Supplementary Material (ESI) for Chemical Communications

First example of an infinite polybromide 2D-network

M. Carla Aragoni,[a] Massimiliano Arca,[*a] Francesco A. Devillanova,[*a] Francesco Isaia,[a] Vito Lippolis,[a] Annalisa Mancini,[a] Luca Pala,[a] Alexandra M.Z. Slawin[b] and J. Derek Woollins[b]

[a] Dipartimento di Chimica Inorganica ed Analitica, S.S. 554 bivio per Sestu, 09042 Monserrato – Cagliari, Italy.E-mail: marca@unica.it; Fax: +39 070 675 4456; Tel: +39 070 675 4483
[b] School of Chemistry, University of St. Andrews, St. Andrews, Fife, UK KY16 9ST

Electronic Supplementary Information (ESI)

Table S1. Crystal data and structure refinement for $\mathbf{1}^{Et}\cdot 5Br_2$.

Empirical formula	C14 H20 Br10 N4 S6	
Formula weight	1235.80	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 12.893(2) Å	α= 90°.
	b = 13.401(2) Å	β= 112.671(3)°.
	c = 10.6610(17) Å	γ = 90°.
Volume	1699.6(5) $Å^3$	
Z	2	
Density (calculated)	2.415 Mg/m^3	
Absorption coefficient	12.173 mm^{-1}	
F(000)	1156	
Crystal size	0.08 x 0.08 x 0.04 mm^3	
Theta range for data collection	1.71 to 23.25°.	
Index ranges	-14<=h<=13, -14<=k<=14, -9<=l<=11	
Reflections collected	8183	
Independent reflections	2379 [R(int) = 0.0680]	
Completeness to theta = 23.25°	97.9 %	
Absorption correction	Sadabs	
Max. and min. transmission	1.00000 and 0.653024	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2379 / 0 / 155	
Goodness-of-fit on F^2	0.849	
Final R indices [I>2sigma(I)]	R1 = 0.0469, wR2 = 0.0909	
R indices (all data)	R1 = 0.1187, wR2 = 0.1095	
Extinction coefficient	0.0008(2)	
Largest diff. peak and hole	0.899 and -0.646 $e.Å^{-3}$	

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $\mathbf{1}^{Et}\cdot 5Br_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	1244(1)	8113(1)	-7404(1)	71(1)
S(1)	1157(2)	6814(2)	-8777(3)	62(1)
C(1)	2364(9)	6177(8)	-7764(10)	45(3)
N(2)	3416(8)	6371(6)	-7616(8)	48(2)
C(3)	4115(8)	5627(7)	-6820(10)	41(3)
S(3)	5544(2)	5592(2)	-6465(3)	58(1)
C(4)	3455(8)	4986(7)	-6458(9)	41(2)
S(4)	3791(2)	3888(2)	-5531(3)	57(1)
N(5)	2362(7)	5366(7)	-7009(8)	46(2)
C(6)	3776(11)	7172(10)	-8313(14)	83(4)
C(7)	4284(13)	7940(11)	-7325(17)	121(6)
C(8)	1390(8)	4937(9)	-6840(12)	67(3)
C(9)	1233(10)	5383(12)	-5616(15)	104(5)
Br(2)	1554(1)	4999(1)	-10574(1)	55(1)
Br(3)	4025(1)	4956(1)	-10153(2)	108(1)
Br(4)	1787(1)	3408(1)	-12572(1)	84(1)
Br(5)	1842(1)	2072(1)	-13984(1)	94(1)

Table S3. Bond lengths [Å] and angles [°] for $\mathbf{1}^{Et}\cdot 5Br_2$.

Br(1)-S(1)	2.249(3)
S(1)-C(1)	1.738(10)
C(1)-N(2)	1.329(12)
C(1)-N(5)	1.354(12)
N(2)-C(3)	1.392(12)
N(2)-C(6)	1.479(14)
C(3)-C(4)	1.365(12)
C(3)-S(3)	1.733(10)
S(3)-S(4)#1	2.084(4)
C(4)-N(5)	1.398(11)
C(4)-S(4)	1.732(10)
S(4)-S(3)#1	2.084(4)
N(5)-C(8)	1.452(12)
C(6)-C(7)	1.437(17)
C(8)-C(9)	1.519(15)
Br(3)-Br(3)#2	2.411(3)
Br(4)-Br(5)	2.358(2)
C(1)-S(1)-Br(1)	99.6(3)
N(2)-C(1)-N(5)	108.8(9)
N(2)-C(1)-S(1)	127.3(9)
N(5)-C(1)-S(1)	123.9(9)
C(1)-N(2)-C(3)	108.8(9)
C(1)-N(2)-C(6)	125.9(10)
C(3)-N(2)-C(6)	124.9(10)
C(4)-C(3)-N(2)	107.3(9)
C(4)-C(3)-S(3)	130.3(8)
N(2)-C(3)-S(3)	122.4(8)
C(3)-S(3)-S(4)#1	101.5(3)
C(3)-C(4)-N(5)	106.8(8)
C(3)-C(4)-S(4)	130.6(8)
N(5)-C(4)-S(4)	122.6(8)
C(4)-S(4)-S(3)#1	102.3(3)
C(1)-N(5)-C(4)	108.0(8)
C(1)-N(5)-C(8)	126.3(9)
C(4)-N(5)-C(8)	125.6(9)
C(7)-C(6)-N(2)	107.1(11)
N(5)-C(8)-C(9)	111.0(10)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z-1 #2 -x+1,-y+1,-z-2.

