Electronic Supplementary Material (ESI) for CrystEngComm.


# Short contacts of the sulphur atoms of a 1,2,3,5-dithiadiazolyl dimer with triphenylstibine: first co-crystal with an aromatic compound

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## Electronic Supplementary Information

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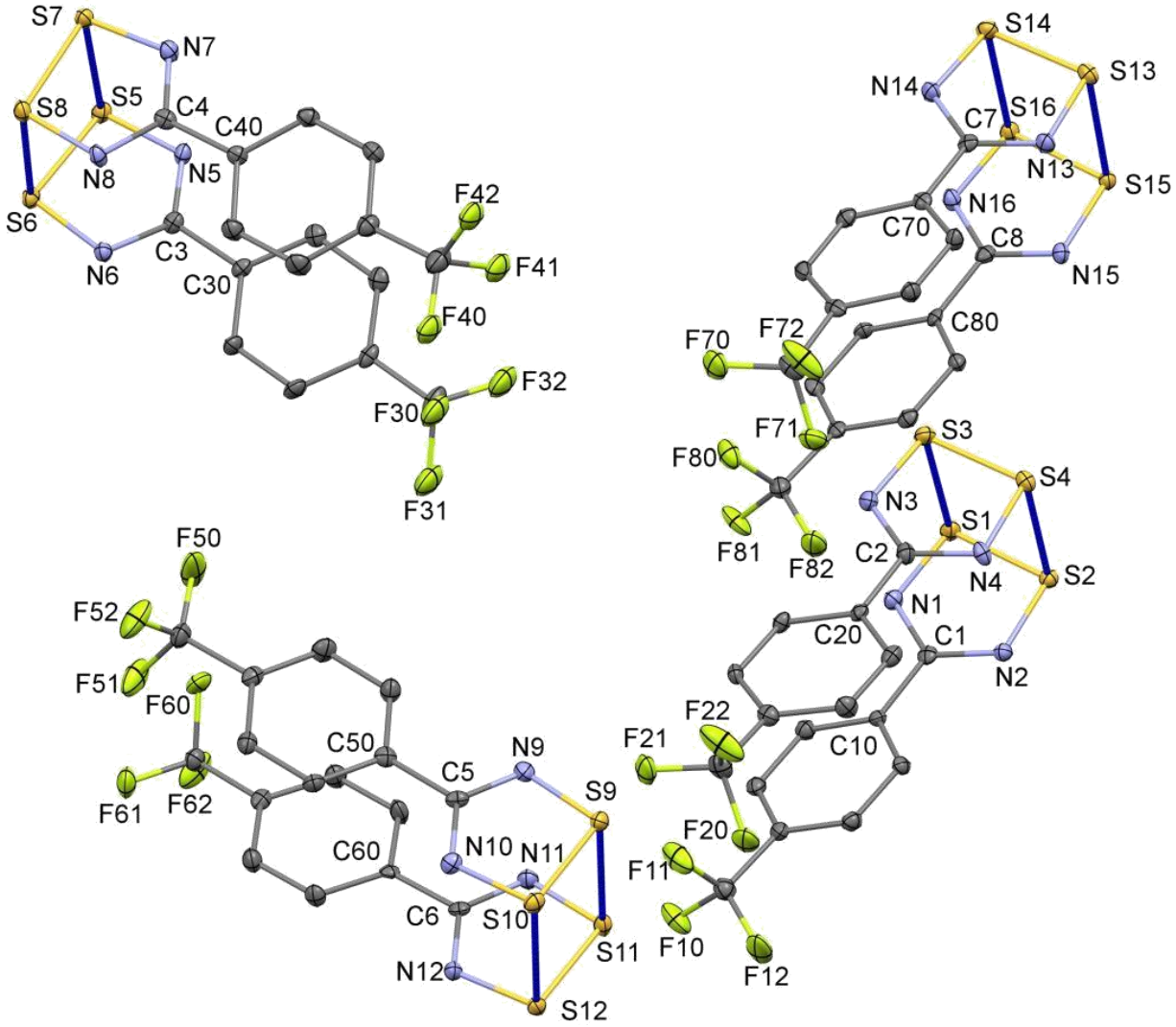



**Figure S1**. The full atom numbering scheme of the structure of **1**. The two-part disorder model for the fluorine atoms in dimer **1iii** is also shown (refined occupancy ratio 67:33).

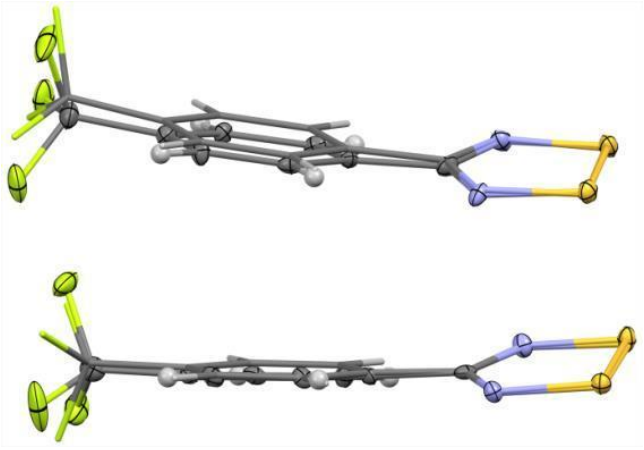


**Figure S2**. An overlay of structures of dtda dimers in **2** shown as a line diagram and one of the four independent dimers in **1**, shown as displacement ellipsoids.

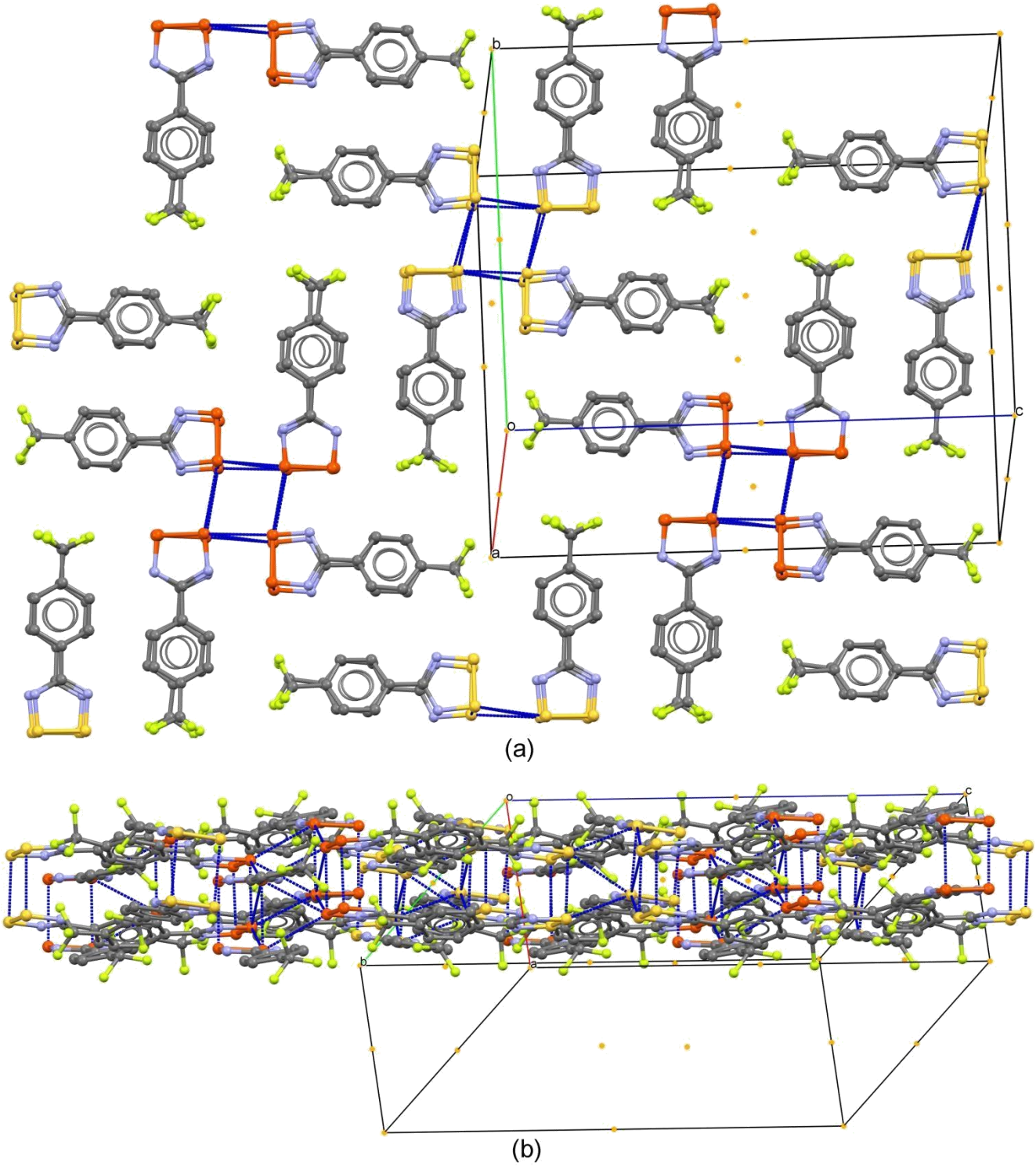



**Figure S3**. Two views of the layer structure in the crystal lattice of **1**. The layer is ~8.3 Å thick and parallel to the (1 1 0) Miller plane. Both unique pin-wheel tetramers lie within the plane which is seen from (a) a top view and

(b) a side view. The unit cell boundaries are shown as are the locations of the sites by yellow dots. The sulfur atoms are false-coloured to orange and yellow to distinguish the first and second (S' in Figure 2) pin-wheels.

