9)	C(10)	-175.81(7)
C(8)	C(9)	C(10)	N(2)	170.96(7)
C(10)	N(2)	C(11)	C(12)	-100.65(8)
C(10)	N(2)	C(14)	C(13)	125.22(8)
C(11)	N(2)	C(10)	C(9)	55.79(10)
C(11)	N(2)	C(14)	C(13)	4.24(9)
C(11)	C(12)	C(13)	C(14)	39.79(9)
C(12)	C(13)	C(14)	N(2)	-27.17(10)
C(14)	N(2)	C(10)	C(9)	-62.57(9)
C(14)	N(2)	C(11)	C(12)	20.60(9)
C(15)	N(1)	C(1)	C(2)	-88.98(8)
C(15)	N(1)	C(4)	C(3)	114.11(8)
C(15)	N(1)	C(5)	C(6)	-179.33(7)
C(16)	N(2)	C(10)	C(9)	176.23(7)
C(16)	N(2)	C(11)	C(12)	139.96(8)
C(16)	N(2)	C(14)	C(13)	-114.86(8)
B(1)	O(1)	B(2)	O(2)	-1.44(13)
B(1)	O(1)	B(2)	O(7)	178.99(8)
B(1)	O(3)	B(3)	O(2)	8.01(13)
B(1)	O(3)	B(3)	O(8)	-173.43(8)
B(1)	O(4)	B(4)	O(5)	-7.59(13)
B(1)	O(4)	B(4)	O(9)	174.53(8)
B(1)	O(6)	B(5)	O(5)	9.46(12)
B(1)	O(6)	B(5)	O(10)	-171.74(8)
B(2)	O(1)	B(1)	O(3)	11.13(11)
B(2)	O(1)	B(1)	O(4)	-107.02(9)
B(2)	O(1)	B(1)	O(6)	132.10(8)
B(2)	O(2)	B(3)	O(3)	3.30(13)
B(2)	O(2)	B(3)	O(8)	-175.36(8)
B(3)	O(2)	B(2)	O(1)	-6.52(13)
B(3)	O(2)	B(2)	O(7)	173.06(8)

Atom	Atom	Atom	Atom	Angle/°
B(3)	O(3)	B(1)	O(1)	-14.40(11)
B(3)	O(3)	B(1)	O(4)	104.31(9)
B(3)	O(3)	B(1)	O(6)	-134.07(8)
B(4)	O(4)	B(1)	O(1)	-95.95(9)
B(4)	O(4)	B(1)	O(3)	143.91(8)
B(4)	O(4)	B(1)	O(6)	22.94(11)
B(4)	O(5)	B(5)	O(6)	8.25(12)
B(4)	O(5)	B(5)	O(10)	-170.52(8)
B(5)	O(5)	B(4)	O(4)	-9.04(12)
B(5)	O(5)	B(4)	O(9)	168.94(8)
B(5)	O(6)	B(1)	O(1)	95.41(9)
B(5)	O(6)	B(1)	O(3)	-143.26(8)
B(5)	O(6)	B(1)	O(4)	-23.80(11)
B(11)	O(11)	B(12)	O(12)	5.94(13)
B(11)	O(11)	B(12)	O(17)	-174.53(8)
B(11)	O(13)	B(13)	O(12)	-6.24(13)
B(11)	O(13)	B(13)	O(18)	175.40(8)
B(11)	O(14)	B(14)	O(15)	10.10(12)
B(11)	O(14)	B(14)	O(19)	-171.29(8)
B(11)	O(16)	B(15)	O(15)	-12.34(12)
B(11)	O(16)	B(15)	O(20)	168.16(8)
B(12)	O(11)	B(11)	O(13)	-17.11(11)
B(12)	O(11)	B(11)	O(14)	101.34(9)
B(12)	O(11)	B(11)	O(16)	-138.01(8)
B(12)	O(12)	B(13)	O(13)	-6.99(13)
B(12)	O(12)	B(13)	O(18)	171.48(8)
B(13)	O(12)	B(12)	O(11)	7.06(13)
B(13)	O(12)	B(12)	O(17)	-172.49(8)
B(13)	O(13)	B(11)	O(11)	17.22(11)
B(13)	O(13)	B(11)	O(14)	-101.39(9)
B(13)	O(13)	B(11)	O(16)	136.60(8)
B(14)	O(14)	B(11)	O(11)	93.12(9)
B(14)	O(14)	B(11)	O(13)	-146.67(7)
B(14)	O(14)	B(11)	O(16)	-25.29(11)
B(14)	O(15)	B(15)	O(16)	-5.41(12)
B(14)	O(15)	B(15)	O(20)	174.07(8)
B(15)	O(15)	B(14)	O(14)	6.37(12)
B(15)	O(15)	B(14)	O(19)	-172.30(8)
B(15)	O(16)	B(11)	O(11)	-92.40(9)
B(15)	O(16)	B(11)	O(13)	146.38(8)
B(15)	O(16)	B(11)	O(14)	26.33(11)

Table S28: Hydrogen Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **2018ncs0571**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H(B)	2329.83	4134.62	1702.75	17
H(C)	4172.46	4194.86	1958.26	17
H(R)	3501.18	2627.49	2684.23	20
H(S)	2210.83	3248.22	3056.35	20
H(1AA)	4298.32	3562.31	4379.61	22
H(2AA)	5487.8	3783.44	3734.76	22
H	5193.58	5615.11	4060.23	14
H(A)	3559.11	5314.53	4284.5	14
H(V)	4253.46	7005.04	2900.41	17
H(W)	3632.94	6432.18	1834.75	17
H(P)	5843.05	5639.01	1865.44	18
H(Q)	6367.82	5768.78	3005.38	18
H(D)	6203.62	7638.93	1861.24	18
H(E)	6702.35	7777.89	2997.54	18
H(F)	8428.47	6719.9	1835.23	17
H(G)	8929.25	6863.93	2974.18	17
H(X)	8600.3	8750.02	1846.78	17
H(Y)	9213.02	8854.18	2980.83	17
H(T)	11426.29	8053.59	2881.94	15
H(U)	10785.29	7740.05	1762.54	15
H(0AA)	11427.5	10019.08	3458.06	20
H(Z)	12611.98	10819.09	3066.81	20
H(5AA)	10499.78	11806.47	2765.67	29
H(6AA)	9327.48	10667.62	2489.56	29
H(3AA)	9459.21	11183.47	999.33	29
H(4AA)	11269.96	11579.05	1359.41	29
H(N)	11435.02	9818.47	722.23	17
H(O)	9815.84	9328.94	883.67	17
H(K)	1216.77	5006.4	2987.64	21
H(L)	1294.61	5816.61	2169.76	21
H(M)	1869.01	6307.96	3262.78	21
H(H)	13206.41	8615.76	1614.82	21
H(I)	13760.45	9017.59	2727.36	21
H(J)	13745.89	9920.89	1967.37	21
H(7)	8518.04	9902.49	4812.83	19
H(8)	8675.79	4891.71	4835.7	20
H(9)	13821.92	8126.51	7838.22	23
H(10)	15636.09	8784.95	4653.04	21
H(17)	6471.88	5070.98	68.81	18
H(18)	6328.53	10089.74	163.9	21
H(19)	1337.03	6775.14	-2976.28	21
H(20)	-659.55	6107.35	148.31	21

Table S29: Hydrogen Bond information for **2018ncs0571**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
O(7)	H(7)	O(1)[1]	0.84	1.94	2.7788(9)	173.0
O(8)	H(8)	O(3)[2]	0.84	1.95	2.7678(9)	164.2
O(9)	H(9)	O(14)[3]	0.84	1.94	2.7691(9)	170.0
O(10)	H(10)	O(7)[4]	0.84	2.10	2.8800(9)	153.9
O(17)	H(17)	O(11)[5]	0.84	1.95	2.7841(9)	173.8
O(18)	H(18)	O(13)[6]	0.84	1.99	2.8040(9)	163.9
O(19)	H(19)	O(4)[7]	0.84	1.92	2.7479(9)	169.0
O(20)	H(20)	O(17)[8]	0.84	2.09	2.8544(9)	150.8

––––

[1]2-x,2-y,1-z; [2]2-x,1-y,1-z; [3]1+x,+y,1+z; [4]1+x,+y,+z; [5]1-x,1-y,-z; [6]1-x,2-y,-z; [7]-1+x,+y,-1+z; [8]-1+x,+y,+z
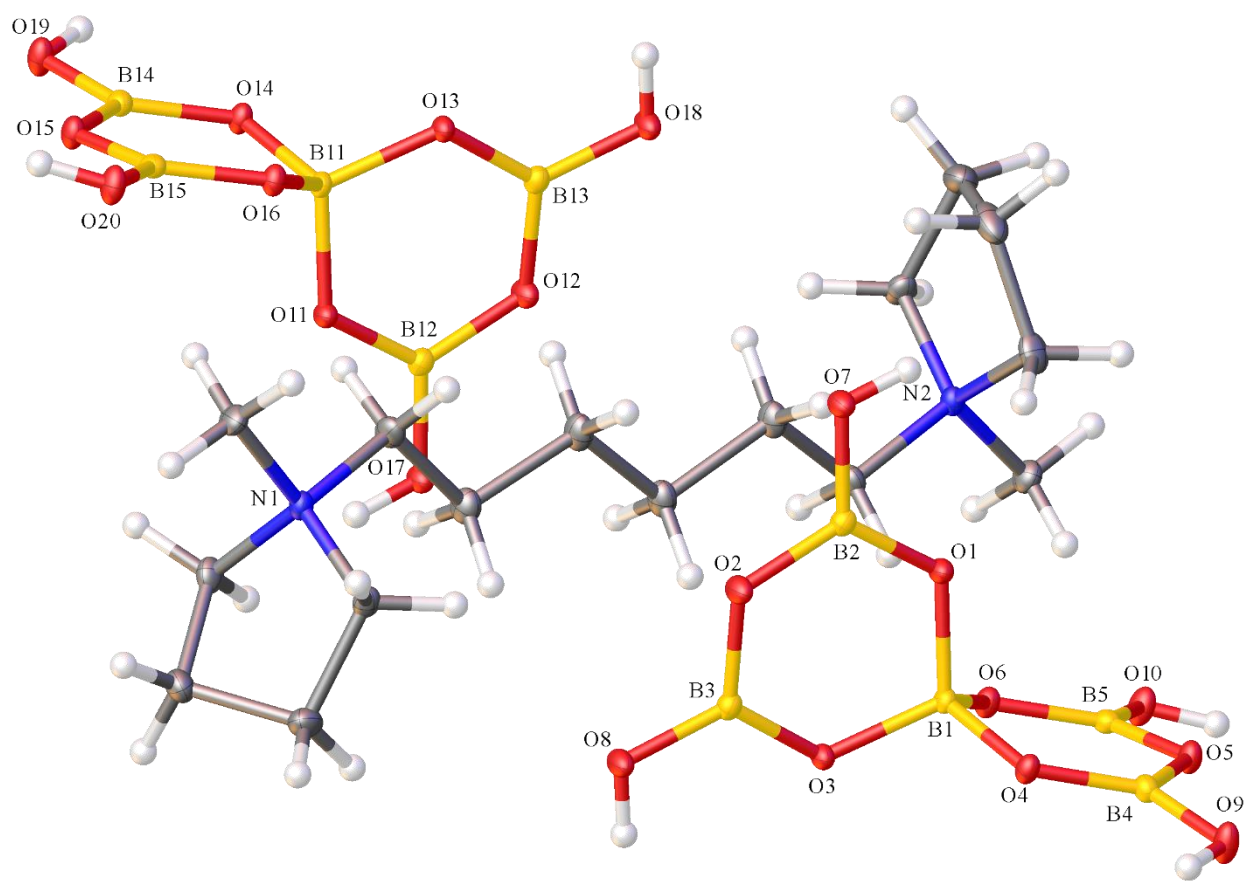
Citations for 4a

CrysAlisPro Software System, Rigaku Oxford Diffraction, (2018).

O.V. Dolomanov and L.J. Bourhis and R.J. Gildea and J.A.K. Howard and H. Puschmann, Olex2: A complete structure solution, refinement and analysis program, *J. Appl. Cryst.*, (2009), **42**, 339-341.

Sheldrick, G.M., Crystal structure refinement with ShelXL, *Acta Cryst.*, (2015), **C27**, 3-8.

Sheldrick, G.M., ShelXT-Integrated space-group and crystal-structure determination, *Acta Cryst.*, (2015), **A71**, 3-8.

Submitted by: **Mike A Beckett / Thomas A. Rixon**

Bangor University

Solved by: **Peter N Horton**

Sample ID: **MAB/TR/AR101 B5 (plate)**
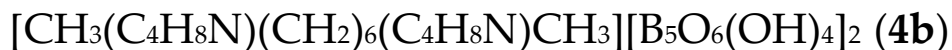
$[CH_3(C_4H_8N)(CH_2)_6(C_4H_8N)CH_3][B_5O_6(OH)_4]_2$ (**4b**)

Crystal Data and Experimental
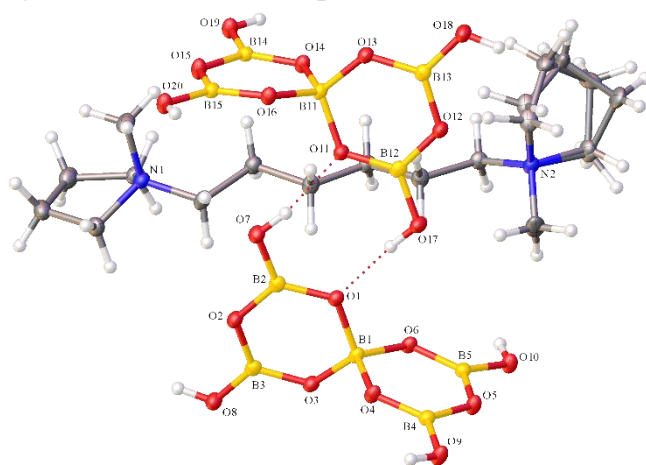
Experimental. Single colourless plate-shaped crystals of **2018ncs0571p** were recrystallised from water. A suitable crystal 0.260×0.120×0.015 mm³ was selected and mounted on a MITIGEN holder in perfluoroether oil on a Rigaku FRE+ diffractometer equipped with VHF Varimax confocal mirrors and an AFC12 goniometer and HyPix 6000 detector. The crystal was kept at a steady $T = 100(2)$ K during data collection. The structure was solved with the **ShelXT** (Sheldrick, 2015) structure solution program using the Intrinsic Phasing solution method and by using **Olex2** (Dolomanov et al., 2009) as the graphical interface. The model was refined with version 2018/3 of **ShelXL** (Sheldrick, 2015) using Least Squares minimisation.

Crystal Data. $C_{16}H_{42}B_{10}N_2O_{20}$, $M_r = 690.61$, triclinic, $P\text{-}1$ (No. 2), a = 9.6641(4) Å, b = 12.7484(4) Å, c = 13.0913(4) Å, $\alpha = 84.415(3)°$, $\beta = 84.541(3)°$, $\gamma = 75.872(3)°$, $V = 1552.40(10)$ Å³, $T = 100(2)$ K, $Z = 2$, $Z' = 1$, $\mu(MoK_\alpha) = 0.124$ mm⁻¹, 35495 reflections measured, 7118 unique ($R_{int} = 0.0398$) which were used in all calculations. The final wR_2 was 0.0929 (all data) and R_1 was 0.0358 (I > 2(I)).

Compound	2018ncs0571p
Formula	$C_{16}H_{42}B_{10}N_2O_{20}$
$D_{calc.}$/ g cm⁻³	1.477
μ/mm⁻¹	0.124
Formula Weight	690.61
Colour	colourless
Shape	plate
Size/mm³	0.260×0.120×0.015
T/K	100(2)
Crystal System	triclinic
Space Group	$P\text{-}1$
a/Å	9.6641(4)
b/Å	12.7484(4)
c/Å	13.0913(4)
α/°	84.415(3)
β/°	84.541(3)
γ/°	75.872(3)
V/Å³	1552.40(10)
Z	2
Z'	1
Wavelength/Å	0.71075
Radiation type	MoK$_\alpha$
Θ_{min}/°	1.652
Θ_{max}/°	27.480
Measured Refl.	35495
Independent Refl.	7118
Reflections with I > 2(I)	6044
R_{int}	0.0398
Parameters	484
Restraints	94
Largest Peak	0.317
Deepest Hole	-0.223
GooF	1.018
wR_2 (all data)	0.0929
wR_2	0.0888
R_1 (all data)	0.0444
R_1	0.0358

Structure Quality Indicators

Reflections:

d min (Mo)	I/σ	Rint	complete 100% (IUCr)
0.77	34.2	3.98%	100%

Refinement:

Shift	Max Peak	Min Peak	GooF
0.001	0.3	-0.2	1.018

A colourless plate-shaped crystal with dimensions 0.260×0.120×0.015 mm^3 was mounted on a MITIGEN holder in perfluoroether oil. Data were collected using a Rigaku FRE+ diffractometer equipped with VHF Varimax confocal mirrors and an AFC12 goniometer and HyPix 6000 detector, equipped with an Oxford Cryosystems low-temperature device operating at T = 100(2) K.

Data were measured using profile data from ω-scans 0.5° per frame for 2.5 s using MoK$_\alpha$ radiation. The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.39.46, 2018). The maximum resolution that was achieved was Θ = 27.480° (0.77 Å).

The diffraction pattern was indexed. The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.39.46, 2018) and the unit cell was refined using **CrysAlisPro** (Rigaku, V1.171.39.46, 2018) on 11779 reflections, 33% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using **CrysAlisPro** (Rigaku, V1.171.39.46, 2018). The final completeness is 100.00 % out to 27.480° in Θ. A multi-scan absorption correction was performed using CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018) using spherical harmonics as implemented in SCALE3 ABSPACK. The absorption coefficient μ of this material is 0.124 mm^{-1} at this wavelength (λ = 0.71075 Å) and the minimum and maximum transmissions are 0.673 and 1.000.

The structure was solved and the space group P-1 (# 2) determined by the **ShelXT** (Sheldrick, 2015) structure solution program using Intrinsic Phasing and refined by Least Squares using version 2018/3 of **ShelXL** (Sheldrick, 2015). All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model. Most hydrogen atom positions were calculated geometrically and refined using the riding model, but some hydrogen atoms were refined freely.

_refine_special_details_: One of the 5-membered ammonium rings is disordered over 2 positions. As such various geometrical (SADI, BUMP) and displacement (RIGU) restraints have been employed. Also due to their overlap there were two uses of EADP constraints

_exptl_absorpt_process_details_: CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018) using spherical harmonicsas implemented in SCALE3 ABSPACK.

