Supporting Information

Crystalline Structure of an Ammonia Borane – Polyethylene Oxide Cocrystal: A Material Investigated for its Hydrogen Storage Potential

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Single crystal X-ray diffraction measurements and analysis

A single crystal of the prepared cocrystal was held in a Hampton Research nylon loop (20 µm thickness, 0.9 mm diameter, shown in Figure S1). The sample was cooled to 150 K and measurements were made using a twin-source SuperNova diffractometer with a micro-focus Cu X-ray beam (50 kV, 0.8 mA), an Atlas (135 mm CCD) detector, and the sample temperature was controlled with an Oxford Instruments Cryojet5. Full spheres of data were collected. The data were processed with the CrysAlisPro software package (version 1.171.38.43) from Rigaku Oxford Diffraction. Crystal structures of each phase were solved and refined by least-squares within the Olex2 program suite[1] using the ShelXS structure solution program and the ShelXL 2014 refinement program[2].

[1] O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard, H. Puschmann, *J. Appl. Crystallogr*. 2009, **42**, 339-341.

[2] G. M. Sheldrick, *Acta Crystallogr.* 2015, **C71**, 3-8.

Table S1: Crystal data and structure refinement for $C_{10}H_{20}O_5 \cdot NH_3BH_3$.

Identification code	xstr0748
Empirical formula	$C_{10}H_{26}BNO_5$
Formula weight	251.13
Temperature / K	150
Crystal system	monoclinic
Space group	$P2_1/n$
a / Å	8.42518(12)
b / Å	8.39871(16)
c / Å	20.2469(3)
α / °	90
β / °	91.5852(13)
γ / °	90
Volume / Å^3	1432.13(4)
Z	4
ρ_{calc} / g cm^{-3}	1.165
μ / mm^{-1}	0.740
F(000)	552.0
Crystal size / mm^3	0.317 × 0.106 × 0.024
Radiation	CuKα (λ = 1.54184)
2θ range for data collection / °	8.738 to 148.816
Index ranges	$-10 \le h \le 10$, $-9 \le k \le 10$, $-25 \le l \le 24$
Reflections collected	21539
Independent reflections	2909 [R_{int} = 0.0257, R_{sigma} = 0.0134]
Data/restraints/parameters	2909/0/156
Goodness-of-fit on F^2	1.030
Final R indexes [I >= 2σ(I)]	R_1 = 0.0587, wR_2 = 0.1684
Final R indexes [all data]	R_1 = 0.0628, wR_2 = 0.1726
Largest diff. peak/hole / e Å^{-3}	0.82/−0.34

Table S2: Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for $C_{10}H_{20}O_5 \cdot NH_3BH_3$. U_{eq} is defined as ⅓ of the trace of the orthogonalised U_{ij} tensor

Atom	x	y	z	U**(eq)**
O(1)	5830.3(15)	4620.2(16)	3491.2(6)	42.6(3)
O(2)	3265.1(14)	5713.3(15)	4294.7(6)	39.7(3)
O(3)	1537.8(15)	8487.6(17)	3956.8(6)	44.3(3)
O(4)	2831.8(14)	10857.8(16)	3146.8(6)	40.4(3)
O(5)	5467.8(17)	9992.6(16)	2350.7(8)	49.9(4)
C(1)	7492(2)	4496(3)	3417.7(10)	47.5(5)
C(2)	5342(3)	3894(3)	4094.4(9)	51.0(5)

C(3)	3620(3)	4086(2)	4170.8(10)	49.9(5)
C(4)	1653(2)	5931(3)	4443.9(10)	48.4(5)
C(5)	1324(2)	7651(3)	4557.4(10)	51.6(5)
C(6)	1077(2)	10121(3)	3986.1(11)	52.1(5)
C(7)	1213(2)	10827(2)	3312.4(11)	48.6(5)
C(8)	3082(2)	11460(2)	2496.8(9)	45.2(5)
C(9)	4813(2)	11545(2)	2388.6(9)	43.3(4)
C(10)	7141(2)	10033(3)	2263.1(11)	52.5(5)
N(1)	5162.6(19)	8321(2)	3675.5(10)	52.2(5)
B(1)	6739(3)	8871(4)	4064.8(16)	65.3(8)
H(1A)	7839	3380	3484	57
H(1B)	8056	5172	3749	57
H(2A)	5917	4393	4474	61
H(2B)	5613	2747	4090	61
H(3A)	3048	3729	3763	60
H(3B)	3265	3421	4543	60
H(4A)	1401	5314	4844	58
H(4B)	970	5535	4073	58
H(5A)	222	7791	4705	62
H(5B)	2057	8072	4906	62
H(6A)	1775	10700	4306	63
H(6B)	−31	10207	4133	63
H(7A)	599	10181	2986	58
H(7B)	776	11921	3307	58
H(8A)	2605	12532	2450	54
H(8B)	2570	10750	2164	54
H(9A)	5006	12128	1973	52
H(9B)	5339	12137	2757	52
H(10A)	7679	9329	2591	63
H(10B)	7540	11131	2334	63
H(1C)	4299	8643	3900	78
H(1D)	5155	7242	3638	78
H(1E)	5134	8766	3265	78
H(1F)	6860	10027	4025	98
H(1G)	7659	8340	3877	98
H(1H)	6667	8584	4532	98