**Table S1**. Intermolecular Contacts in **1** less than ($\Sigma r_{vdW}$ – 0.1 Å) (except for inter-slice distances)

| **Atom1** | **Atom2** | **Symm. op. 1** | **Symm. op. 2** | **Length** | **Length < $\Sigma r_{vdW}$** |
|---|---|---|---|---|---|
| Intra-Dimer | | | | | |
| S2 | S4 | x,y,z | x,y,z | 3.031(2) | -0.569 |
| S6 | S8 | x,y,z | -1+x,y,z | 3.031(2) | -0.569 |
| S14 | S16 | x,y,z | 1+x,y,z | 3.043(2) | -0.557 |
| S13 | S15 | x,y,z | 1+x,y,z | 3.046(2) | -0.554 |
| S1 | S3 | x,y,z | x,y,z | 3.054(2) | -0.546 |
| S9 | S11 | x,y,z | x,y,z | 3.076(2) | -0.524 |
| S10 | S12 | x,y,z | x,y,z | 3.110(2) | -0.490 |
| S6 | S7 | x,y,z | -1+x,y,z | 3.170(2) | -0.430 |
| | | | *Mean(s.u.)* | *3.07(5)* | *-0.53(5)* |
| Dimer-to- neighbour | | | | | |
| S15 | S10 | x,y,z | 1-x,1-y,1-z | 3.247(2) | -0.353 |
| S6 | S2 | x,y,z | -1+x,1+y,z | 3.259(2) | -0.341 |
| S15 | S12 | x,y,z | -1+x,y,1+z | 3.271(2) | -0.329 |
| N15 | S10 | x,y,z | 1-x,1-y,1-z | 3.130(4) | -0.220 |
| S7 | S4 | -1+x,y,z | -1+x,1+y,z | 3.387(2) | -0.213 |
| S13 | S10 | -1+x,y,z | -1+x,y,1+z | 3.391(2) | -0.209 |
| S7 | S2 | -1+x,y,z | -x,1-y,1-z | 3.399(2) | -0.201 |
| S6 | N2 | x,y,z | 3.156(4) | 3.156(4) | -0.194 |
| S6 | S4 | x,y,z | -x,1-y,1-z | 3.419(2) | -0.181 |
| S13 | S12 | -1+x,y,z | -1+x,y,1+z | 3.437(2) | -0.163 |
| N15 | S9 | x,y,z | 1-x,1-y,1-z | 3.225(4) | -0.125 |
| S16 | N12 | x,y,z | -1+x,y,1+z | 3.230(4) | -0.120 |
| N7 | S3 | -1+x,y,z | -x,1-y,1-z | 3.241(4) | -0.109 |
| | | | *S-S Mean(s.u.)* | 3.35(8) | -0.25(7) |
| | | | *S-N Mean(s.u.)* | 3.23(5) | -0.19(8) |
| Inter-slice | | | | | |
| S14 | S10 | x,y,z | 1-x,1-y,1-z | 3.711(2) | +0.111 |
| S3 | S6 | x,y,z | x,1+y,z | 3.765(2) | +0.165 |

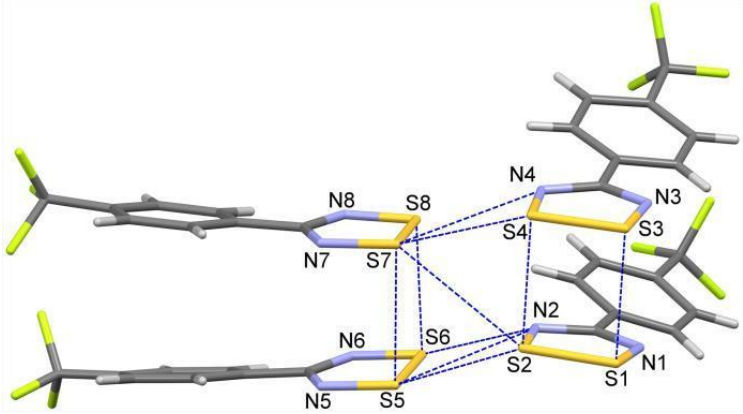



**Figure S4**. Detailed view of the end-to-side contacts of one DTDA dimer with another in the same layer in the structure of **1**. This group is part of the left-hand cluster shown in Figure 2*a.*

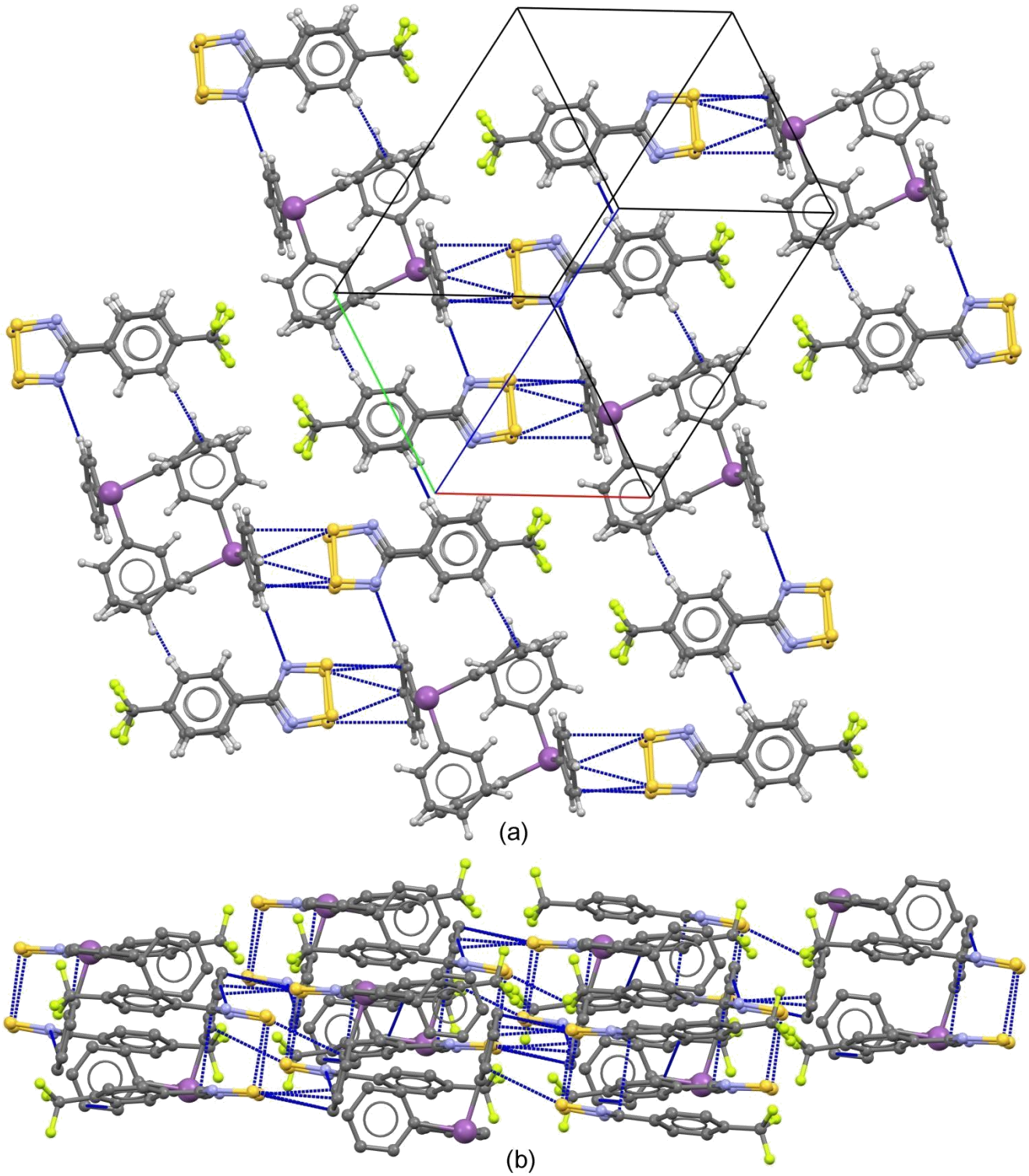



**Figure S5**. Two views of the layer structure in the crystal lattice of **2**. The slice, which is ~8.0 Å thick and lies along the (1 1 0) Miller plane, contains the centrosymmetric $(DTDA)_2(Ph_3Sb)_2$. It is presented from (a) a top view and (b) a side view. The unit cell boundaries are also shown. In this structure, the closest inter-layer contacts are S···F at 3.231 (9) Å due to an offset packing that precludes any long-range S···S interactions.

**Table S2**. Intermolecular Contacts in **2** less than $\Sigma r_{vdW}$ (except for intermolecular $Ph_3Sb$)

| Atom1 | Atom2 | Symm. op. 1 | Symm. op. 2 | Length | Length- $\Sigma r_{vdW}$ |
|---|---|---|---|---|---|
| Intra-Dimer | | | | | |
| S1 | S11 | x,y,z | x,y,z | 3.044(1) | -0.556 |
| S2 | S12 | x,y,z | x,y,z | 3.0913(9) | -0.509 |
| C1 | C11 | x,y,z | x,y,z | 3.380(3) | -0.02 |
| | | | *Mean(s.u.)* | *3.068(1)* | *-0.53* |
| Dimer-to-end neighbour | | | | | |
| S11 | C44 | x,y,z | x,y,z | 3.168(3) | -0.332 |
| S11 | C43 | x,y,z | x,y,z | 3.360(3) | -0.14 |
| S12 | C43 | x,y,z | x,y,z | 3.388(4) | -0.112 |
| S1 | C45 | x,y,z | x,y,z | 3.458(3) | -0.042 |
| S12 | C42 | x,y,z | x,y,z | 3.464(3) | -0.036 |
| | | | *Mean(s.u.)* | *3.37(11)* | *-0.13(11)* |
| Dimer to side neighbour | | | | | |
| H16 | C34 | x,y,z | 1-x,1-y,1-z | 2.742 | -0.158 |
| N1 | H44 | x,y,z | 1-x,1-y,1-z | 2.686 | -0.064 |
| | | | | | |
| Inter-slice | | | | | |
| F2 | S12 | x,y,z | -1+x,y,z | 3.231(9) | -0.039 |
| | | | | | |
| Inter $Ph_3Sb$ | | | | | |
| H22 | C43 | x,y,z | 2-x,-y,1-z | 2.915 | +0.015 |
| C32 | H41 | x,y,z | 2-x,-y,1-z | 2.932 | +0.032 |
| H22 | C44 | x,y,z | 2-x,-y,1-z | 2.995 | +0.095 |