There is a single molecule in the asymmetric unit, which is represented by the reported sum formula. In other words: Z is 2 and Z' is 1.
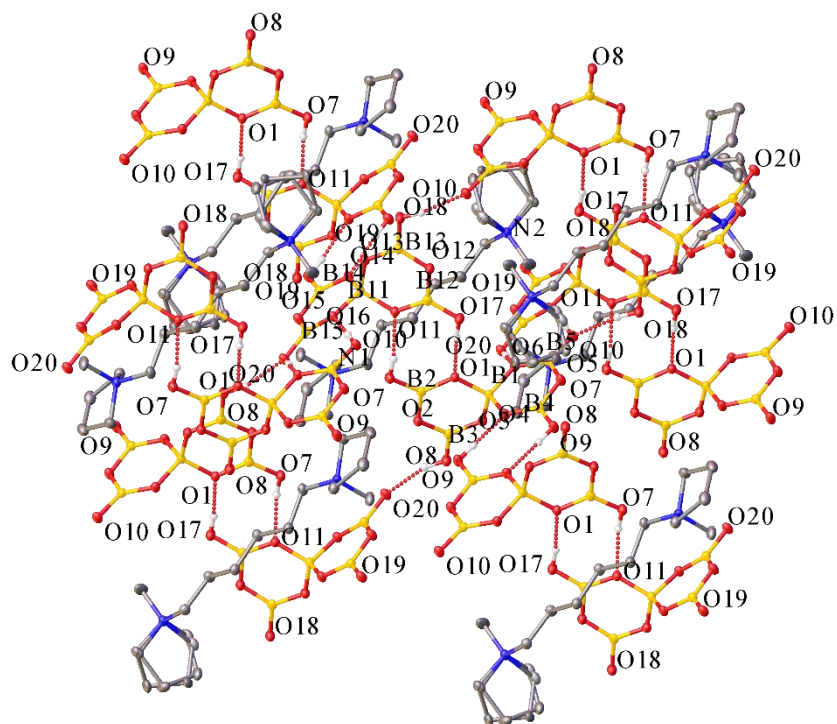
Figure S36: The following hydrogen bonding interactions with a maximum D-D distance of 2.94 Å and a minimum angle of 120 ° are present in **2018ncs0571p**: O7–O11: 2.751 Å, O8–O20_*1: 2.817 Å, O9–O4*_2: 2.703 Å, O10–O16_*3: 2.734 Å, O17–O1: 2.789 Å, O18–O10*_6: 2.872 Å, O19–O14_*4: 2.736 Å, O20–O6*_5: 2.791 Å.
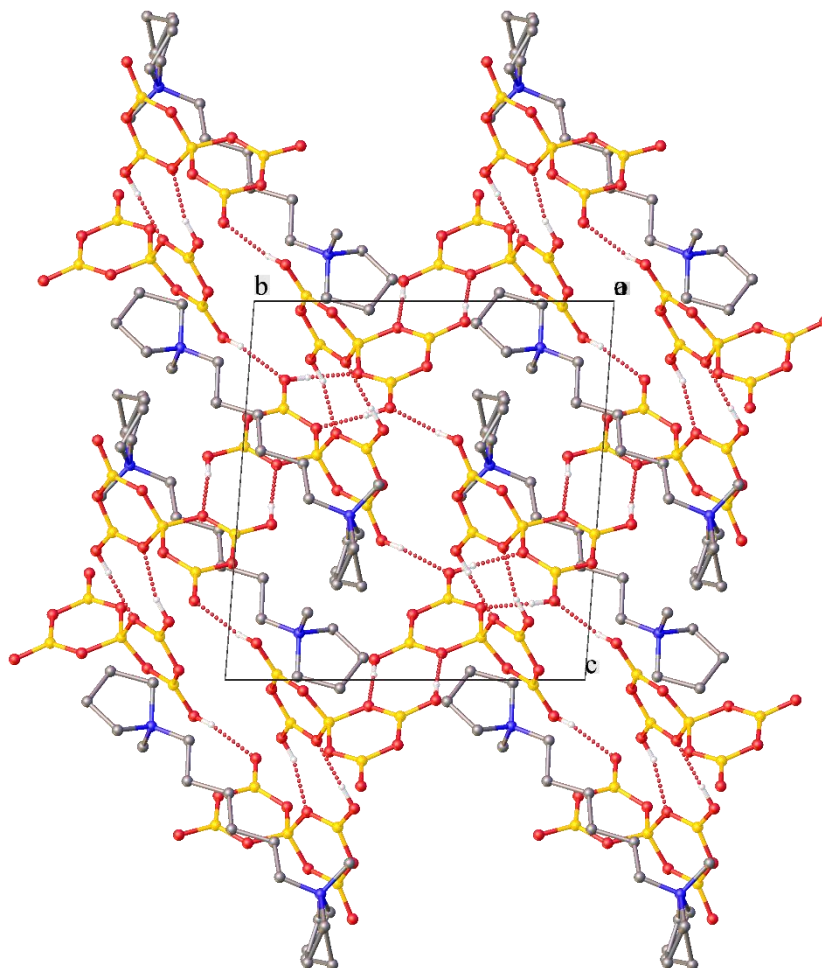
Figure S37: Packing diagram of 2018ncs0571p.

Reflection Statistics

Total reflections (after filtering)	35495	Unique reflections	7118
Completeness	1.0	Mean I/σ	20.28
hkl$_{max}$ collected	(12, 16, 16)	hkl$_{min}$ collected	(-12, -16, -16)
hkl$_{max}$ used	(12, 16, 16)	hkl$_{min}$ used	(-12, -16, 0)
Lim d$_{max}$ collected	100.0	Lim d$_{min}$ collected	0.36
d$_{max}$ used	12.33	d$_{min}$ used	0.77
Friedel pairs	6236	Friedel pairs merged	1
Inconsistent equivalents	1	R$_{int}$	0.0398
R$_{sigma}$	0.0292	Intensity transformed	0
Omitted reflections	0	Omitted by user (OMIT hkl)	0
Multiplicity	(2756, 3910, 3288, 2153, 1048, 191, 7, 1)	Maximum multiplicity	10
Removed systematic absences	0	Filtered off (Shel/OMIT)	0

Table S30: Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for **2018ncs0571p**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
N(1)	5887.9(11)	11951.6(8)	1230.6(8)	16.2(2)
N(2)	10427.5(11)	6893.5(8)	5661.7(8)	15.3(2)
C(1)	6551.1(13)	12882.1(10)	1335.8(10)	18.7(2)
C(2)	5809.2(14)	13785.8(11)	578.6(11)	24.9(3)
C(3)	5253.9(14)	13204.9(12)	-225.1(10)	25.2(3)
C(4)	5780.8(14)	12000.3(11)	82.8(10)	23.5(3)
C(5)	6795.8(13)	10876.0(10)	1607.1(10)	18.2(2)
C(6)	7290.8(13)	10834.1(10)	2679.5(9)	17.8(2)
C(7)	8005.6(13)	9658.1(10)	3002.6(10)	18.3(2)
C(8)	8839.5(13)	9501.1(10)	3954.0(9)	16.6(2)
C(9)	9283.5(13)	8294.8(10)	4287.6(9)	18.3(2)
C(10)	10164.7(13)	8060.5(9)	5217.3(9)	15.7(2)
C(15)	4424.9(13)	12145.0(11)	1769.3(11)	24.0(3)
C(16)	11129.3(17)	6153.2(11)	4852.0(11)	30.2(3)
C(11)	11350(2)	6735.5(17)	6562.8(16)	17.1(3)
C(12)	10308.3(18)	7105.0(18)	7482.9(11)	23.2(5)
C(13)	8815(2)	7139.7(19)	7150.4(17)	20.9(4)
C(14)	9089.7(18)	6598.7(17)	6148.0(15)	20.7(4)
C(11A)	11466(19)	6709(13)	6520(12)	17.1(3)
C(12A)	10679(13)	6412(13)	7528(9)	25(4)
C(13A)	9228(18)	7156(16)	7388(12)	20(4)
C(14A)	8992(15)	6886(17)	6338(13)	20.7(4)
O(1)	8784.9(8)	7326.7(7)	1608.7(6)	14.05(17)
O(2)	8035.5(9)	8688.8(7)	249.3(6)	16.39(18)
O(3)	10509.2(9)	7814.4(7)	296.0(6)	15.76(18)
O(4)	10434.8(9)	5956.1(7)	700.0(6)	15.70(18)
O(5)	12356.5(9)	5067.6(7)	1720.7(7)	16.64(18)
O(6)	11293.3(9)	6949.4(7)	1901.0(6)	14.16(17)
O(7)	6349.8(9)	8308.2(7)	1533.6(7)	17.28(18)
O(8)	9802.9(9)	9209.9(7)	-963.6(7)	19.00(19)
O(9)	11625.2(9)	4102.9(7)	538.5(7)	18.19(18)
O(10)	13305.4(9)	6000.0(7)	2811.7(7)	16.39(18)
B(1)	10249.2(14)	7025.0(10)	1120.1(10)	13.4(2)
B(2)	7734.6(14)	8086.1(11)	1143.4(10)	14.6(3)
B(3)	9445.8(14)	8570.9(11)	-136.9(10)	14.9(3)
B(4)	11438.0(14)	5056.6(11)	970.1(10)	14.0(2)
B(5)	12291.9(14)	6032.1(11)	2143.3(10)	13.4(2)
O(11)	5852.8(9)	7440.2(7)	3502.5(6)	14.25(17)
O(12)	6495.5(9)	6059.5(7)	4850.4(6)	16.64(18)
O(13)	4200.8(9)	7288.7(7)	4996.6(6)	14.89(17)
O(14)	4519.6(9)	9027.5(7)	4318.8(6)	14.62(17)
O(15)	2894.9(9)	9936.2(7)	3073.6(7)	17.01(18)
O(16)	3334.8(9)	8009.1(7)	3360.9(6)	14.22(17)
O(17)	8090.0(9)	6138.8(7)	3405.4(7)	18.42(18)
O(18)	4874.3(9)	5949.4(8)	6323.8(7)	19.48(19)
O(19)	3823.4(9)	10955.4(7)	4103.3(7)	16.84(18)
O(20)	1814.5(9)	8979.5(7)	2082.9(7)	17.04(18)
B(11)	4475.6(14)	7934.4(10)	4058.8(10)	13.4(2)
B(12)	6794.0(14)	6560.4(11)	3898.4(10)	14.5(3)
B(13)	5183.3(14)	6435.9(11)	5388.3(10)	14.4(3)
B(14)	3773.9(14)	9956.9(11)	3846.3(10)	13.6(2)
B(15)	2700.0(14)	8951.0(11)	2839.5(10)	14.0(2)

Table S31: Anisotropic Displacement Parameters (×10⁴) **2018ncs0571p**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11}+ ... +2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	13.3(5)	17.3(5)	17.2(5)	-1.8(4)	-1.2(4)	-1.7(4)
N(2)	15.3(5)	13.6(5)	17.2(5)	-3.8(4)	-3.5(4)	-1.8(4)
C(1)	17.6(6)	16.6(6)	21.6(6)	-2.0(5)	-1.0(5)	-3.8(5)
C(2)	22.4(6)	21.6(7)	27.9(7)	4.8(5)	-0.2(5)	-3.1(5)
C(3)	20.3(6)	31.7(7)	20.1(6)	5.7(5)	-1.5(5)	-2.5(5)
C(4)	22.4(6)	28.6(7)	17.8(6)	-3.7(5)	-5.8(5)	-0.7(5)
C(5)	17.1(6)	13.8(6)	23.2(6)	-3.1(5)	-5.0(5)	-0.8(5)
C(6)	17.3(6)	16.1(6)	19.7(6)	-2.7(5)	-3.1(4)	-2.0(5)
C(7)	18.8(6)	14.8(6)	22.0(6)	-2.0(5)	-5.4(5)	-3.7(5)
C(8)	16.9(6)	14.0(6)	19.1(6)	-2.6(4)	-1.7(4)	-3.5(4)
C(9)	21.2(6)	14.7(6)	19.8(6)	-3.2(5)	-5.0(5)	-4.0(5)
C(10)	17.5(6)	12.9(5)	18.1(6)	-2.6(4)	-2.2(4)	-5.3(4)
C(15)	13.7(6)	26.3(7)	28.2(7)	4.9(5)	2.0(5)	-1.7(5)
C(16)	43.7(9)	19.3(7)	22.0(7)	-10.5(5)	-8.9(6)	9.2(6)
C(11)	14.0(7)	20.7(6)	17.0(6)	-2.8(5)	-4.7(5)	-3.2(5)
C(12)	20.3(9)	34.5(12)	17.3(7)	-7.4(7)	0.8(6)	-9.9(8)
C(13)	13.2(8)	23.6(9)	24.0(10)	2.0(8)	1.0(7)	-2.8(7)
C(14)	19.6(7)	20.6(10)	26.1(9)	2.6(6)	-7.7(6)	-12.1(7)
C(11A)	14.0(7)	20.7(6)	17.0(6)	-2.8(5)	-4.7(5)	-3.2(5)
C(12A)	14(6)	31(10)	27(5)	-2(6)	-8(4)	2(6)
C(13A)	21(8)	19(7)	16(6)	-4(5)	-6(5)	6(7)
C(14A)	19.6(7)	20.6(10)	26.1(9)	2.6(6)	-7.7(6)	-12.1(7)
O(1)	13.1(4)	13.5(4)	15.7(4)	-2.8(3)	-1.6(3)	-2.5(3)
O(2)	13.6(4)	16.3(4)	18.0(4)	-1.4(3)	-2.3(3)	-0.8(3)
O(3)	12.9(4)	16.3(4)	17.3(4)	-2.3(3)	-1.1(3)	-1.6(3)
O(4)	15.6(4)	13.6(4)	18.8(4)	-6.5(3)	-6.1(3)	-1.2(3)
O(5)	17.1(4)	12.8(4)	20.9(4)	-4.0(3)	-7.7(3)	-1.6(3)
O(6)	13.8(4)	12.7(4)	16.8(4)	-5.3(3)	-3.6(3)	-2.2(3)
O(7)	13.7(4)	18.9(4)	18.7(4)	-0.5(3)	-2.2(3)	-2.9(3)
O(8)	16.2(4)	19.0(4)	19.3(4)	1.1(3)	-0.9(3)	-0.6(4)
O(9)	17.9(4)	14.2(4)	23.1(5)	-7.1(3)	-8.4(3)	-0.4(3)
O(10)	17.9(4)	13.0(4)	19.6(4)	-3.7(3)	-7.5(3)	-2.9(3)
B(1)	12.7(6)	12.0(6)	16.3(6)	-4.5(5)	-3.7(5)	-1.8(5)
B(2)	15.0(6)	13.4(6)	16.9(6)	-5.2(5)	-3.6(5)	-4.0(5)
B(3)	15.3(6)	14.2(6)	15.9(6)	-5.7(5)	-2.5(5)	-2.6(5)
B(4)	13.1(6)	14.5(6)	15.3(6)	-3.5(5)	-1.2(5)	-3.7(5)
B(5)	12.2(6)	14.0(6)	14.6(6)	-2.7(5)	-1.1(4)	-3.6(5)
O(11)	13.7(4)	13.6(4)	15.0(4)	-1.8(3)	-1.4(3)	-2.0(3)
O(12)	13.9(4)	16.7(4)	17.6(4)	-0.9(3)	-1.5(3)	-0.4(3)
O(13)	13.2(4)	14.6(4)	16.5(4)	-1.9(3)	-1.4(3)	-2.1(3)
O(14)	15.0(4)	12.5(4)	17.5(4)	-3.7(3)	-5.8(3)	-2.5(3)
O(15)	20.1(4)	12.5(4)	19.9(4)	-1.8(3)	-9.0(3)	-3.5(3)
O(16)	14.5(4)	12.5(4)	17.0(4)	-3.3(3)	-5.0(3)	-3.3(3)
O(17)	15.8(4)	17.3(4)	19.6(4)	-0.2(3)	0.5(3)	-0.1(3)
O(18)	16.1(4)	19.7(5)	19.4(4)	1.8(3)	-1.3(3)	0.7(3)
O(19)	19.8(4)	13.1(4)	19.1(4)	-2.8(3)	-7.7(3)	-3.7(3)
O(20)	20.0(4)	12.8(4)	20.0(4)	-2.4(3)	-8.8(3)	-4.0(3)
B(11)	12.4(6)	11.5(6)	16.7(6)	-2.7(5)	-3.4(5)	-2.1(5)
B(12)	14.9(6)	12.4(6)	17.9(6)	-4.8(5)	-2.9(5)	-4.6(5)
B(13)	14.0(6)	14.1(6)	17.1(6)	-4.6(5)	-4.0(5)	-4.5(5)
B(14)	12.5(6)	15.2(6)	14.1(6)	-3.0(5)	-1.2(5)	-4.2(5)
B(15)	12.8(6)	14.2(6)	15.5(6)	-2.7(5)	-1.6(5)	-3.4(5)

Table S32: Bond Lengths in Å for **2018ncs0571p**.

Atom	Atom	Length/Å
N(1)	C(1)	1.5022(16)
N(1)	C(4)	1.5102(16)
N(1)	C(5)	1.5022(15)
N(1)	C(15)	1.4936(15)
N(2)	C(10)	1.5138(15)
N(2)	C(16)	1.4899(16)
N(2)	C(11)	1.514(2)
N(2)	C(14)	1.5007(19)
N(2)	C(11A)	1.541(15)
N(2)	C(14A)	1.575(13)
C(1)	C(2)	1.5277(18)
C(2)	C(3)	1.548(2)
C(3)	C(4)	1.5221(19)
C(5)	C(6)	1.5178(17)
C(6)	C(7)	1.5264(17)
C(7)	C(8)	1.5182(17)
C(8)	C(9)	1.5239(16)
C(9)	C(10)	1.5159(16)
C(11)	C(12)	1.528(2)
C(12)	C(13)	1.535(2)
C(13)	C(14)	1.513(2)
C(11A)	C(12A)	1.521(15)
C(12A)	C(13A)	1.505(13)
C(13A)	C(14A)	1.500(13)
O(1)	B(1)	1.4729(15)
O(1)	B(2)	1.3644(16)
O(2)	B(2)	1.3836(16)
O(2)	B(3)	1.3854(16)

Atom	Atom	Length/Å
O(3)	B(1)	1.4492(15)
O(3)	B(3)	1.3521(16)
O(4)	B(1)	1.4830(15)
O(4)	B(4)	1.3504(16)
O(5)	B(4)	1.3886(15)
O(5)	B(5)	1.3822(15)
O(6)	B(1)	1.4839(15)
O(6)	B(5)	1.3544(15)
O(7)	B(2)	1.3584(16)
O(8)	B(3)	1.3596(16)
O(9)	B(4)	1.3554(15)
O(10)	B(5)	1.3650(15)
O(11)	B(11)	1.4813(15)
O(11)	B(12)	1.3537(16)
O(12)	B(12)	1.3824(16)
O(12)	B(13)	1.3903(15)
O(13)	B(11)	1.4503(15)
O(13)	B(13)	1.3515(16)
O(14)	B(11)	1.4778(15)
O(14)	B(14)	1.3532(16)
O(15)	B(14)	1.3875(15)
O(15)	B(15)	1.3815(16)
O(16)	B(11)	1.4773(15)
O(16)	B(15)	1.3581(16)
O(17)	B(12)	1.3635(16)
O(18)	B(13)	1.3589(16)
O(19)	B(14)	1.3609(16)
O(20)	B(15)	1.3615(15)

Table S33: Bond Angles in ° for **2018ncs0571p**.

Atom	Atom	Atom	Angle/°
C(1)	N(1)	C(4)	102.36(10)
C(5)	N(1)	C(1)	112.86(9)
C(5)	N(1)	C(4)	110.53(9)
C(15)	N(1)	C(1)	110.29(10)
C(15)	N(1)	C(4)	109.76(10)
C(15)	N(1)	C(5)	110.74(10)
C(10)	N(2)	C(11)	109.68(11)
C(10)	N(2)	C(11A)	109.7(6)
C(10)	N(2)	C(14A)	102.0(8)
C(16)	N(2)	C(10)	109.88(10)
C(16)	N(2)	C(11)	110.66(12)
C(16)	N(2)	C(14)	111.23(13)
C(16)	N(2)	C(11A)	106.9(6)
C(16)	N(2)	C(14A)	127.7(7)
C(14)	N(2)	C(10)	112.73(11)
C(14)	N(2)	C(11)	102.47(12)
C(11A)	N(2)	C(14A)	99.6(9)
N(1)	C(1)	C(2)	104.49(10)
C(1)	C(2)	C(3)	105.43(11)
C(4)	C(3)	C(2)	104.89(10)
N(1)	C(4)	C(3)	104.02(10)
N(1)	C(5)	C(6)	114.99(10)

Atom	Atom	Atom	Angle/°
C(5)	C(6)	C(7)	108.23(10)
C(8)	C(7)	C(6)	115.00(10)
C(7)	C(8)	C(9)	109.27(10)
C(10)	C(9)	C(8)	112.81(10)
N(2)	C(10)	C(9)	113.75(9)
N(2)	C(11)	C(12)	105.05(15)
C(11)	C(12)	C(13)	105.67(14)
C(14)	C(13)	C(12)	104.88(13)
N(2)	C(14)	C(13)	103.83(13)
C(12A)	C(11A)	N(2)	108.2(10)
C(13A)	C(12A)	C(11A)	99.7(11)
C(14A)	C(13A)	C(12A)	99.6(11)
C(13A)	C(14A)	N(2)	105.9(10)
B(2)	O(1)	B(1)	121.41(10)
B(2)	O(2)	B(3)	119.06(10)
B(3)	O(3)	B(1)	122.95(10)
B(4)	O(4)	B(1)	124.67(9)
B(5)	O(5)	B(4)	119.01(10)
B(5)	O(6)	B(1)	123.36(9)
O(1)	B(1)	O(4)	107.66(9)
O(1)	B(1)	O(6)	109.43(9)
O(3)	B(1)	O(1)	112.00(9)

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O(3)	B(1)	O(4)	109.42(9)	B(15)	O(16)	B(11)	122.87(9)
O(3)	B(1)	O(6)	108.25(9)	O(13)	B(11)	O(11)	111.82(9)
O(4)	B(1)	O(6)	110.08(9)	O(13)	B(11)	O(14)	109.17(10)
O(1)	B(2)	O(2)	121.28(11)	O(13)	B(11)	O(16)	109.44(9)
O(7)	B(2)	O(1)	122.45(11)	O(14)	B(11)	O(11)	108.72(9)
O(7)	B(2)	O(2)	116.25(11)	O(16)	B(11)	O(11)	107.32(9)
O(3)	B(3)	O(2)	120.96(11)	O(16)	B(11)	O(14)	110.35(9)
O(3)	B(3)	O(8)	117.96(11)	O(11)	B(12)	O(12)	120.95(11)
O(8)	B(3)	O(2)	121.07(11)	O(11)	B(12)	O(17)	122.61(11)
O(4)	B(4)	O(5)	120.73(11)	O(17)	B(12)	O(12)	116.42(11)
O(4)	B(4)	O(9)	123.36(11)	O(13)	B(13)	O(12)	120.93(11)
O(9)	B(4)	O(5)	115.90(11)	O(13)	B(13)	O(18)	118.97(11)
O(6)	B(5)	O(5)	121.97(11)	O(18)	B(13)	O(12)	120.09(11)
O(6)	B(5)	O(10)	122.39(11)	O(14)	B(14)	O(15)	121.11(11)
O(10)	B(5)	O(5)	115.63(11)	O(14)	B(14)	O(19)	122.59(11)
B(12)	O(11)	B(11)	122.79(10)	O(19)	B(14)	O(15)	116.30(11)
B(12)	O(12)	B(13)	119.47(10)	O(16)	B(15)	O(15)	121.23(11)
B(13)	O(13)	B(11)	123.40(9)	O(16)	B(15)	O(20)	122.14(11)
B(14)	O(14)	B(11)	123.60(9)	O(20)	B(15)	O(15)	116.61(11)
B(15)	O(15)	B(14)	119.10(10)				

Table S34: Torsion Angles in ° for **2018ncs0571p**.

