Electronic Supplementary Material (ESI) for ChemComm.

Electronic Supporting Information

Triazine based eccentric Piedfort units towards a single source hydrogen bonded network

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Experimental Section

Materials

Cyanuric chloride (Avra Synthesis), aniline (Rankem), isopropylamine (Alfa Aesar), isopropyl (TCI) and thiol 4-methylthiophenol (TCI) were used as received from commercial sources. Solvents were distilled under dry nitrogen atmosphere using conventional methods.

Methods

Elemental analyses were carried out on a Perkin-Elmer CHNS/O analyzer. NMR spectra were recorded on JEOL 500 MHz or JEOL 400 MHz spectrometers. Temperature was kept constant using a variable temperature unit within the error limit of ±1 K. The software MestReNova was used for the processing of the NMR spectra. Tetramethylsilane (TMS) or the deuterated solvent residual peaks were used for calibration. Mass spectrometry experiments were performed on a Waters-Q-ToF-Premier-HAB213 equipped with an electrospray interface. Spectra were collected by constant infusion of the sample dissolved in methanol or acetonitrile with 0.1% formic acid.

Crystal Structure Determinations

Single-crystal X-ray data were collected on a Bruker SMART APEX CCD diffractometer using graphite-monochromated Mo Kα radiation (λ = 0.71069 Å). The linear absorption coefficients, the scattering factors for the atoms, and the anomalous dispersion corrections were taken from International Tables for X-ray Crystallography. Data integration and reduction were conducted with SAINT. An empirical absorption correction was applied to the collected reflections with SADABS using XPREP. Structures were determined by dual-space methods using SHELXTL[1] and refined on F^2 by a full-matrix least-squares technique using the SHELXL-2018 program package.[2] The lattice parameters and structural data are listed at the end of this Supporting Information. All the crystallographic data have been deposited in The Cambridge Crystallographic Data Centre; CCDC-1454811 **(1)**, 1454814 **(2)** and 1495815 **(3)** contain the crystallographic data.

N^2,N^4,N^6-triisopropyl-1,3,5-triazine-2,4,6-triamine (1).

A 250 ml heavy-walled high-pressure tube (Ace Glass, Inc.) was charged with cyanuric chloride (1.0 g, 5.42 mmol) at 0-5 °C. Isopropylamine (17.30 g, 292 mmol) was slowly added at same temperature. The resulting reaction mixture was stirred at 120 °C for 48 h and cooled to room temperature. The chloroform (20 ml) was added and washed two times with 50 ml saturated $NaHCO_3$ solution and followed with 25 ml of distilled water. The

organic layer was dried over $MgSO_4$ and evaporated to dryness. White solid was obtained (1.0 g, 73%). Colourless needles of diffraction quality grew from $CHCl_3$ solution within a day at room temperature. ^1H NMR (500 MHz, $CDCl_3$): δ = 4.61 (bs, 3H, NH), 4.09 (bs, 3H, CH), 1.15 (d, 18H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.5, 42.1, 23.1; ESI-MS: m/z = 253.2141 (calcd 253.2141) = $[M+H]^+$.

6-chloro-*N*^{2},*N*^{4}-diisopropyl-1,3,5-triazine-2,4-diamine.

A 250 ml round bottom flask was charged with isopropylamine (4.72 g, 80 mmol) in 15 ml of THF. The solution was treated with DIPEA (12.91 g, 100 mmol) slowly at room temperature. The reaction mixture was kept at 0 °C and added the solution of cyanuric chloride (3.68 g, 20 mmol) in 50 ml of THF through dropping funnel over 2 hrs. The resulting white precipitate was kept at room temperature for 1 hrs and further refluxed for 4 hrs. After that cooled down to room temperature and evaporated under reduced pressure to dryness. The white residue was dissolved in $CHCl_3$ (40 ml) and washed with distilled water (3 x 20 ml). The organic layer was dried over $MgSO_4$ and evaporated the solvent under reduced pressure to get white solid. (3.0 g, 66%). X-ray quality crystals were grown in $CHCl_3$ solution. ^1H NMR (500 MHz, $CDCl_3$): δ = 5.17 (bs, 2H, NH), 4.16 – 4.09 (m, 2H, CH), 1.20 (d, 12H, CH_3); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 168.57, 165.13, 43.06, 22.72.

N^2,*N*^{4}-diisopropyl-6-(isopropylthio)-1,3,5-triazine-2,4-diamine (2).

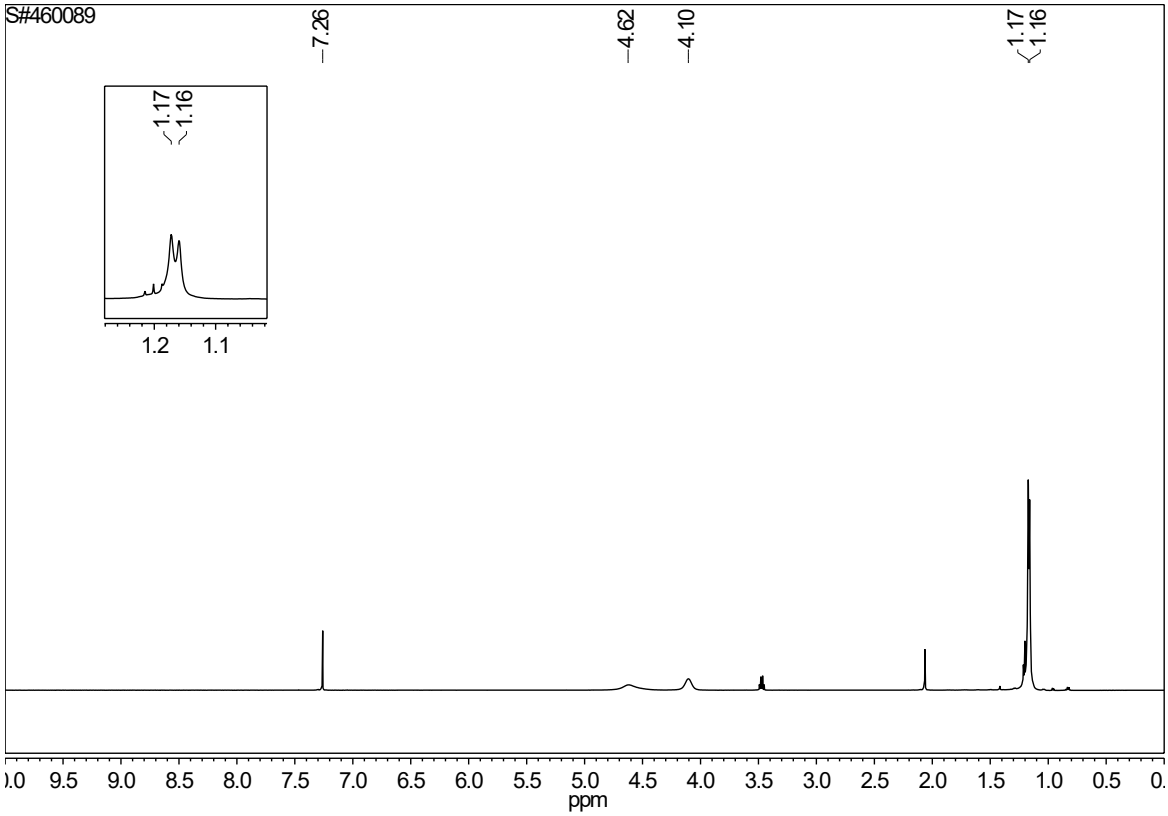
A 60 ml heavy-walled high-pressure tube (Ace Glass, Inc.) was charged with suspension of sodium ethoxide in 8 ml of ethanol and isopropyl thiol (0.304 g, 4 mmol) was added slowly at room temperature with continuous stirring. The solution of 6-chloro-N^2,N^4-diisopropyl-1,3,5-triazine-2,4-diamine (0.470 g, 2.0 mmol) in 15 mL of toluene was added slowly at room temperature in high pressure tube; the flask was sealed with a Teflon screw cap. The reaction mixture was heated at 80 °C for 24 h. The yellow homogeneous solution was cooled down to room temperature and the solvent was removed. The resulting yellow liquid was dissolved in 20 ml of chloroform and washed with the distilled water (3 x 10 ml). The organic layer was collected, dried over $MgSO_4$ and evaporated the solvent. The yellow crude mixture was purified by column chromatography. (Eluent: 9:1 Hexane : ethyl acetate). Off-white solid was obtained. X-ray quality crystals were grown in dimethyl sulfoxide solution. (0.2 g, 57%). ^1H NMR (400 MHz, $CDCl_3$): δ 4.84 (bs, 2H, NH), 4,14 (bs, 2H, CH), 3.86 (bs, 1H CH), 1.37 (d, 6H, CH_3), 1.17 (d, 12H, CH_3); ^{13}C NMR (400 MHz, $CDCl_3$): δ 171.28, 163.81, 49.55, 42.49, 34.53, 23.03. ESI-MS: m/z = 270.1741 (calcd. 269.1674) =

$[M+H]^+$. Elemental analysis calculated for $C_{12}H_{23}N_5S$ 0.1 DMSO: C 52.86, H 8.58, N 25.26; found: C 52.64, H 8.65, N 25.34.

N^2,N^4-diisopropyl-6-(p-tolylthio)-1,3,5-triazine-2,4-diamine (3).

In 100 ml round bottom flask, 4-methylthiophenol (0.243 g, 1.95 mmol) was dissolved in 5 ml of toluene under nitrogen atmosphere and stirred until it was dissolved. DIPEA was added slowly in stirred toluene solution. Then toluene solution (10 ml) of Synthesis of 6-chloro-N^2,N^4-diisopropyl-1,3,5-triazine-2,4-diamine (0.3 g, 1.30 mmol) was added slowly to pre stirred solution through dropping funnel and refluxed for 48 h under nitrogen atmosphere. The reaction mixture was cooled down to room temperature and the solvent was removed. The resulting yellow oily residue was dissolved in $CHCl_3$ (15 ml) and washed with distill. water (3 x 10 ml). The organic layer was collected, dried over $MgSO_4$ and evaporated the solvent. The yellow sticky residue was purified by column chromatography. (Eluent: 9:1 hexane:ethyl acetate).Pale yellow solid was obtained. X-ray quality crystals were grown in dimethyl sulfoxide solution. (0.2 g, 48%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, 2H), 7.17 (d, 2H), 4.83 (bs, 2H, NH), 3.87 (bs, 2H, CH), 2.36 (s, 3H CH_3), 1.10 (d, 12H, CH_3); ^{13}C NMR (400 MHz, $CDCl_3$): δ 171.11, 163.84, 138.90, 135.55, 129.38, 125.76, 42.51, 22.83, 21.15. ESI-MS: m/z = 318.1750 (calcd 317.1674) Elemental analysis calculated for $C_{16}H_{23}N_5S$•0.5 DMSO: C 57.27, H 7.35, N 19.64; found: C 57.43, H 7.31, N 19.75.

Figure S 1. ^{1}H NMR of N^2,N^4,N^6-triisopropyl-1,3,5-triazine-2,4,6-triamine **(1)**



^{1}H NMR (500 MHz, $CDCl_3$): δ = 4.61 (bs, 3H, NH), 4.09 (bs, 3H, CH), 1.15 (d, 18H, CH_3)

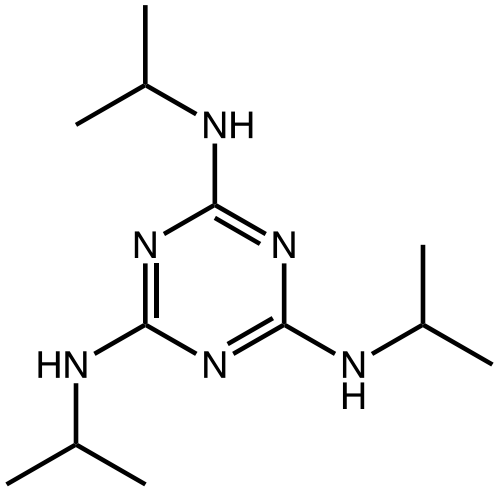
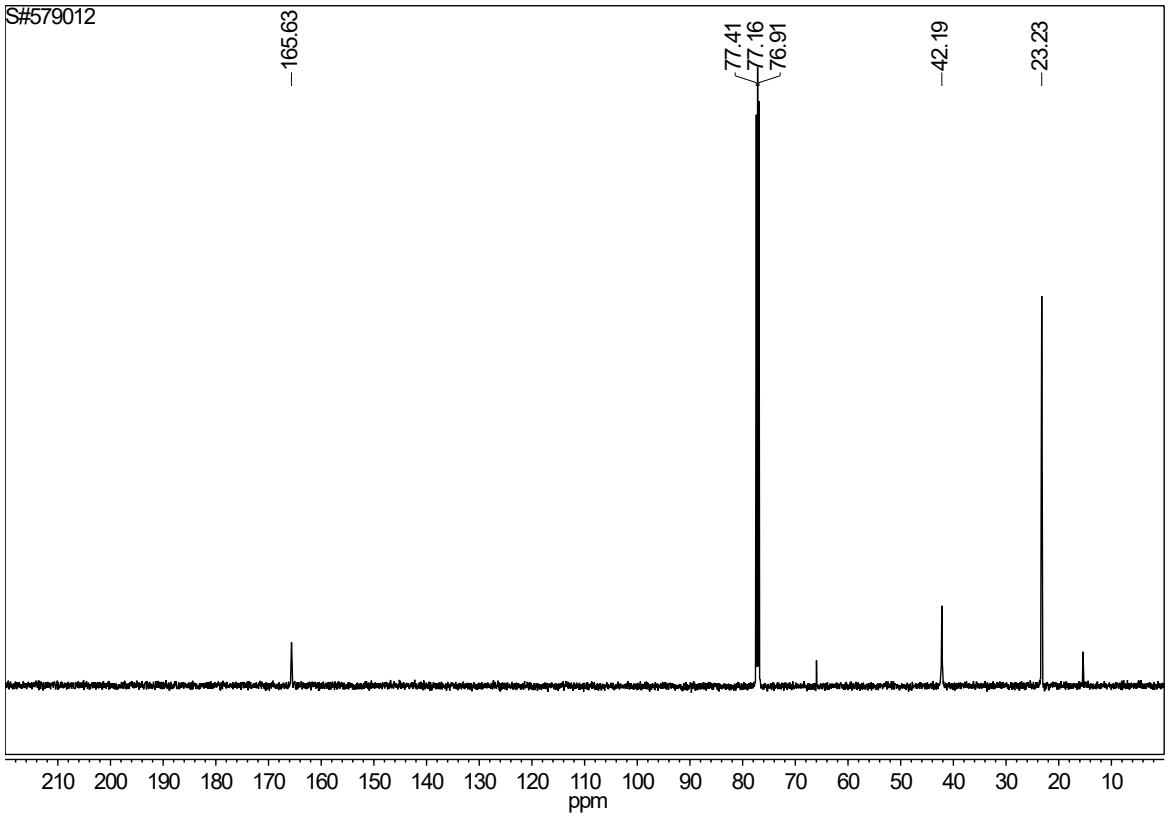