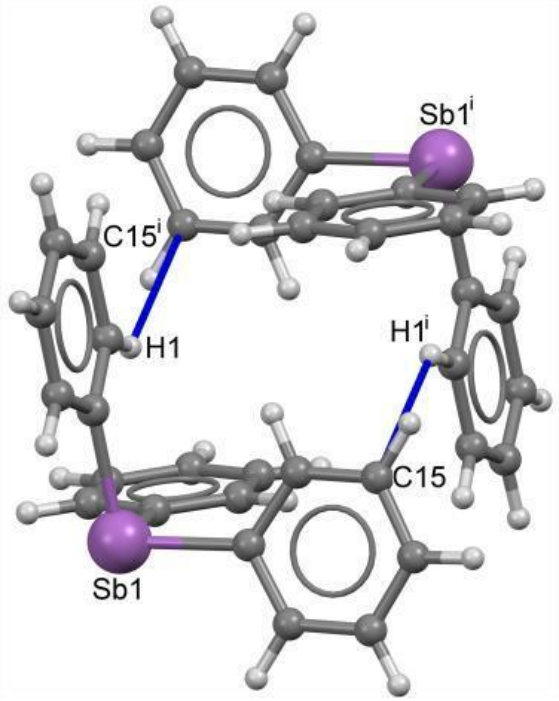


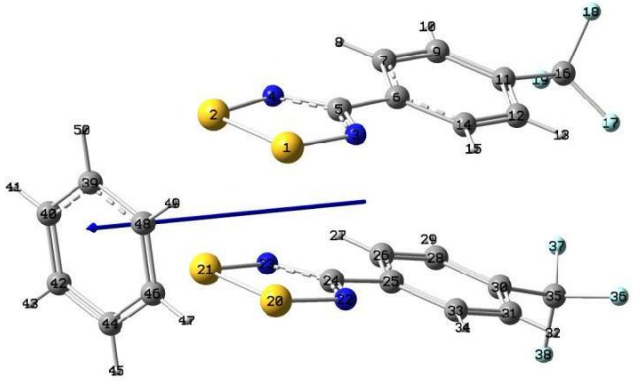

**Figure S6**. Depiction of one of the centrosymmetric pseudo-cuboidal units found in the monoclinic form of **3** (refcode: ZZEHEA02)[26] which may be compared to **1** in Figure 1. [Symmetry code: (i), 1-x,-y,1-z.]

**Table S3**. Summary of Natural Population Analysis in a model system for **2.**

| Atom | No | Charge | Core | Valence | Rydberg | Total |
|---|---|---|---|---|---|---|
| S | 1 | 0.46284 | 9.99912 | 5.46648 | 0.0716 | 15.53716 |
| S | 2 | 0.46687 | 9.99913 | 5.46319 | 0.0708 | 15.53313 |
| N | 3 | -0.71607 | 1.9994 | 5.656 | 0.0607 | 7.71607 |
| N | 4 | -0.7048 | 1.99938 | 5.64458 | 0.0608 | 7.7048 |
| C | 5 | 0.49662 | 1.99911 | 3.46804 | 0.0362 | 5.50338 |
| C | 6 | -0.11434 | 1.99907 | 4.0725 | 0.0428 | 6.11434 |
| C | 7 | -0.13465 | 1.99903 | 4.11892 | 0.0167 | 6.13465 |
| H | 8 | 0.19407 | 0 | 0.80359 | 0.0023 | 0.80593 |
| C | 9 | -0.15825 | 1.99903 | 4.14237 | 0.0169 | 6.15825 |
| H | 10 | 0.19013 | 0 | 0.80713 | 0.0027 | 0.80987 |
| C | 11 | -0.10664 | 1.99896 | 4.08368 | 0.024 | 6.10664 |
| C | 12 | -0.15995 | 1.99902 | 4.1445 | 0.0164 | 6.15995 |
| H | 13 | 0.19026 | 0 | 0.80697 | 0.0028 | 0.80974 |
| C | 14 | -0.12985 | 1.99903 | 4.11327 | 0.0176 | 6.12985 |
| H | 15 | 0.19433 | 0 | 0.80314 | 0.0025 | 0.80567 |
| C | 16 | 1.01014 | 1.99921 | 2.93246 | 0.0582 | 4.98986 |
| F | 17 | -0.32332 | 1.99989 | 7.3104 | 0.013 | 9.32332 |
| F | 18 | -0.31723 | 1.99989 | 7.30622 | 0.0111 | 9.31723 |
| F | 19 | -0.34664 | 1.99991 | 7.33546 | 0.0113 | 9.34664 |
| S | 20 | 0.46868 | 9.99912 | 5.46016 | 0.072 | 15.53132 |
| S | 21 | 0.46922 | 9.99913 | 5.46051 | 0.0712 | 15.53078 |
| N | 22 | -0.71008 | 1.9994 | 5.65088 | 0.0598 | 7.71008 |
| N | 23 | -0.70609 | 1.99939 | 5.64654 | 0.0602 | 7.70609 |
| C | 24 | 0.49605 | 1.99911 | 3.46982 | 0.035 | 5.50395 |
| C | 25 | -0.10808 | 1.99906 | 4.06609 | 0.0429 | 6.10808 |
| C | 26 | -0.12959 | 1.99903 | 4.11333 | 0.0172 | 6.12959 |
| H | 27 | 0.19504 | 0 | 0.80251 | 0.0025 | 0.80496 |
| C | 28 | -0.14988 | 1.99902 | 4.13402 | 0.0168 | 6.14988 |
| H | 29 | 0.19302 | 0 | 0.80421 | 0.0028 | 0.80698 |
| C | 30 | -0.12312 | 1.99894 | 4.10079 | 0.0234 | 6.12312 |
| C | 31 | -0.14986 | 1.99903 | 4.13436 | 0.0165 | 6.14986 |
| H | 32 | 0.19027 | 0 | 0.80703 | 0.0027 | 0.80973 |
| C | 33 | -0.14171 | 1.99902 | 4.12629 | 0.0164 | 6.14171 |
| H | 34 | 0.1955 | 0 | 0.8022 | 0.0023 | 0.8045 |
| C | 35 | 1.01492 | 1.99915 | 2.92881 | 0.0571 | 4.98508 |
| F | 36 | -0.33182 | 1.9999 | 7.32075 | 0.0112 | 9.33182 |
| F | 37 | -0.34173 | 1.99991 | 7.33036 | 0.0115 | 9.34173 |
| F | 38 | -0.33275 | 1.9999 | 7.32284 | 0.01 | 9.33275 |
| C | 39 | -0.20323 | 1.99924 | 4.18559 | 0.0184 | 6.20323 |
| C | 40 | -0.17295 | 1.99912 | 4.1565 | 0.0173 | 6.17295 |
| H | 41 | 0.17907 | 0 | 0.81852 | 0.0024 | 0.82093 |
| C | 42 | -0.16968 | 1.99915 | 4.15528 | 0.0153 | 6.16968 |
| H | 43 | 0.1798 | 0 | 0.81784 | 0.0024 | 0.8202 |

| | | | | | | |
|---|---|---|---|---|---|---|
| C | 44 | -0.17936 | 1.99916 | 4.16384 | 0.0164 | 6.17936 |
| H | 45 | 0.18021 | 0 | 0.81732 | 0.0025 | 0.81979 |
| C | 46 | -0.1818 | 1.99913 | 4.16438 | 0.0183 | 6.1818 |
| H | 47 | 0.18112 | 0 | 0.81648 | 0.0024 | 0.81888 |
| C | 48 | -0.18725 | 1.99912 | 4.17028 | 0.0178 | 6.18725 |
| H | 49 | 0.17939 | 0 | 0.81825 | 0.0024 | 0.82061 |
| H | 50 | 0.21321 | 0 | 0.78525 | 0.0015 | 0.78679 |
| * Tot | al * | 0.01003 | 103.9732 | 186.8299 | 1.1868 | 291.99 |

Determined by a PBEPBE/6-311+g(2df,2p) DFT calculations in Gaussian W03. The numbering scheme for the DFT calculation is shown below.



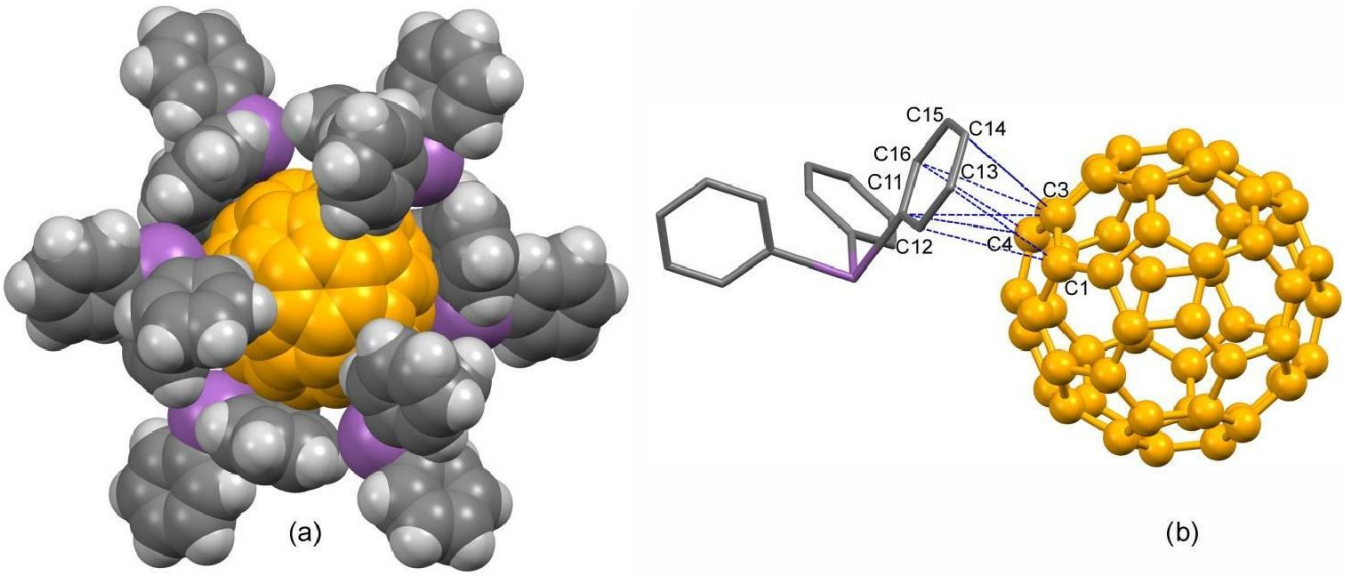



**Figure S7**. Another view of the $C_{60}$·$6Ph_3Sb$ structure which shares such a close similarity in the interaction of a negative charge region above the Ph ring and the positively charged 6:5 junction point in the fullerene (the latter is rendered orange for contrast).