Atom	Atom	Atom	Atom	Angle/°
N(1)	C(1)	C(2)	C(3)	-23.13(13)
N(1)	C(5)	C(6)	C(7)	171.89(10)
N(2)	C(11)	C(12)	C(13)	16.2(2)
N(2)	C(11A)	C(12A)	C(13A)	-38.3(13)
C(1)	N(1)	C(4)	C(3)	-42.65(12)
C(1)	N(1)	C(5)	C(6)	51.16(13)
C(1)	C(2)	C(3)	C(4)	-3.06(13)
C(2)	C(3)	C(4)	N(1)	27.99(13)
C(4)	N(1)	C(1)	C(2)	40.60(12)
C(4)	N(1)	C(5)	C(6)	165.10(11)
C(5)	N(1)	C(1)	C(2)	159.41(10)
C(5)	N(1)	C(4)	C(3)	-163.09(10)
C(5)	C(6)	C(7)	C(8)	167.57(10)
C(6)	C(7)	C(8)	C(9)	170.81(10)
C(7)	C(8)	C(9)	C(10)	178.22(10)
C(8)	C(9)	C(10)	N(2)	171.20(10)
C(10)	N(2)	C(11)	C(12)	83.52(16)
C(10)	N(2)	C(14)	C(13)	-74.84(16)
C(10)	N(2)	C(11A)	C(12A)	116.7(9)
C(10)	N(2)	C(14A)	C(13A)	-90.6(13)
C(15)	N(1)	C(1)	C(2)	-76.14(12)
C(15)	N(1)	C(4)	C(3)	74.47(13)
C(15)	N(1)	C(5)	C(6)	-73.04(13)
C(16)	N(2)	C(10)	C(9)	56.47(13)
C(16)	N(2)	C(11)	C(12)	-155.10(14)
C(16)	N(2)	C(14)	C(13)	161.23(14)
C(16)	N(2)	C(11A)	C(12A)	-124.2(9)
C(16)	N(2)	C(14A)	C(13A)	142.2(11)
C(11)	N(2)	C(10)	C(9)	178.32(12)
C(11)	N(2)	C(14)	C(13)	42.97(18)
C(11)	C(12)	C(13)	C(14)	10.0(2)
C(12)	C(13)	C(14)	N(2)	-32.86(19)
C(14)	N(2)	C(10)	C(9)	-68.20(14)

Atom	Atom	Atom	Atom	Angle/°
C(14)	N(2)	C(11)	C(12)	-36.44(19)
C(11A)	N(2)	C(10)	C(9)	173.7(7)
C(11A)	N(2)	C(14A)	C(13A)	22.0(15)
C(11A)	C(12A)	C(13A)	C(14A)	50.4(15)
C(12A)	C(13A)	C(14A)	N(2)	-46.3(17)
C(14A)	N(2)	C(10)	C(9)	-81.4(6)
C(14A)	N(2)	C(11A)	C(12A)	10.2(13)
B(1)	O(1)	B(2)	O(2)	-7.16(17)
B(1)	O(1)	B(2)	O(7)	173.99(10)
B(1)	O(3)	B(3)	O(2)	5.44(17)
B(1)	O(3)	B(3)	O(8)	-174.40(10)
B(1)	O(4)	B(4)	O(5)	2.82(18)
B(1)	O(4)	B(4)	O(9)	-176.86(11)
B(1)	O(6)	B(5)	O(5)	-4.07(17)
B(1)	O(6)	B(5)	O(10)	175.36(10)
B(2)	O(1)	B(1)	O(3)	16.46(14)
B(2)	O(1)	B(1)	O(4)	-103.88(12)
B(2)	O(1)	B(1)	O(6)	136.50(10)
B(2)	O(2)	B(3)	O(3)	5.65(17)
B(2)	O(2)	B(3)	O(8)	-174.51(11)
B(3)	O(2)	B(2)	O(1)	-4.66(17)
B(3)	O(2)	B(2)	O(7)	174.26(10)
B(3)	O(3)	B(1)	O(1)	-15.72(15)
B(3)	O(3)	B(1)	O(4)	103.58(12)
B(3)	O(3)	B(1)	O(6)	-136.46(11)
B(4)	O(4)	B(1)	O(1)	-121.03(11)
B(4)	O(4)	B(1)	O(3)	117.03(12)
B(4)	O(4)	B(1)	O(6)	-1.81(15)
B(4)	O(5)	B(5)	O(6)	4.75(17)
B(4)	O(5)	B(5)	O(10)	-174.72(10)
B(5)	O(5)	B(4)	O(4)	-4.08(17)
B(5)	O(5)	B(4)	O(9)	175.61(10)
B(5)	O(6)	B(1)	O(1)	120.51(11)
B(5)	O(6)	B(1)	O(3)	-117.17(11)
B(5)	O(6)	B(1)	O(4)	2.39(15)
B(11)	O(11)	B(12)	O(12)	1.30(17)
B(11)	O(11)	B(12)	O(17)	-176.83(10)
B(11)	O(13)	B(13)	O(12)	-6.25(17)
B(11)	O(13)	B(13)	O(18)	173.45(10)
B(11)	O(14)	B(14)	O(15)	0.38(17)
B(11)	O(14)	B(14)	O(19)	179.55(10)
B(11)	O(16)	B(15)	O(15)	-12.13(17)
B(11)	O(16)	B(15)	O(20)	169.63(11)
B(12)	O(11)	B(11)	O(13)	-7.00(15)
B(12)	O(11)	B(11)	O(14)	113.60(11)
B(12)	O(11)	B(11)	O(16)	-127.03(11)
B(12)	O(12)	B(13)	O(13)	-0.46(17)
B(12)	O(12)	B(13)	O(18)	179.84(11)
B(13)	O(12)	B(12)	O(11)	2.84(17)
B(13)	O(12)	B(12)	O(17)	-178.92(10)
B(13)	O(13)	B(11)	O(11)	9.47(15)
B(13)	O(13)	B(11)	O(14)	-110.87(12)
B(13)	O(13)	B(11)	O(16)	128.25(11)
B(14)	O(14)	B(11)	O(11)	107.59(12)
B(14)	O(14)	B(11)	O(13)	-130.18(11)
B(14)	O(14)	B(11)	O(16)	-9.86(15)
B(14)	O(15)	B(15)	O(16)	0.97(17)
B(14)	O(15)	B(15)	O(20)	179.30(10)
B(15)	O(15)	B(14)	O(14)	4.84(17)
B(15)	O(15)	B(14)	O(19)	-174.37(10)
B(15)	O(16)	B(11)	O(11)	-102.66(12)
B(15)	O(16)	B(11)	O(13)	135.80(11)

Atom	Atom	Atom	Atom	Angle/°
B(15)	O(16)	B(11)	O(14)	15.65(15)

Table S35: Hydrogen Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **2018ncs0571p**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H(1A)	7595.44	12680.26	1155.83	22
H(1B)	6378.38	13110.09	2047.59	22
H(2A)	6491.75	14202.19	243.38	30
H(2B)	5005.73	14289.35	936.09	30
H(3A)	4195.96	13414.19	-204.03	30
H(3B)	5645.68	13388.88	-928	30
H(4A)	5093.69	11589.6	-81.43	28
H(4B)	6725.13	11701.7	-274.24	28
H(5A)	7649.51	10685.74	1118.91	22
H(5B)	6245.06	10316.49	1598.63	22
H(6A)	6463.23	11105.19	3168.82	21
H(6B)	7978.03	11296.72	2679.29	21
H(7A)	7258.44	9240.52	3130.06	22
H(7B)	8664.51	9349.33	2422.92	22
H(8A)	8238.13	9894.69	4516.62	20
H(8B)	9699.47	9797.1	3800.22	20
H(9A)	8414.35	8011.9	4451.36	22
H(9B)	9846.83	7906.32	3708.16	22
H(10A)	11098.71	8240.84	5018.72	19
H(10B)	9666.67	8539.11	5757.6	19
H(15A)	3973.65	11563.2	1646.89	36
H(15B)	3845.31	12843.65	1504.61	36
H(15C)	4496	12155.11	2509.9	36
H(16A)	10471.04	6204.75	4314.27	45
H(16B)	11379.13	5405.35	5159.25	45
H(16C)	11999.48	6362.41	4549.91	45
H(11A)	12067.56	7179.23	6436.85	20
H(11B)	11854.18	5963.87	6682.05	20
H(12A)	10366.61	7831.74	7654.02	28
H(12B)	10527.3	6587.54	8093.38	28
H(13A)	8288.33	6740.73	7674	25
H(13B)	8252.33	7897.84	7048.26	25
H(14A)	9234.83	5802.25	6274.2	25
H(14B)	8282.58	6878.65	5706.16	25
H(11C)	12312.52	6116.34	6361.78	20
H(11D)	11795.88	7376.97	6574.9	20
H(12C)	10641.01	5639.29	7593.59	30
H(12D)	11113.63	6574.6	8131.34	30
H(13C)	8496.08	6979.8	7914.43	24
H(13D)	9246.2	7927.59	7399.07	24
H(14C)	8185	7433.34	6045.15	25
H(14D)	8774.87	6163.45	6370.97	25
H(7)	6240(18)	7922(14)	2111(13)	26
H(8)	9116(19)	9716(14)	-1190(13)	28
H(9)	10984(18)	4119(14)	126(13)	27
H(10)	13281(17)	6647(14)	3014(12)	25
H(17)	8244(18)	6526(14)	2835(13)	28
H(18)	5528(19)	5405(15)	6521(13)	29
H(19)	4340(18)	10912(13)	4579(13)	25
H(20)	1676(17)	8356(15)	2014(12)	26

Table S36: Hydrogen Bond information for **2018ncs0571p**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
O(7)	H(7)	O(11)	0.873(17)	1.898(17)	2.7509(12)	165.2(16)
O(8)	H(8)	O(20)[1]	0.858(18)	2.008(18)	2.8169(12)	156.8(16)
O(9)	H(9)	O(4)[2]	0.855(18)	1.851(18)	2.7035(12)	174.9(17)
O(10)	H(10)	O(16)[3]	0.885(18)	1.851(18)	2.7342(12)	175.7(15)
O(17)	H(17)	O(1)	0.875(17)	1.918(18)	2.7893(12)	174.1(16)
O(18)	H(18)	O(10)[4]	0.854(18)	2.035(19)	2.8721(12)	166.5(16)
O(19)	H(19)	O(14)[5]	0.823(18)	1.916(18)	2.7362(12)	174.0(17)
O(20)	H(20)	O(6)[6]	0.851(18)	1.940(18)	2.7907(12)	177.6(16)

––––

[1]1-x,2-y,-z; [2]2-x,1-y,-z; [3]1+x,+y,+z; [4]2-x,1-y,1-z; [5]1-x,2-y,1-z; [6]-1+x,+y,+z

Table S37: Atomic Occupancies for all atoms that are not fully occupied in **2018ncs0571p**.

Atom	Occupancy
C(11)	0.900(5)
H(11A)	0.900(5)
H(11B)	0.900(5)
C(12)	0.900(5)
H(12A)	0.900(5)
H(12B)	0.900(5)
C(13)	0.900(5)
H(13A)	0.900(5)
H(13B)	0.900(5)
C(14)	0.900(5)
H(14A)	0.900(5)
H(14B)	0.900(5)
C(11A)	0.100(5)
H(11C)	0.100(5)
H(11D)	0.100(5)
C(12A)	0.100(5)
H(12C)	0.100(5)
H(12D)	0.100(5)
C(13A)	0.100(5)
H(13C)	0.100(5)
H(13D)	0.100(5)
C(14A)	0.100(5)
H(14C)	0.100(5)
H(14D)	0.100(5)
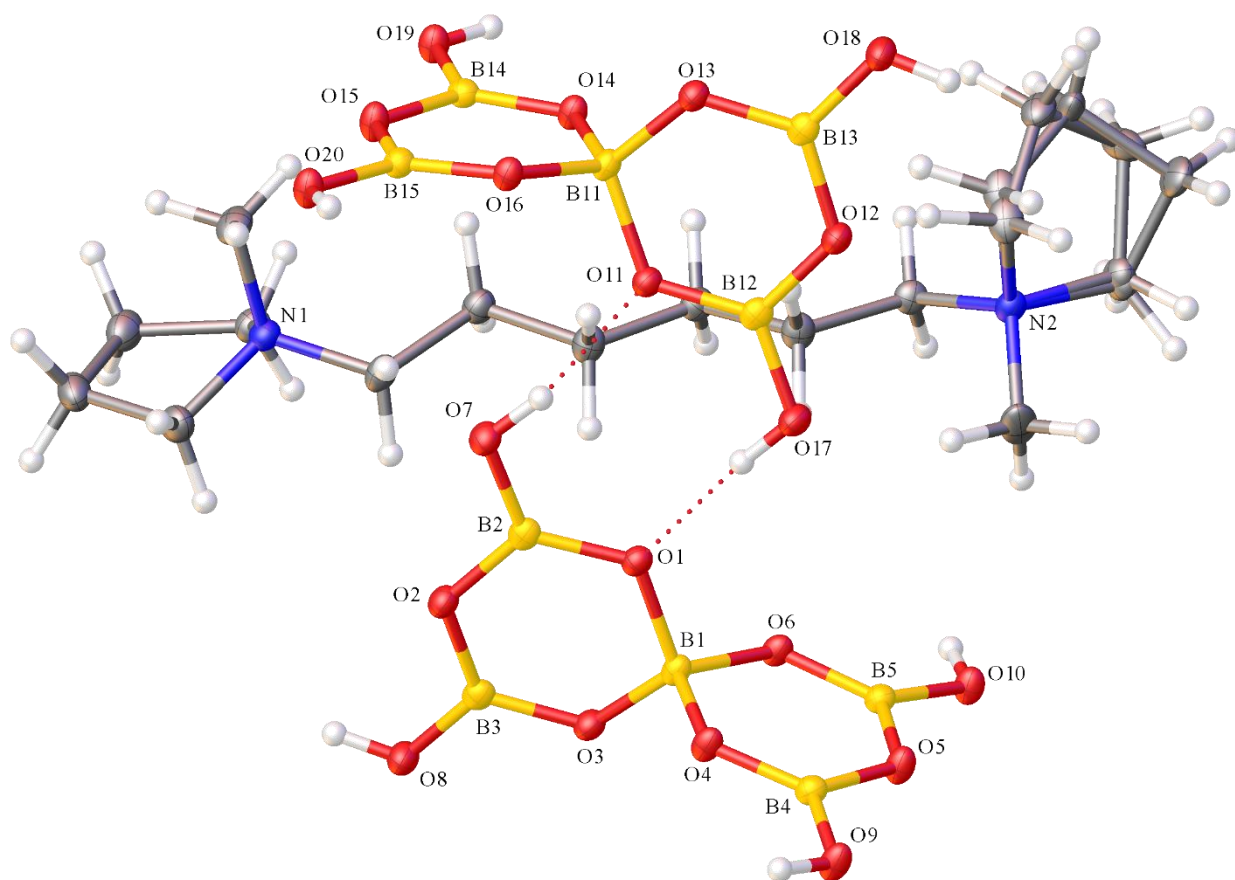
Citations for 4b

CrysAlisPro Software System, Rigaku Oxford Diffraction, (2018).

O.V. Dolomanov and L.J. Bourhis and R.J. Gildea and J.A.K. Howard and H. Puschmann, Olex2: A complete structure solution, refinement and analysis program, *J. Appl. Cryst.*, (2009), **42**, 339-341.

Sheldrick, G.M., Crystal structure refinement with ShelXL, *Acta Cryst.*, (2015), **C27**, 3-8.

Sheldrick, G.M., ShelXT-Integrated space-group and crystal-structure determination, *Acta Cryst.*, (2015), **A71**, 3-8.
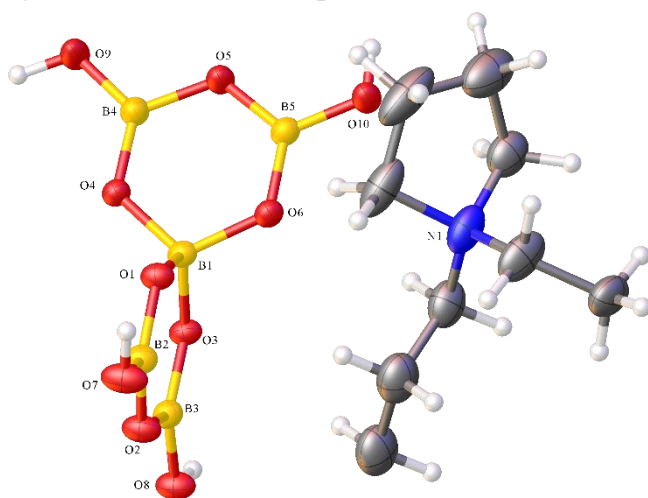
Submitted by: **Mike A Beckett / Thomas A. Rixon**

Bangor University

Solved by: **Peter N Horton**

Sample ID: **MAB/TAR/AR104 B5**

$[C_2H_5(C_4H_8N)(CH_2)_6(C_4H_8N)C_2H_5][B_5O_6(OH)_4]_2$ (**5**).

Crystal Data and Experimental

Experimental. Single colourless plate-shaped crystals of **2018ncs0474** were recrystallised from water. A suitable crystal 0.15×0.07×0.02 mm^3 was selected and mounted on a MITIGEN holder in perfluoroether oil on a Rigaku 007HF diffractometer equipped with Varimax confocal mirrors and an AFC11 goniometer and HyPix 6000 detector. The crystal was kept at a steady $T = 100(2)$ K during data collection. The structure was solved with the **ShelXT** (Sheldrick, 2015) structure solution program using the Intrinsic Phasing solution method and by using **Olex2** (Dolomanov et al., 2009) as the graphical interface. The model was refined with version 2018/3 of **ShelXL** (Sheldrick, 2015) using Least Squares minimisation.

Crystal Data. $C_{18}H_{46}B_{10}N_2O_{20}$, $M_r = 718.67$, monoclinic, $P2_1/c$ (No. 14), a = 10.06420(10) Å, b = 11.55510(10) Å, c = 15.7004(2) Å, $\beta = 107.6290(10)°$, $\alpha = \gamma = 90°$, $V = 1740.10(3)$ Å^3, $T = 100(2)$ K, $Z = 2$, $Z' = 0.5$, $\mu(CuK_\alpha) = 0.976$ mm^{-1}, 25011 reflections measured, 3183 unique ($R_{int} = 0.0306$) which were used in all calculations. The final wR_2 was 0.1290 (all data) and R_1 was 0.0417 (I > 2(I)).