Table S3: Anisotropic displacement parameters (Å^2×10^3) for $C_{10}H_{20}O_5{\cdot}NH_3BH_3$. Anisotropic displacement factor exponent has the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	44.7(7)	43.7(7)	39.4(7)	-3.0(5)	−0.7(5)	2.8(5)
O(2)	39.0(7)	39.0(7)	41.4(7)	−1.5(5)	3.5(5)	−8.1(5)
O(3)	38.7(7)	49.8(8)	44.7(7)	−12.0(6)	6.0(5)	−1.0(5)
O(4)	32.3(6)	43.2(7)	45.2(7)	−2.6(5)	−6.7(5)	4.2(5)
O(5)	42.8(7)	40.9(8)	66.1(9)	1.1(6)	5.5(6)	0.1(6)
C(1)	43.4(10)	50.3(11)	48.6(11)	−4.2(8)	−3.6(8)	4.5(8)
C(2)	69.6(13)	46.9(11)	36.6(9)	4.1(8)	3.5(9)	11.5(10)
C(3)	68.4(13)	38.0(10)	43.6(10)	0.3(8)	7.9(9)	−10.7(9)
C(4)	38.5(9)	64.7(13)	42.2(10)	3.5(9)	4.0(7)	−15.9(9)
C(5)	33.2(9)	78.5(15)	43.4(10)	−11.5(10)	6.9(7)	−0.8(9)
C(6)	31.3(9)	55.4(12)	70.0(13)	−27.5(10)	8.1(8)	−2.8(8)
C(7)	31.4(9)	42.5(10)	71.4(13)	−14.4(9)	−7.1(8)	4.8(7)
C(8)	52.8(11)	38.8(10)	43.1(10)	0.3(7)	−11.9(8)	9.6(8)
C(9)	54.6(11)	35.1(9)	40.1(9)	2.4(7)	−2.5(8)	0.1(8)
C(10)	41.8(10)	58.4(13)	57.2(12)	−0.7(10)	2.2(8)	3.0(9)
N(1)	34.8(8)	46.7(10)	74.7(12)	19.3(8)	−4.1(7)	−4.5(7)
B(1)	57.8(15)	55.1(15)	81.4(19)	10.3(14)	−26.1(13)	−13.7(12)

Table S4: Bond Lengths for $C_{10}H_{20}O_5{\cdot}NH_3BH_3$

Atom	—	Atom	Length / Å	Atom	—	Atom	Length / Å
O(1)	—	C(1)	1.416(2)	O(5)	—	C(10)	1.426(2)
O(1)	—	C(2)	1.435(2)	C(1)	—	C(10)^1	1.491(3)
O(2)	—	C(3)	1.423(2)	C(2)	—	C(3)	1.472(3)
O(2)	—	C(4)	1.412(2)	C(4)	—	C(5)	1.490(3)
O(3)	—	C(5)	1.420(3)	C(6)	—	C(7)	1.495(3)
O(3)	—	C(6)	1.427(3)	C(8)	—	C(9)	1.482(3)
O(4)	—	C(7)	1.414(2)	C(10)	—	C(1)^2	1.491(3)
O(4)	—	C(8)	1.431(2)	N(1)	—	B(1)	1.594(3)
O(5)	—	C(9)	1.419(2)				

$^1x, y, 1½–z$ and ^{2} x, y, ½–z

Table S5: Selected bond angles for $C_{10}H_{20}O_5 \cdot NH_3BH_3$

Atom — Atom — Atom	Angle / ˚
C(1) — O(1) — C(2)	111.43(15)
C(4) — O(2) — C(3)	111.73(15)
C(5) — O(3) — C(6)	113.49(16)
C(7) — O(4) — C(8)	113.10(15)
C(9) — O(5) — C(10)	111.82(15)
O(1) — C(1) — C(10)[1]	107.79(16)
O(1) — C(2) — C(3)	110.44(16)
O(2) — C(3) — C(2)	109.62(16)
O(2) — C(4) — C(5)	110.05(15)
O(3) — C(5) — C(4)	108.62(15)
O(3) — C(6) — C(7)	108.39(15)
O(4) — C(7) — C(6)	108.81(15)
O(4) — C(8) — C(9)	108.89(14)
O(5) — C(9) — C(8)	110.42(15)
O(5) — C(10) — C(1)[2]	109.79(17)