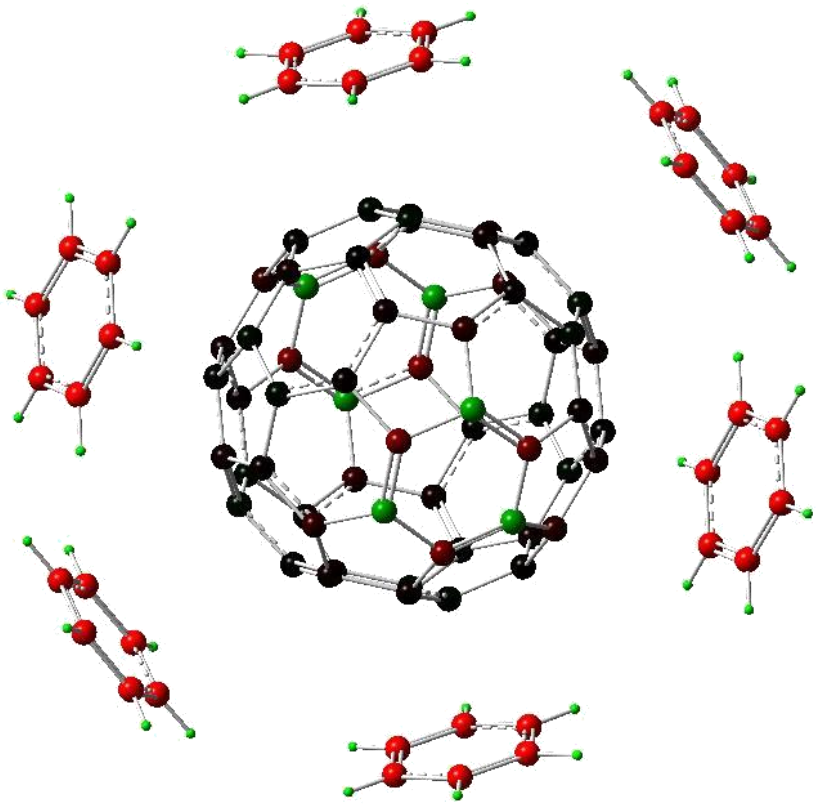


**Figure S8.** PBEPBE/6-311+g(2df,2p) DFT calculated NPA charge distribution in $C_{60}$ : 6 $C_6H_6$ at the geometry of the $Ph_3Sb$ phenyl rings in YIKVET. Each benzene ring is centred over a 6:5 ring junction in $C_{60}$.

**Table S4**. Summary of Natural Population Analysis in a model system for **4** where six $C_6H_6$ replace six $Ph_3Sb$.

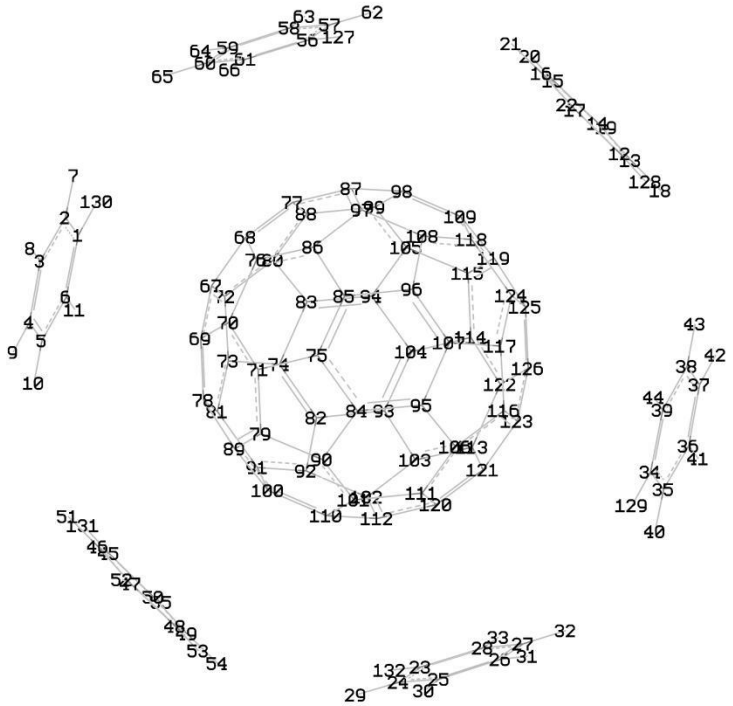
| Atom | No | Charge | Core | Valence | Rydberg | Total |
|---|---|---|---|---|---|---|
| C benzene | 1 | -0.19977 | 1.99922 | 4.18313 | 0.01742 | 6.19977 |
| C benzene | 2 | -0.17842 | 1.99915 | 4.16298 | 0.0163 | 6.17842 |
| C benzene | 3 | -0.18049 | 1.99916 | 4.16537 | 0.01596 | 6.18049 |
| C benzene | 4 | -0.18135 | 1.99917 | 4.16622 | 0.01596 | 6.18135 |
| C benzene | 5 | -0.17535 | 1.99917 | 4.16005 | 0.01614 | 6.17535 |
| C benzene | 6 | -0.18165 | 1.99914 | 4.16623 | 0.01628 | 6.18165 |
| H | 7 | 0.17935 | 0 | 0.81835 | 0.0023 | 0.82065 |
| H | 8 | 0.18311 | 0 | 0.81467 | 0.00222 | 0.81689 |
| H | 9 | 0.18198 | 0 | 0.81577 | 0.00224 | 0.81802 |
| H | 10 | 0.18045 | 0 | 0.81724 | 0.00231 | 0.81955 |
| H | 11 | 0.18262 | 0 | 0.81524 | 0.00215 | 0.81738 |
| C | 12 | -0.19982 | 1.99922 | 4.18318 | 0.01742 | 6.19982 |
| C | 13 | -0.17841 | 1.99915 | 4.16296 | 0.0163 | 6.17841 |
| C | 14 | -0.1805 | 1.99916 | 4.16538 | 0.01596 | 6.1805 |
| C | 15 | -0.18132 | 1.99917 | 4.16619 | 0.01596 | 6.18132 |
| C | 16 | -0.17534 | 1.99917 | 4.16004 | 0.01614 | 6.17534 |
| C | 17 | -0.18166 | 1.99914 | 4.16624 | 0.01628 | 6.18166 |
| H | 18 | 0.17935 | 0 | 0.81835 | 0.0023 | 0.82065 |
| H | 19 | 0.18312 | 0 | 0.81466 | 0.00222 | 0.81688 |

| | | | | | | |
|---|---|---|---|---|---|---|
| H | 20 | 0.18198 | 0 | 0.81578 | 0.00224 | 0.81802 |
| H | 21 | 0.18044 | 0 | 0.81724 | 0.00231 | 0.81956 |
| H | 22 | 0.18262 | 0 | 0.81523 | 0.00215 | 0.81738 |
| C | 23 | -0.19979 | 1.99922 | 4.18315 | 0.01742 | 6.19979 |
| C | 24 | -0.17838 | 1.99915 | 4.16293 | 0.0163 | 6.17838 |
| C | 25 | -0.18048 | 1.99916 | 4.16535 | 0.01596 | 6.18048 |
| C | 26 | -0.18136 | 1.99917 | 4.16623 | 0.01596 | 6.18136 |
| C | 27 | -0.17538 | 1.99917 | 4.16008 | 0.01614 | 6.17538 |
| C | 28 | -0.1817 | 1.99914 | 4.16628 | 0.01628 | 6.1817 |
| H | 29 | 0.17934 | 0 | 0.81835 | 0.0023 | 0.82066 |
| H | 30 | 0.18313 | 0 | 0.81465 | 0.00222 | 0.81687 |
| H | 31 | 0.18199 | 0 | 0.81577 | 0.00224 | 0.81801 |
| H | 32 | 0.18046 | 0 | 0.81723 | 0.00231 | 0.81954 |
| H | 33 | 0.18262 | 0 | 0.81524 | 0.00215 | 0.81738 |
| C | 34 | -0.19978 | 1.99922 | 4.18314 | 0.01742 | 6.19978 |
| C | 35 | -0.17843 | 1.99915 | 4.16298 | 0.0163 | 6.17843 |
| C | 36 | -0.18048 | 1.99916 | 4.16536 | 0.01596 | 6.18048 |
| C | 37 | -0.18136 | 1.99917 | 4.16623 | 0.01596 | 6.18136 |
| C | 38 | -0.17538 | 1.99917 | 4.16008 | 0.01614 | 6.17538 |
| C | 39 | -0.18162 | 1.99914 | 4.16621 | 0.01628 | 6.18162 |
| H | 40 | 0.17934 | 0 | 0.81835 | 0.0023 | 0.82066 |
| H | 41 | 0.18309 | 0 | 0.81469 | 0.00222 | 0.81691 |
| H | 42 | 0.18199 | 0 | 0.81577 | 0.00224 | 0.81801 |
| H | 43 | 0.18044 | 0 | 0.81724 | 0.00231 | 0.81956 |
| H | 44 | 0.1826 | 0 | 0.81525 | 0.00215 | 0.8174 |
| C | 45 | -0.19982 | 1.99922 | 4.18317 | 0.01742 | 6.19982 |
| C | 46 | -0.1784 | 1.99915 | 4.16296 | 0.0163 | 6.1784 |
| C | 47 | -0.18048 | 1.99916 | 4.16536 | 0.01596 | 6.18048 |
| C | 48 | -0.18132 | 1.99917 | 4.16619 | 0.01596 | 6.18132 |
| C | 49 | -0.17535 | 1.99917 | 4.16005 | 0.01614 | 6.17535 |
| C | 50 | -0.18165 | 1.99914 | 4.16623 | 0.01628 | 6.18165 |
| H | 51 | 0.17935 | 0 | 0.81835 | 0.0023 | 0.82065 |
| H | 52 | 0.1831 | 0 | 0.81468 | 0.00222 | 0.8169 |
| H | 53 | 0.18199 | 0 | 0.81576 | 0.00224 | 0.81801 |
| H | 54 | 0.18044 | 0 | 0.81724 | 0.00231 | 0.81956 |
| H | 55 | 0.18263 | 0 | 0.81523 | 0.00215 | 0.81737 |
| C | 56 | -0.19981 | 1.99922 | 4.18317 | 0.01742 | 6.19981 |
| C | 57 | -0.17838 | 1.99915 | 4.16293 | 0.0163 | 6.17838 |
| C | 58 | -0.18045 | 1.99916 | 4.16532 | 0.01596 | 6.18045 |
| C | 59 | -0.18135 | 1.99917 | 4.16622 | 0.01596 | 6.18135 |
| C | 60 | -0.17537 | 1.99917 | 4.16007 | 0.01614 | 6.17537 |
| C | 61 | -0.18169 | 1.99914 | 4.16628 | 0.01628 | 6.18169 |
| H | 62 | 0.17934 | 0 | 0.81835 | 0.0023 | 0.82066 |
| H | 63 | 0.1831 | 0 | 0.81468 | 0.00222 | 0.8169 |
| H | 64 | 0.182 | 0 | 0.81575 | 0.00224 | 0.818 |
| H | 65 | 0.18046 | 0 | 0.81723 | 0.00231 | 0.81954 |