Compound	2018ncs0474
Formula	$C_{18}H_{46}B_{10}N_2O_{20}$
$D_{calc.}$/ g cm^{-3}	1.372
μ/mm^{-1}	0.976
Formula Weight	718.67
Colour	colourless
Shape	plate
Size/mm^3	0.15×0.07×0.02
T/K	100(2)
Crystal System	monoclinic
Space Group	$P2_1/c$
a/Å	10.06420(10)
b/Å	11.55510(10)
c/Å	15.7004(2)
α/°	90
β/°	107.6290(10)
γ/°	90
V/Å^3	1740.10(3)
Z	2
Z'	0.5
Wavelength/Å	1.54184
Radiation type	CuK_α
Θ_{min}/°	4.610
Θ_{max}/°	68.237
Measured Refl.	25011
Independent Refl.	3183
Reflections with I > 2(I)	2928
R_{int}	0.0306
Parameters	243
Restraints	0
Largest Peak	0.253
Deepest Hole	-0.300
GooF	1.067
wR_2 (all data)	0.1290
wR_2	0.1259
R_1 (all data)	0.0439
R_1	0.0417

Structure Quality Indicators

Reflections:

d min (Cu)	I/σ	Rint	complete 100% (IUCr)
0.83	61.8	3.06%	100%

Refinement:

Shift	Max Peak	Min Peak	GooF
0.000	0.2	-0.3	1.067

A colourless plate-shaped crystal with dimensions 0.15×0.07×0.02 mm^3 was mounted on a MITIGEN holder in perfluoroether oil. Data were collected using an Rigaku 007HF equipped with Varimax confocal mirrors and an AFC11 goniometer and HyPix 6000 detector diffractometer equipped with an Oxford Cryosystems low-temperature device operating at T = 100(2) K.

Data were measured using profile data from ω-scans of 0.5 ° per frame for 1.0/4.0 s using CuK$_\alpha$ radiation (Rotating anode, 40.0 kV, 30.0 mA). The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.39.46, 2018). The maximum resolution that was achieved was Θ = 68.237° (0.83 Å).

The diffraction pattern was indexed. The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.39.46, 2018) and the unit cell was refined using **CrysAlisPro** (Rigaku, V1.171.39.46, 2018) on 15705 reflections, 63% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using **CrysAlisPro** (Rigaku, V1.171.39.46, 2018). The final completeness is 100.00 % out to 68.237° in Θ. A multi-scan absorption correction was performed using CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018) using spherical harmonicsas implemented in SCALE3 ABSPACK. The absorption coefficient μ of this material is 0.976 mm^{-1} at this wavelength (λ = 1.542Å) and the minimum and maximum transmissions are 0.786 and 1.000.

The structure was solved and the space group $P2_1/c$ (# 14) determined by the **ShelXT** (Sheldrick, 2015) structure solution program using Intrinsic Phasing and refined by Least Squares using version 2018/3 of **ShelXL** (Sheldrick, 2015). All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model. Most hydrogen atom positions were calculated geometrically and refined using the riding model, but some hydrogen atoms were refined freely.

_exptl_absorpt_process_details: CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018) using spherical harmonicsas implemented in SCALE3 ABSPACK.

The value of Z' is 0.5. This means that only half of the formula unit is present in the asymmetric unit, with the other half consisting of symmetry equivalent atoms.
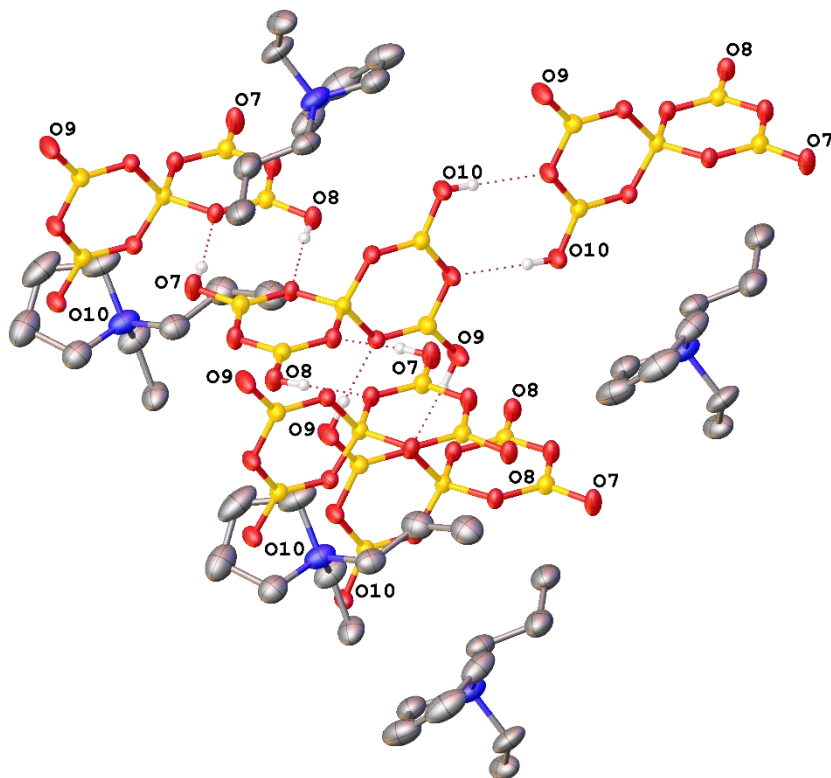
Figure S38: The following hydrogen bonding interactions with a maximum D-D distance of 2.9 Å and a minimum angle of 120 ° are present in **2018ncs0474**: O7–O3_*1: 2.788 Å, O8–O1_*2: 2.749 Å, O9–O4_*3: 2.737 Å, O10–O5_*4: 2.736 Å.
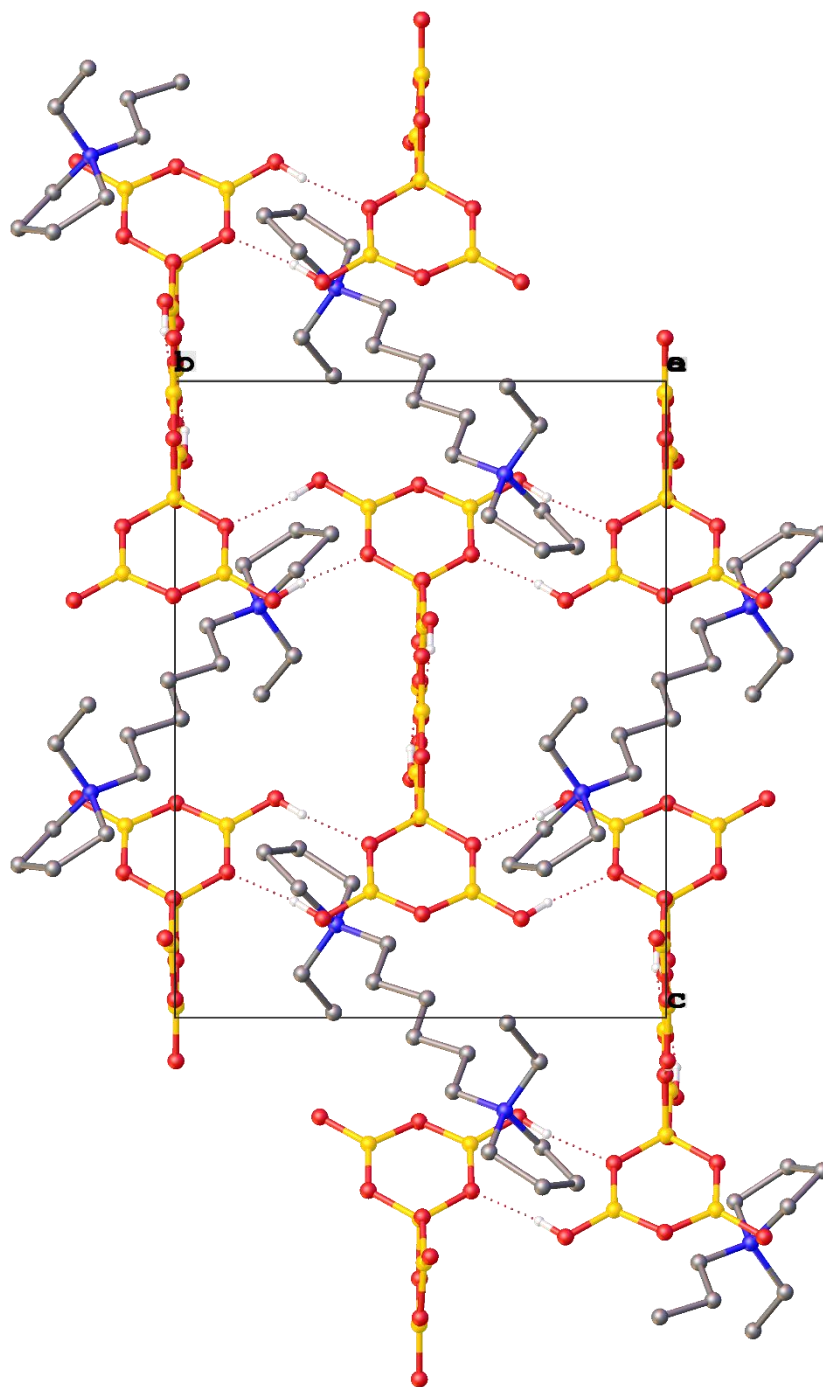
Figure S39: Packing diagram of 2018ncs0474.

Reflection Statistics

Total reflections (after filtering)	25725	Unique reflections	3183
Completeness	1.0	Mean I/σ	41.14
hkl_{max} collected	(12, 13, 16)	hkl_{min} collected	(-12, -13, -18)
hkl_{max} used	(11, 13, 18)	hkl_{min} used	(-12, 0, 0)
Lim d_{max} collected	100.0	Lim d_{min} collected	0.77
d_{max} used	14.96	d_{min} used	0.83
Friedel pairs	3623	Friedel pairs merged	1
Inconsistent equivalents	10	R_{int}	0.0306
R_{sigma}	0.0162	Intensity transformed	0
Omitted reflections	0	Omitted by user (OMIT hkl)	0
Multiplicity	(3054, 2706, 1820, 1110, 592, 332, 164, 79, 46, 18, 3)	Maximum multiplicity	22
Removed systematic absences	714	Filtered off (Shel/OMIT)	0

Table S38: Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{Å}^2 \times 10^3$) for **2018ncs0474**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
N(1)	8950.6(16)	3285.3(12)	1460.6(9)	43.1(4)
C(1)	8341(3)	2525.5(16)	2034.6(13)	59.2(6)
C(2)	9545(3)	1823.8(19)	2597.2(14)	74.8(7)
C(3)	10854(3)	2520.4(19)	2657.2(14)	68.3(6)
C(4)	10343.2(19)	3604.3(16)	2109.0(11)	47.9(4)
C(5)	8065.6(19)	4335.5(15)	1156.3(11)	45.3(4)
C(6)	6607(2)	4049.6(18)	563.6(12)	55.1(5)
C(7)	5737(2)	5130.6(18)	302.3(14)	56.9(5)
C(8)	9136(2)	2582.6(15)	689.6(12)	52.6(5)
C(9)	9781.2(19)	3231.9(19)	83.4(10)	50.4(5)
O(1)	5048.2(9)	3965.8(7)	2720.0(5)	22.5(2)
O(2)	3309.1(11)	5030.4(7)	1630.2(7)	26.9(3)
O(3)	5100.0(9)	6062.1(7)	2713.1(5)	22.0(2)
O(4)	5733.3(10)	5022.3(7)	4098.7(6)	21.5(2)
O(5)	8206.6(10)	5008.2(8)	4710.9(6)	28.3(3)
O(6)	7207.5(10)	4964.6(7)	3128.0(6)	22.4(2)
O(7)	3332.4(11)	3009.4(9)	1556.8(7)	33.4(3)
O(8)	3401.5(10)	7049.0(8)	1557.3(6)	27.4(2)
O(9)	6859.1(11)	5024.1(10)	5677.2(7)	38.0(3)
O(10)	9613.0(11)	4804.0(9)	3750.5(7)	29.5(3)
B(1)	5773.0(16)	5008.2(11)	3162.8(10)	20.2(3)
B(2)	3916.5(16)	3990.2(12)	1982.8(10)	24.0(3)
B(3)	3956.4(15)	6058.0(12)	1981.0(9)	22.1(3)
B(4)	6877.4(17)	5027.8(12)	4818.8(11)	24.9(3)
B(5)	8329.0(17)	4926.8(11)	3851.2(10)	22.1(3)

Table S39: Anisotropic Displacement Parameters (×10^4) **2018ncs0474**. The anisotropic displacement factor exponent takes the form: *-2π^2[h^{2}a*^{2} × U$_{11}$+ ... +2hka* × b* × U$_{12}$]*

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	65.7(9)	35.5(7)	38.6(7)	-4.5(6)	31.7(7)	-2.3(6)
C(1)	101.5(16)	46.0(10)	45.5(10)	-6.5(8)	45.3(11)	-17.3(10)
C(2)	140(2)	51.4(12)	50.8(11)	8.2(9)	56.2(14)	11.0(13)
C(3)	96.6(17)	66.2(14)	51.4(11)	8.4(10)	36.1(11)	30.4(12)
C(4)	61.2(11)	49.3(10)	39.7(9)	-4.4(7)	24.9(8)	4.5(8)
C(5)	56.3(10)	44.0(9)	44.6(9)	-5.4(7)	28.7(8)	0.7(8)
C(6)	60.4(11)	66.6(12)	44.1(10)	-11.2(9)	24.7(9)	-5.9(9)
C(7)	60.0(13)	71.9(13)	45.8(11)	-12.4(9)	26.4(10)	-1.0(9)
C(8)	88.3(14)	41.4(9)	38.1(9)	-6.2(7)	33.9(9)	5.5(9)
C(9)	56.9(10)	69.0(12)	30.5(8)	3.1(8)	21.1(8)	11.2(9)
O(1)	23.8(5)	20.2(5)	20.0(5)	-0.6(3)	1.7(4)	-0.7(3)
O(2)	26.9(5)	22.0(5)	25.1(5)	-0.7(3)	-2.1(4)	0.2(3)
O(3)	24.3(5)	20.4(5)	18.4(5)	0.8(3)	2.3(4)	-0.3(3)
O(4)	21.0(5)	26.4(5)	16.9(5)	0.4(3)	5.3(4)	0.2(3)
O(5)	20.3(5)	47.6(7)	16.5(5)	-0.5(3)	4.9(4)	0.3(4)
O(6)	22.6(5)	28.3(5)	16.0(5)	-0.7(3)	5.5(4)	-0.2(3)
O(7)	34.7(6)	21.7(5)	32.7(5)	-2.3(4)	-6.6(4)	-0.4(4)
O(8)	29.8(5)	20.7(5)	25.5(5)	0.5(4)	-0.8(4)	0.8(4)
O(9)	22.5(5)	74.6(9)	17.2(5)	1.0(4)	6.4(4)	2.3(4)
O(10)	21.9(5)	48.1(6)	18.3(5)	-2.4(4)	6.0(4)	-0.5(4)
B(1)	21.8(7)	20.7(7)	17.0(7)	-0.2(5)	4.2(6)	-0.4(5)
B(2)	25.0(7)	23.3(7)	22.1(7)	-0.5(5)	4.9(6)	0.1(5)
B(3)	23.7(7)	23.2(7)	19.2(7)	-0.3(5)	6.3(5)	0.6(5)
B(4)	22.0(8)	33.3(8)	19.3(7)	0.5(5)	6.3(6)	0.8(5)
B(5)	23.7(7)	24.6(7)	17.9(7)	-0.7(5)	6.1(6)	-1.0(5)

Table S40: Bond Lengths in Å for **2018ncs0474**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N(1)	C(1)	1.514(2)	O(3)	B(1)	1.4665(15)
N(1)	C(4)	1.507(2)	O(3)	B(3)	1.3582(17)
N(1)	C(5)	1.495(2)	O(4)	B(1)	1.4821(17)
N(1)	C(8)	1.5154(19)	O(4)	B(4)	1.3475(19)
C(1)	C(2)	1.502(3)	O(5)	B(4)	1.3986(19)
C(2)	C(3)	1.522(4)	O(5)	B(5)	1.3950(18)
C(3)	C(4)	1.518(3)	O(6)	B(1)	1.4621(18)
C(5)	C(6)	1.518(3)	O(6)	B(5)	1.3375(19)
C(6)	C(7)	1.509(3)	O(7)	B(2)	1.3562(17)
C(7)	C(7)[1]	1.529(4)	O(8)	B(3)	1.3567(17)
C(8)	C(9)	1.505(2)	O(9)	B(4)	1.3534(19)
O(1)	B(1)	1.4694(15)	O(10)	B(5)	1.3561(19)
O(1)	B(2)	1.3562(17)			
O(2)	B(2)	1.3855(17)	----		
O(2)	B(3)	1.3858(16)	[1]1-x,1-y,-z		

Table S41: Bond Angles in ° for **2018ncs0474**.

Atom	Atom	Atom	Angle/°
C(1)	N(1)	C(8)	109.56(13)
C(4)	N(1)	C(1)	101.60(14)
C(4)	N(1)	C(8)	110.87(14)
C(5)	N(1)	C(1)	110.56(14)
C(5)	N(1)	C(4)	111.06(13)
C(5)	N(1)	C(8)	112.66(13)
C(2)	C(1)	N(1)	105.25(17)
C(1)	C(2)	C(3)	106.24(16)
C(4)	C(3)	C(2)	105.25(18)
N(1)	C(4)	C(3)	105.49(16)
N(1)	C(5)	C(6)	113.03(15)
C(7)	C(6)	C(5)	111.12(17)
C(6)	C(7)	C(7)[1]	112.3(2)
C(9)	C(8)	N(1)	114.78(15)
B(2)	O(1)	B(1)	123.71(10)
B(2)	O(2)	B(3)	119.15(11)
B(3)	O(3)	B(1)	123.64(10)
B(4)	O(4)	B(1)	124.00(11)
B(5)	O(5)	B(4)	119.09(12)
B(5)	O(6)	B(1)	123.94(11)

Atom	Atom	Atom	Angle/°
O(1)	B(1)	O(4)	108.63(10)
O(3)	B(1)	O(1)	111.23(11)
O(3)	B(1)	O(4)	108.72(10)
O(6)	B(1)	O(1)	107.96(10)
O(6)	B(1)	O(3)	109.15(10)
O(6)	B(1)	O(4)	111.15(11)
O(1)	B(2)	O(2)	120.92(11)
O(7)	B(2)	O(1)	122.01(12)
O(7)	B(2)	O(2)	117.07(12)
O(3)	B(3)	O(2)	121.01(11)
O(8)	B(3)	O(2)	117.02(11)
O(8)	B(3)	O(3)	121.97(12)
O(4)	B(4)	O(5)	120.26(12)
O(4)	B(4)	O(9)	124.72(13)
O(9)	B(4)	O(5)	115.00(13)
O(6)	B(5)	O(5)	121.34(13)
O(6)	B(5)	O(10)	119.55(13)
O(10)	B(5)	O(5)	119.10(13)

––––
[1]1-x,1-y,-z

Table S42: Torsion Angles in ° for **2018ncs0474**.