Figure S 2. ^{13}C NMR of N^2,N^4,N^6-triisopropyl-1,3,5-triazine-2,4,6-triamine **(1)**



^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.5, 42.1, 23.1

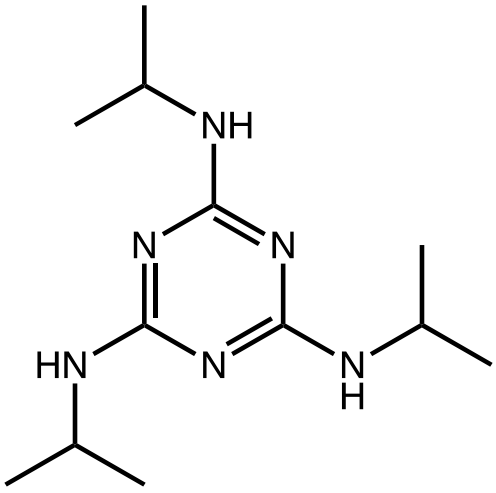
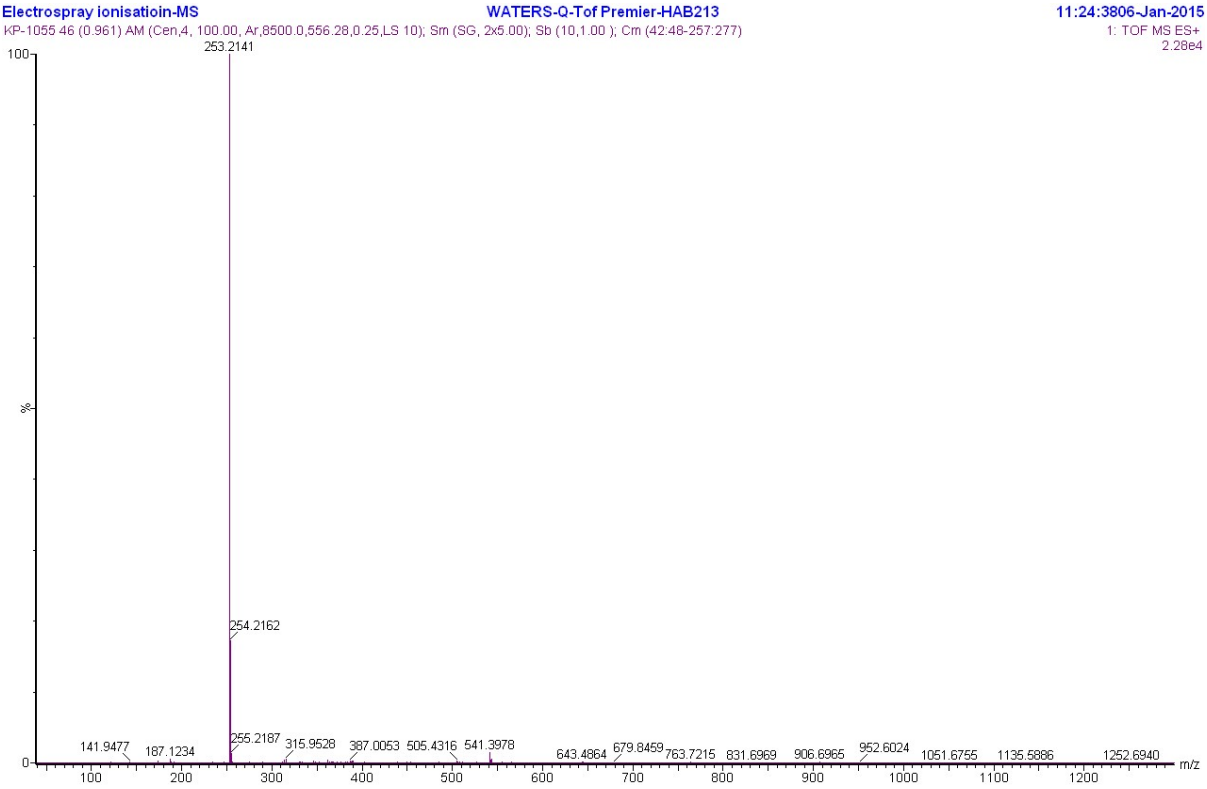


Figure S 3. ESI-MS of N^2,N^4,N^6-triisopropyl-1,3,5-triazine-2,4,6-triamine **(1)**



Calculated m/z for $[MH]^+$ = 252.2141; observed m/z is 253.2141.

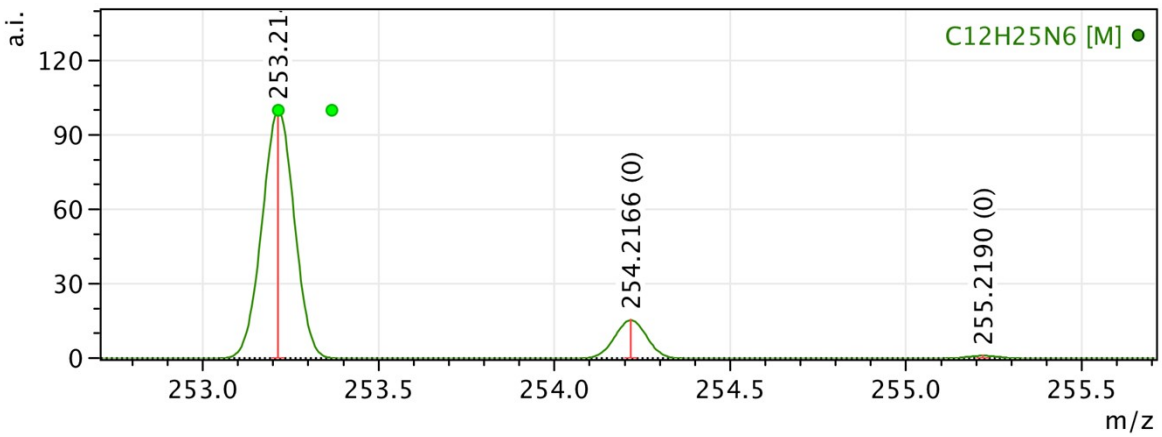
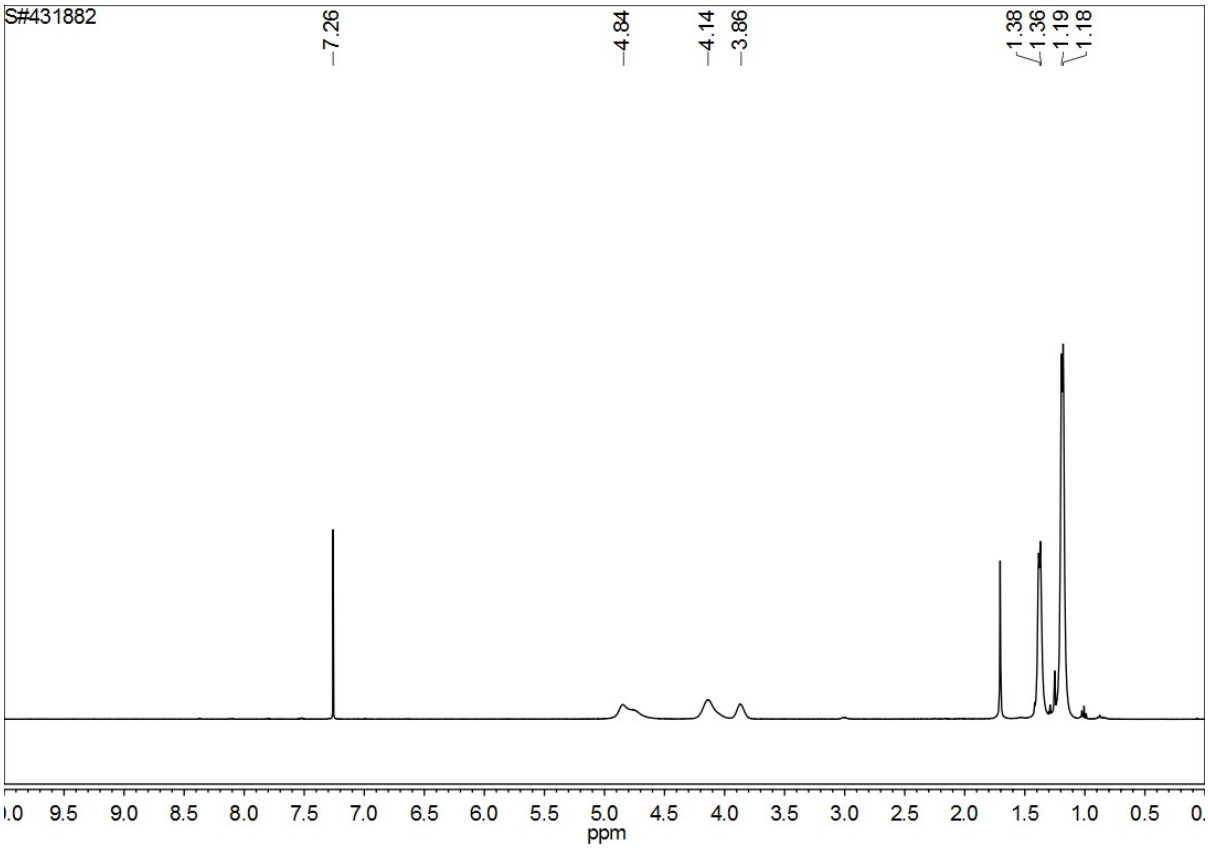


Figure S 4. [1]H NMR of diisopropyl-6-(isopropylthio)-1,3,5-triazine-2,4-diamine **(2)**



[1]H NMR (400 MHz, $CDCl_3$): δ 4.84 (bs, 2H, NH), 4,14 (bs, 2H, CH), 3.86 (bs, 1H CH), 1.37 (d, 6H, CH_3), 1.17 (d, 12H, CH_3).

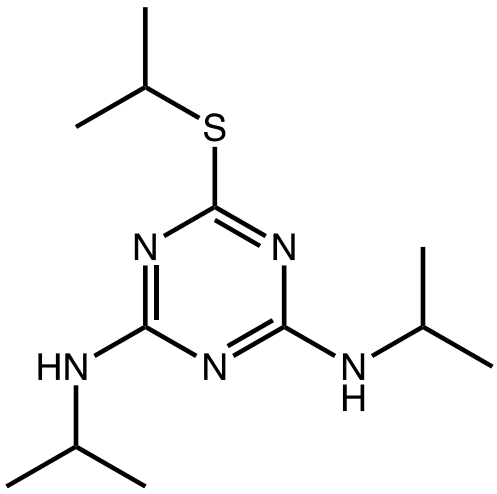
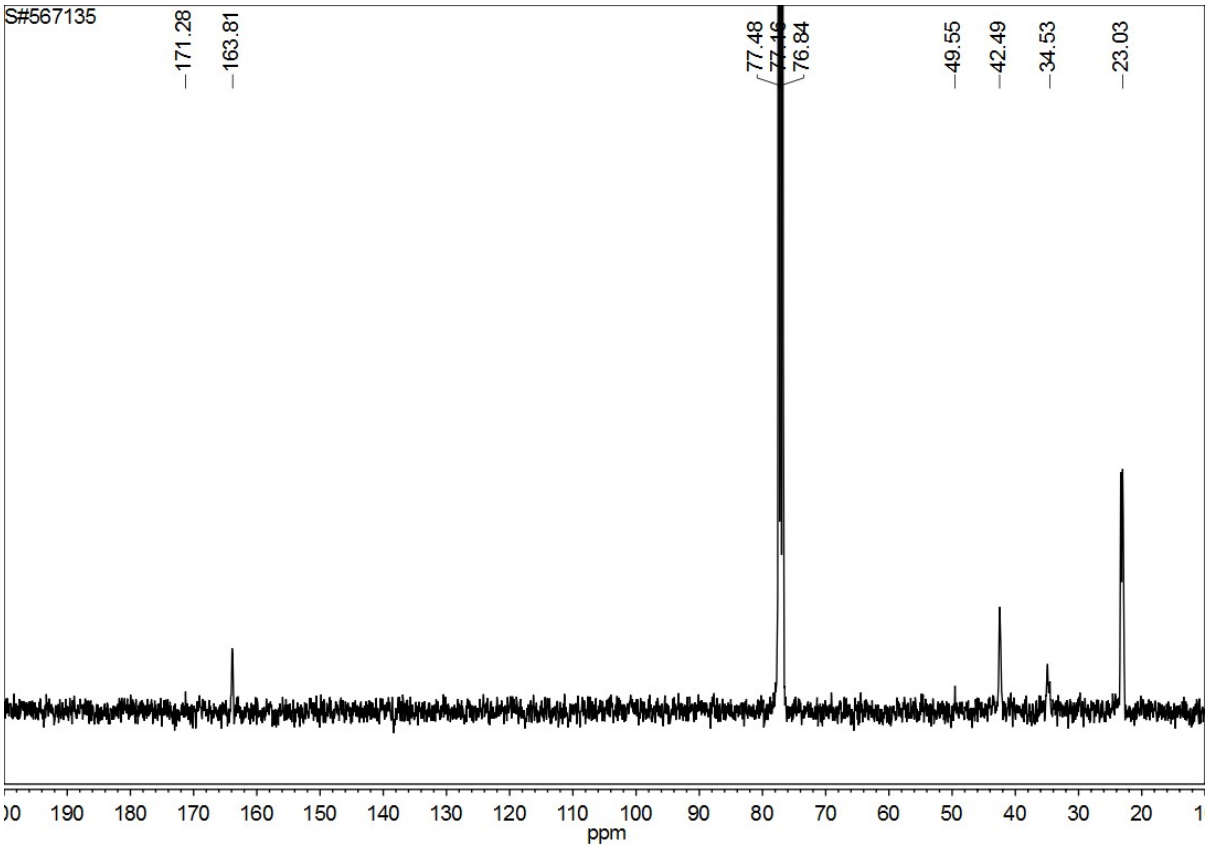


Figure S 5. ^{13}C NMR of diisopropyl-6-(isopropylthio)-1,3,5-triazine-2,4-diamine **(2)**



^{13}C NMR (400 MHz, $CDCl_3$): δ 171.28, 163.81, 49.55, 42.49, 34.53, 23.03.