| | | | | | | |
|---|---|---|---|---|---|---|
| H | 66 | 0.18262 | 0 | 0.81523 | 0.00215 | 0.81738 |
| C 6:5 junct. | 67 | 0.0019 | 1.99857 | 3.97641 | 0.02312 | 5.9981 |
| C | 68 | -0.01059 | 1.99859 | 3.98873 | 0.02328 | 6.01059 |
| C 6:5 junct. | 69 | 0.00839 | 1.99857 | 3.96869 | 0.02435 | 5.99161 |
| C adj. to 6:5 | 70 | 0.00674 | 1.9986 | 3.97142 | 0.02324 | 5.99326 |
| C | 71 | -0.00562 | 1.99859 | 3.98517 | 0.02185 | 6.00562 |
| C | 72 | -0.01102 | 1.99857 | 3.99137 | 0.02107 | 6.01102 |
| C | 73 | -0.03324 | 1.99867 | 4.00493 | 0.02964 | 6.03324 |
| C | 74 | 0.11304 | 1.9986 | 3.86035 | 0.02802 | 5.88696 |
| C | 75 | -0.08405 | 1.99875 | 4.04732 | 0.03798 | 6.08405 |
| C | 76 | -0.00436 | 1.99859 | 3.98388 | 0.0219 | 6.00436 |
| C | 77 | 0.00805 | 1.99857 | 3.96904 | 0.02435 | 5.99195 |
| C | 78 | -0.01078 | 1.99859 | 3.98893 | 0.02327 | 6.01078 |
| C | 79 | -0.01122 | 1.99857 | 3.99156 | 0.02108 | 6.01122 |
| C | 80 | -0.00651 | 1.99859 | 3.98607 | 0.02185 | 6.00651 |
| C | 81 | -0.00465 | 1.99859 | 3.98418 | 0.02189 | 6.00465 |
| C | 82 | -0.08264 | 1.99875 | 4.04592 | 0.03797 | 6.08264 |
| C | 83 | -0.08219 | 1.99875 | 4.04546 | 0.03798 | 6.08219 |
| C | 84 | 0.11401 | 1.9986 | 3.85935 | 0.02804 | 5.88599 |
| C | 85 | 0.11488 | 1.9986 | 3.85849 | 0.02802 | 5.88512 |
| C | 86 | -0.03415 | 1.99867 | 4.00584 | 0.02964 | 6.03415 |
| C | 87 | 0.00098 | 1.99857 | 3.97734 | 0.02312 | 5.99902 |
| C | 88 | 0.00678 | 1.9986 | 3.97138 | 0.02323 | 5.99322 |
| C | 89 | 0.00154 | 1.99857 | 3.97677 | 0.02312 | 5.99846 |
| C | 90 | -0.03326 | 1.99867 | 4.00493 | 0.02966 | 6.03326 |
| C | 91 | 0.00618 | 1.9986 | 3.97199 | 0.02323 | 5.99382 |
| C | 92 | -0.00571 | 1.99859 | 3.98527 | 0.02185 | 6.00571 |
| C | 93 | 0.11491 | 1.9986 | 3.85846 | 0.02802 | 5.88509 |
| C | 94 | 0.11397 | 1.9986 | 3.85939 | 0.02804 | 5.88603 |
| C | 95 | -0.08223 | 1.99875 | 4.0455 | 0.03798 | 6.08223 |
| C | 96 | -0.08263 | 1.99875 | 4.04591 | 0.03797 | 6.08263 |
| C | 97 | -0.01 | 1.99857 | 3.99033 | 0.02109 | 6.01 |
| C | 98 | -0.01033 | 1.99859 | 3.98848 | 0.02327 | 6.01033 |
| C | 99 | -0.00466 | 1.99859 | 3.98417 | 0.02189 | 6.00466 |
| C | 100 | 0.0085 | 1.99857 | 3.96858 | 0.02435 | 5.9915 |
| C | 101 | -0.00468 | 1.99859 | 3.98419 | 0.02189 | 6.00468 |
| C | 102 | -0.00996 | 1.99857 | 3.9903 | 0.02109 | 6.00996 |
| C | 103 | -0.03417 | 1.99867 | 4.00586 | 0.02964 | 6.03417 |
| C | 104 | -0.08407 | 1.99875 | 4.04734 | 0.03798 | 6.08407 |
| C | 105 | -0.03333 | 1.99867 | 4.005 | 0.02966 | 6.03333 |
| C | 106 | -0.00653 | 1.99859 | 3.98609 | 0.02185 | 6.00653 |
| C | 107 | 0.11297 | 1.9986 | 3.86042 | 0.02802 | 5.88703 |
| C | 108 | -0.00571 | 1.99859 | 3.98527 | 0.02185 | 6.00571 |
| C | 109 | 0.00842 | 1.99857 | 3.96866 | 0.02434 | 5.99158 |
| C | 110 | -0.01039 | 1.99859 | 3.98854 | 0.02327 | 6.01039 |
| C | 111 | 0.00685 | 1.9986 | 3.97132 | 0.02323 | 5.99315 |

| | | | | | | |
|---|---|---|---|---|---|---|
| C | 112 | 0.00101 | 1.99857 | 3.9773 | 0.02312 | 5.99899 |
| C | 113 | -0.00443 | 1.99859 | 3.98395 | 0.0219 | 6.00443 |
| C | 114 | -0.00563 | 1.99859 | 3.98519 | 0.02185 | 6.00563 |
| C | 115 | -0.01123 | 1.99857 | 3.99158 | 0.02108 | 6.01123 |
| C | 116 | -0.01102 | 1.99857 | 3.99138 | 0.02107 | 6.01102 |
| C | 117 | -0.03321 | 1.99867 | 4.0049 | 0.02964 | 6.03321 |
| C | 118 | 0.00618 | 1.9986 | 3.97198 | 0.02323 | 5.99382 |
| C | 119 | 0.00157 | 1.99857 | 3.97675 | 0.02311 | 5.99843 |
| C | 120 | 0.00809 | 1.99857 | 3.969 | 0.02434 | 5.99191 |
| C | 121 | -0.01056 | 1.99859 | 3.98869 | 0.02328 | 6.01056 |
| C | 122 | 0.0067 | 1.9986 | 3.97146 | 0.02323 | 5.9933 |
| C | 123 | 0.00175 | 1.99857 | 3.97656 | 0.02312 | 5.99825 |
| C | 124 | -0.00465 | 1.99859 | 3.98418 | 0.02189 | 6.00465 |
| C | 125 | -0.01086 | 1.99859 | 3.98901 | 0.02327 | 6.01086 |
| C | 126 | 0.00846 | 1.99857 | 3.96864 | 0.02434 | 5.99154 |
| H | 127 | 0.20794 | 0 | 0.79028 | 0.00178 | 0.79206 |
| H | 128 | 0.20793 | 0 | 0.79029 | 0.00178 | 0.79207 |
| H | 129 | 0.20793 | 0 | 0.79029 | 0.00178 | 0.79207 |
| H | 130 | 0.20793 | 0 | 0.79028 | 0.00178 | 0.79207 |
| H | 131 | 0.20793 | 0 | 0.79029 | 0.00178 | 0.79207 |
| H | 132 | 0.20794 | 0 | 0.79028 | 0.00178 | 0.79206 |
| * Total | | 0.00195 | 191.8866 | 417.9184 | 2.19301 | 611.9981 |

Determined by a PBEPBE/6-311+g(2df,2p) DFT calculations in Gaussian W03. The numbering scheme for the DFT calculation is shown below.



**Details of the Crystal Structure Refinement of 1**

There are four independent dimers (i.e. eight different monomeric dithiadiazolyl radicals comprise the asymmetric unit of this structure.) Of these, one dimer displays $CF_3$ group (rotational) disorder, specifically involving the C(36)F3 and C(46)F3 $CF_3$ groups, also identifiable as the two heterocycles containing S6 S8. This disorder could be described adequately a two-part disorder model but to get reasonable geometries (employing SADI restraints) it was found necessary to include the $CF_3$ carbon atoms in the model. EADP restraints were required to keep the displacement ellipsoids of these two C atoms well behaved, namely C36/C36A and C46/C46A. In addition, a strong tendency for the fluorine atoms to go oblate or NPD was solved by applying the newly developed RIGU restraints in ShelXL-2014, which have been applied globally to this structure. Refined occupancies for the C(36) set is 0.70 (major) and 0.30 (minor); for C(46) is 0.60 (major) and 0.40 (minor).