Atom	Atom	Atom	Atom	Angle/°
N(1)	C(1)	C(2)	C(3)	24.62(19)
N(1)	C(5)	C(6)	C(7)	177.85(14)
C(1)	N(1)	C(4)	C(3)	38.33(16)
C(1)	N(1)	C(5)	C(6)	-63.12(17)
C(1)	N(1)	C(8)	C(9)	-178.07(17)
C(1)	C(2)	C(3)	C(4)	-0.7(2)
C(2)	C(3)	C(4)	N(1)	-23.67(18)
C(4)	N(1)	C(1)	C(2)	-38.73(17)
C(4)	N(1)	C(5)	C(6)	-175.12(13)
C(4)	N(1)	C(8)	C(9)	-66.7(2)
C(5)	N(1)	C(1)	C(2)	-156.68(15)
C(5)	N(1)	C(4)	C(3)	155.92(13)
C(5)	N(1)	C(8)	C(9)	58.4(2)
C(5)	C(6)	C(7)	C(7)[1]	179.5(2)
C(8)	N(1)	C(1)	C(2)	78.58(18)
C(8)	N(1)	C(4)	C(3)	-78.02(16)
C(8)	N(1)	C(5)	C(6)	59.83(18)
B(1)	O(1)	B(2)	O(2)	-4.4(2)
B(1)	O(1)	B(2)	O(7)	175.41(12)
B(1)	O(3)	B(3)	O(2)	2.78(19)
B(1)	O(3)	B(3)	O(8)	-176.80(11)
B(1)	O(4)	B(4)	O(5)	-0.65(18)
B(1)	O(4)	B(4)	O(9)	-179.02(12)
B(1)	O(6)	B(5)	O(5)	-4.55(18)
B(1)	O(6)	B(5)	O(10)	175.28(10)
B(2)	O(1)	B(1)	O(3)	0.93(17)
B(2)	O(1)	B(1)	O(4)	120.56(12)
B(2)	O(1)	B(1)	O(6)	-118.80(12)
B(2)	O(2)	B(3)	O(3)	-6.1(2)
B(2)	O(2)	B(3)	O(8)	173.53(11)
B(3)	O(2)	B(2)	O(1)	6.8(2)

Atom	Atom	Atom	Atom	Angle/°
B(3)	O(2)	B(2)	O(7)	-172.94(12)
B(3)	O(3)	B(1)	O(1)	-0.15(17)
B(3)	O(3)	B(1)	O(4)	-119.72(12)
B(3)	O(3)	B(1)	O(6)	118.88(12)
B(4)	O(4)	B(1)	O(1)	120.43(12)
B(4)	O(4)	B(1)	O(3)	-118.39(12)
B(4)	O(4)	B(1)	O(6)	1.78(15)
B(4)	O(5)	B(5)	O(6)	5.65(18)
B(4)	O(5)	B(5)	O(10)	-174.18(11)
B(5)	O(5)	B(4)	O(4)	-3.06(18)
B(5)	O(5)	B(4)	O(9)	175.46(11)
B(5)	O(6)	B(1)	O(1)	-118.21(12)
B(5)	O(6)	B(1)	O(3)	120.75(12)
B(5)	O(6)	B(1)	O(4)	0.84(15)

––––

[1]1-x,1-y,-z

Table S43: Hydrogen Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **2018ncs0474**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H(1A)	7933.65	3002.99	2415.88	71
H(1B)	7606.62	2014.98	1657.56	71
H(2A)	9581.54	1062.01	2316.57	90
H(2B)	9452.79	1696.22	3200.22	90
H(3A)	11352.13	2721.88	3285.88	82
H(3B)	11491.86	2075.64	2408.6	82
H(4A)	10241.29	4250.42	2498.61	57
H(4B)	11004.57	3839.52	1785.32	57
H(5A)	8523.85	4846.77	823.83	54
H(5B)	7998.1	4766.18	1686.77	54
H(6A)	6664.81	3650.02	18.2	66
H(6B)	6152.72	3518.58	885.2	66
H(7A)	6200.4	5663.37	-12.29	68
H(7B)	5678.23	5525.79	849.26	68
H(8A)	9727.37	1903.18	935.4	63
H(8B)	8212.68	2290.06	328.87	63
H(9A)	9940.5	2699.68	-361.83	76
H(9B)	10671.94	3566.47	437.69	76
H(9C)	9151.35	3852.43	-220.45	76
H(7)	3790(20)	2434(19)	1827(14)	55(6)
H(8)	3870(20)	7587(19)	1807(14)	49(6)
H(9)	5980(30)	5041(15)	5758(15)	56(6)
H(10)	10230(30)	4797(18)	4217(17)	54(6)

Table S44: Hydrogen Bond information for **2018ncs0474**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
O(7)	H(7)	O(3)[1]	0.85(2)	1.95(2)	2.7875(13)	172(2)
O(8)	H(8)	O(1)[2]	0.81(2)	1.95(2)	2.7487(13)	174(2)
O(9)	H(9)	O(4)[3]	0.93(3)	1.81(3)	2.7368(15)	176.4(17)
O(10)	H(10)	O(5)[4]	0.81(3)	1.94(3)	2.7364(15)	172(2)

––––

[1]1-x,-1/2+y,1/2-z; [2]1-x,1/2+y,1/2-z; [3]1-x,1-y,1-z; [4]2-x,1-y,1
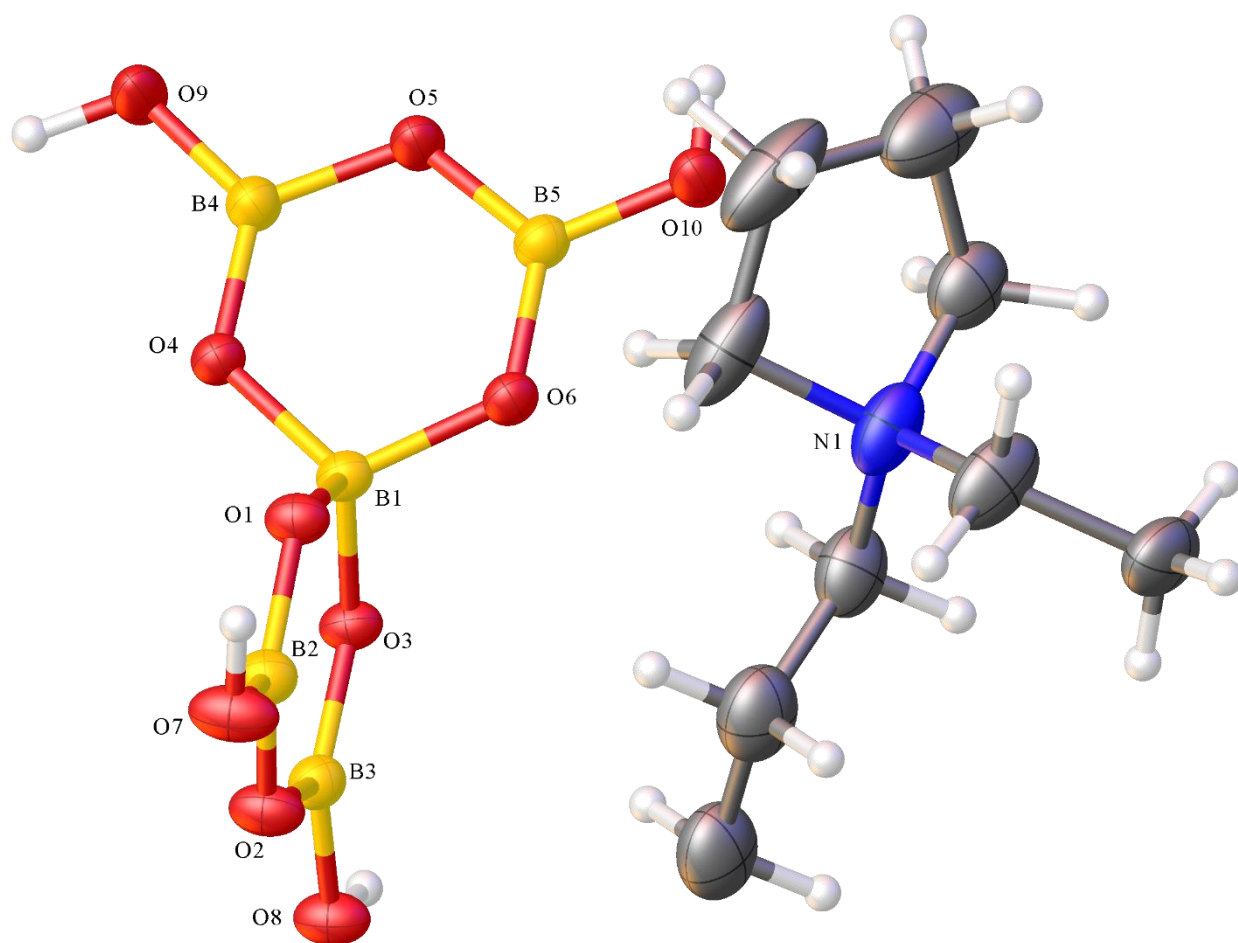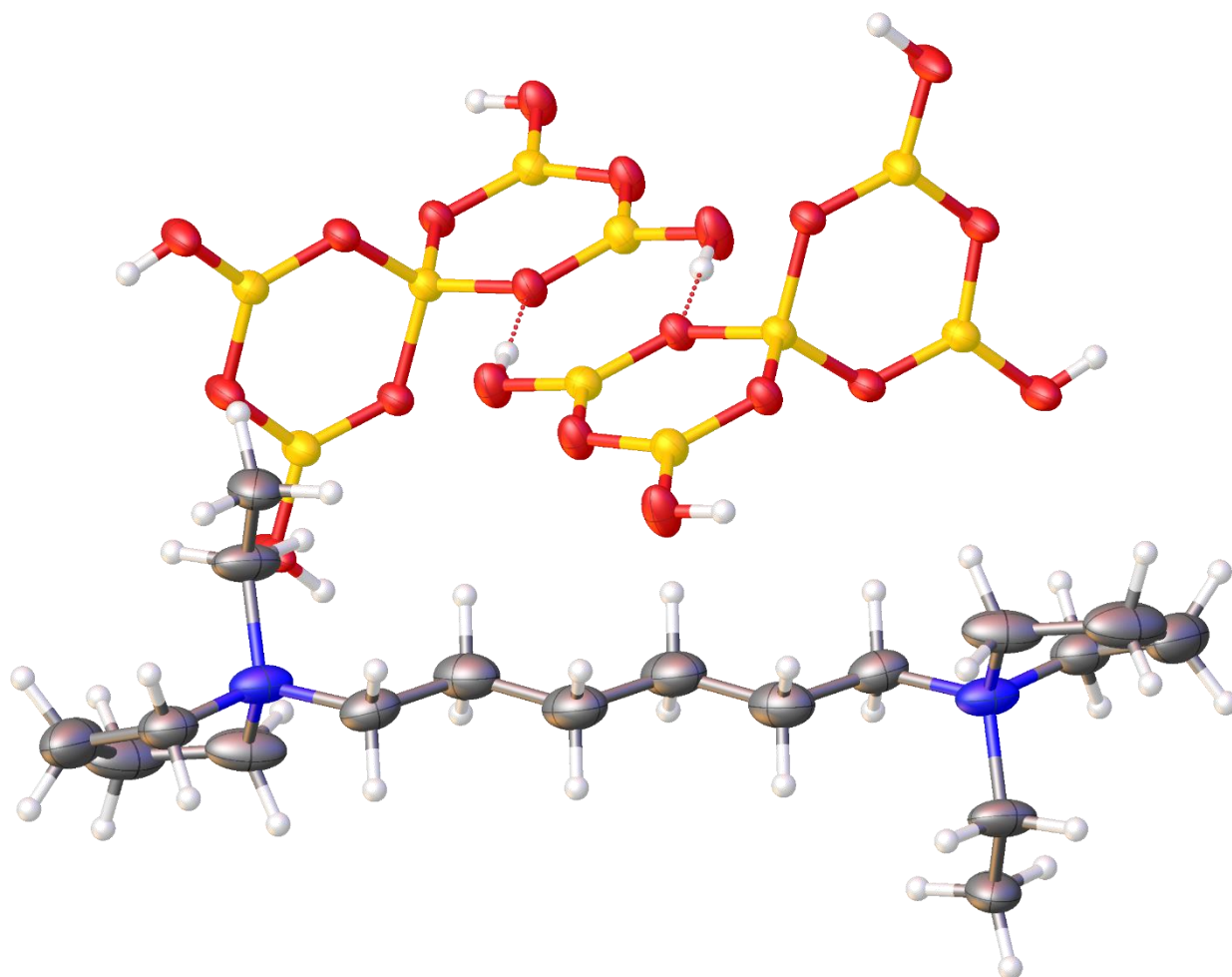
Citations for 5

CrysAlisPro Software System, Rigaku Oxford Diffraction, (2018).

O.V. Dolomanov and L.J. Bourhis and R.J. Gildea and J.A.K. Howard and H. Puschmann, Olex2: A complete structure solution, refinement and analysis program, *J. Appl. Cryst.*, (2009), **42**, 339-341.
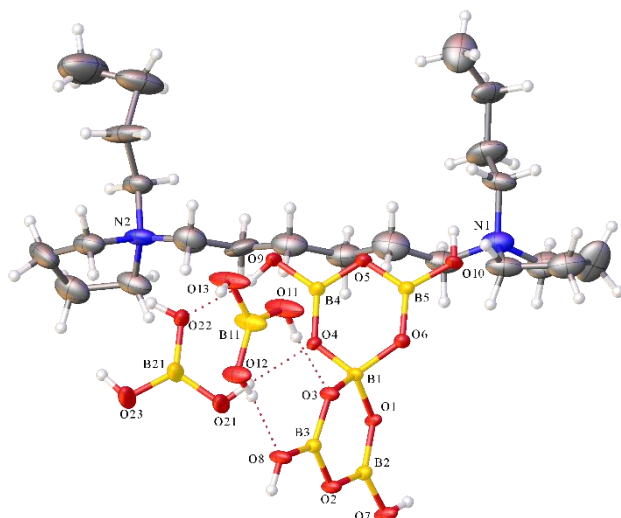
Sheldrick, G.M., Crystal structure refinement with ShelXL, *Acta Cryst.*, (2015), **C27**, 3-8.

Sheldrick, G.M., ShelXT-Integrated space-group and crystal-structure determination, *Acta Cryst.*, (2015), **A71**, 3-8.

Submitted by: **Mike A Beckett**

Bangor University

Solved by: **Peter N Horton**

Sample ID: **MAB/TAR/AR103 B5**

$[C_4H_9(C_4H_8N)(CH_2)_6(C_4H_8N)C_4H_9]$ $[B_5O_6(OH)_4]_2 \cdot 4B(OH)_3$ (**6**).

Crystal Data and Experimental

Experimental. Single colourless plate-shaped crystals of **2018ncs0542** were recrystallised from water. A suitable crystal 0.160×0.075×0.025 mm³ was selected and mounted on a MITIGEN holder in perfluoroether oil on a Rigaku FRE+ diffractometer equipped with VHF Varimax confocal mirrors and an AFC12 goniometer and HyPix 6000 detector. The crystal was kept at a steady $T =$ 100(2) K during data collection. The structure was solved with the **ShelXT** (Sheldrick, 2015) structure solution program using the Intrinsic Phasing solution method and by using **Olex2** (Dolomanov et al., 2009) as the graphical interface. The model was refined with version 2018/3 of **ShelXL** (Sheldrick, 2015) using Least Squares minimisation.

Crystal Data. $C_{22}H_{66}B_{14}N_2O_{32}$, $M_r = 1022.10$, monoclinic, Cc (No. 9), a = 9.7545(3) Å, b = 15.4363(4) Å, c = 17.0325(5) Å, $\beta =$ 101.851(3)°, $\alpha = \gamma =$ 90°, $V =$ 2509.98(13) Å³, $T =$ 100(2) K, $Z =$ 2, $Z' =$ 0.5, $\mu(MoK_\alpha) =$ 0.117, 9481 reflections measured, 9481 unique (R_{int} = .) which were used in all calculations. The final wR_2 was 0.1483 (all data) and R_1 was 0.0532 (I > 2(I)).

Compound	2018ncs0542
Formula	$C_{22}H_{66}B_{14}N_2O_{32}$
$D_{calc.}$/ g cm^{-3}	1.352
μ/mm^{-1}	0.117
Formula Weight	1022.10
Colour	colourless
Shape	plate
Size/mm³	0.160×0.075×0.025
T/K	100(2)
Crystal System	monoclinic
Flack Parameter	-2.2(7)
Hooft Parameter	-3.0(3)
Space Group	Cc
a/Å	9.7545(3)
b/Å	15.4363(4)
c/Å	17.0325(5)
α/°	90
β/°	101.851(3)
γ/°	90
V/Å³	2509.98(13)
Z	2
Z'	0.5
Wavelength/Å	0.71075
Radiation type	MoK$_\alpha$
Θ_{min}/°	2.444
Θ_{max}/°	27.518
Measured Refl.	9481
Independent Refl.	9481
Reflections with I > 2(I)	8213
R_{int}	.
Parameters	437
Restraints	617
Largest Peak	0.260
Deepest Hole	-0.247
GooF	1.052
wR_2 (all data)	0.1483
wR_2	0.1443
R_1 (all data)	0.0607
R_1	0.0532

Structure Quality Indicators

Reflections:

d min (Mo)	I/σ	Rint	complete
0.77	68.0	Merged!	100%

Refinement:

Shift	Max Peak	Min Peak	GooF	Flack
0.000	0.3	-0.2	1.052	.1(10)

A colourless plate-shaped crystal with dimensions 0.160×0.075×0.025 mm^3 was mounted on a MITIGEN holder in perfluoroether oil. Data were collected using an Rigaku FRE+ diffractometer equipped with VHF Varimax confocal mirrors and an AFC12 goniometer and HyPix 6000 detector equipped with an Oxford Cryosystems low-temperature device operating at T = 100(2) K.

Data were measured using profile data from ω-scans of 0.5° per frame for 10.0 s using MoK$_\alpha$ radiation. The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.39.46, 2018). The maximum resolution that was achieved was Θ = 27.518° (0.77 Å).

The diffraction pattern was indexed The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.39.46, 2018) and the unit cell was refined using **CrysAlisPro** (Rigaku, V1.171.39.46, 2018) on 12567 reflections, 133% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using **CrysAlisPro** (Rigaku, V1.171.39.46, 2018). The final completeness is 100.00 % out to 27.518° in Θ. A multi-scan absorption correction was performed using CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018) using spherical harmonics as implemented in SCALE3 ABSPACK. The absorption coefficient μ of this material is 0.117 mm^{-1} at this wavelength (λ = 0.71075 Å) and the minimum and maximum transmissions are 0.370 and 1.000.

The structure was solved and the space group Cc (# 9) determined by the **ShelXT** (Sheldrick, 2015) structure solution program using Intrinsic Phasing and refined by Least Squares using version 2018/3 of **ShelXL** (Sheldrick, 2015). All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model. Hydrogen atom positions were calculated geometrically and refined using the riding model.

_refine_special_details_: The diammonium cation lies disordered within a channel. The disorder is a half moelcule slippage covering the distance between the two butyl groups. As such various geometrical (SADI) and displacement (RIGU, DELU) restraints have been employed.

_exptl_absorpt_process_details_: CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018) using spherical harmonicsas implemented in SCALE3 ABSPACK.

_twin_special_details_: Component 2 rotated by -179.7963° around [0.00 -0.00 1.00] (reciprocal) or [0.34 - 0.00 0.94] (direct)

The value of Z' is 0.5. This means that only half of the formula unit is present in the asymmetric unit, with the other half consisting of symmetry equivalent atoms.

The Flack parameter was refined to -2.2(7). Determination of absolute structure using Bayesian statistics on Bijvoet differences using the Olex2 results in -3.0(3). Note: The Flack parameter is used to determine chirality of the crystal studied, the value should be near 0, a value of 1 means that the stereochemistry is wrong and the model should be inverted. A value of 0.5 means that the crystal consists of a racemic mixture of the two enantiomers.

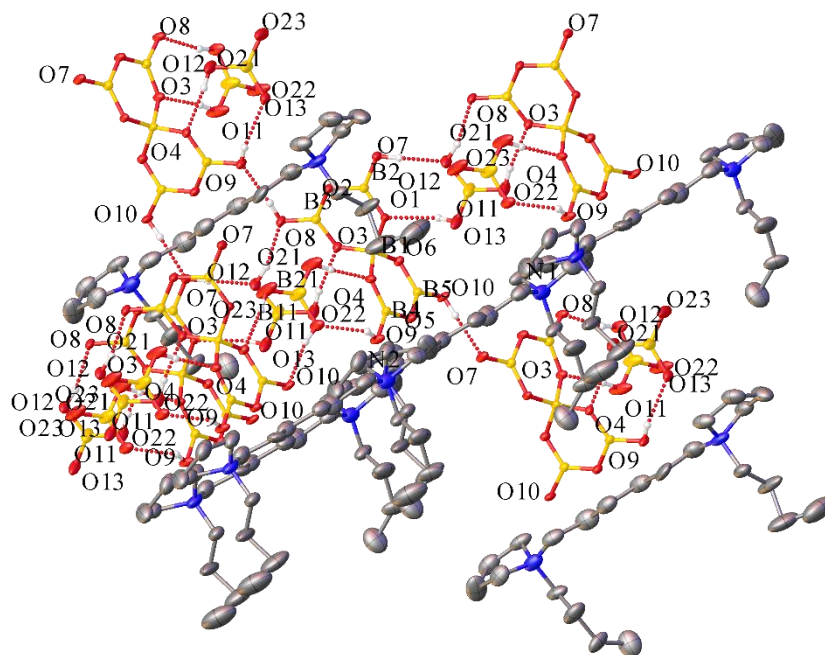**Figure S40**: The following hydrogen bonding interactions with a maximum D-D distance of 2.9 Å and a minimum angle of 120 ° are present in **2018ncs0542**: O7–O12_*1: 2.673 Å*, *O8–O9_*4: 2.653 Å, O9–O22: 2.698 Å, O10–O7_*5: 2.697 Å, O11–O3: 2.709 Å, O12–O8: 2.705 Å, O13–O1_*2: 2.737 Å, O21–O4: 2.74 Å, O22–O10_*3: 2.749 Å*, *O23–O6_*3: 2.748 Å.
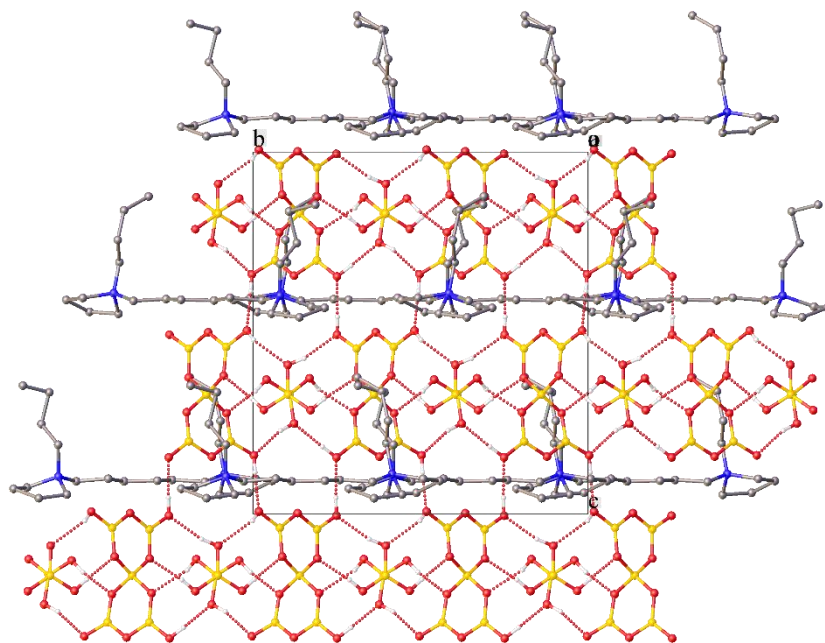

Figure S41: Packing diagram of 2018ncs0542.