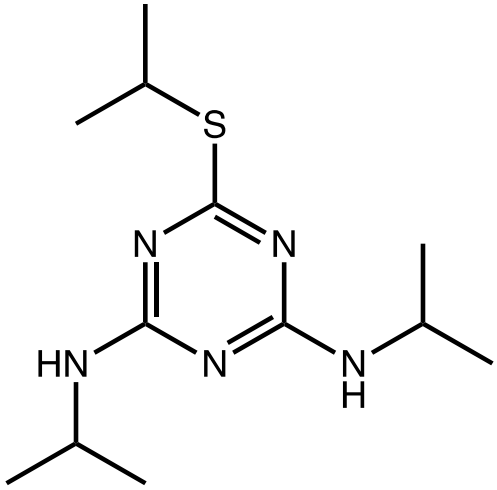
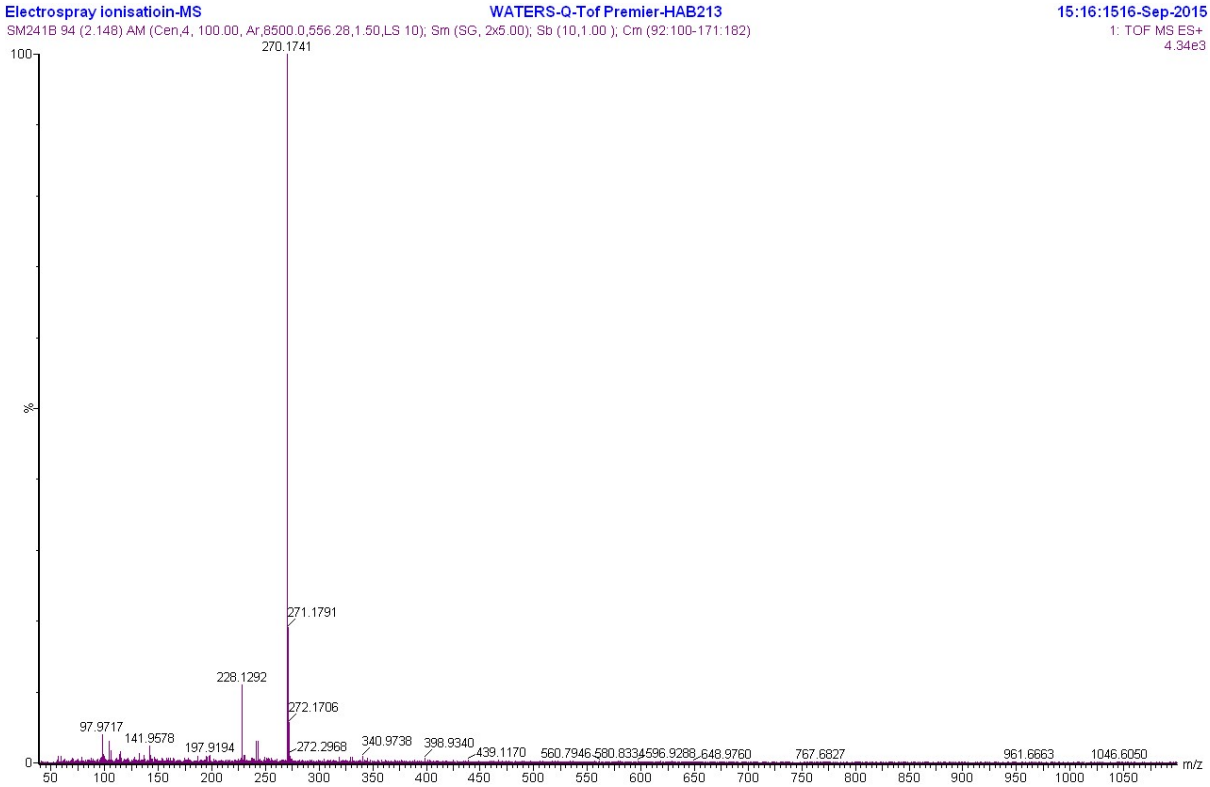


Figure S 6. ESI-MS of diisopropyl-6-(isopropylthio)-1,3,5-triazine-2,4-diamine **(2)**



Calculated m/z for $[MH]^+$ = 270.1752; observed m/z is 270.1741.

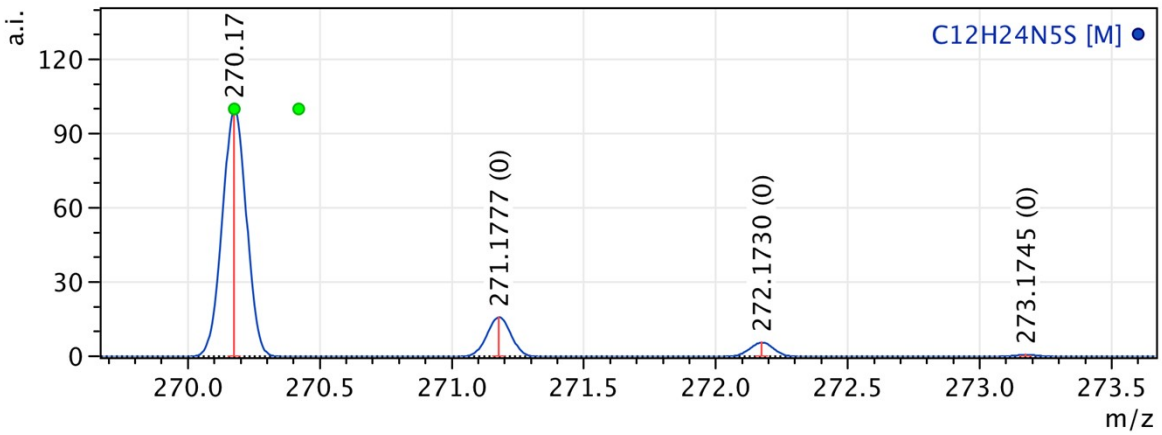
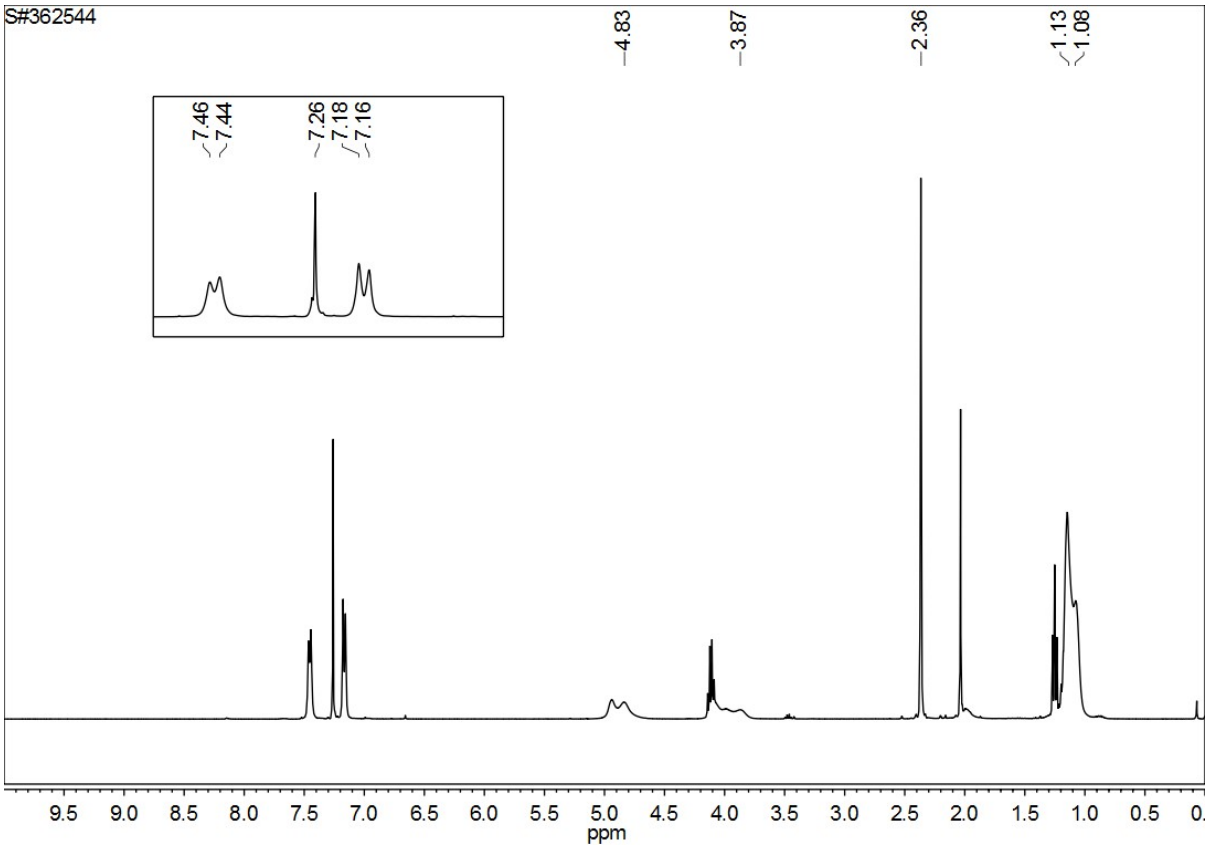


Figure S 7. ^{1}H NMR of N^{2},N^{4}-diisopropyl-6-(p-tolylthio)-1,3,5-triazine-2,4-diamine **(3)**



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, 2H), 7.17 (d, 2H), 4.83 (bs, 2H, NH), 3.87 (bs, 2H, CH), 2.36 (s, 3H CH_3), 1.10 (d, 12H, CH_3).

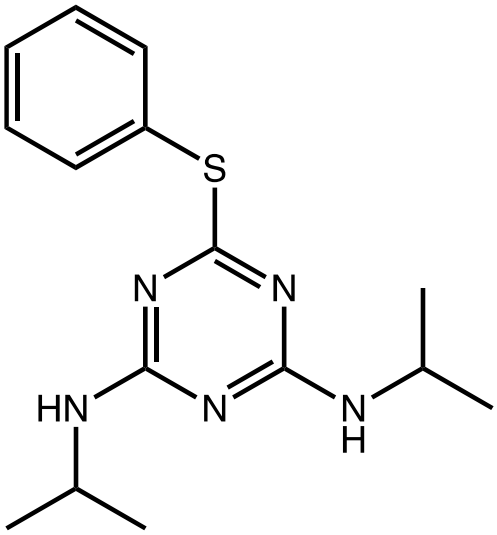
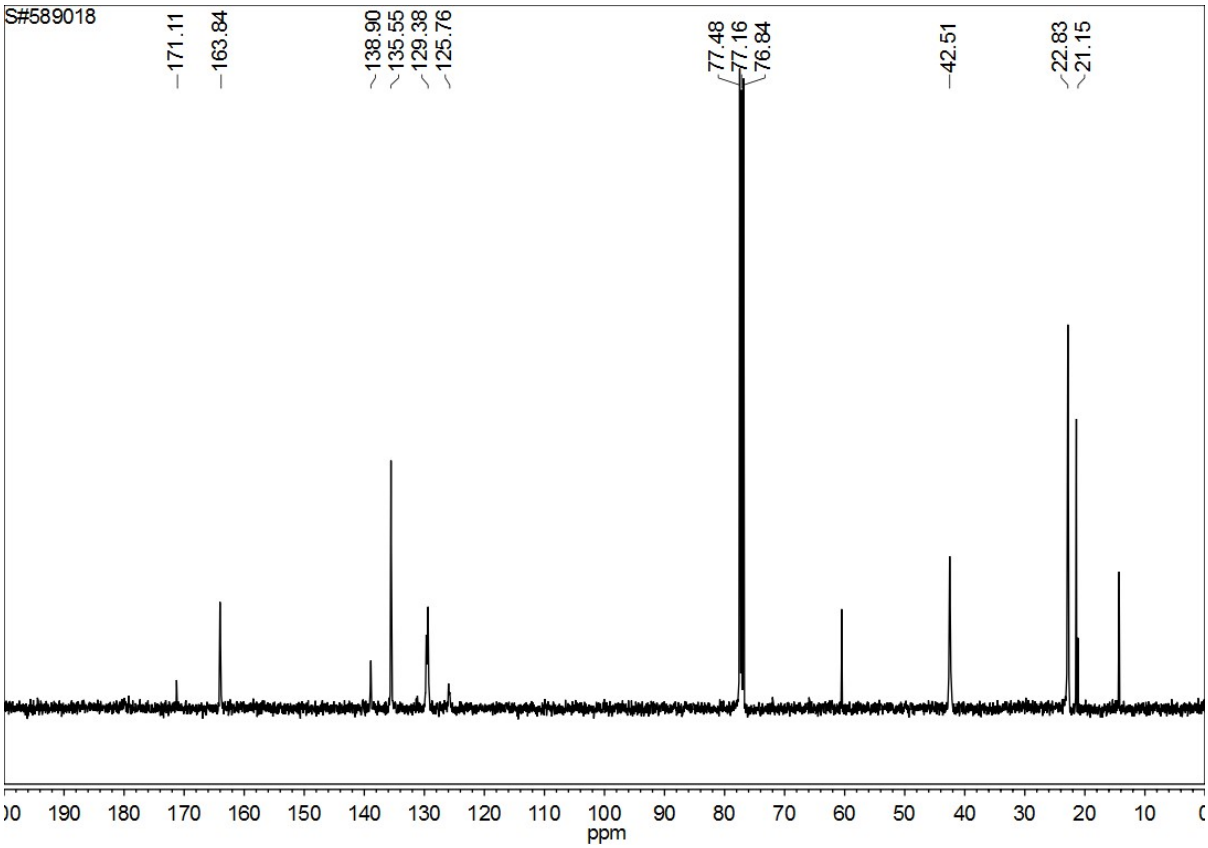


Figure S 8. ^{13}C NMR of N^2,N^4-diisopropyl-6-(p-tolylthio)-1,3,5-triazine-2,4-diamine **(3)**



^{13}C NMR (400 MHz, $CDCl_3$): δ 171.11, 163.84, 138.90, 135.55, 129.38, 125.76, 42.51, 22.83, 21.15.