Table S6: Selected torsion angles for $C_{10}H_{20}O_5 \cdot NH_3BH_3$

A	B	C	D	Angle/˚
O(1)	C(2)	C(3)	O(2)	−69.5(2)
O(2)	C(4)	C(5)	O(3)	65.15(19)
O(3)	C(6)	C(7)	O(4)	−66.42(19)
O(4)	C(8)	C(9)	O(5)	68.79(19)
O(5)	C(10)	C(1)[1]	O(1)[1]	177.56(16)
C(1)	O(1)	C(2)	C(3)	178.08(16)
C(2)	O(1)	C(1)	C(10)[2]	170.91(17)
C(3)	O(2)	C(4)	C(5)	−178.92(16)
C(4)	O(2)	C(3)	C(2)	−173.47(15)
C(5)	O(3)	C(6)	C(7)	−174.77(15)
C(6)	O(3)	C(5)	C(4)	173.16(15)
C(7)	O(4)	C(8)	C(9)	176.24(15)
C(8)	O(4)	C(7)	C(6)	177.73(15)
C(9)	O(5)	C(10)	C(1)[1]	−109.60(19)
C(10)	O(5)	C(9)	C(8)	−178.54(16)

[1] 1½−x, ½+y, ½−z; [2] 1½−x, y−½, ½−z

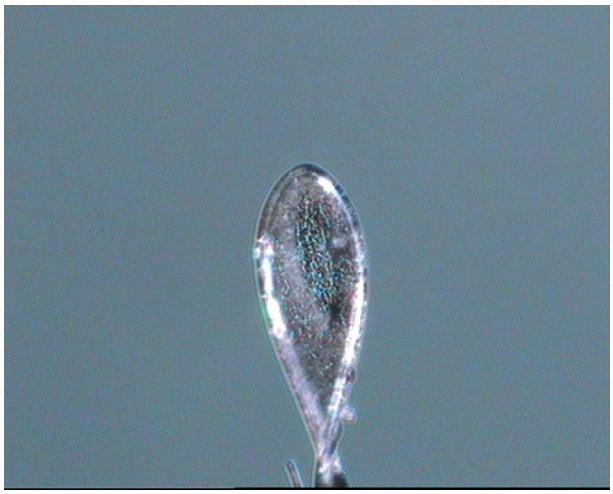

Figure S1: Crystal used for the single crystal study held in Hampton Research nylon loop suspended in a film of amorphous material formed by AB dissolved in PEO.