The SADI restraints on 1,2 C–F and 1,3 F–F distances permitted the two $CF_3$ groups to rotate apart without requiring the two rotamers to be staggered. The combination of this approach provided a successful disorder model which is depicted in Figure S9. In the final difference Fourier map, the highest peak of 0.47 $e/Å^3$ at (0.3534 0.4237 0.0106) bridges between S13 and S14, whilst the deepest hole of -0.46 $e/Å^3$ is at (0.5938 0.4677 0.0595) is about 0.85 Å from S10. Thus they are associated with the heaviest atoms in the unit cell and are *not* associated with the $CF_3$ groups. This is considered a positive indication of the adequacy of the disorder models applied to the trifluoromethyl groups.

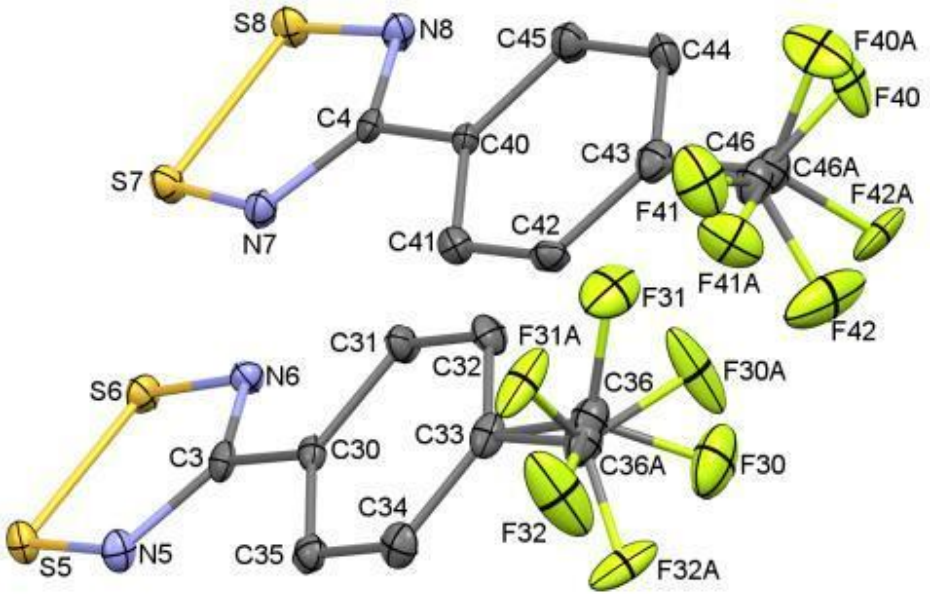



**Figure S9**. Depiction of the two-component disorder models developed for the $CF_3$ groups of the dimer involving S6 S8. The remaining three DTDA dimers exhibited ordered $CF_3$ groups in the structure at 173 K.

**Table S5**. Crystal structure report for **1**

**Table S5A.** Crystal data and structure refinement for **1**

| | | |
|---|---|---|
| Empirical formula | C8 H4 F3 N2 S2 | |
| Formula weight | 249.25 | |
| Temperature | 173(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Triclinic | |
| Space group | P-1 | |
| Unit cell dimensions | a = 9.4916(9) Å | α= 91.5790(10)°. |
| | b = 18.1887(17) Å | β= 97.3290(10)°. |
| | c = 22.275(2) Å | γ = 102.7550(10)°. |
| Volume | 3713.8(6) $Å^3$ | |
| Z | 16 | |
| Density (calculated) | 1.783 $Mg/m^3$ | |
| Absorption coefficient | 0.583 $mm^{-1}$ | |
| F(000) | 2000 | |
| Crystal size | 0.180 x 0.100 x 0.040 $mm^3$ | |
| Theta range for data collection | 1.847 to 26.220°. | |
| Index ranges | -11<=h<=11, -22<=k<=22, -27<=l<=27 | |
| Reflections collected | 39140 | |
| Independent reflections | 14828 [R(int) = 0.0812] | |
| Completeness to theta = 25.500° | 99.6 % | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 14828 / 1034 / 1137 | |
| Goodness-of-fit on $F^2$ | 0.973 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0540, wR2 = 0.0930 | |
| R indices (all data) | R1 = 0.1396, wR2 = 0.1185 | |
| Extinction coefficient | n/a | |
| Largest diff. peak and hole | 0.470 and -0.456 $e.Å^{-3}$ | |

**Table S5B** Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters (Å$^2$x $10^3$) for **1**. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 5498(1) | 2298(1) | 4562(1) | 18(1) |
| S(2) | 6117(1) | 1267(1) | 4535(1) | 15(1) |
| N(1) | 5510(4) | 2409(2) | 3842(2) | 17(1) |
| N(2) | 6146(4) | 1237(2) | 3805(2) | 14(1) |
| C(1) | 5847(4) | 1845(3) | 3532(2) | 14(1) |
| C(10) | 5825(4) | 1904(3) | 2873(2) | 12(1) |
| C(11) | 5856(5) | 2598(3) | 2625(2) | 16(1) |
| C(12) | 5761(4) | 2661(3) | 2005(2) | 16(1) |
| C(13) | 5646(5) | 2029(3) | 1628(2) | 14(1) |
| C(14) | 5654(5) | 1339(3) | 1870(2) | 16(1) |
| C(15) | 5749(5) | 1273(3) | 2490(2) | 15(1) |
| C(16) | 5469(5) | 2092(3) | 958(2) | 21(1) |
| F(11) | 4070(3) | 2010(2) | 719(1) | 37(1) |
| F(10) | 6143(3) | 2776(2) | 798(1) | 33(1) |
| F(12) | 5988(4) | 1593(2) | 666(1) | 49(1) |
| S(3) | 2314(1) | 1435(1) | 4395(1) | 19(1) |
| S(4) | 2958(1) | 412(1) | 4403(1) | 18(1) |
| N(3) | 2132(4) | 1460(2) | 3658(2) | 18(1) |
| N(4) | 2902(4) | 321(2) | 3668(2) | 16(1) |
| C(2) | 2450(5) | 872(3) | 3372(2) | 16(1) |
| C(20) | 2253(4) | 828(3) | 2703(2) | 13(1) |
| C(21) | 2040(5) | 1451(3) | 2389(2) | 14(1) |
| C(22) | 1748(5) | 1405(3) | 1760(2) | 14(1) |
| C(24) | 1934(5) | 105(3) | 1762(2) | 19(1) |
| C(23) | 1682(5) | 726(3) | 1450(2) | 18(1) |
| C(25) | 2235(5) | 162(3) | 2385(2) | 17(1) |
| C(26) | 1267(5) | 628(3) | 781(2) | 23(1) |
| F(20) | 2298(3) | 421(2) | 498(1) | 35(1) |
| F(21) | 1015(3) | 1249(2) | 524(1) | 34(1) |
| F(22) | 54(3) | 88(2) | 630(1) | 50(1) |
| S(5) | -1507(1) | 10264(1) | 4339(1) | 17(1) |
| S(6) | -1683(1) | 10295(1) | 3396(1) | 17(1) |

| | | | | |
|---|---|---|---|---|
| N(5) | -750(4) | 9538(2) | 4370(2) | 16(1) |
| N(6) | -861(4) | 9612(2) | 3318(2) | 15(1) |
| C(3) | -481(5) | 9297(3) | 3832(2) | 15(1) |
| C(30) | 266(5) | 8675(2) | 3803(2) | 14(1) |
| C(34) | 1108(5) | 7628(3) | 4239(2) | 20(1) |
| C(32) | 1475(5) | 7935(3) | 3222(2) | 20(1) |
| C(33) | 1634(5) | 7487(3) | 3709(2) | 17(1) |
| C(31) | 803(5) | 8518(3) | 3273(2) | 18(1) |
| C(35) | 437(5) | 8228(3) | 4292(2) | 17(1) |
| C(36) | 2331(8) | 6838(4) | 3611(3) | 24(1) |
| F(30) | 1398(5) | 6254(3) | 3298(3) | 45(2) |
| F(31) | 3436(5) | 6986(2) | 3283(2) | 39(2) |
| F(32) | 2835(7) | 6575(3) | 4120(2) | 59(2) |
| C(36A) | 2258(15) | 6802(8) | 3700(6) | 24(1) |
| F(30A) | 2155(15) | 6535(7) | 3130(5) | 41(4) |
| F(31A) | 3670(10) | 7012(5) | 3918(5) | 34(3) |
| F(32A) | 1675(10) | 6234(5) | 4012(5) | 28(3) |
| S(7) | 5251(1) | 9278(1) | 4169(1) | 17(1) |
| S(8) | 5280(1) | 9281(1) | 3237(1) | 17(1) |
| N(7) | 5918(4) | 8522(2) | 4256(2) | 17(1) |
| N(8) | 5933(4) | 8531(2) | 3202(2) | 16(1) |
| C(4) | 6183(5) | 8229(3) | 3735(2) | 14(1) |
| C(43) | 7730(5) | 6209(3) | 3697(2) | 20(1) |
| C(40) | 6747(5) | 7531(2) | 3740(2) | 13(1) |
| C(45) | 7169(5) | 7284(3) | 3210(2) | 20(1) |
| C(42) | 7347(5) | 6460(3) | 4231(2) | 17(1) |
| C(44) | 7649(5) | 6619(3) | 3187(2) | 22(1) |
| C(41) | 6859(5) | 7120(3) | 4255(2) | 18(1) |
| C(46) | 8221(10) | 5480(5) | 3708(4) | 24(1) |
| F(40) | 8545(11) | 5295(6) | 3158(4) | 32(2) |
| F(41) | 9412(10) | 5489(5) | 4104(3) | 35(2) |
| F(42) | 7212(8) | 4895(3) | 3839(6) | 50(3) |
| C(46A) | 8131(14) | 5464(7) | 3607(6) | 24(1) |
| F(40A) | 9090(15) | 5465(11) | 3217(6) | 34(3) |
| F(41A) | 8715(17) | 5259(8) | 4127(5) | 36(3) |
| F(42A) | 6987(9) | 4914(5) | 3396(8) | 38(3) |
| S(9) | 7104(1) | 4824(1) | 1760(1) | 18(1) |