Reflection Statistics

Total reflections (after filtering)	13711	Unique reflections	5725
Completeness	0.993	Mean I/σ	7.22
hkl_{max} collected	(12, 20, 22)	hkl_{min} collected	(-12, -20, -22)
hkl_{max} used	(12, 20, 22)	hkl_{min} used	(-12, 0, -22)
Lim d_{max} collected	100.0	Lim d_{min} collected	0.36
d_{max} used	8.33	d_{min} used	0.77
Friedel pairs	2582	Friedel pairs merged	0
Inconsistent equivalents	0	R_{int}	0.0
R_{sigma}	0.0147	Intensity transformed	0
Omitted reflections	0	Omitted by user (OMIT hkl)	0
Multiplicity	(5487, 1721, 184)	Maximum multiplicity	0
Removed systematic absences	0	Filtered off (Shel/OMIT)	0

Table S45: Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **2018ncs0542**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
N(1)	7200(20)	9153(15)	3899(12)	47(4)
N(2)	11901(19)	4193(14)	3966(11)	44(3)
C(1)	6238(11)	9011(9)	4470(6)	51(3)
C(2)	5418(15)	9877(10)	4413(9)	60(3)
C(3)	6320(20)	10573(15)	4082(19)	83(8)
C(4)	7637(12)	10076(8)	4030(8)	55(3)
C(5)	8539(12)	8565(9)	4113(7)	53(3)
C(6)	8299(17)	7591(12)	4060(13)	67(4)
C(7)	9687(13)	7145(8)	4083(8)	56(3)
C(8)	9565(12)	6158(8)	4101(8)	55(3)
C(9)	10920(20)	5682(12)	4028(12)	58(4)
C(10)	10710(15)	4731(9)	4096(9)	57(3)
C(11)	11777(12)	3261(7)	4271(7)	51(2)
C(12)	13207(18)	2861(11)	4304(12)	69(6)
C(13)	14223(14)	3614(8)	4565(8)	62(3)
C(14)	13302(12)	4448(8)	4464(7)	54(3)
C(15)	6419(15)	8985(10)	3050(7)	49(3)
C(16)	7382(18)	9034(11)	2429(7)	75(4)
C(17)	6511(14)	8846(12)	1609(9)	56(4)
C(18)	5624(17)	8113(12)	1227(13)	81(5)
C(19)	12033(16)	4190(9)	3086(7)	47(3)
C(20)	10950(20)	3689(11)	2516(7)	93(6)
C(21)	10850(30)	3924(18)	1638(11)	104(8)
C(22)	11590(30)	3140(20)	1454(13)	130(9)
O(1)	7267(3)	6975.1(16)	7189.2(14)	19.4(6)
O(2)	7249(3)	6227.1(17)	8414.7(15)	22.8(6)
O(3)	5668(3)	5789.5(17)	7219.4(16)	22.3(6)
O(4)	6946(3)	5873.6(16)	6179.1(16)	20.5(6)
O(5)	5398(3)	6334.6(19)	4978.8(15)	22.8(6)
O(6)	5124(3)	6918.3(17)	6234.4(15)	20.2(5)
O(7)	8516(3)	7510.6(18)	8437.6(17)	29.1(7)
O(8)	5833(3)	4969.8(17)	8380.8(16)	26.1(6)
O(9)	6929(3)	5179.1(19)	4934.7(16)	30.7(7)
O(10)	3819(3)	7490.2(18)	5047.9(16)	25.8(6)
O(11)	4073(5)	4594(3)	6287(2)	67.7(15)
O(12)	4159(4)	3773(2)	7501.3(19)	40.2(9)
O(13)	2839(6)	3311(3)	6251(2)	69.5(16)
B(1)	6244(5)	6392(2)	6701(3)	17.8(6)
B(2)	7664(5)	6909(3)	7997(3)	19.6(8)
B(3)	6266(4)	5660(3)	7997(3)	18.5(8)

Atom	x	y	z	U_{eq}
B(4)	6447(5)	5800(3)	5382(3)	20.2(9)
B(5)	4791(4)	6911(3)	5426(2)	19.1(8)
B(11)	3712(8)	3902(4)	6691(4)	48.3(17)
O(21)	8586(6)	4677(3)	7125(2)	67.8(15)
O(22)	8414(4)	3962(2)	5881.9(18)	36.3(8)
O(23)	9487(6)	3282(3)	7134(2)	74.7(17)
B(21)	8825(8)	3954(4)	6702(3)	46.8(16)

Table S46: Anisotropic Displacement Parameters (×10^4) **2018ncs0542**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11}+ \ldots +2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	49(7)	60(7)	32(6)	-1(5)	8(5)	-22(5)
N(2)	55(8)	55(6)	18(5)	3(5)	-1(5)	-19(5)
C(1)	43(5)	85(7)	26(5)	0(5)	6(4)	-23(5)
C(2)	49(7)	87(9)	47(8)	-10(7)	15(6)	-26(6)
C(3)	108(14)	71(9)	94(19)	-4(10)	80(15)	-15(9)
C(4)	46(6)	66(6)	54(7)	-12(5)	12(5)	-27(5)
C(5)	41(5)	83(7)	34(6)	2(6)	3(5)	-17(5)
C(6)	54(7)	84(9)	62(12)	4(8)	10(7)	-26(6)
C(7)	61(6)	69(6)	37(6)	-2(5)	5(5)	-26(5)
C(8)	50(6)	73(6)	40(6)	3(5)	4(5)	-28(5)
C(9)	89(10)	58(7)	23(7)	-17(6)	5(8)	-20(6)
C(10)	61(7)	68(7)	43(8)	8(7)	12(6)	-11(5)
C(11)	65(6)	51(6)	33(5)	5(4)	0(5)	-18(5)
C(12)	80(9)	54(7)	57(12)	16(8)	-24(8)	-2(7)
C(13)	61(6)	71(7)	49(7)	22(6)	3(6)	-14(5)
C(14)	52(6)	64(6)	40(6)	15(5)	-8(5)	-24(5)
C(15)	51(7)	67(8)	24(5)	6(5)	-4(5)	-21(6)
C(16)	96(10)	97(11)	37(6)	-12(6)	25(6)	-26(8)
C(17)	46(8)	92(10)	32(6)	-20(6)	13(6)	11(7)
C(18)	50(7)	78(10)	114(14)	-6(9)	16(8)	-13(7)
C(19)	63(8)	50(7)	25(5)	3(4)	2(5)	-11(6)
C(20)	156(16)	89(10)	23(5)	0(6)	-5(7)	-74(11)
C(21)	98(16)	170(20)	35(8)	10(10)	-2(10)	5(15)
C(22)	120(20)	210(30)	56(11)	-14(14)	12(12)	35(19)
O(1)	32.7(14)	13.3(12)	10.6(12)	-0.1(9)	0.6(11)	-5.8(10)
O(2)	35.1(14)	19.2(13)	11.4(12)	2.0(10)	-1.5(11)	-8.0(11)
O(3)	31.9(15)	18.6(12)	13.6(13)	2.6(10)	-1.6(11)	-7.2(11)
O(4)	30.1(14)	16.5(12)	12.7(12)	-3.2(9)	-0.9(11)	3.2(10)
O(5)	33.6(14)	21.9(13)	11.0(12)	-1.2(10)	0.2(11)	6.3(11)
O(6)	27.4(13)	19.2(12)	12.9(12)	0.3(10)	1.9(10)	3.2(10)
O(7)	50.1(17)	21.7(14)	13.3(13)	0.1(10)	1.7(12)	-15.8(13)
O(8)	38.2(15)	23.9(13)	12.5(12)	4.9(11)	-3.5(11)	-11.1(12)
O(9)	46.4(18)	28.5(15)	13.0(13)	-4.9(11)	-3.3(12)	18.9(13)
O(10)	39.4(15)	26.9(15)	8.8(12)	0.6(10)	-0.3(11)	11.4(12)
O(11)	106(4)	54(2)	29(2)	18.5(17)	-20(2)	-56(2)
O(12)	60(2)	30.7(16)	23.7(17)	6.0(13)	-6.8(15)	-28.1(15)
O(13)	108(4)	59(2)	26.1(18)	18.0(17)	-22(2)	-63(3)
B(1)	26.5(16)	14.6(15)	11.0(14)	2.2(17)	0.7(13)	-2.4(18)
B(2)	27(2)	16.1(18)	15(2)	0.4(15)	2.8(17)	-3.2(15)
B(3)	23.4(19)	16.2(18)	14.6(18)	-0.2(14)	1.1(16)	-1.2(15)
B(4)	31(2)	16.7(18)	12.9(18)	0.7(14)	4.4(17)	0.6(15)
B(5)	27(2)	16.2(18)	12.9(19)	-0.1(15)	1.7(17)	-1.8(15)
B(11)	66(4)	43(3)	27(3)	12(2)	-12(3)	-32(3)
O(21)	119(4)	53(2)	21.7(18)	-8.3(16)	-9(2)	59(3)
O(22)	60(2)	26.7(15)	19.0(16)	-3.5(12)	0.4(15)	20.4(15)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(23)	139(4)	59(2)	20.5(18)	-1.5(18)	3(2)	68(3)
B(21)	76(4)	39(3)	22(3)	-2(2)	1(3)	33(3)

Table S47: Bond Lengths in Å for **2018ncs0542**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N(1)	C(1)	1.50(2)	C(21)	C(22)	1.47(3)
N(1)	C(4)	1.49(2)	O(1)	B(1)	1.469(5)
N(1)	C(5)	1.57(2)	O(1)	B(2)	1.355(5)
N(1)	C(15)	1.51(2)	O(2)	B(2)	1.376(5)
N(2)	C(10)	1.48(2)	O(2)	B(3)	1.383(5)
N(2)	C(11)	1.54(2)	O(3)	B(1)	1.471(5)
N(2)	C(14)	1.50(2)	O(3)	B(3)	1.348(5)
N(2)	C(19)	1.532(19)	O(4)	B(1)	1.465(5)
C(1)	C(2)	1.551(18)	O(4)	B(4)	1.350(5)
C(2)	C(3)	1.56(2)	O(5)	B(4)	1.383(5)
C(3)	C(4)	1.52(2)	O(5)	B(5)	1.381(5)
C(5)	C(6)	1.52(2)	O(6)	B(1)	1.459(5)
C(6)	C(7)	1.51(2)	O(6)	B(5)	1.349(5)
C(7)	C(8)	1.529(16)	O(7)	B(2)	1.364(5)
C(8)	C(9)	1.54(2)	O(8)	B(3)	1.362(5)
C(9)	C(10)	1.49(2)	O(9)	B(4)	1.366(5)
C(11)	C(12)	1.515(19)	O(10)	B(5)	1.365(5)
C(12)	C(13)	1.533(18)	O(11)	B(11)	1.356(7)
C(13)	C(14)	1.558(17)	O(12)	B(11)	1.374(7)
C(15)	C(16)	1.555(17)	O(13)	B(11)	1.362(7)
C(16)	C(17)	1.505(18)	O(21)	B(21)	1.374(6)
C(17)	C(18)	1.49(2)	O(22)	B(21)	1.372(6)
C(19)	C(20)	1.494(16)	O(23)	B(21)	1.356(6)
C(20)	C(21)	1.52(2)			

Table S48: Bond Angles in ° for **2018ncs0542**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C(1)	N(1)	C(5)	111.5(15)	C(10)	C(9)	C(8)	109.5(15)
C(1)	N(1)	C(15)	109.5(14)	N(2)	C(10)	C(9)	114.7(15)
C(4)	N(1)	C(1)	103.8(13)	C(12)	C(11)	N(2)	104.9(11)
C(4)	N(1)	C(5)	108.7(14)	C(11)	C(12)	C(13)	103.7(12)
C(4)	N(1)	C(15)	112.3(16)	C(12)	C(13)	C(14)	105.7(10)
C(15)	N(1)	C(5)	110.8(14)	N(2)	C(14)	C(13)	106.4(12)
C(10)	N(2)	C(11)	110.8(14)	N(1)	C(15)	C(16)	112.7(12)
C(10)	N(2)	C(14)	114.8(15)	C(17)	C(16)	C(15)	108.5(12)
C(10)	N(2)	C(19)	112.0(14)	C(18)	C(17)	C(16)	135.9(17)
C(14)	N(2)	C(11)	100.3(12)	C(20)	C(19)	N(2)	116.6(14)
C(14)	N(2)	C(19)	107.7(13)	C(19)	C(20)	C(21)	114.1(15)
C(19)	N(2)	C(11)	110.8(14)	C(22)	C(21)	C(20)	94.5(18)
N(1)	C(1)	C(2)	102.5(12)	B(2)	O(1)	B(1)	123.3(3)
C(1)	C(2)	C(3)	107.2(12)	B(2)	O(2)	B(3)	117.7(3)
C(4)	C(3)	C(2)	102.6(15)	B(3)	O(3)	B(1)	122.8(3)
N(1)	C(4)	C(3)	105.8(13)	B(4)	O(4)	B(1)	122.6(3)
C(6)	C(5)	N(1)	116.6(13)	B(5)	O(5)	B(4)	117.9(3)
C(7)	C(6)	C(5)	108.8(12)	B(5)	O(6)	B(1)	123.2(3)
C(6)	C(7)	C(8)	112.4(11)	O(1)	B(1)	O(3)	110.2(3)
C(7)	C(8)	C(9)	113.8(11)	O(4)	B(1)	O(1)	109.3(4)

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O(4)	B(1)	O(3)	107.6(3)	O(4)	B(4)	O(9)	121.7(4)
O(6)	B(1)	O(1)	108.2(3)	O(9)	B(4)	O(5)	116.5(3)
O(6)	B(1)	O(3)	110.1(4)	O(6)	B(5)	O(5)	121.8(3)
O(6)	B(1)	O(4)	111.4(3)	O(6)	B(5)	O(10)	118.3(3)
O(1)	B(2)	O(2)	122.0(3)	O(10)	B(5)	O(5)	119.8(3)
O(1)	B(2)	O(7)	121.4(3)	O(11)	B(11)	O(12)	124.3(5)
O(7)	B(2)	O(2)	116.6(3)	O(11)	B(11)	O(13)	116.5(5)
O(3)	B(3)	O(2)	121.7(3)	O(13)	B(11)	O(12)	119.3(5)
O(3)	B(3)	O(8)	118.2(3)	O(22)	B(21)	O(21)	118.7(4)
O(8)	B(3)	O(2)	120.0(3)	O(23)	B(21)	O(21)	116.8(5)
O(4)	B(4)	O(5)	121.8(3)	O(23)	B(21)	O(22)	124.4(5)

Table S49: Torsion Angles in ° for **2018ncs0542**.

Atom	Atom	Atom	Atom	Angle/°
N(1)	C(1)	C(2)	C(3)	-21.1(18)
N(1)	C(5)	C(6)	C(7)	166.2(14)
N(1)	C(15)	C(16)	C(17)	179.2(15)
N(2)	C(11)	C(12)	C(13)	35.8(16)
N(2)	C(19)	C(20)	C(21)	162.3(17)
C(1)	N(1)	C(4)	C(3)	-43(2)
C(1)	N(1)	C(5)	C(6)	62.8(19)
C(1)	N(1)	C(15)	C(16)	-174.5(14)
C(1)	C(2)	C(3)	C(4)	-4(2)
C(2)	C(3)	C(4)	N(1)	28(2)
C(4)	N(1)	C(1)	C(2)	38.7(15)
C(4)	N(1)	C(5)	C(6)	176.7(14)
C(4)	N(1)	C(15)	C(16)	70.7(17)
C(5)	N(1)	C(1)	C(2)	155.6(13)
C(5)	N(1)	C(4)	C(3)	-162.0(16)
C(5)	N(1)	C(15)	C(16)	-51(2)
C(5)	C(6)	C(7)	C(8)	175.4(12)
C(6)	C(7)	C(8)	C(9)	173.5(14)
C(7)	C(8)	C(9)	C(10)	176.9(13)
C(8)	C(9)	C(10)	N(2)	175.0(14)
C(10)	N(2)	C(11)	C(12)	-165.7(14)
C(10)	N(2)	C(14)	C(13)	153.5(13)
C(10)	N(2)	C(19)	C(20)	-72(2)
C(11)	N(2)	C(10)	C(9)	164.5(14)
C(11)	N(2)	C(14)	C(13)	34.8(13)
C(11)	N(2)	C(19)	C(20)	51.7(19)
C(11)	C(12)	C(13)	C(14)	-13.4(16)
C(12)	C(13)	C(14)	N(2)	-14.0(15)
C(14)	N(2)	C(10)	C(9)	51.8(19)
C(14)	N(2)	C(11)	C(12)	-44.0(15)
C(14)	N(2)	C(19)	C(20)	160.5(15)
C(15)	N(1)	C(1)	C(2)	-81.4(16)
C(15)	N(1)	C(4)	C(3)	75.0(19)
C(15)	N(1)	C(5)	C(6)	-59(2)
C(15)	C(16)	C(17)	C(18)	-59(3)
C(19)	N(2)	C(10)	C(9)	-71(2)
C(19)	N(2)	C(11)	C(12)	69.5(16)
C(19)	N(2)	C(14)	C(13)	-81.1(14)
C(19)	C(20)	C(21)	C(22)	104(2)
B(1)	O(1)	B(2)	O(2)	7.2(6)
B(1)	O(1)	B(2)	O(7)	-173.8(4)
B(1)	O(3)	B(3)	O(2)	16.1(6)

Atom	Atom	Atom	Atom	Angle/°
B(1)	O(3)	B(3)	O(8)	-166.7(3)
B(1)	O(4)	B(4)	O(5)	12.6(6)
B(1)	O(4)	B(4)	O(9)	-165.8(4)
B(1)	O(6)	B(5)	O(5)	6.8(5)
B(1)	O(6)	B(5)	O(10)	-173.7(3)
B(2)	O(1)	B(1)	O(3)	4.1(5)
B(2)	O(1)	B(1)	O(4)	-114.0(4)
B(2)	O(1)	B(1)	O(6)	124.5(4)
B(2)	O(2)	B(3)	O(3)	-3.6(6)
B(2)	O(2)	B(3)	O(8)	179.3(4)
B(3)	O(2)	B(2)	O(1)	-7.9(6)
B(3)	O(2)	B(2)	O(7)	173.1(4)
B(3)	O(3)	B(1)	O(1)	-15.5(5)
B(3)	O(3)	B(1)	O(4)	103.6(4)
B(3)	O(3)	B(1)	O(6)	-134.8(4)
B(4)	O(4)	B(1)	O(1)	-129.6(4)
B(4)	O(4)	B(1)	O(3)	110.7(4)
B(4)	O(4)	B(1)	O(6)	-10.0(5)
B(4)	O(5)	B(5)	O(6)	-4.8(5)
B(4)	O(5)	B(5)	O(10)	175.7(3)
B(5)	O(5)	B(4)	O(4)	-4.8(5)
B(5)	O(5)	B(4)	O(9)	173.7(3)
B(5)	O(6)	B(1)	O(1)	120.7(4)
B(5)	O(6)	B(1)	O(3)	-118.8(4)
B(5)	O(6)	B(1)	O(4)	0.5(5)