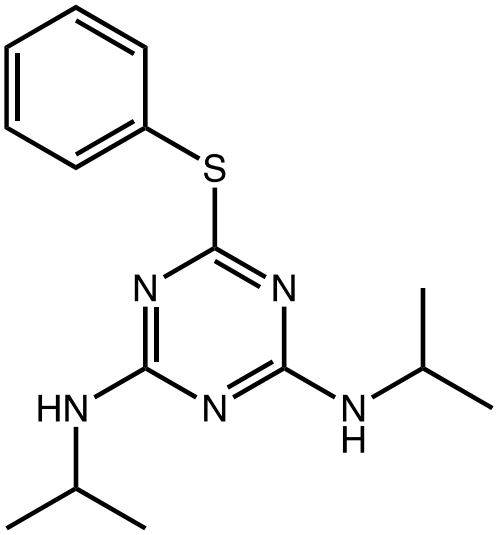
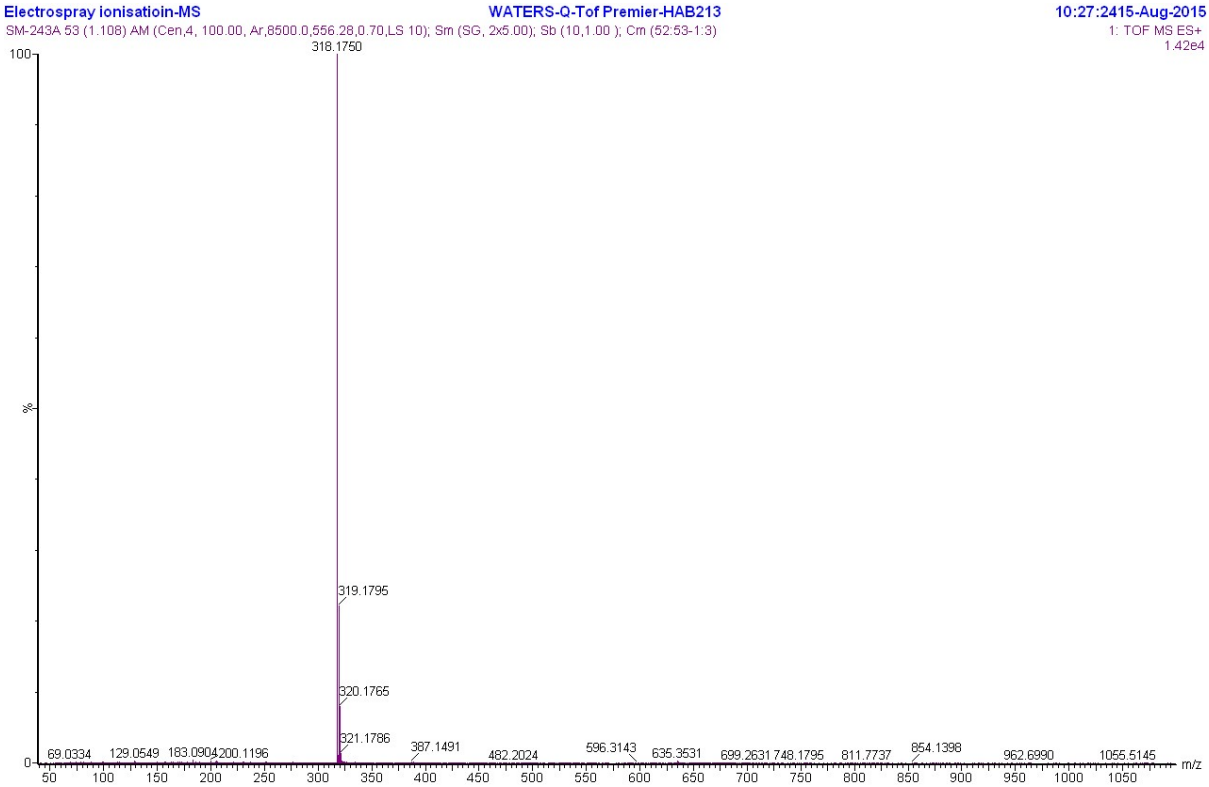


Figure S 9. ESI-MS of N^2,N^4-diisopropyl-6-(p-tolylthio)-1,3,5-triazine-2,4-diamine **(3)**



Calculated m/z for $[MH]^+$ = 318.1752; observed m/z is 318.1750.

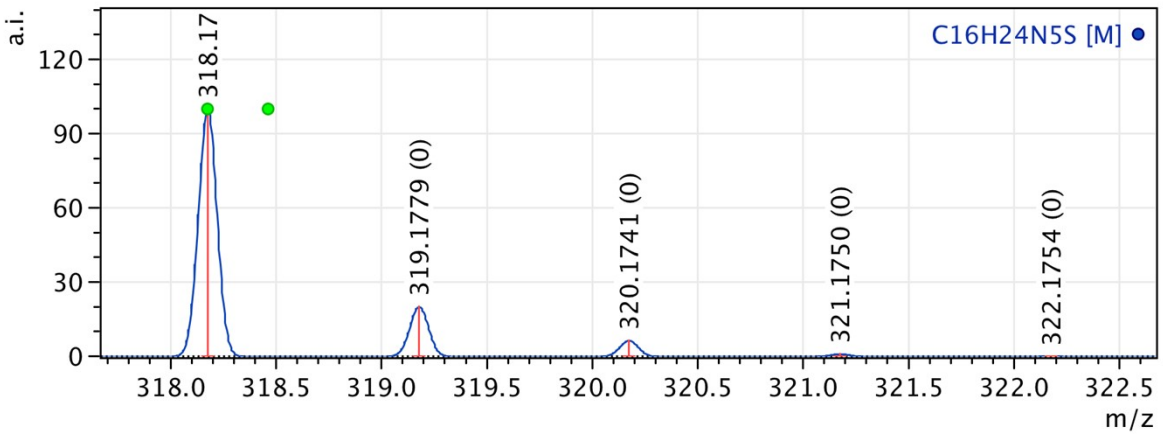


Figure S 10. TGA of **1·0.125CHCl$_3$**.

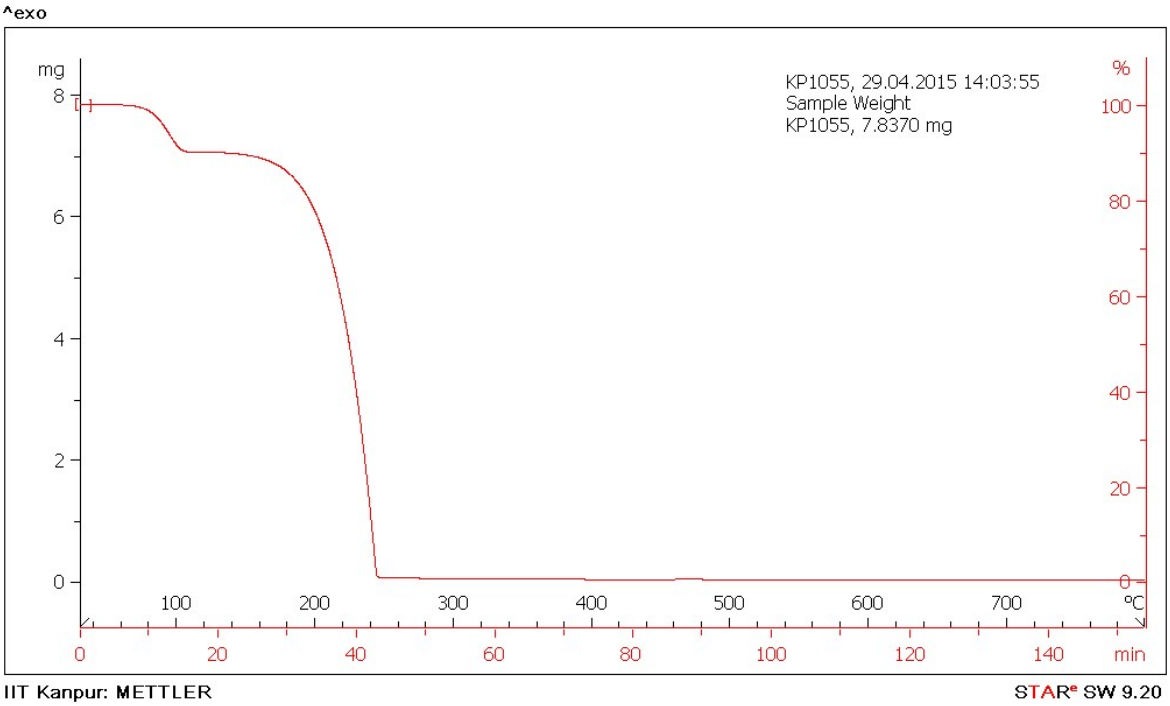


Figure S 11. SEM snapshots of **1** showing highly ordered rod like structures.

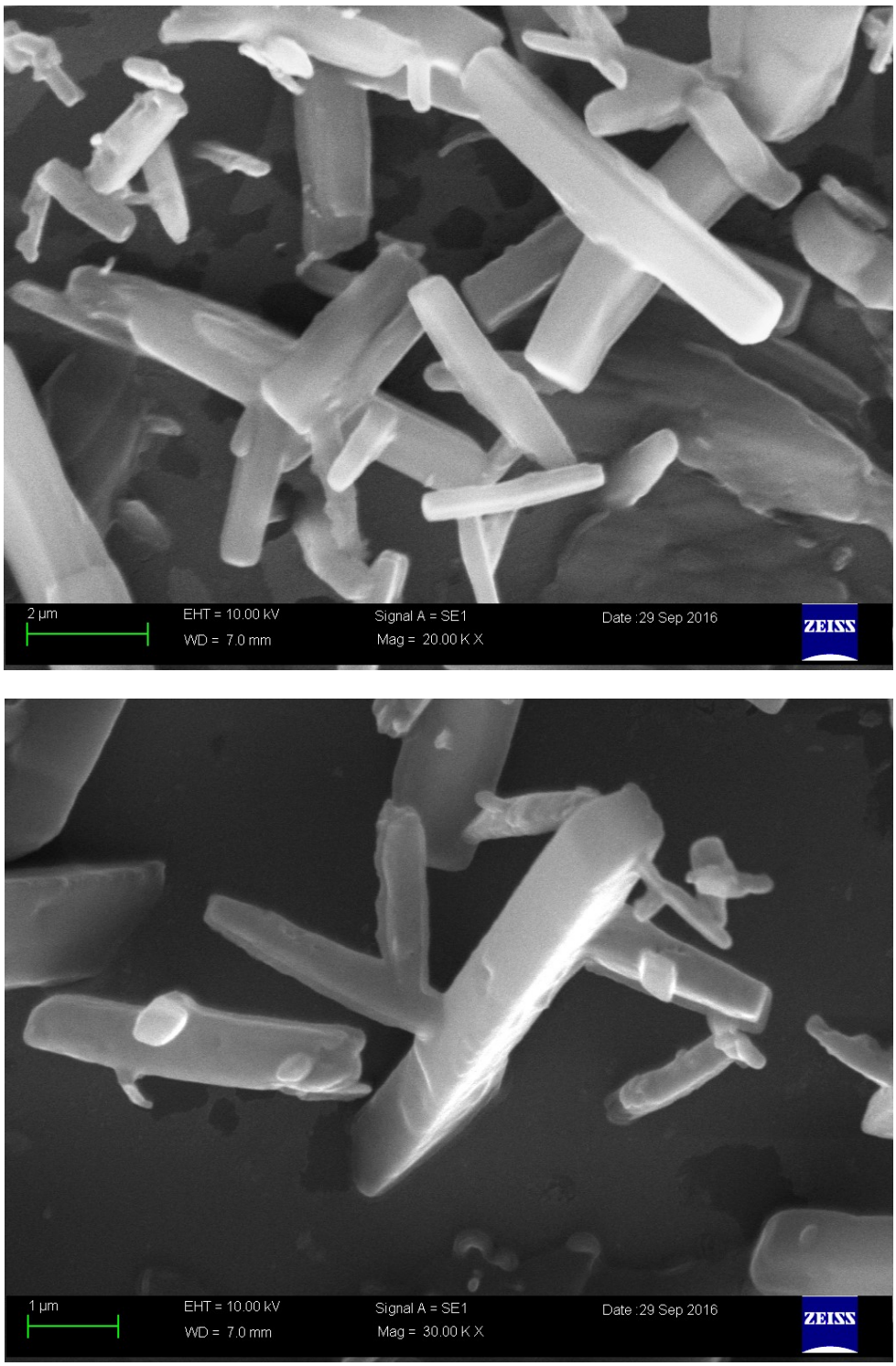
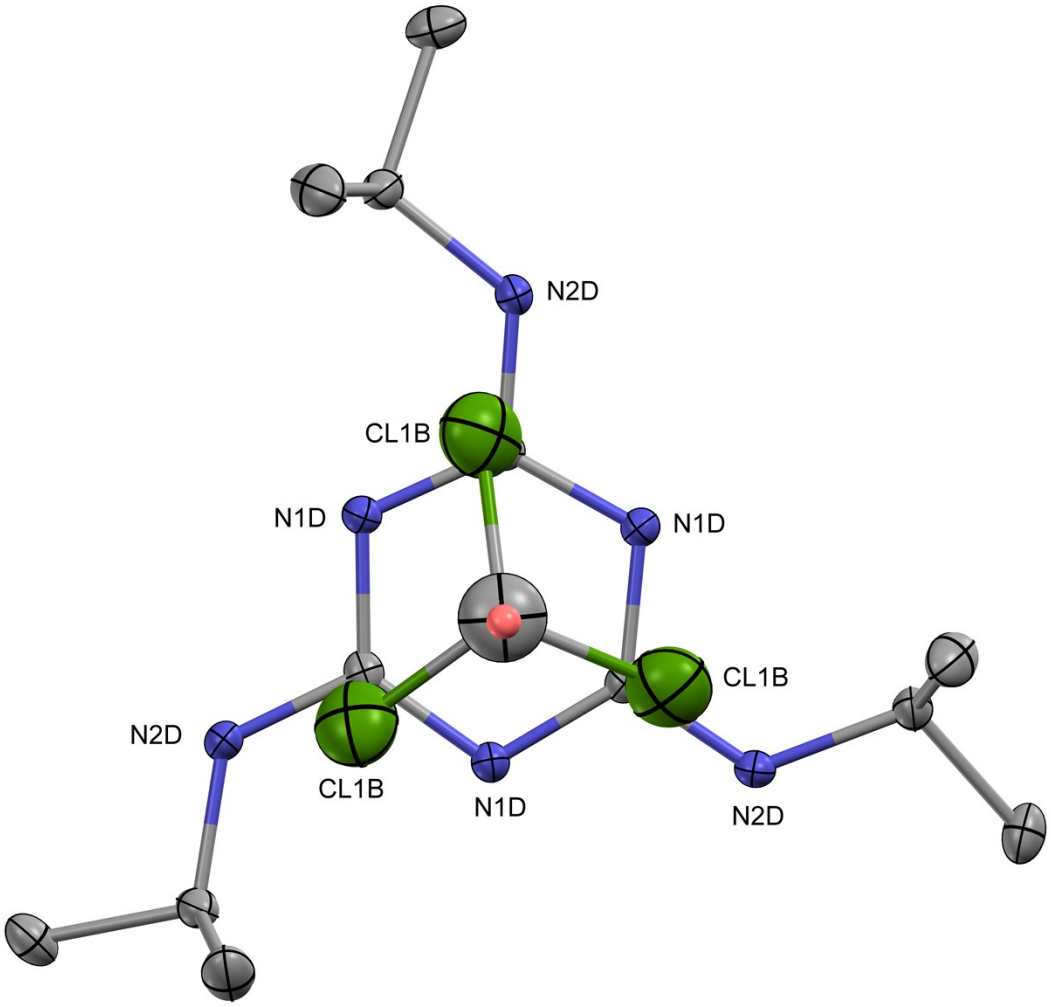