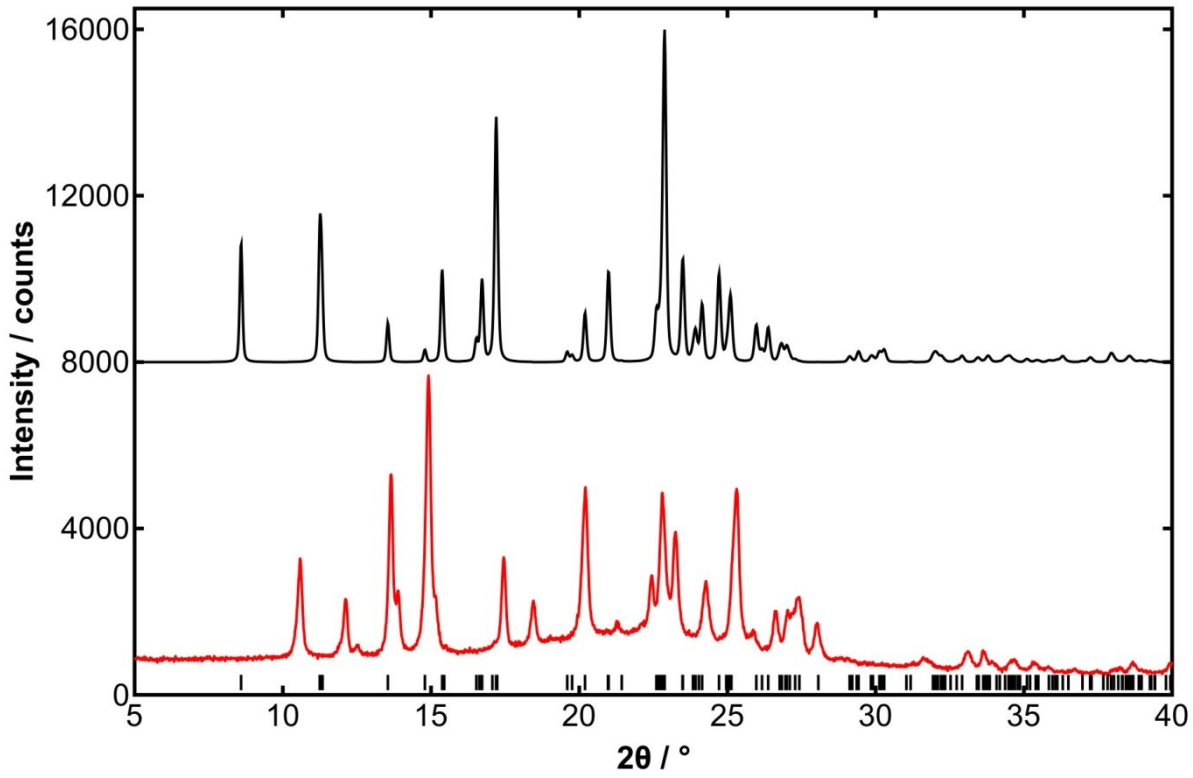

Figure S2: Powder X-ray diffraction pattern from the single cocrystal (black), and the powder X-ray diffraction pattern for the phase-pure cocrystal phase in a 30 wt% AB power sample, which was described in previous work[3].

[3] A. R. Ploszajski, M. Billing, A. S. Nathanson, M. Vickers and S. M. Bennington. *Int. J. Hydrogen Energy*, 2018, 43, 5645–5656.

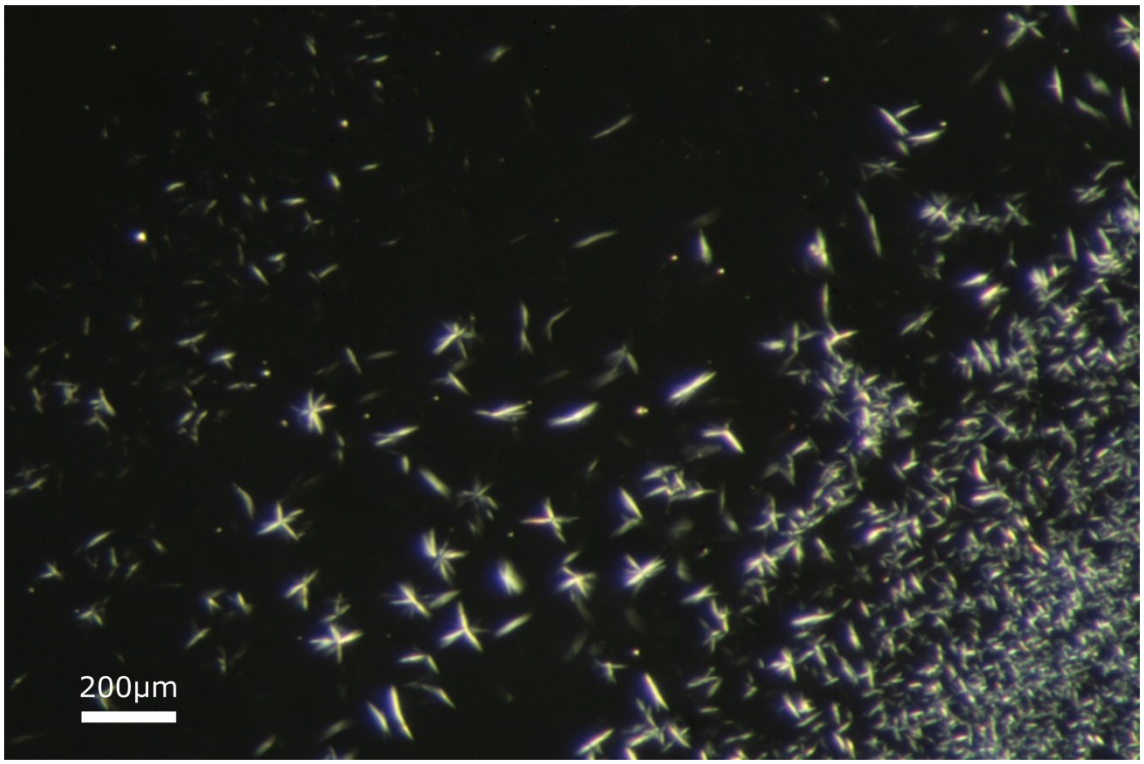

Figure S3: Optical micrograph of single crystals of the 30 wt% AB cocrystals which have the powder diffraction pattern matching a previously-found crystalline phase (Fig. S2), but have proven too small to isolate.