Table S4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for $\mathbf{1}^{Et}\cdot 5Br_2$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + \cdots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	79(1)	73(1)	61(1)	4(1)	27(1)	22(1)
S(1)	60(2)	62(2)	50(2)	3(2)	7(1)	12(2)
C(1)	45(8)	55(7)	31(7)	-5(6)	9(5)	0(6)
N(2)	49(7)	47(6)	48(6)	4(5)	18(5)	-6(5)
C(3)	43(7)	41(6)	41(6)	2(5)	19(5)	10(6)
S(3)	48(2)	81(2)	50(2)	11(2)	25(1)	0(2)
C(4)	39(7)	43(6)	38(6)	4(6)	13(5)	-5(6)
S(4)	52(2)	50(2)	61(2)	8(2)	13(2)	-7(1)
N(5)	30(6)	76(7)	37(5)	-3(5)	17(4)	-6(5)
C(6)	64(9)	79(11)	96(12)	19(9)	20(8)	3(7)
C(7)	144(16)	89(11)	147(17)	57(12)	76(13)	26(11)
C(8)	29(7)	85(9)	81(9)	33(8)	14(6)	10(6)
C(9)	51(9)	162(14)	118(13)	13(11)	55(9)	19(8)
Br(2)	57(1)	51(1)	53(1)	-1(1)	18(1)	-5(1)
Br(3)	131(1)	127(1)	82(1)	-45(1)	59(1)	-64(1)
Br(4)	101(1)	70(1)	68(1)	3(1)	17(1)	-15(1)
Br(5)	92(1)	123(1)	65(1)	-16(1)	28(1)	-2(1)

Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for $\mathbf{1}^{Et}\cdot 5Br_2$.

	x	y	z	U(eq)
H(6A)	4314	6916	-8666	100
H(6B)	3135	7436	-9065	100
H(7A)	4530	8475	-7742	181
H(7B)	4917	7669	-6587	181
H(7C)	3743	8188	-6986	181
H(8A)	1487	4220	-6723	80
H(8B)	723	5060	-7651	80
H(9A)	589	5086	-5522	155
H(9B)	1121	6090	-5739	155
H(9C)	1889	5255	-4811	155

Table S6. Torsion angles [°] for $\mathbf{1}^{Et}\cdot 5Br_2$.

Br(1)-S(1)-C(1)-N(2)	-81.0(9)
Br(1)-S(1)-C(1)-N(5)	100.7(8)
N(5)-C(1)-N(2)-C(3)	4.2(10)
S(1)-C(1)-N(2)-C(3)	-174.2(7)
N(5)-C(1)-N(2)-C(6)	177.4(10)
S(1)-C(1)-N(2)-C(6)	-1.0(15)
C(1)-N(2)-C(3)-C(4)	-1.5(11)
C(6)-N(2)-C(3)-C(4)	-174.8(10)
C(1)-N(2)-C(3)-S(3)	177.1(7)
C(6)-N(2)-C(3)-S(3)	3.8(14)
C(4)-C(3)-S(3)-S(4)#1	-78.4(9)
N(2)-C(3)-S(3)-S(4)#1	103.4(8)
N(2)-C(3)-C(4)-N(5)	-1.7(10)
S(3)-C(3)-C(4)-N(5)	179.9(7)
N(2)-C(3)-C(4)-S(4)	177.4(7)
S(3)-C(3)-C(4)-S(4)	-1.1(15)
C(3)-C(4)-S(4)-S(3)#1	81.0(9)
N(5)-C(4)-S(4)-S(3)#1	-100.1(7)
N(2)-C(1)-N(5)-C(4)	-5.3(10)
S(1)-C(1)-N(5)-C(4)	173.2(7)
N(2)-C(1)-N(5)-C(8)	176.9(9)
S(1)-C(1)-N(5)-C(8)	-4.6(14)
C(3)-C(4)-N(5)-C(1)	4.3(10)
S(4)-C(4)-N(5)-C(1)	-174.9(7)
C(3)-C(4)-N(5)-C(8)	-177.9(9)
S(4)-C(4)-N(5)-C(8)	2.9(13)
C(1)-N(2)-C(6)-C(7)	105.7(12)
C(3)-N(2)-C(6)-C(7)	-82.2(14)
C(1)-N(5)-C(8)-C(9)	-91.0(12)
C(4)-N(5)-C(8)-C(9)	91.5(13)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z-1 #2 -x+1,-y+1,-z-2

Table S7. Experimental and calculated (for C14 H20 Br10 N4 S6) elemental analysis for $\mathbf{1}^{Et}\cdot 5Br_2$.

	Exp. (%)	Calcd. (%)
C	13.40	13.61
H	1.95	1.63
N	4.60	4.53
S	14.97	15.57