Figure S 12. ORTEP (top view) of N^2,N^4,N^6-triisopropyl-1,3,5-triazine-2,4,6-triamine **(1)**



CCDC-1454811. Selected bond distances (Å) and angles (°): N(1A)-C(2A), 1.339(4); N(1A)-C(1A), 1.346(4); N(2A)-C(3A), 1.332(4); N(2A)-C(2A), 1.355(4); N(3A)-C(1A), 1.335(4); N(3A)-C(3A), 1.354(4); N(4A)-C(1A), 1.346(4); N(4A)-C(4A), 1.466(4); N(4A)-H(4A), 0.86(4); N(5A)-C(2A), 1.340(4); N(5A)-C(7A), 1.456(4); N(5A)-H(5A), 0.84(4); N(6A)-C(3A), 1.346(4); N(6A)-C(10A), 1.461(4); C(4A)-C(6A), 1.519(5); C(4A)-C(5A), 1.522(5); C(2A)-N(1A)-C(1A), 114.5(3); C(3A)-N(2A)-C(2A), 113.8(3); C(1A)-N(3A)-C(3A), 113.8(3); C(1A)-N(4A)-C(4A), 124.5(3); C(1A)-N(4A)-H(4A), 113(2); C(4A)-N(4A)-H(4A), 121(2); C(2A)-N(5A)-C(7A), 124.2(3); C(2A)-N(5A)-H(5A), 117(3); C(7A)-N(5A)-H(5A), 119(3); C(3A)-N(6A)-C(10A), 125.0(3); C(3A)-N(6A)-H(6A), 118(2); C(10A)-N(6A)-H(6A), 117(2); N(3A)-C(1A)-N(4A), 118.8(3); N(3A)-C(1A)-N(1A), 125.8(3); N(4A)-C(1A)-N(1A), 115.4(3); N(1A)-C(2A)-N(5A), 117.9(3); N(1A)-C(2A)-N(2A), 125.5(3); N(5A)-C(2A)-N(2A), 116.7(3); N(2A)-C(3A)-N(6A), 119.4(3); N(2A)-C(3A)-N(3A), 126.4(3); N(6A)-C(3A)-N(3A), 114.2(3); N(4A)-C(4A)-C(6A), 107.9(3); N(4A)-C(4A)-C(5A), 111.2(3); C(6A)-C(4A)-C(5A), 111.7(3).

Figure S 13. ORTEP (side view) of N^2,N^4,N^6-triisopropyl-1,3,5-triazine-2,4,6-triamine **(1)**

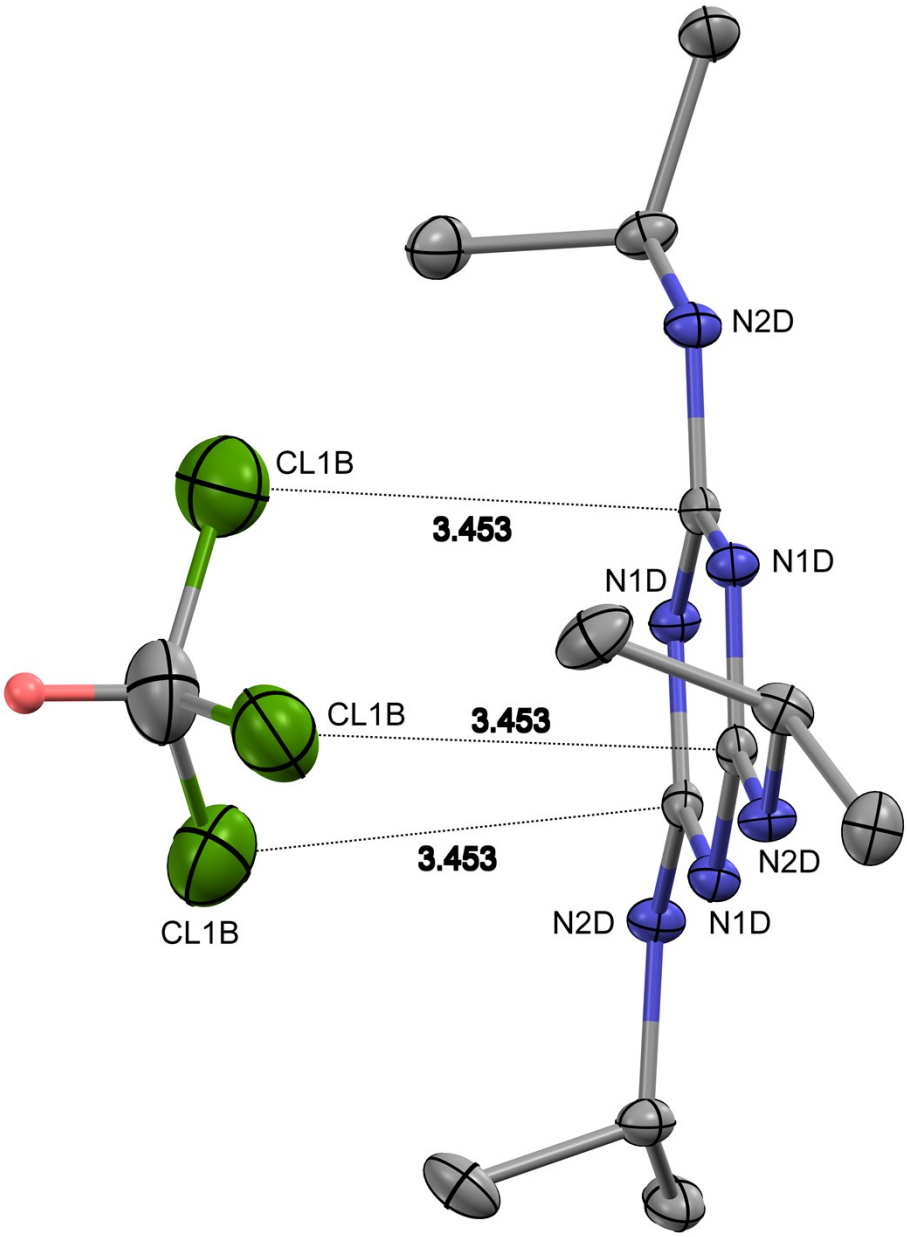


Figure S 14. Superstar network of **1** seen from side (‘a’ axis)

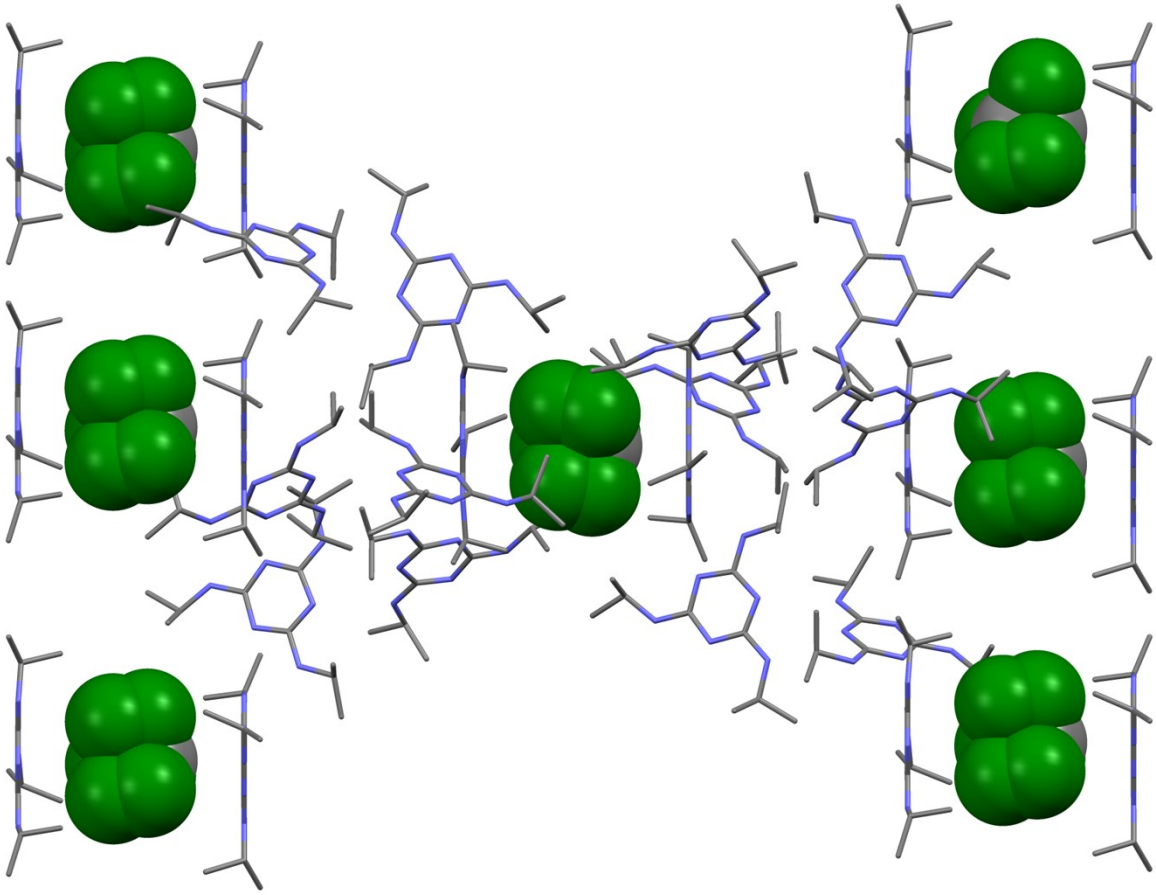


Figure S 15. Superstar network of **1** seen from top (‘c’ axis)