| | | | | |
|---|---|---|---|---|
| S(10) | 6761(1) | 4735(1) | 814(1) | 18(1) |
| N(9) | 6330(4) | 5533(2) | 1806(2) | 18(1) |
| N(10) | 5972(4) | 5441(2) | 749(2) | 16(1) |
| C(5) | 5829(5) | 5759(3) | 1275(2) | 13(1) |
| C(51) | 4931(5) | 6735(3) | 1817(2) | 21(1) |
| C(52) | 4240(5) | 7333(3) | 1824(2) | 23(1) |
| C(50) | 5083(5) | 6397(3) | 1274(2) | 15(1) |
| C(53) | 3702(5) | 7587(3) | 1285(2) | 17(1) |
| C(54) | 3832(5) | 7241(3) | 735(2) | 18(1) |
| C(55) | 4525(5) | 6649(3) | 730(2) | 17(1) |
| C(56) | 2989(5) | 8235(3) | 1277(2) | 23(1) |
| F(51) | 3721(3) | 8829(2) | 1030(2) | 45(1) |
| F(50) | 2782(4) | 8461(2) | 1819(1) | 63(1) |
| F(52) | 1671(3) | 8078(2) | 940(2) | 44(1) |
| S(11) | 10195(1) | 5808(1) | 1777(1) | 18(1) |
| S(12) | 9879(1) | 5740(1) | 836(1) | 16(1) |
| N(11) | 9582(4) | 6572(2) | 1832(2) | 18(1) |
| N(12) | 9211(4) | 6494(2) | 770(2) | 15(1) |
| C(6) | 9147(5) | 6836(3) | 1302(2) | 14(1) |
| C(63) | 7705(5) | 8893(3) | 1409(2) | 15(1) |
| C(60) | 8625(4) | 7545(2) | 1326(2) | 12(1) |
| C(61) | 8695(5) | 7912(3) | 1887(2) | 23(1) |
| C(62) | 8241(5) | 8579(3) | 1928(2) | 22(1) |
| C(64) | 7662(5) | 8528(3) | 850(2) | 21(1) |
| C(66) | 7254(5) | 9617(3) | 1482(2) | 20(1) |
| C(65) | 8104(5) | 7857(3) | 806(2) | 17(1) |
| F(60) | 6275(3) | 9571(2) | 1868(1) | 30(1) |
| F(61) | 6665(3) | 9844(2) | 965(1) | 32(1) |
| F(62) | 8373(3) | 10178(2) | 1718(1) | 39(1) |
| S(13) | 7679(1) | 5352(1) | 9463(1) | 18(1) |
| S(14) | 7063(1) | 6380(1) | 9448(1) | 19(1) |
| N(13) | 7534(4) | 5226(2) | 8728(2) | 17(1) |
| N(14) | 6797(4) | 6382(2) | 8714(2) | 18(1) |
| C(7) | 7081(4) | 5783(3) | 8431(2) | 13(1) |
| C(70) | 6842(4) | 5715(3) | 7762(2) | 13(1) |
| C(73) | 6366(5) | 5592(3) | 6506(2) | 16(1) |
| C(71) | 6700(5) | 5019(3) | 7454(2) | 16(1) |

| | | | | |
|---|---|---|---|---|
| C(74) | 6520(5) | 6288(3) | 6808(2) | 17(1) |
| C(72) | 6449(5) | 4962(3) | 6832(2) | 17(1) |
| C(75) | 6749(4) | 6345(3) | 7427(2) | 16(1) |
| C(76) | 6128(5) | 5494(3) | 5827(2) | 24(1) |
| F(70) | 5719(3) | 6088(2) | 5569(1) | 35(1) |
| F(71) | 7341(3) | 5437(2) | 5603(1) | 32(1) |
| F(72) | 5139(3) | 4874(2) | 5629(1) | 48(1) |
| S(15) | 850(1) | 6191(1) | 9513(1) | 15(1) |
| S(16) | 240(1) | 7223(1) | 9546(1) | 18(1) |
| N(15) | 845(4) | 6155(2) | 8776(2) | 15(1) |
| N(16) | 212(4) | 7327(2) | 8825(2) | 16(1) |
| C(8) | 521(5) | 6759(3) | 8511(2) | 15(1) |
| C(80) | 489(4) | 6820(3) | 7847(2) | 13(1) |
| C(83) | 310(4) | 6951(3) | 6610(2) | 15(1) |
| C(81) | 342(4) | 6185(3) | 7462(2) | 14(1) |
| C(84) | 468(5) | 7583(3) | 6988(2) | 17(1) |
| C(85) | 569(5) | 7525(3) | 7605(2) | 14(1) |
| C(86) | 127(5) | 7027(3) | 5935(2) | 20(1) |
| C(82) | 254(4) | 6249(3) | 6844(2) | 15(1) |
| F(80) | -1256(3) | 7021(2) | 5708(1) | 27(1) |
| F(82) | 505(3) | 6485(2) | 5627(1) | 40(1) |
| F(81) | 900(3) | 7689(2) | 5778(1) | 32(1) |

**Table S5C.** Bond lengths [Å] and angles [°] for **1**

| | | | |
|---|---|---|---|
| S(1)-N(1) | 1.623(4) | C(23)-C(26) | 1.488(6) |
| S(1)-S(2) | 2.0880(17) | C(25)-H(25) | 0.9500 |
| S(2)-N(2) | 1.628(4) | C(26)-F(21) | 1.335(5) |
| N(1)-C(1) | 1.338(5) | C(26)-F(22) | 1.336(5) |
| N(2)-C(1) | 1.343(5) | C(26)-F(20) | 1.342(5) |
| C(1)-C(10) | 1.474(6) | S(5)-N(5) | 1.637(4) |
| C(10)-C(11) | 1.387(6) | S(5)-S(6) | 2.0881(17) |
| C(10)-C(15) | 1.396(6) | S(6)-N(6) | 1.623(4) |
| C(11)-C(12) | 1.384(6) | N(5)-C(3) | 1.337(5) |
| C(11)-H(11) | 0.9500 | N(6)-C(3) | 1.339(5) |
| C(12)-C(13) | 1.381(6) | C(3)-C(30) | 1.467(6) |
| C(12)-H(12) | 0.9500 | C(30)-C(35) | 1.390(6) |
| C(13)-C(14) | 1.380(6) | C(30)-C(31) | 1.391(6) |
| C(13)-C(16) | 1.491(6) | C(34)-C(33) | 1.377(6) |
| C(14)-C(15) | 1.382(6) | C(34)-C(35) | 1.390(6) |
| C(14)-H(14) | 0.9500 | C(34)-H(34) | 0.9500 |
| C(15)-H(15) | 0.9500 | C(32)-C(31) | 1.363(6) |
| C(16)-F(12) | 1.317(5) | C(32)-C(33) | 1.387(6) |
| C(16)-F(11) | 1.340(5) | C(32)-H(32) | 0.9500 |
| C(16)-F(10) | 1.348(5) | C(33)-C(36A) | 1.494(16) |
| S(3)-N(3) | 1.631(4) | C(33)-C(36) | 1.498(9) |
| S(3)-S(4) | 2.0841(18) | C(31)-H(31) | 0.9500 |
| S(4)-N(4) | 1.634(4) | C(35)-H(35) | 0.9500 |
| N(3)-C(2) | 1.339(6) | C(36)-F(32) | 1.318(8) |
| N(4)-C(2) | 1.332(5) | C(36)-F(30) | 1.337(8) |
| C(2)-C(20) | 1.477(6) | C(36)-F(31) | 1.337(8) |
| C(20)-C(25) | 1.382(6) | C(36A)-F(32A) | 1.319(13) |
| C(20)-C(21) | 1.387(6) | C(36A)-F(30A) | 1.331(14) |
| C(21)-C(22) | 1.390(6) | C(36A)-F(31A) | 1.332(14) |
| C(21)-H(21) | 0.9500 | S(7)-N(7) | 1.641(4) |
| C(22)-C(23) | 1.383(6) | S(7)-S(8) | 2.0803(17) |
| C(22)-H(22) | 0.9500 | S(8)-N(8) | 1.622(4) |
| C(24)-C(25) | 1.378(6) | N(7)-C(4) | 1.338(5) |
| C(24)-C(23) | 1.392(6) | N(8)-C(4) | 1.338(5) |
| C(24)-H(24) | 0.9500 | C(4)-C(40) | 1.484(6) |

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|---|---|---|---|
| C(43)-C(44) | 1.380(6) | C(56)-F(52) | 1.342(5) |
| C(43)-C(42) | 1.382(6) | S(11)-N(11) | 1.628(4) |
| C(43)-C(46) | 1.499(10) | S(11)-S(12) | 2.0758(17) |
| C(43)-C(46A) | 1.502(14) | S(12)-N(12) | 1.636(4) |
| C(40)-C(45) | 1.387(6) | N(11)-C(6) | 1.339(5) |
| C(40)-C(41) | 1.391(6) | N(12)-C(6) | 1.338(5) |
| C(45)-C(44) | 1.386(6) | C(6)-C(60) | 1.482(6) |
| C(45)-H(45) | 0.9500 | C(63)-C(64) | 1.386(6) |
| C(42)-C(41) | 1.382(6) | C(63)-C(62) | 1.389(6) |
| C(42)-H(42) | 0.9500 | C(63)-C(66) | 1.484(6) |
| C(44)-H(44) | 0.9500 | C(60)-C(65) | 1.387(6) |
| C(41)-H(41) | 0.9500 | C(60)-C(61) | 1.387(6) |
| C(46)-F(42) | 1.332(10) | C(61)-C(62) | 1.379(6) |
| C(46)-F(41) | 1.339(9) | C(61)-H(61) | 0.9500 |
| C(46)-F(40) | 1.351(10) | C(62)-H(62) | 0.9500 |
| C(46A)-F(41A) | 1.317(13) | C(64)-C(65) | 1.379(6) |
| C(46A)-F(42A) | 1.329(13) | C(64)-H(64) | 0.9500 |
| C(46A)-F(40A) | 1.336(13) | C(66)-F(61) | 1.331(5) |
| S(9)-N(9) | 1.626(4) | C(66)-F(60) | 1.336(5) |
| S(9)-S(10) | 2.0884(18) | C(66)-F(62) | 1.340(5) |
| S(10)-N(10) | 1.625(4) | C(65)-H(65) | 0.9500 |
| N(9)-C(5) | 1.329(5) | S(13)-N(13) | 1.629(4) |
| N(10)-C(5) | 1.328(5) | S(13)-S(14) | 2.0785(18) |
| C(5)-C(50) | 1.486(6) | S(14)-N(14) | 1.623(4) |
| C(51)-C(50) | 1.380(6) | N(13)-C(7) | 1.344(5) |
| C(51)-C(52) | 1.389(6) | N(14)-C(7) | 1.337(5) |
| C(51)-H(51) | 0.9500 | C(7)-C(70) | 1.477(6) |
| C(52)-C(53) | 1.377(6) | C(70)-C(71) | 1.394(6) |
| C(52)-H(52) | 0.9500 | C(70)-C(75) | 1.397(6) |
| C(50)-C(55) | 1.391(6) | C(73)-C(72) | 1.384(6) |
| C(53)-C(54) | 1.395(6) | C(73)-C(74) | 1.387(6) |
| C(53)-C(56) | 1.482(6) | C(73)-C(76) | 1.501(6) |
| C(54)-C(55) | 1.380(6) | C(71)-C(72) | 1.374(6) |
| C(54)-H(54) | 0.9500 | C(71)-H(71) | 0.9500 |
| C(55)-H(55) | 0.9500 | C(74)-C(75) | 1.366(6) |
| C(56)-F(50) | 1.318(5) | C(74)-H(74) | 0.9500 |
| C(56)-F(51) | 1.323(5) | C(72)-H(72) | 0.9500 |