Table S50: Hydrogen Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for **2018ncs0542**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H(1A)	6771.57	8907.12	5022.52	62
H(1B)	5601.44	8515.67	4300.47	62
H(2A)	5268.69	10050.12	4948.67	72
H(2B)	4493.56	9814.08	4047.59	72
H(3A)	5832.91	10784.38	3547.85	99
H(3B)	6526.03	11071.48	4453.21	99
H(4A)	8039.15	10292.61	3578.92	66
H(4B)	8347.33	10139.86	4533.62	66
H(5A)	9173.32	8720.46	3749.56	64
H(5B)	9031.27	8705.93	4665.76	64
H(6A)	7892.19	7393.4	4516.14	80
H(6B)	7635.77	7445.31	3555.81	80
H(7A)	10367.78	7339.89	4564.61	67
H(7B)	10051.91	7318.12	3604.9	67
H(8A)	8801.59	5972.86	3655.37	66
H(8B)	9301.67	5984.83	4609.81	66
H(9A)	11156	5816.94	3504.45	69
H(9B)	11697.74	5877.52	4458.03	69
H(10A)	10535.18	4604.4	4637.31	68
H(10B)	9862.1	4558.51	3700.56	68
H(11A)	11541.73	3265.27	4808.94	61
H(11B)	11044.97	2935.48	3897.86	61
H(12A)	13293.49	2637.34	3771.9	83
H(12B)	13381.4	2382.24	4698.67	83
H(13A)	14929.91	3644.01	4224.17	74
H(13B)	14712.51	3542.78	5131.06	74

Atom	x	y	z	U_{eq}
H(14A)	13195.05	4668.13	4993.78	65
H(14B)	13737.73	4906.11	4190.08	65
H(15A)	5658.12	9417.36	2906.63	59
H(15B)	5982.61	8404.16	3023.67	59
H(16A)	7803.69	9617.78	2436.68	90
H(16B)	8147.36	8604.12	2563.84	90
H(17A)	5868.86	9348.87	1507.42	67
H(17B)	7180.48	8931.2	1251	67
H(18A)	5481.07	8163.26	642.11	121
H(18B)	4715.21	8131.83	1387.34	121
H(18C)	6091.55	7563.46	1399.65	121
H(19A)	12002.84	4798.13	2897.63	56
H(19B)	12965.46	3955.23	3057.44	56
H(20A)	10027.29	3787.27	2654.62	111
H(20B)	11168.94	3063.41	2587.32	111
H(21A)	9876.51	3955.52	1331.48	125
H(21B)	11362.45	4464.1	1567.04	125
H(22A)	11463.66	2678.35	1824.97	195
H(22B)	12588.5	3269.93	1512.44	195
H(22C)	11202.31	2959.96	901.93	195
H(7)	8632.73	7927.24	8140.85	44
H(8)	6322.09	4933.82	8846.97	39
H(9)	7560.55	4889.06	5230.28	46
H(10)	3796.98	7480.37	4552.45	39
H(11)	4610.45	4918.51	6606.37	102
H(12)	4710.46	4172.04	7694.97	60
H(13)	2545.11	2967	6560.67	104
H(21)	8133.53	5042.57	6808.27	102
H(22)	8574.95	3475.36	5698.39	54
H(23)	9623.31	2885.05	6820.58	112

Table S51: Hydrogen Bond information for **2018ncs0542**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
O(7)	H(7)	O(12)[1]	0.84	1.84	2.672(4)	171.2
O(8)	H(8)	O(9)[2]	0.84	1.83	2.653(3)	164.6
O(9)	H(9)	O(22)	0.84	1.90	2.697(4)	158.9
O(10)	H(10)	O(7)[3]	0.84	1.86	2.698(3)	173.0
O(11)	H(11)	O(3)	0.84	1.88	2.708(4)	170.8
O(12)	H(12)	O(8)	0.84	1.89	2.706(4)	165.1
O(13)	H(13)	O(1)[4]	0.84	1.92	2.737(4)	164.0
O(21)	H(21)	O(4)	0.84	1.90	2.740(5)	172.8
O(22)	H(22)	O(10)[5]	0.84	1.92	2.750(4)	167.1
O(23)	H(23)	O(6)[5]	0.84	1.91	2.748(5)	171.8

––––

[1]1/2+x,1/2+y,+z; [2]+x,1-y,1/2+z; [3]-1/2+x,3/2-y,-1/2+z; [4]-1/2+x,-1/2+y,+z; [5]1/2+x,-1/2+y,+z

Table S52: Atomic Occupancies for all atoms that are not fully occupied in **2018ncs0542**.

Atom	Occupancy	Atom	Occupancy
N(1)	0.5	C(20)	0.5
N(2)	0.5	H(20A)	0.5
C(1)	0.5	H(20B)	0.5
H(1A)	0.5	C(21)	0.5
H(1B)	0.5	H(21A)	0.5
C(2)	0.5	H(21B)	0.5
H(2A)	0.5	C(22)	0.5
H(2B)	0.5	H(22A)	0.5
C(3)	0.5	H(22B)	0.5
H(3A)	0.5	H(22C)	0.5
H(3B)	0.5		
C(4)	0.5		
H(4A)	0.5		
H(4B)	0.5		
C(5)	0.5		
H(5A)	0.5		
H(5B)	0.5		
C(6)	0.5		
H(6A)	0.5		
H(6B)	0.5		
C(7)	0.5		
H(7A)	0.5		
H(7B)	0.5		
C(8)	0.5		
H(8A)	0.5		
H(8B)	0.5		
C(9)	0.5		
H(9A)	0.5		
H(9B)	0.5		
C(10)	0.5		
H(10A)	0.5		
H(10B)	0.5		
C(11)	0.5		
H(11A)	0.5		
H(11B)	0.5		
C(12)	0.5		
H(12A)	0.5		
H(12B)	0.5		
C(13)	0.5		
H(13A)	0.5		
H(13B)	0.5		
C(14)	0.5		
H(14A)	0.5		
H(14B)	0.5		
C(15)	0.5		
H(15A)	0.5		
H(15B)	0.5		
C(16)	0.5		
H(16A)	0.5		
H(16B)	0.5		
C(17)	0.5		
H(17A)	0.5		
H(17B)	0.5		
C(18)	0.5		
H(18A)	0.5		
H(18B)	0.5		
H(18C)	0.5		
C(19)	0.5		
H(19A)	0.5		
H(19B)	0.5		
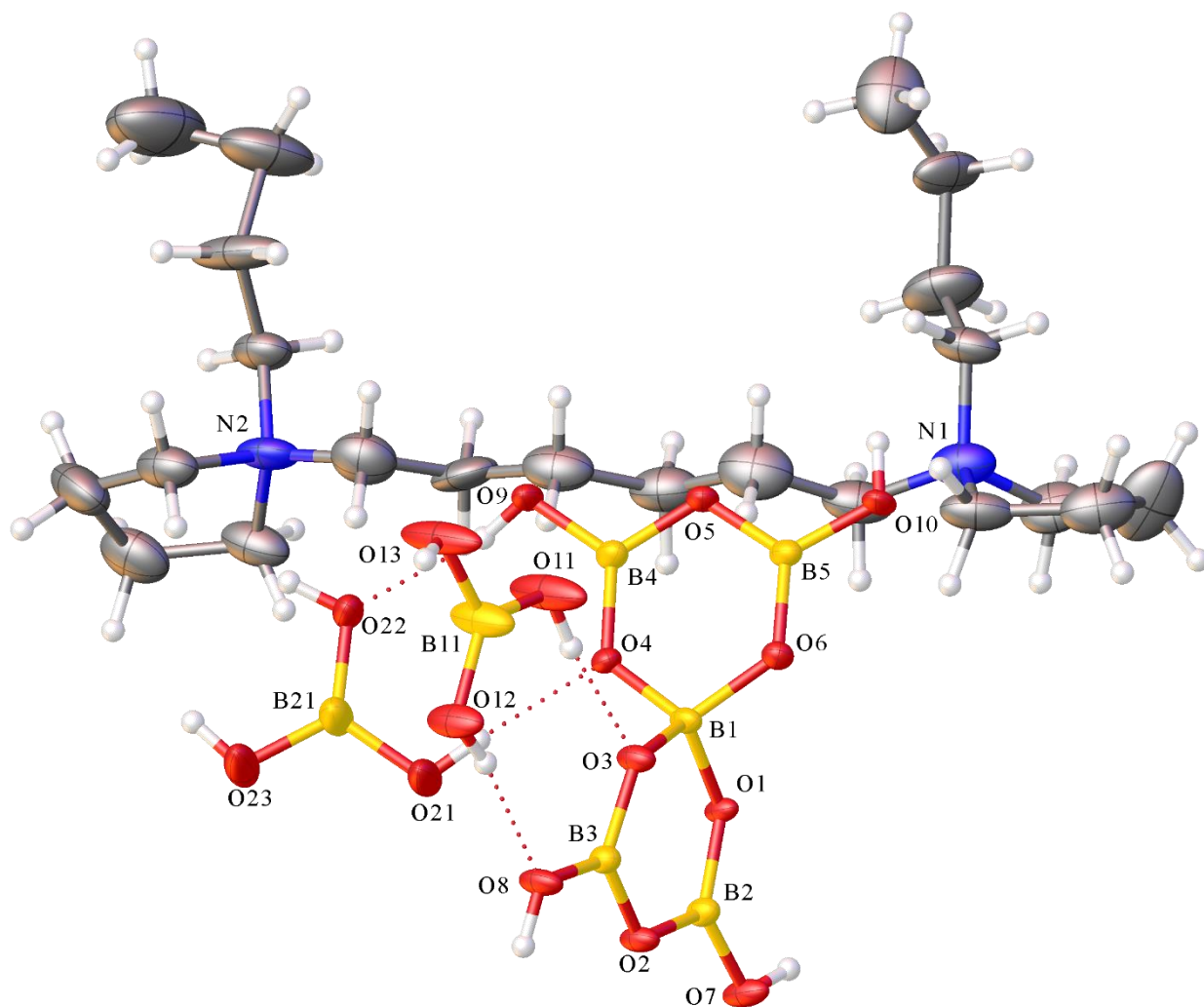
Citations for 6

CrysAlisPro Software System, Rigaku Oxford Diffraction, (2018).

O.V. Dolomanov and L.J. Bourhis and R.J. Gildea and J.A.K. Howard and H. Puschmann, Olex2: A complete structure solution, refinement and analysis program, *J. Appl. Cryst.*, (2009), **42**, 339-341.
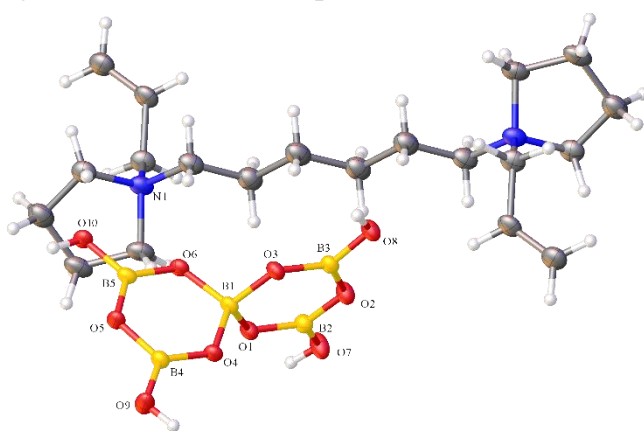
Sheldrick, G.M., Crystal structure refinement with ShelXL, *Acta Cryst.*, (2015), **C27**, 3-8.

Sheldrick, G.M., ShelXT-Integrated space-group and crystal-structure determination, *Acta Cryst.*, (2015), **A71**, 3-8.

Submitted by: **Mike A Beckett**

Bangor University

Solved by: **Peter N Horton**

Sample ID: **MAB/TR/AR102 B5**

$[C_3H_5(C_4H_8N)(CH_2)_6(C_4H_8N)C_3H_5][B_5O_6(OH)_4]_2$ (**7**).

Crystal Data and Experimental

Experimental. Single colourless plate-shaped crystals of **2018ncs0572** were recrystallised from water. A suitable crystal 0.230×0.140×0.010 mm³ was selected and mounted on a MITIGEN holder in perfluoroether oil on a Rigaku FRE+ diffractometer equipped with VHF Varimax confocal mirrors and an AFC12 goniometer and HyPix 6000 detector. The crystal was kept at a steady $T = 100(2)$ K during data collection. The structure was solved with the **ShelXT** (Sheldrick, 2015) structure solution program using the Intrinsic Phasing solution method and by using **Olex2** (Dolomanov et al., 2009) as the graphical interface. The model was refined with version 2018/3 of **ShelXL** (Sheldrick, 2015) using Least Squares minimisation.

Crystal Data. $C_{20}H_{46}B_{10}N_2O_{20}$, $M_r = 742.69$, monoclinic, $P2_1/c$ (No. 14), a = 10.0823(5) Å, b = 11.6950(4) Å, c = 15.7836(6) Å, β = 105.717(5)°, $\alpha = \gamma = 90$°, $V = 1791.50(13)$ Å³, $T = 100(2)$ K, $Z = 2$, $Z' = 0.5$, $\mu(MoK_\alpha) = 0.113$ mm⁻¹, 22100 reflections measured, 4090 unique ($R_{int} = 0.0653$) which were used in all calculations. The final wR_2 was 0.1324 (all data) and R_1 was 0.0449 (I > 2(I)).

Compound	2018ncs0572
Formula	$C_{20}H_{46}B_{10}N_2O_{20}$
$D_{calc.}$/ g cm⁻³	1.377
μ/mm⁻¹	0.113
Formula Weight	742.69
Colour	colourless
Shape	plate
Size/mm³	0.230×0.140×0.010
T/K	100(2)
Crystal System	monoclinic
Space Group	$P2_1/c$
a/Å	10.0823(5)
b/Å	11.6950(4)
c/Å	15.7836(6)
α/°	90
β/°	105.717(5)
γ/°	90
V/Å³	1791.50(13)
Z	2
Z'	0.5
Wavelength/Å	0.71075
Radiation type	MoK$_\alpha$
Θ_{min}/°	2.098
Θ_{max}/°	27.485
Measured Refl.	22100
Independent Refl.	4090
Reflections with I > 2(I)	3129
R_{int}	0.0653
Parameters	247
Restraints	0
Largest Peak	0.279
Deepest Hole	-0.310
GooF	1.039
wR_2 (all data)	0.1324
wR_2	0.1219
R_1 (all data)	0.0611
R_1	0.0449

Structure Quality Indicators

Reflections:

d min (Mo)	I/σ	Rint	complete 100% (IUCr)
0.77	23.1	6.53%	100%

Refinement:

Shift	Max Peak	Min Peak	GooF
0.001	0.3	-0.3	1.039

A colourless plate-shaped crystal with dimensions $0.230 \times 0.140 \times 0.010$ mm^3 was mounted on a MITIGEN holder in perfluoroether oil. Data were collected using an Rigaku FRE+ diffractometer equipped with VHF Varimax confocal mirrors and an AFC12 goniometer and HyPix 6000 detector equipped with an Oxford Cryosystems low-temperature device operating at T = 100(2) K.

Data were measured using profile data from ω-scans 0.5° per frame for 9.0 s using MoK$_\alpha$ radiation. The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.39.46, 2018). The maximum resolution that was achieved was Θ = 27.485° (0.77 Å).

The diffraction pattern was indexed. The total number of runs and images was based on the strategy calculation from the program **CrysAlisPro** (Rigaku, V1.171.39.46, 2018) and the unit cell was refined using **CrysAlisPro** (Rigaku, V1.171.39.46, 2018) on 6300 reflections, 29% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using **CrysAlisPro** (Rigaku, V1.171.39.46, 2018). The final completeness is 99.90 % out to 27.485° in Θ. A multi-scan absorption correction was performed using CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018) using spherical harmonicsas implemented in SCALE3 ABSPACK.. The absorption coefficient μ of this material is 0.113 mm^{-1} at this wavelength (λ = 0.71075 Å) and the minimum and maximum transmissions are 0.132 and 1.000.

The structure was solved and the space group $P2_1/c$ (# 14) determined by the **ShelXT** (Sheldrick, 2015) structure solution program using Intrinsic Phasing and refined by Least Squares using version 2018/3 of **ShelXL** (Sheldrick, 2015). All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model. Most hydrogen atom positions were calculated geometrically and refined using the riding model, but some hydrogen atoms were refined freely.

_exptl_absorpt_process_details_: CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018) using spherical harmonicsas implemented in SCALE3 ABSPACK.

The value of Z' is 0.5. This means that only half of the formula unit is present in the asymmetric unit, with the other half consisting of symmetry equivalent atoms.
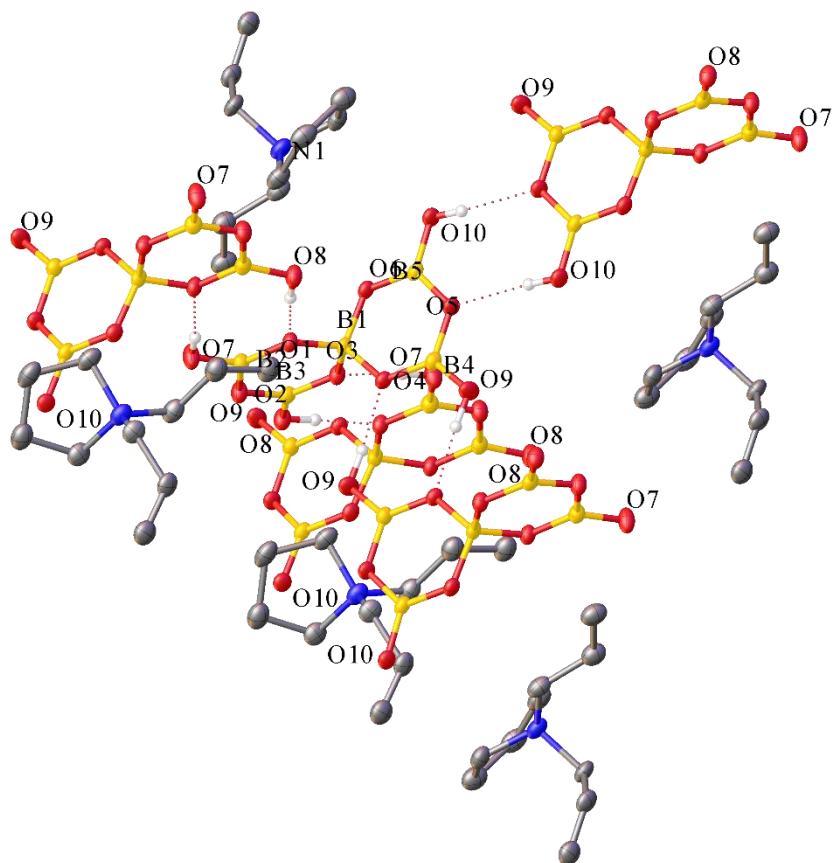
Figure S42: The following hydrogen bonding interactions with a maximum D-D distance of 2.82 Å and a minimum angle of 120 ° are present in **2018ncs0572**: O10–O5_*3: 2.798 Å, O8–O1_*1: 2.75 Å, O9–O4_*4: 2.733 Å, *O7–O3_*2: 2.728 Å.
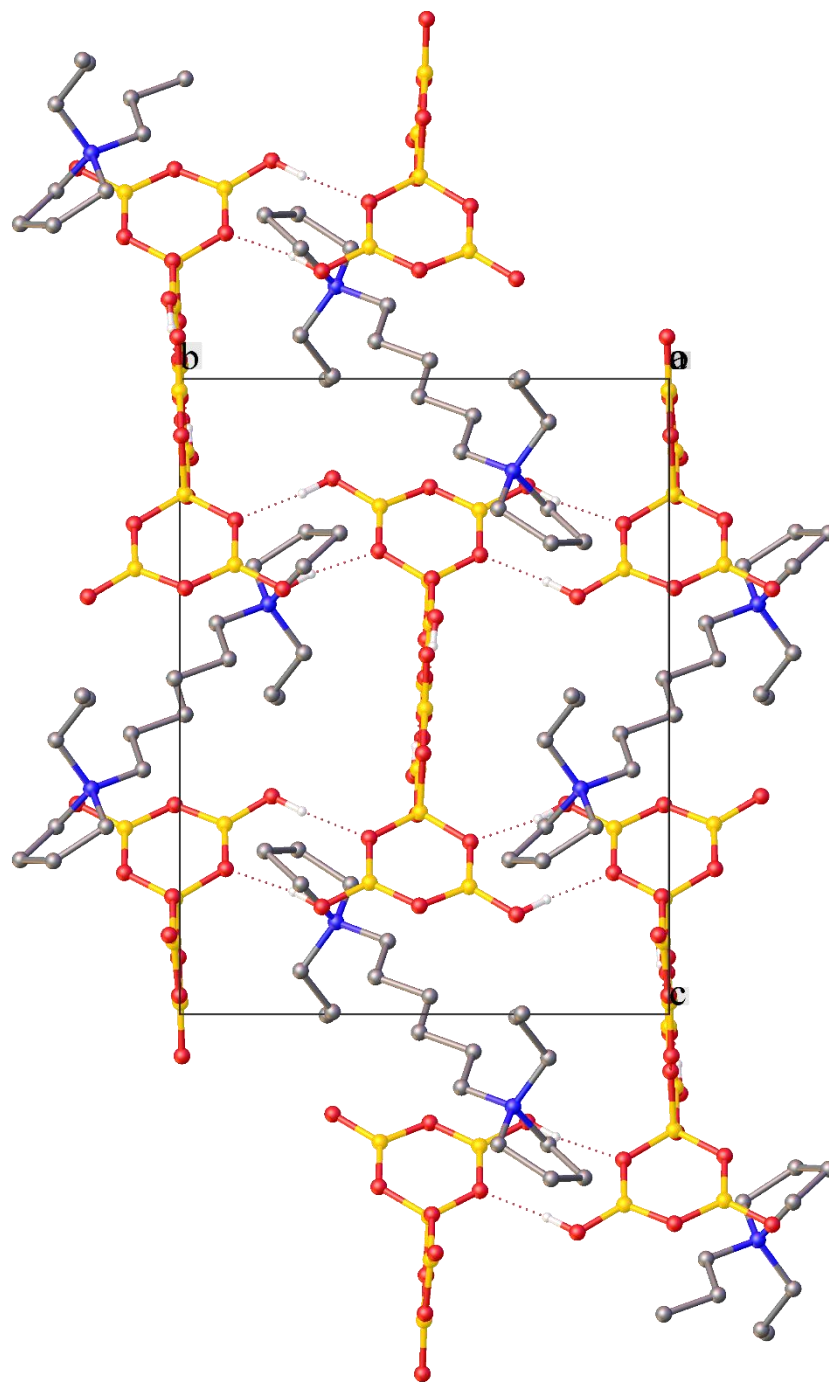
Figure S43: Packing diagram of 2018ncs0572.