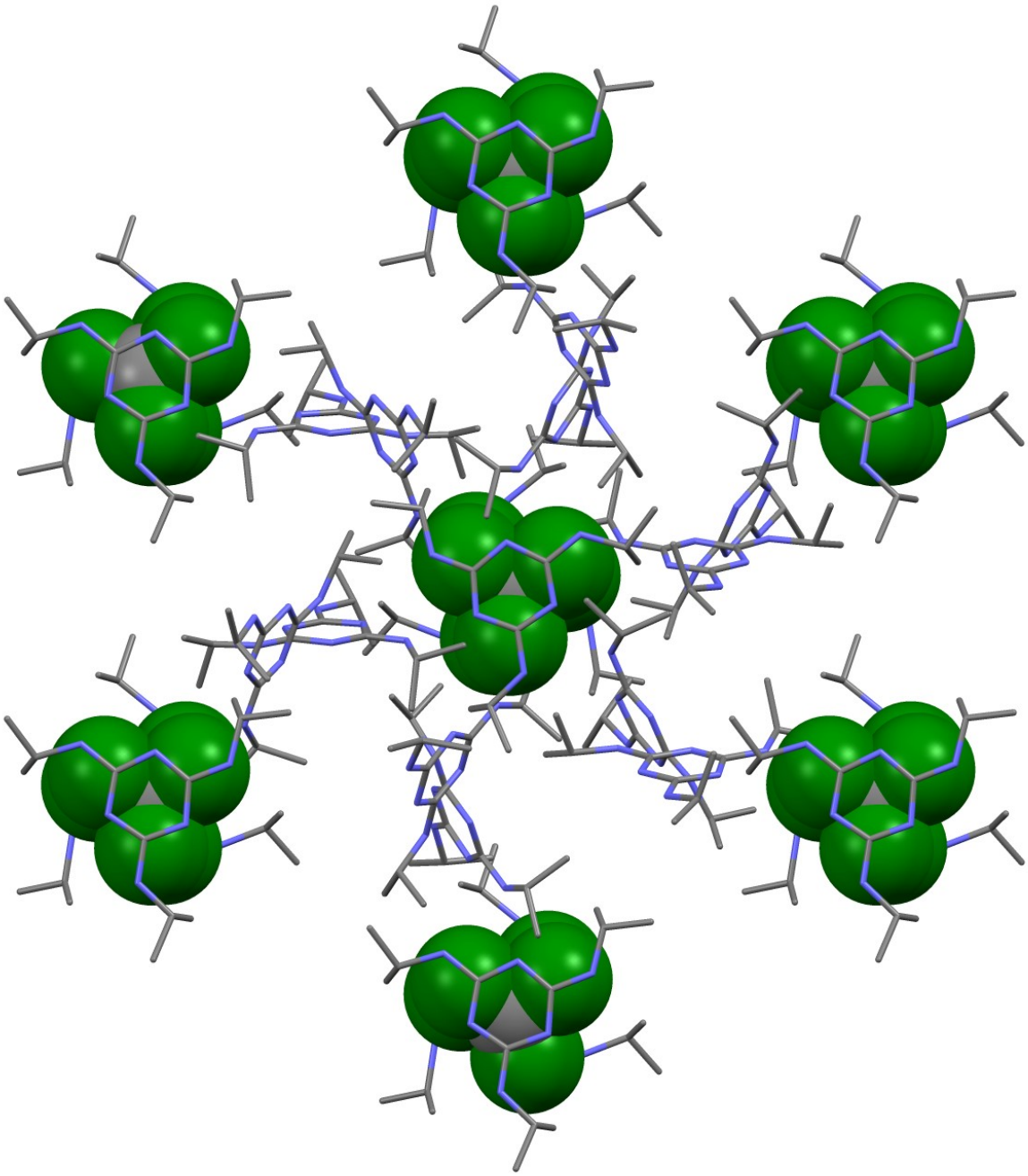


Figure S 16. Piedfort dimer formed by **1** seen from side

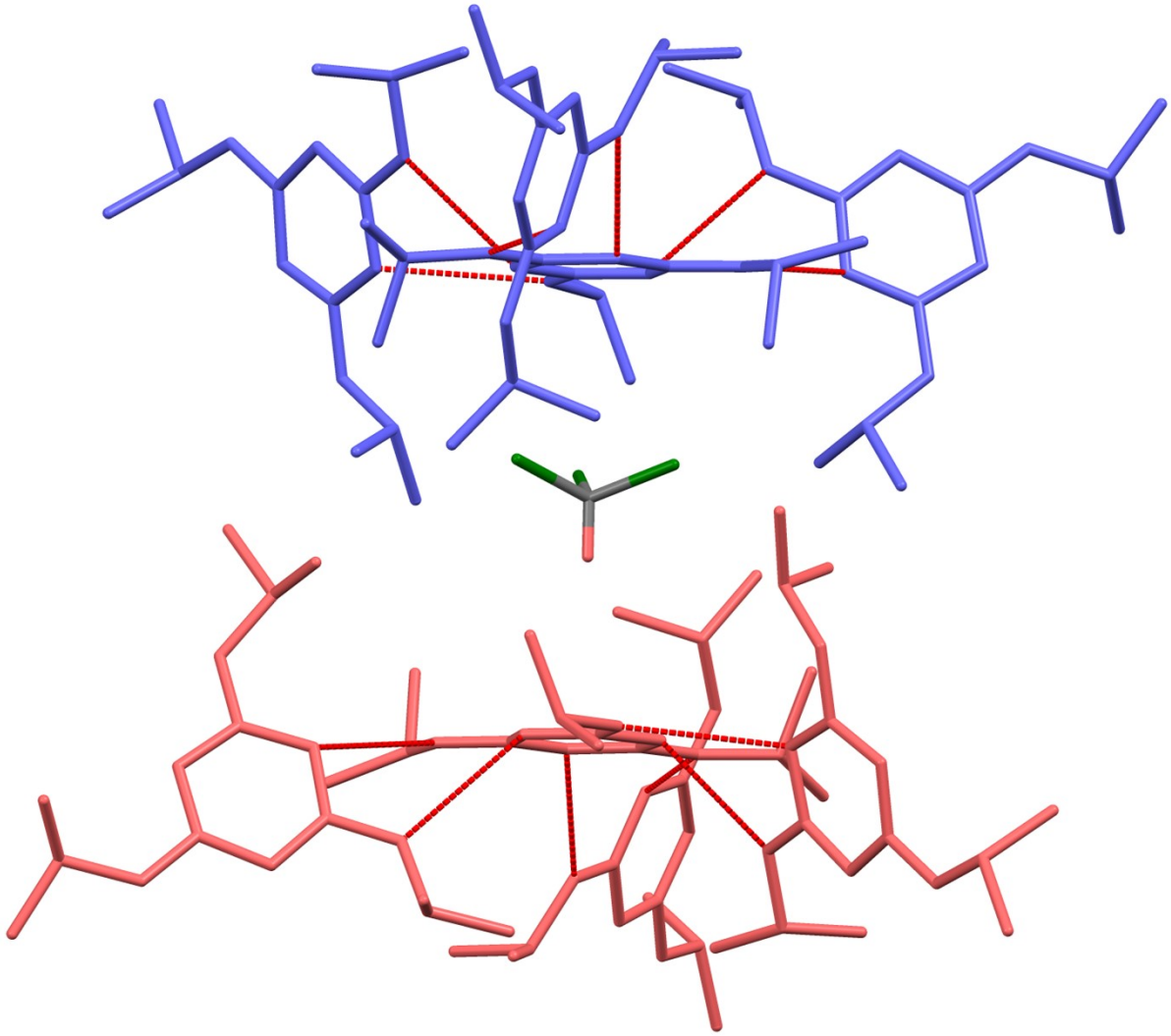


Figure S 17. Piedfort dimer formed by **1** seen from side (space fill model)

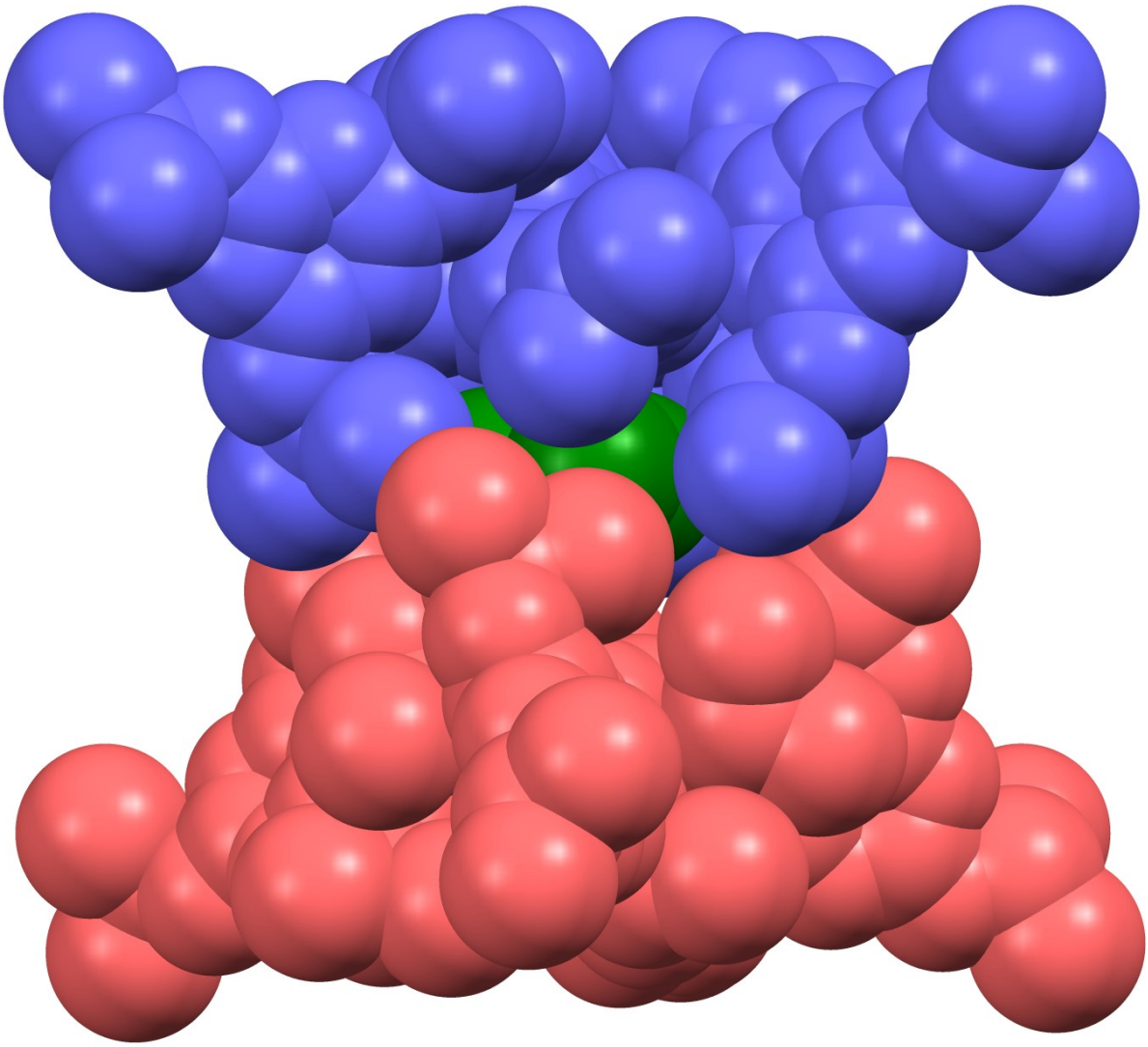


Figure S 18. Piedfort dimer formed by **1** seen from top

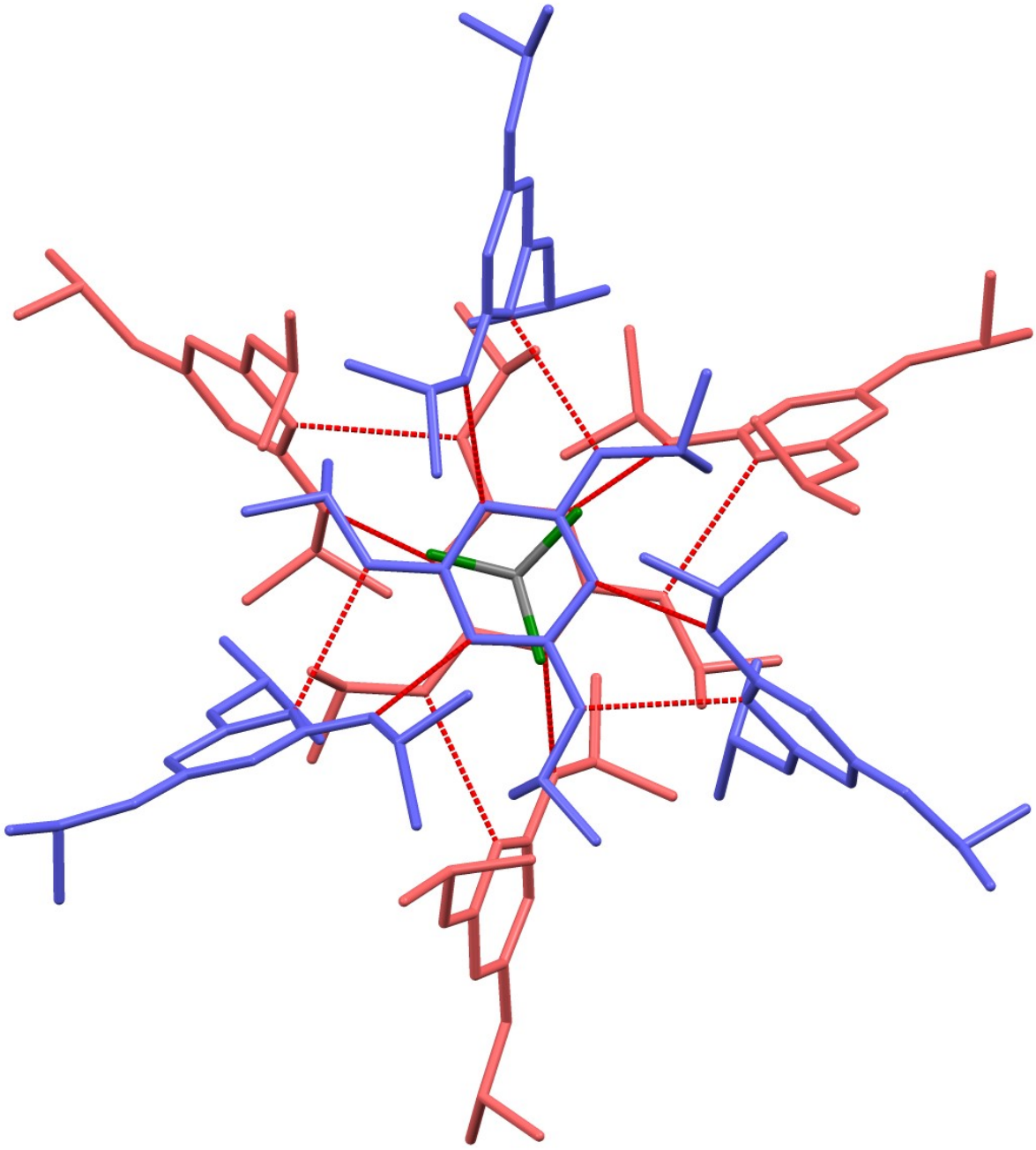


Figure S 19. Piedfort dimer formed by **1** seen from top (space fill model)

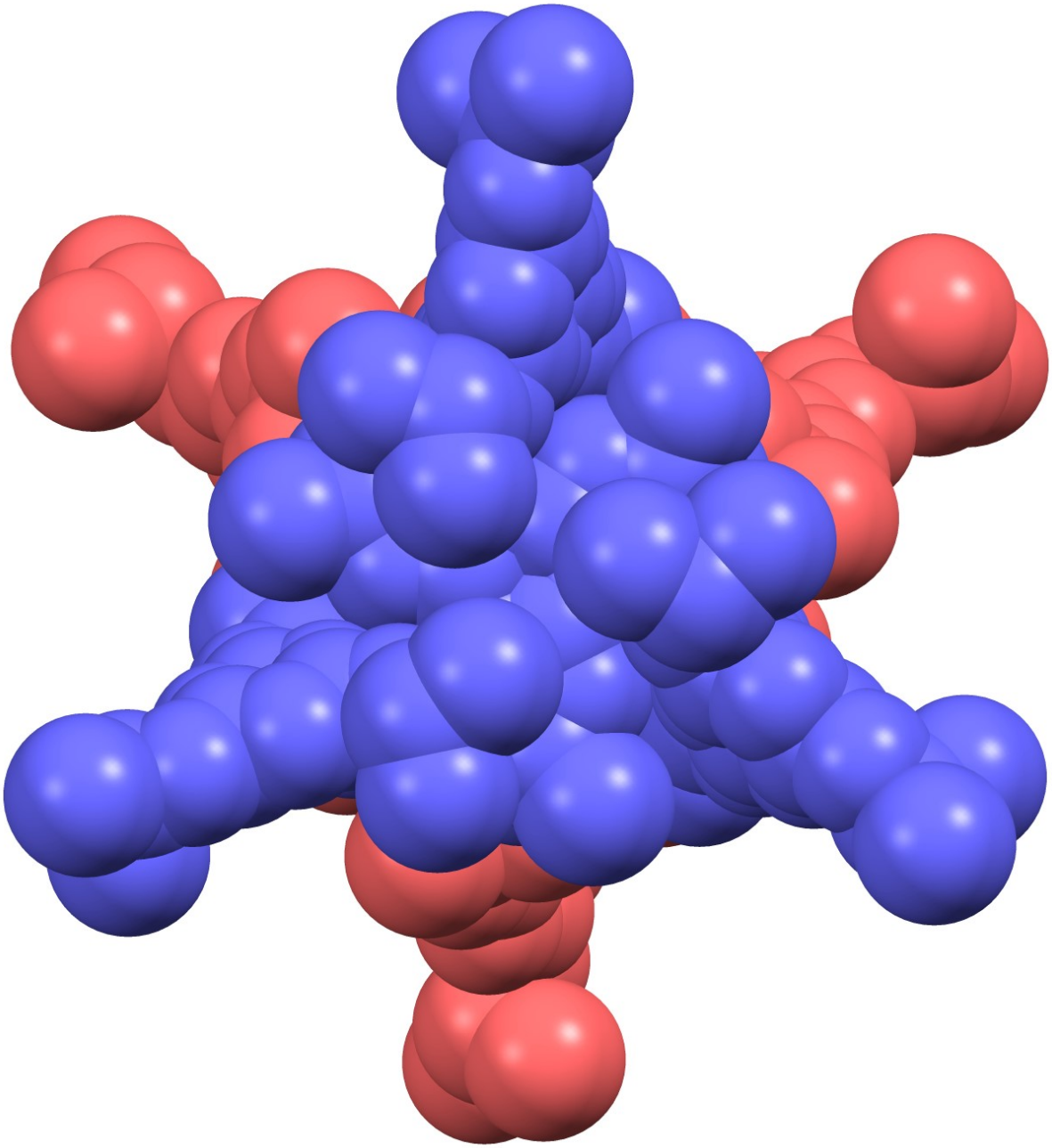
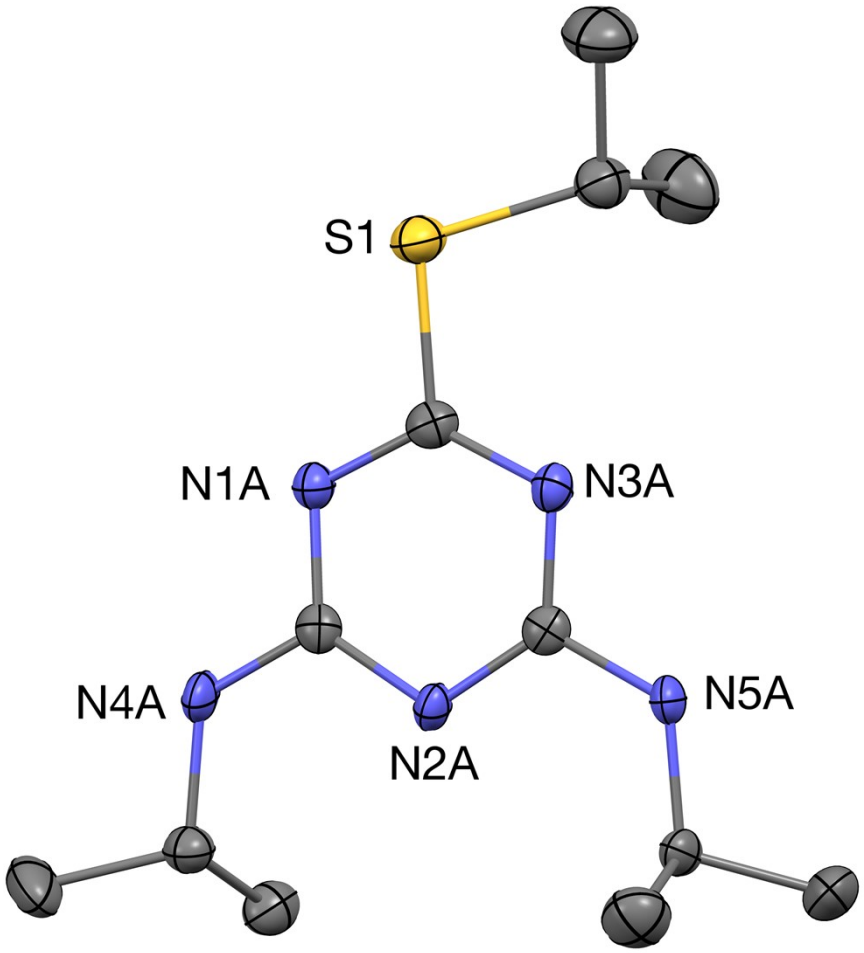


Figure S 20. ORTEP of N^2,N^4-diisopropyl-6-(isopropylthio)-1,3,5-triazine-2,4-diamine **(2)**



CCDC-1454814. Selected bond distances (Å) and angles (°): S(1)-C(1A), 1.759(3); S(1)-C(4A), 1.828(3); N(1A)-C(1A), 1.333(3); N(1A)-C(2A), 1.350(3); N(2A)-C(3A), 1.336(3); N(2A)-C(2A), 1.348(3); N(3A)-C(1A), 1.330(3); N(3A)-C(3A), 1.356(3); N(4A)-C(2A), 1.332(3); N(4A)-C(7A), 1.458(3); N(5A)-C(3A), 1.344(3); N(5A)-C(10A), 1.452(3); C(4A)-C(5A), 1.506(4); C(4A)-C(6A), 1.525(4); C(7A)-C(8A), 1.508(4); C(7A)-C(9A), 1.526(4); C(10A)-C(11A), 1.513(4); C(10A)-C(12A), 1.518(4); C(1A)-S(1)-C(4A), 103.36(13); C(1A)-N(1A)-C(2A), 114.0(2); C(3A)-N(2A)-C(2A), 113.7(2); C(1A)-N(3A)-C(3A), 112.9(2); C(2A)-N(4A)-C(7A), 124.2(2); C(3A)-N(5A)-C(10A), 123.7(2); N(3A)-C(1A)-N(1A), 127.1(3); N(3A)-C(1A)-S(1), 119.8(2); N(1A)-C(1A)-S(1), 113.1(2); N(4A)-C(2A)-N(2A), 117.7(2); N(4A)-C(2A)-N(1A), 117.0(2); N(2A)-C(2A)-N(1A), 125.3(2); N(2A)-C(3A)-N(5A), 117.5(2); N(2A)-C(3A)-N(3A), 126.5(2); N(5A)-C(3A)-N(3A), 115.9(2); C(5A)-C(4A)-C(6A), 111.6(3); C(5A)-C(4A)-S(1), 111.4(2); C(6A)-C(4A)-S(1), 107.3(2).

Figure S 21. Packing diagram of **2** seen thru ‘b’ axis showing the tape motifs

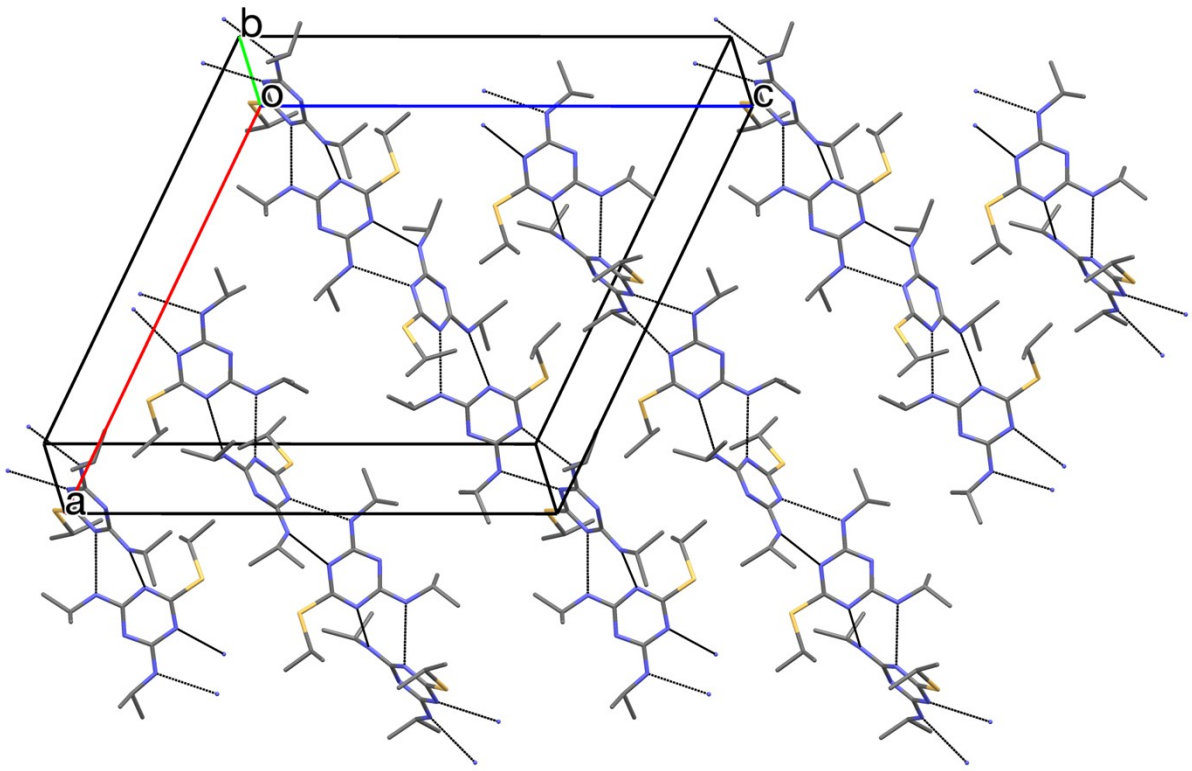


Figure S 22. Packing diagram of **2** seen thru tape motif.

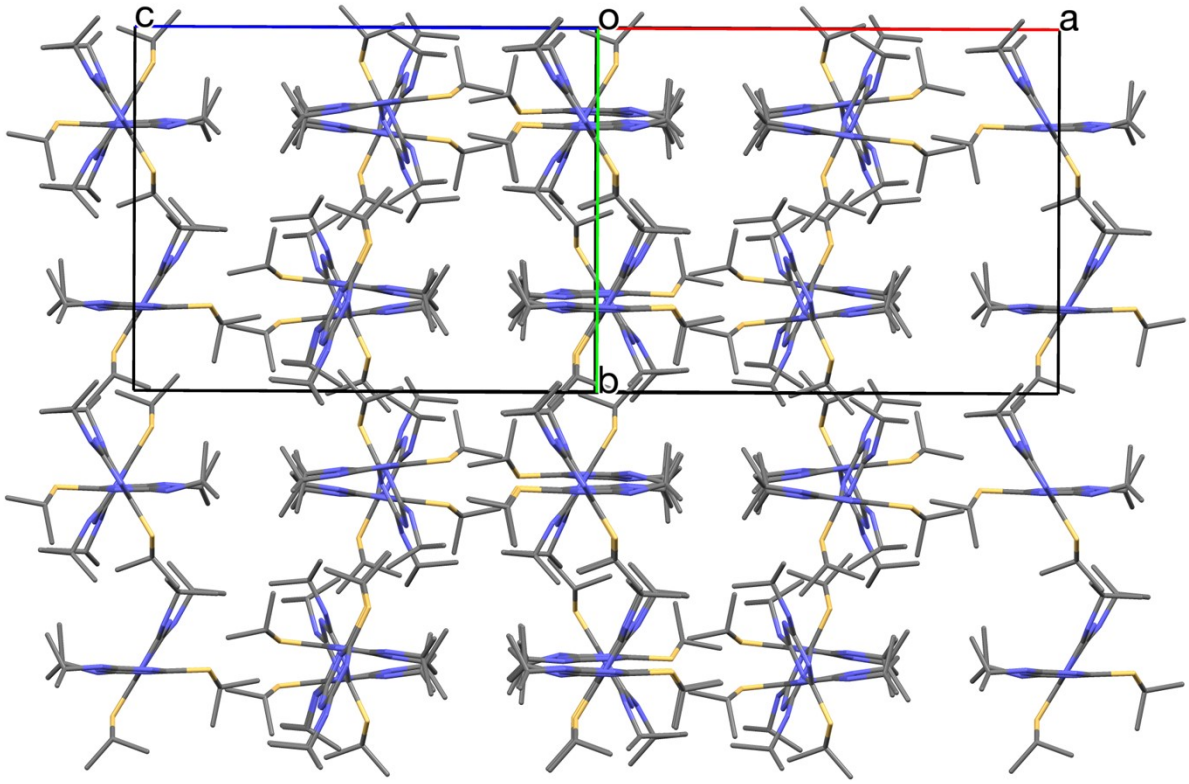


Figure S 23. Packing diagram of **2** thru tape motif.