Reflection Statistics

Total reflections (after filtering)	22675	Unique reflections	4090
Completeness	0.997	Mean I/σ	13.35
hkl$_{max}$ collected	(13, 14, 19)	hkl$_{min}$ collected	(-12, -15, -20)
hkl$_{max}$ used	(12, 15, 20)	hkl$_{min}$ used	(-13, 0, 0)
Lim d$_{max}$ collected	100.0	Lim d$_{min}$ collected	0.36
d$_{max}$ used	9.71	d$_{min}$ used	0.77
Friedel pairs	5291	Friedel pairs merged	1
Inconsistent equivalents	3	R$_{int}$	0.0653
R$_{sigma}$	0.0433	Intensity transformed	0
Omitted reflections	0	Omitted by user (OMIT hkl)	0
Multiplicity	(6399, 4903, 1564, 417, 22)	Maximum multiplicity	11
Removed systematic absences	575	Filtered off (Shel/OMIT)	0

Table S53: Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **2018ncs0572**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
N(1)	8784.6(14)	1808.1(10)	6451.4(8)	25.3(3)
C(1)	8171.4(18)	2530.6(13)	7046.7(11)	30.5(4)
C(2)	9354.5(19)	3261.5(14)	7568.7(11)	35.1(4)
C(3)	10673.4(19)	2586.6(15)	7587.7(12)	36.7(4)
C(4)	10183.9(17)	1509.9(14)	7046.2(10)	29.0(3)
C(5)	7942.6(17)	753.7(13)	6149.9(11)	29.4(4)
C(6)	6502.4(18)	997.4(14)	5587.6(11)	33.7(4)
C(7)	5727(2)	-108.3(14)	5283.5(12)	34.8(4)
C(8)	8934.3(17)	2534.1(12)	5683.9(10)	27.1(3)
C(9)	9481.8(18)	1880.5(14)	5038.4(10)	28.9(3)
C(10)	10749.2(18)	1992.3(15)	4989.3(11)	33.7(4)
O(1)	5132.9(10)	1132.6(8)	7772.5(6)	19.2(2)
O(2)	3412.9(11)	107.0(8)	6707.3(7)	23.5(2)
O(3)	5198.9(10)	-934.5(8)	7711.7(6)	19.6(2)
O(4)	5767.2(10)	30.8(7)	9104.3(7)	18.6(2)
O(5)	8204.2(11)	12.0(8)	9702.3(7)	21.0(2)
O(6)	7279.0(10)	150.3(8)	8147.1(6)	19.2(2)
O(7)	3398.3(11)	2101.3(9)	6691.9(7)	28.7(3)
O(8)	3527.3(11)	-1876.5(8)	6569.9(7)	23.6(2)
O(9)	6816.3(12)	-43.4(10)	10654.5(7)	28.9(3)
O(10)	9654.0(11)	220.7(9)	8753.5(7)	22.1(2)
B(1)	5851.4(17)	94.8(12)	8180.6(10)	17.6(3)
B(2)	4001.2(17)	1123.7(13)	7072.1(11)	20.8(3)
B(3)	4065.8(17)	-913.0(13)	7008.4(10)	19.2(3)
B(4)	6877.5(17)	-9.3(12)	9809.6(11)	19.2(3)
B(5)	8366.0(17)	128.7(12)	8858.0(10)	17.7(3)

Table S54: Anisotropic Displacement Parameters (×10⁴) **2018ncs0572**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + ... + 2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N(1)	36.0(7)	23.9(6)	19.0(6)	2.0(5)	12.6(6)	-0.6(5)
C(1)	45.2(10)	28.8(8)	22.5(8)	0.5(6)	18.0(7)	1.0(6)
C(2)	53.8(11)	32.5(8)	24.0(8)	-4.9(6)	19.0(8)	-6.1(7)
C(3)	45.4(11)	42.3(10)	24.0(8)	-3.1(7)	12.4(8)	-7.7(7)
C(4)	37.3(9)	31.9(8)	18.9(7)	5.0(6)	9.5(7)	1.6(6)
C(5)	41.1(10)	25.7(7)	24.6(8)	-0.1(6)	14.5(7)	-4.6(6)
C(6)	40.1(10)	36.7(9)	26.4(9)	-0.8(7)	12.6(8)	-4.6(7)
C(7)	43.1(10)	37.8(9)	27.0(9)	-2.7(7)	15.3(8)	-7.5(7)
C(8)	37.1(9)	27.5(8)	18.7(8)	7.0(6)	11.0(7)	-0.1(6)
C(9)	38.5(9)	34.7(8)	13.8(7)	0.8(6)	7.5(6)	-2.6(6)
C(10)	39.7(10)	43.0(9)	18.7(8)	0.8(6)	8.6(7)	0.2(7)
O(1)	23.4(5)	18.3(5)	14.2(5)	0.7(3)	2.0(4)	0.8(4)
O(2)	26.2(6)	20.6(5)	19.6(5)	1.5(4)	-0.9(4)	-0.6(4)
O(3)	24.0(5)	19.4(5)	13.5(5)	-0.8(4)	1.7(4)	0.0(4)
O(4)	22.0(5)	22.6(5)	11.9(5)	0.3(3)	5.8(4)	0.1(3)
O(5)	21.2(6)	31.9(5)	10.4(5)	0.9(4)	4.9(4)	-0.5(4)
O(6)	23.4(5)	23.3(5)	10.9(5)	1.3(3)	5.1(4)	0.5(4)
O(7)	31.1(6)	20.5(5)	26.6(6)	2.2(4)	-5.7(5)	-0.1(4)
O(8)	28.7(6)	20.4(5)	18.1(5)	-0.5(4)	0.1(4)	-1.3(4)
O(9)	23.4(6)	51.6(7)	12.7(5)	0.7(4)	6.5(5)	-2.5(4)
O(10)	23.1(6)	32.3(5)	11.5(5)	1.2(4)	5.6(4)	0.9(4)
B(1)	22.4(8)	19.9(7)	9.8(7)	0.7(5)	3.1(6)	0.3(5)
B(2)	22.5(8)	22.6(7)	16.5(8)	1.5(6)	4.0(7)	0.9(6)
B(3)	22.4(8)	22.2(7)	13.1(7)	0.7(5)	4.6(6)	-1.3(6)
B(4)	22.2(9)	21.8(7)	13.2(8)	0.4(5)	4.2(6)	-0.5(5)
B(5)	24.8(8)	17.0(7)	11.9(7)	0.7(5)	5.7(6)	0.9(5)

Table S55: Bond Lengths in Å for **2018ncs0572**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N(1)	C(1)	1.5145(18)	O(2)	B(3)	1.3830(18)
N(1)	C(4)	1.509(2)	O(3)	B(1)	1.4717(17)
N(1)	C(5)	1.4996(19)	O(3)	B(3)	1.3604(19)
N(1)	C(8)	1.5199(17)	O(4)	B(1)	1.4850(17)
C(1)	C(2)	1.516(2)	O(4)	B(4)	1.349(2)
C(2)	C(3)	1.540(3)	O(5)	B(4)	1.3933(19)
C(3)	C(4)	1.527(2)	O(5)	B(5)	1.3924(18)
C(5)	C(6)	1.510(3)	O(6)	B(1)	1.4560(19)
C(6)	C(7)	1.520(2)	O(6)	B(5)	1.340(2)
C(7)	C(7)[1]	1.518(4)	O(7)	B(2)	1.3562(18)
C(8)	C(9)	1.494(2)	O(8)	B(3)	1.3565(18)
C(9)	C(10)	1.308(2)	O(9)	B(4)	1.3521(19)
O(1)	B(1)	1.4697(17)	O(10)	B(5)	1.3569(19)
O(1)	B(2)	1.357(2)	----		
O(2)	B(2)	1.3828(18)	[1]1-x,-y,1-z		

Table S56: Bond Angles in ° for **2018ncs0572**.

Atom	Atom	Atom	Angle/°
C(1)	N(1)	C(8)	109.15(11)
C(4)	N(1)	C(1)	102.30(12)
C(4)	N(1)	C(8)	110.31(11)
C(5)	N(1)	C(1)	111.38(12)
C(5)	N(1)	C(4)	111.16(12)
C(5)	N(1)	C(8)	112.11(12)
N(1)	C(1)	C(2)	104.97(13)
C(1)	C(2)	C(3)	105.65(13)
C(4)	C(3)	C(2)	105.39(14)
N(1)	C(4)	C(3)	105.29(13)
N(1)	C(5)	C(6)	113.76(13)
C(5)	C(6)	C(7)	110.83(14)
C(7)[1]	C(7)	C(6)	112.10(18)
C(9)	C(8)	N(1)	113.11(12)
C(10)	C(9)	C(8)	122.47(15)
B(2)	O(1)	B(1)	123.83(11)
B(2)	O(2)	B(3)	119.21(12)
B(3)	O(3)	B(1)	123.87(11)
B(4)	O(4)	B(1)	123.82(12)
B(5)	O(5)	B(4)	118.90(12)
B(5)	O(6)	B(1)	124.14(11)

Atom	Atom	Atom	Angle/°
O(1)	B(1)	O(3)	110.79(12)
O(1)	B(1)	O(4)	108.63(11)
O(3)	B(1)	O(4)	108.41(10)
O(6)	B(1)	O(1)	108.78(11)
O(6)	B(1)	O(3)	109.26(11)
O(6)	B(1)	O(4)	110.97(12)
O(1)	B(2)	O(2)	121.14(12)
O(7)	B(2)	O(1)	122.09(13)
O(7)	B(2)	O(2)	116.77(14)
O(3)	B(3)	O(2)	120.85(12)
O(8)	B(3)	O(2)	116.98(13)
O(8)	B(3)	O(3)	122.18(13)
O(4)	B(4)	O(5)	120.56(13)
O(4)	B(4)	O(9)	124.44(14)
O(9)	B(4)	O(5)	114.97(14)
O(6)	B(5)	O(5)	121.48(13)
O(6)	B(5)	O(10)	119.30(13)
O(10)	B(5)	O(5)	119.22(14)

––––
[1]1-x,-y,1-z

Table S57: Torsion Angles in ° for **2018ncs0572**.

Atom	Atom	Atom	Atom	Angle/°
N(1)	C(1)	C(2)	C(3)	-25.57(16)
N(1)	C(5)	C(6)	C(7)	178.23(12)
N(1)	C(8)	C(9)	C(10)	-105.08(18)
C(1)	N(1)	C(4)	C(3)	-38.34(14)
C(1)	N(1)	C(5)	C(6)	63.76(16)
C(1)	N(1)	C(8)	C(9)	-177.72(14)
C(1)	C(2)	C(3)	C(4)	1.80(17)
C(2)	C(3)	C(4)	N(1)	22.73(15)
C(4)	N(1)	C(1)	C(2)	39.51(14)
C(4)	N(1)	C(5)	C(6)	177.14(12)
C(4)	N(1)	C(8)	C(9)	70.63(16)
C(5)	N(1)	C(1)	C(2)	158.34(13)
C(5)	N(1)	C(4)	C(3)	-157.32(12)
C(5)	N(1)	C(8)	C(9)	-53.83(17)
C(5)	C(6)	C(7)	C(7)[1]	179.97(17)
C(8)	N(1)	C(1)	C(2)	-77.35(15)
C(8)	N(1)	C(4)	C(3)	77.68(14)
C(8)	N(1)	C(5)	C(6)	-58.87(16)
B(1)	O(1)	B(2)	O(2)	2.1(2)
B(1)	O(1)	B(2)	O(7)	-177.35(13)
B(1)	O(3)	B(3)	O(2)	-2.6(2)
B(1)	O(3)	B(3)	O(8)	177.17(12)
B(1)	O(4)	B(4)	O(5)	0.23(19)
B(1)	O(4)	B(4)	O(9)	178.54(12)
B(1)	O(6)	B(5)	O(5)	2.11(19)
B(1)	O(6)	B(5)	O(10)	-178.00(11)
B(2)	O(1)	B(1)	O(3)	1.55(18)
B(2)	O(1)	B(1)	O(4)	-117.42(14)
B(2)	O(1)	B(1)	O(6)	121.67(13)
B(2)	O(2)	B(3)	O(3)	6.4(2)

| Atom	Atom	Atom	Atom	Angle/°
B(2)	O(2)	B(3)	O(8)	-173.37(12)
B(3)	O(2)	B(2)	O(1)	-6.2(2)
B(3)	O(2)	B(2)	O(7)	173.32(13)
B(3)	O(3)	B(1)	O(1)	-1.32(18)
B(3)	O(3)	B(1)	O(4)	117.79(13)
B(3)	O(3)	B(1)	O(6)	-121.16(13)
B(4)	O(4)	B(1)	O(1)	-121.64(13)
B(4)	O(4)	B(1)	O(3)	117.90(13)
B(4)	O(4)	B(1)	O(6)	-2.10(16)
B(4)	O(5)	B(5)	O(6)	-4.12(18)
B(4)	O(5)	B(5)	O(10)	175.99(12)
B(5)	O(5)	B(4)	O(4)	2.94(18)
B(5)	O(5)	B(4)	O(9)	-175.53(11)
B(5)	O(6)	B(1)	O(1)	120.38(13)
B(5)	O(6)	B(1)	O(3)	-118.56(13)
B(5)	O(6)	B(1)	O(4)	0.93(16)

––––

[1]1-x,-y,1-z

Table S58: Hydrogen Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{Å}^2 \times 10^3$) for **2018ncs0572**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H(1A)	7806.48	2041.43	7443.64	37
H(1B)	7415.34	3015.2	6695.93	37
H(2A)	9276.28	3388.39	8173.44	42
H(2B)	9359.64	4012.82	7280.67	42
H(3A)	11289.27	3041.36	7324.9	44
H(3B)	11177.16	2385.65	8199.55	44
H(4A)	10827.75	1302.42	6696.05	35
H(4B)	10116.67	859.66	7434.12	35
H(5A)	7882.37	311.33	6672.98	35
H(5B)	8422.51	271.67	5810.09	35
H(6A)	6550.47	1453.53	5068.35	40
H(6B)	5999.72	1451.77	5930.16	40
H(7A)	6235.15	-561.13	4943.52	42
H(7B)	5685.77	-563.32	5804.73	42
H(8A)	8023.31	2856.32	5375.87	33
H(8B)	9561.51	3180.91	5914.84	33
H(9)	8886.71	1364.34	4648.92	35
H(10A)	11359.72	2504.27	5373.26	40
H(10B)	11059.54	1561.54	4569.78	40
H(7)	3820(20)	2677(18)	6929(15)	43
H(8)	3990(20)	-2494(16)	6806(14)	35
H(9A)	5920(30)	-42(16)	10706(16)	43
H(10)	10220(20)	172(15)	9213(16)	33

Table S59: Hydrogen Bond information for **2018ncs0572**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
O(7)	H(7)	O(3)[1]	0.83(2)	1.90(2)	2.7280(14)	171(2)
O(8)	H(8)	O(1)[2]	0.89(2)	1.87(2)	2.7502(14)	175(2)
O(9)	H(9A)	O(4)[3]	0.93(2)	1.81(2)	2.7326(15)	176(2)
O(10)	H(10)	O(5)[4]	0.80(3)	2.01(3)	2.7975(16)	174(2)

––––

[1]1-x,1/2+y,3/2-z; [2]1-x,-1/2+y,3/2-z; [3]1-x,-y,2-z; [4]2-x,-y,2-z
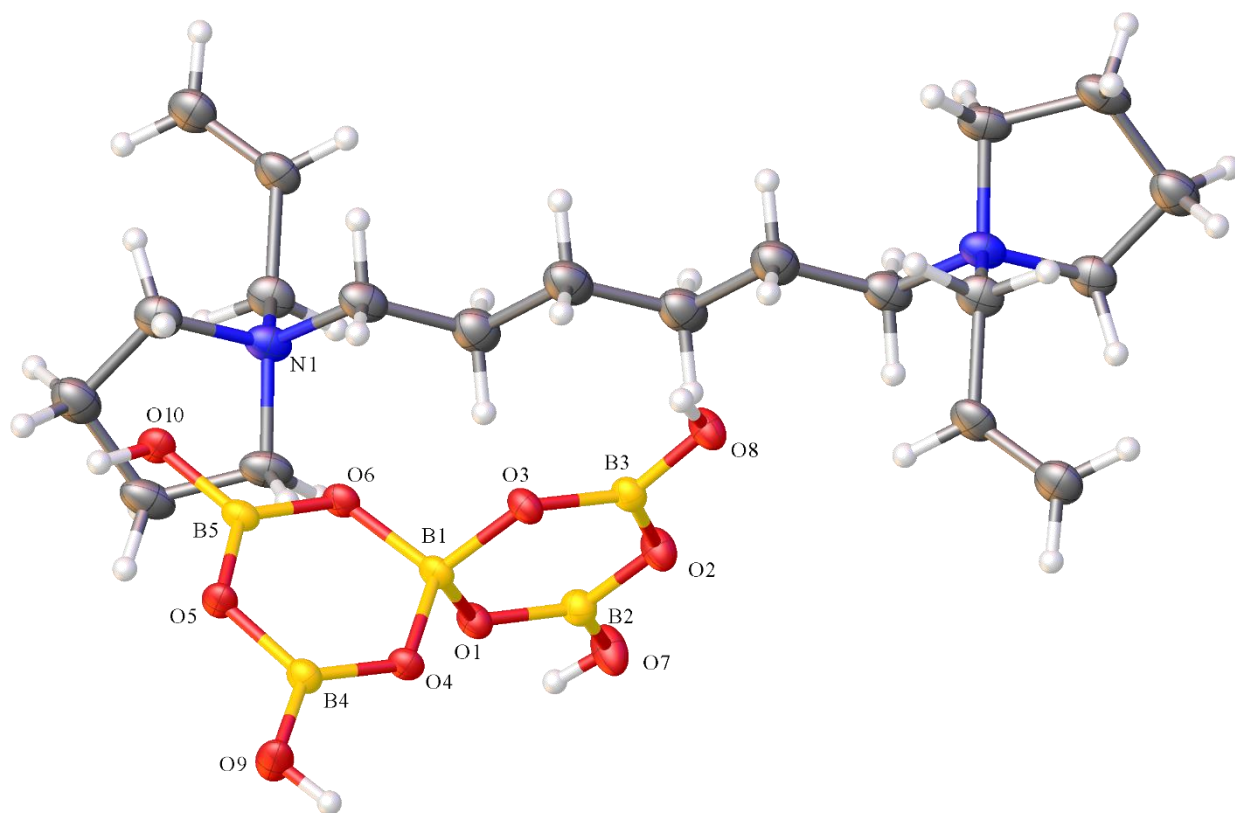
Citations for 7

CrysAlisPro Software System, Rigaku Oxford Diffraction, (2018).

O.V. Dolomanov and L.J. Bourhis and R.J. Gildea and J.A.K. Howard and H. Puschmann, Olex2: A complete structure solution, refinement and analysis program, *J. Appl. Cryst.*, (2009), **42**, 339-341.

Sheldrick, G.M., Crystal structure refinement with ShelXL, *Acta Cryst.*, (2015), **C27**, 3-8.

Sheldrick, G.M., ShelXT-Integrated space-group and crystal-structure determination, *Acta Cryst.*, (2015), **A71**, 3-8.