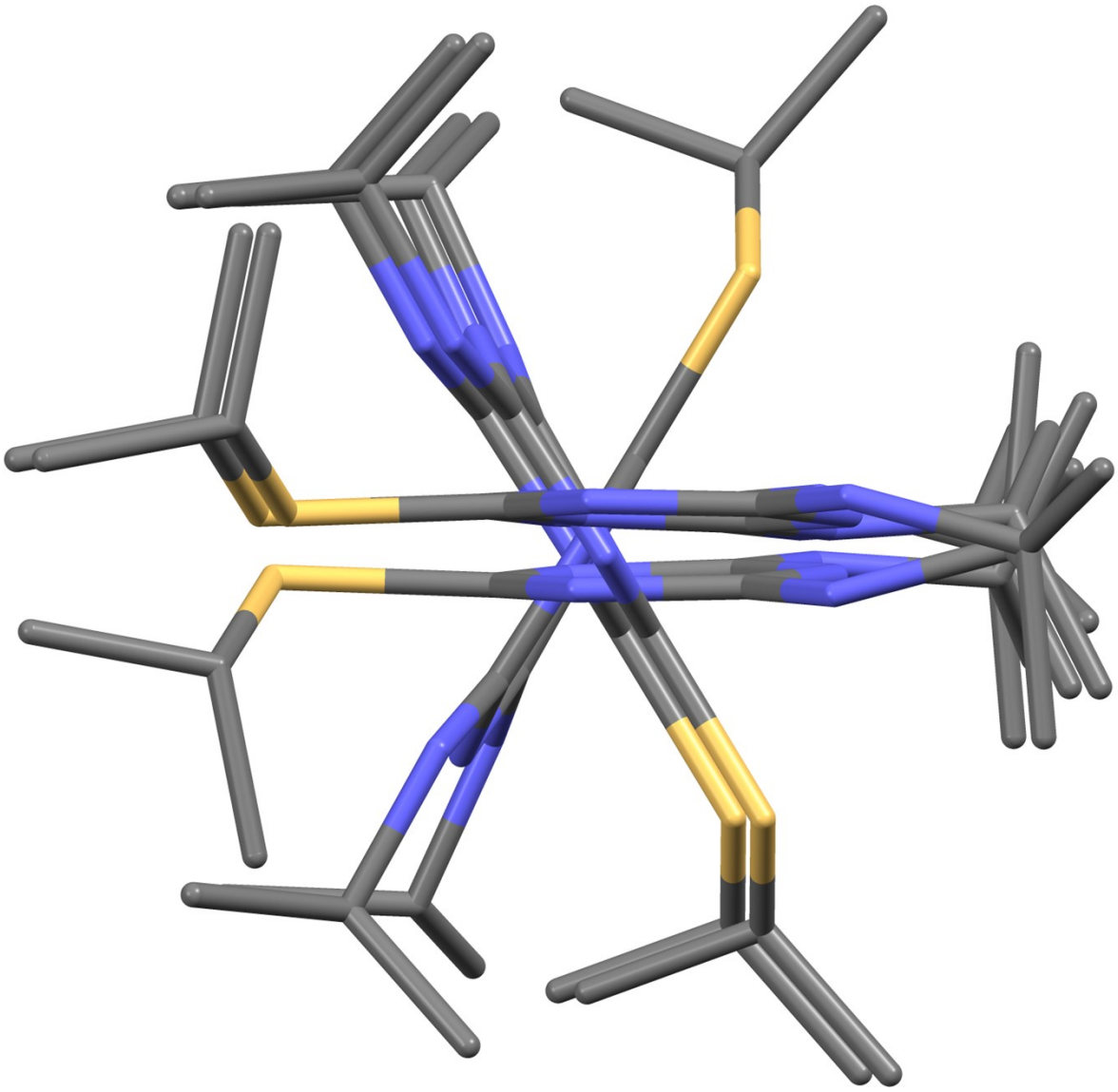
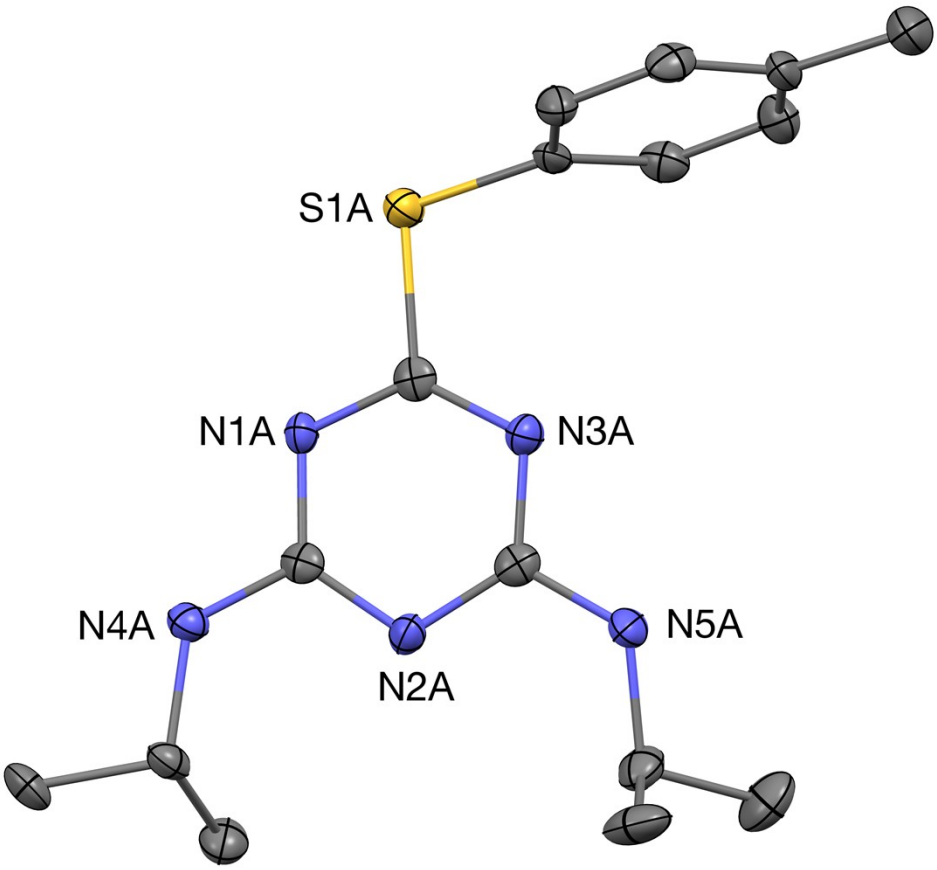


Figure S 24. ORTEP of N^2,N^4-diisopropyl-6-(p-tolylthio)-1,3,5-triazine-2,4-diamine **(3)**



CCDC-1454815. Selected bond distances (Å) and angles (°): S(1A)-C(4A), 1.768(6); S(1A)-C(1A), 1.779(6); N(1A)-C(1A), 1.331(7); N(1A)-C(2A), 1.355(7); N(2A)-C(2A), 1.341(7); N(2A)-C(3A), 1.357(7); N(3A)-C(1A), 1.310(7); N(3A)-C(3A), 1.349(7); N(4A)-C(2A), 1.344(7); N(4A)-C(11A), 1.469(7); N(5A)-C(3A), 1.335(7); N(5A)-C(14A), 1.463(7); C(4A)-S(1A)-C(1A), 102.1(3); C(1A)-N(1A)-C(2A), 113.2(5); C(2A)-N(2A)-C(3A), 114.2(5); C(1A)-N(3A)-C(3A), 113.4(5); C(2A)-N(4A)-C(11A), 124.4(5); C(3A)-N(5A)-C(14A), 124.6(5); N(3A)-C(1A)-N(1A), 128.4(5); N(3A)-C(1A)-S(1A), 121.0(4); N(1A)-C(1A)-S(1A), 110.6(4); N(2A)-C(2A)-N(4A), 118.6(5); N(2A)-C(2A)-N(1A), 125.2(5); N(4A)-C(2A)-N(1A), 116.2(5); N(5A)-C(3A)-N(3A), 116.3(5); N(5A)-C(3A)-N(2A), 118.5(5); N(3A)-C(3A)-N(2A), 125.2(5); C(5A)-C(4A)-C(9A), 119.1(5); C(5A)-C(4A)-S(1A), 121.4(5); C(9A)-C(4A)-S(1A), 119.4(4); C(4A)-C(5A)-C(6A), 119.3(6); C(7A)-C(6A)-C(5A), 121.7(6); C(6A)-C(7A)-C(8A), 118.9(6); C(6A)-C(7A)-C(10A), 120.9(6); C(8A)-C(7A)-C(10A), 120.2(6); C(9A)-C(8A)-C(7A), 120.2(6); C(8A)-C(9A)-C(4A), 120.8(6); N(4A)-C(11A)-C(12A), 109.1(5); N(4A)-C(11A)-C(13A), 111.5(5); C(12A)-C(11A)-C(13A), 111.6(5); N(5A)-C(14A)-C(16A), 111.2(5); N(5A)-C(14A)-C(15A), 108.3(5); C(16A)-C(14A)-C(15A), 111.5(5).

Figure S 25. Packing of **3** showing the 2D network with H-bonds and π···π stacks.

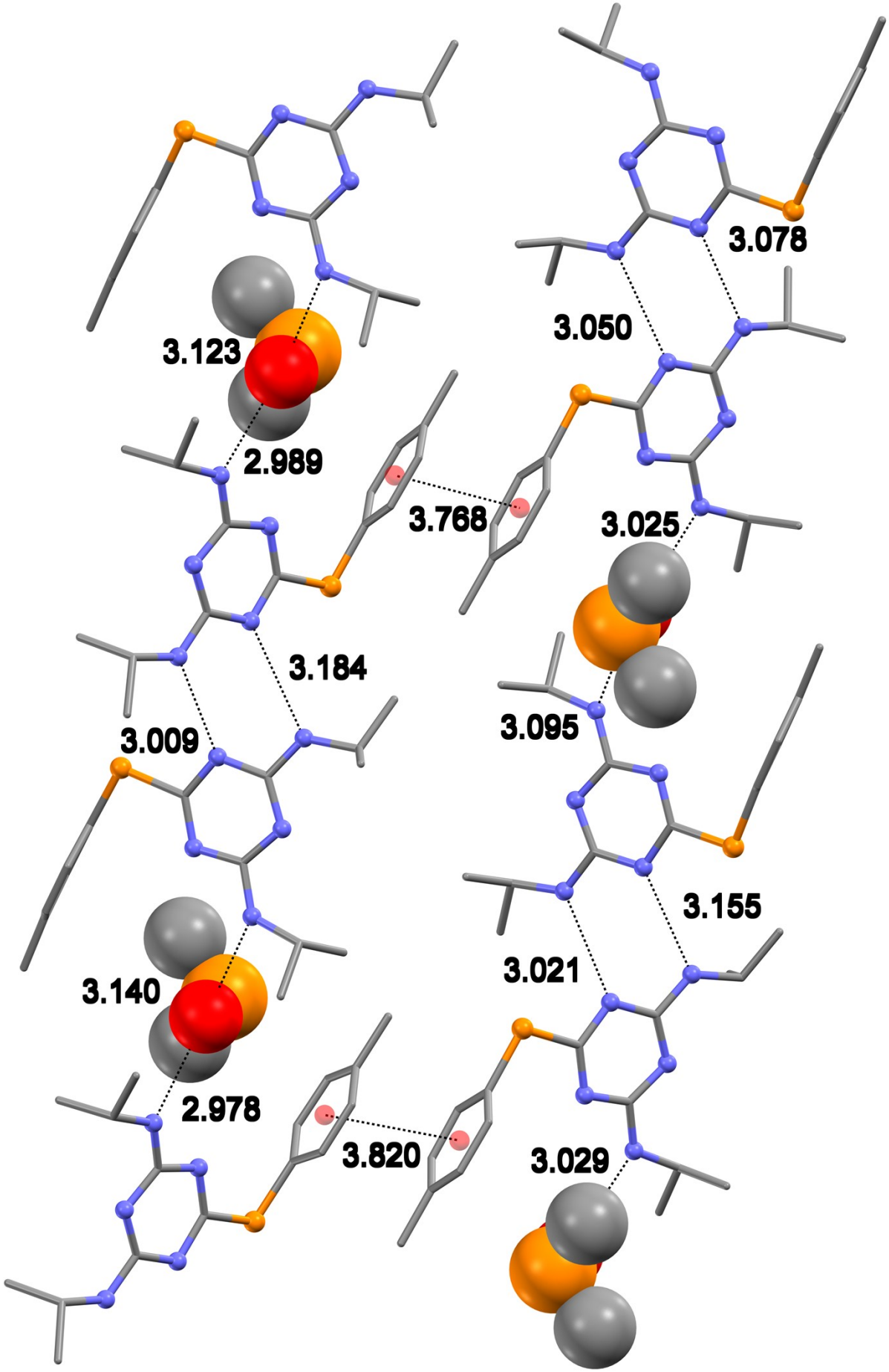


Table S 1. Crystal data for **1-3**

Identification code	8mar_a_sq CCDC-1454811	27auga CCDC-1454814	3sepe CCDC-1454815
Empirical formula	C97 H193 Cl3 N48	C12 H23 N5 S	C34 H52 N10 O S3
Formula weight	2138.33	269.41	713.03
Temperature	123(2) K	123(2) K	123(2) K
Wavelength	0.71073 Å	0.71073 Å	0.71073 Å
Crystal system	Trigonal	Monoclinic	Triclinic
Space group	R -3 c :H	P 21/n	P 1
Unit cell dimensions	a = 22.1441(7) Å α = 90°	a = 21.0568(9) Å α = 90°	a = 17.0555(7) Å α = 81.0580(10)°.
	b = 22.1441(7) Å β = 90°.	b = 14.4050(6) Å β = 114.4290(10)°	b = 21.6567(8) Å β = 79.5800(10)°.
	c = 96.226(5) Å γ = 120°.	c = 22.7028(9) Å γ = 90°	c = 23.7689(9) Å γ = 66.8910(10)°.
Volume	40864(3) $Å^3$	6269.8(5) $Å^3$	6269.8(5) $Å^3$
Z	12	16	16
Density (calculated)	1.043 Mg/m^3	1.142 Mg/m^3	1.142 Mg/m^3
Absorption coefficient	0.124 mm^{-1}	0.200 mm^{-1}	0.200 mm^{-1}
F(000)	13944	2336	2336
Crystal size	0.21 x 0.18 x 0.08 mm^3	0.27 x 0.18 x 0.11 mm^3	0.27 x 0.18 x 0.11 mm^3
Theta range for data collection	2.166 to 25.263°.	2.228 to 25.252°.	2.228 to 25.252°.
Index ranges	-26<=h<=26, -26<=k<=26, -115<=l<=115	-25<=h<=25, -17<=k<=17, -27<=l<=26	-25<=h<=25, - 17<=k<=17, - 27<=l<=26
Reflections collected	161522	50360	50360
Independent reflections	8242 [R(int) = 0.1403]	11345 [R(int) = 0.0959]	11345 [R(int) = 0.0959]
Completeness to theta = 25.263°	99.9 %	99.8 %	99.8 %
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents	Semi-empirical from equivalents
Max. and min. transmission	0.7457 and 0.6158	0.7457 and 0.6053	0.7457 and 0.6338
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	8242 / 20 / 499	11345 / 0 / 673	77502 / 93 / 3599
Goodness-of-fit on F^2	1.020	1.007	0.989
Final R indices [I>2sigma(I)]	R1 = 0.0769, wR2 = 0.1859	R1 = 0.0585, wR2 = 0.1065	R1 = 0.0825, wR2 = 0.1150
R indices (all data)	R1 = 0.1304, wR2 = 0.2172	R1 = 0.1270, wR2 = 0.1266	R1 = 0.1826, wR2 = 0.1433
Largest diff. peak and hole	1.594 and -1.722 $e.Å^{-3}$	0.384 and -0.383 $e.Å^{-3}$	0.527 and -0.486 $e.Å^{-3}$

1. G. M. Sheldrick, *Acta Crystallogr A Found Adv*, 2015, **71**, 3-8.
2. G. M. Sheldrick, *Acta Crystallogr C Struct Chem*, 2015, **71**, 3-8.