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|---|---|---|---|
| C(75)-H(75) | 0.9500 | C(10)-C(11)-H(11) | 119.7 |
| C(76)-F(72) | 1.321(5) | C(13)-C(12)-C(11) | 119.7(4) |
| C(76)-F(71) | 1.334(5) | C(13)-C(12)-H(12) | 120.1 |
| C(76)-F(70) | 1.346(6) | C(11)-C(12)-H(12) | 120.1 |
| S(15)-N(15) | 1.641(4) | C(14)-C(13)-C(12) | 120.2(4) |
| S(15)-S(16) | 2.0846(17) | C(14)-C(13)-C(16) | 120.2(4) |
| S(16)-N(16) | 1.619(4) | C(12)-C(13)-C(16) | 119.5(4) |
| N(15)-C(8) | 1.337(6) | C(13)-C(14)-C(15) | 120.3(4) |
| N(16)-C(8) | 1.337(6) | C(13)-C(14)-H(14) | 119.8 |
| C(8)-C(80) | 1.484(6) | C(15)-C(14)-H(14) | 119.8 |
| C(80)-C(81) | 1.391(6) | C(14)-C(15)-C(10) | 119.9(4) |
| C(80)-C(85) | 1.395(6) | C(14)-C(15)-H(15) | 120.1 |
| C(83)-C(84) | 1.376(6) | C(10)-C(15)-H(15) | 120.1 |
| C(83)-C(82) | 1.385(6) | F(12)-C(16)-F(11) | 106.9(4) |
| C(83)-C(86) | 1.505(6) | F(12)-C(16)-F(10) | 106.3(4) |
| C(81)-C(82) | 1.377(6) | F(11)-C(16)-F(10) | 104.7(4) |
| C(81)-H(81) | 0.9500 | F(12)-C(16)-C(13) | 113.5(4) |
| C(84)-C(85) | 1.376(6) | F(11)-C(16)-C(13) | 112.4(4) |
| C(84)-H(84) | 0.9500 | F(10)-C(16)-C(13) | 112.4(4) |
| C(85)-H(85) | 0.9500 | N(3)-S(3)-S(4) | 94.18(15) |
| C(86)-F(82) | 1.323(5) | N(4)-S(4)-S(3) | 94.89(15) |
| C(86)-F(80) | 1.342(5) | C(2)-N(3)-S(3) | 114.5(3) |
| C(86)-F(81) | 1.345(5) | C(2)-N(4)-S(4) | 113.8(3) |
| C(82)-H(82) | 0.9500 | N(4)-C(2)-N(3) | 122.6(4) |
| | | N(4)-C(2)-C(20) | 119.1(4) |
| N(1)-S(1)-S(2) | 94.35(15) | N(3)-C(2)-C(20) | 118.3(4) |
| N(2)-S(2)-S(1) | 94.57(14) | C(25)-C(20)-C(21) | 119.6(4) |
| C(1)-N(1)-S(1) | 114.9(3) | C(25)-C(20)-C(2) | 120.4(4) |
| C(1)-N(2)-S(2) | 114.4(3) | C(21)-C(20)-C(2) | 120.0(4) |
| N(1)-C(1)-N(2) | 121.7(4) | C(20)-C(21)-C(22) | 120.9(4) |
| N(1)-C(1)-C(10) | 117.2(4) | C(20)-C(21)-H(21) | 119.6 |
| N(2)-C(1)-C(10) | 121.1(4) | C(22)-C(21)-H(21) | 119.6 |
| C(11)-C(10)-C(15) | 119.2(4) | C(23)-C(22)-C(21) | 118.8(4) |
| C(11)-C(10)-C(1) | 119.5(4) | C(23)-C(22)-H(22) | 120.6 |
| C(15)-C(10)-C(1) | 121.3(4) | C(21)-C(22)-H(22) | 120.6 |
| C(12)-C(11)-C(10) | 120.6(4) | C(25)-C(24)-C(23) | 119.9(4) |
| C(12)-C(11)-H(11) | 119.7 | C(25)-C(24)-H(24) | 120.1 |

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|---|---|---|---|
| C(23)-C(24)-H(24) | 120.1 | C(30)-C(35)-C(34) | 119.8(5) |
| C(22)-C(23)-C(24) | 120.6(4) | C(30)-C(35)-H(35) | 120.1 |
| C(22)-C(23)-C(26) | 121.1(4) | C(34)-C(35)-H(35) | 120.1 |
| C(24)-C(23)-C(26) | 118.2(4) | F(32)-C(36)-F(30) | 106.3(6) |
| C(24)-C(25)-C(20) | 120.3(4) | F(32)-C(36)-F(31) | 106.5(6) |
| C(24)-C(25)-H(25) | 119.9 | F(30)-C(36)-F(31) | 103.3(6) |
| C(20)-C(25)-H(25) | 119.9 | F(32)-C(36)-C(33) | 113.2(6) |
| F(21)-C(26)-F(22) | 106.5(4) | F(30)-C(36)-C(33) | 112.0(6) |
| F(21)-C(26)-F(20) | 106.1(4) | F(31)-C(36)-C(33) | 114.8(6) |
| F(22)-C(26)-F(20) | 106.2(4) | F(32A)-C(36A)-F(30A) | 107.1(11) |
| F(21)-C(26)-C(23) | 113.8(4) | F(32A)-C(36A)-F(31A) | 107.4(11) |
| F(22)-C(26)-C(23) | 111.3(4) | F(30A)-C(36A)-F(31A) | 107.7(11) |
| F(20)-C(26)-C(23) | 112.4(4) | F(32A)-C(36A)-C(33) | 117.0(11) |
| N(5)-S(5)-S(6) | 94.68(15) | F(30A)-C(36A)-C(33) | 109.4(11) |
| N(6)-S(6)-S(5) | 94.13(14) | F(31A)-C(36A)-C(33) | 107.8(10) |
| C(3)-N(5)-S(5) | 113.9(3) | N(7)-S(7)-S(8) | 94.95(15) |
| C(3)-N(6)-S(6) | 115.1(3) | N(8)-S(8)-S(7) | 94.51(14) |
| N(5)-C(3)-N(6) | 122.0(4) | C(4)-N(7)-S(7) | 113.2(3) |
| N(5)-C(3)-C(30) | 119.3(4) | C(4)-N(8)-S(8) | 114.5(3) |
| N(6)-C(3)-C(30) | 118.7(4) | N(7)-C(4)-N(8) | 122.8(4) |
| C(35)-C(30)-C(31) | 118.9(4) | N(7)-C(4)-C(40) | 119.3(4) |
| C(35)-C(30)-C(3) | 121.5(4) | N(8)-C(4)-C(40) | 117.9(4) |
| C(31)-C(30)-C(3) | 119.6(4) | C(44)-C(43)-C(42) | 120.5(4) |
| C(33)-C(34)-C(35) | 120.1(5) | C(44)-C(43)-C(46) | 122.9(6) |
| C(33)-C(34)-H(34) | 120.0 | C(42)-C(43)-C(46) | 116.5(5) |
| C(35)-C(34)-H(34) | 120.0 | C(44)-C(43)-C(46A) | 115.2(6) |
| C(31)-C(32)-C(33) | 119.5(5) | C(42)-C(43)-C(46A) | 124.0(7) |
| C(31)-C(32)-H(32) | 120.2 | C(45)-C(40)-C(41) | 119.6(4) |
| C(33)-C(32)-H(32) | 120.2 | C(45)-C(40)-C(4) | 118.3(4) |
| C(34)-C(33)-C(32) | 120.3(4) | C(41)-C(40)-C(4) | 122.1(4) |
| C(34)-C(33)-C(36A) | 114.7(7) | C(44)-C(45)-C(40) | 120.3(4) |
| C(32)-C(33)-C(36A) | 124.9(7) | C(44)-C(45)-H(45) | 119.9 |
| C(34)-C(33)-C(36) | 123.2(5) | C(40)-C(45)-H(45) | 119.9 |
| C(32)-C(33)-C(36) | 116.4(5) | C(41)-C(42)-C(43) | 120.0(4) |
| C(32)-C(31)-C(30) | 121.4(4) | C(41)-C(42)-H(42) | 120.0 |
| C(32)-C(31)-H(31) | 119.3 | C(43)-C(42)-H(42) | 120.0 |
| C(30)-C(31)-H(31) | 119.3 | C(43)-C(44)-C(45) | 119.6(5) |

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| C(43)-C(44)-H(44) | 120.2 | C(55)-C(54)-H(54) | 120.1 |
| C(45)-C(44)-H(44) | 120.2 | C(53)-C(54)-H(54) | 120.1 |
| C(42)-C(41)-C(40) | 119.9(4) | C(54)-C(55)-C(50) | 119.9(5) |
| C(42)-C(41)-H(41) | 120.0 | C(54)-C(55)-H(55) | 120.0 |
| C(40)-C(41)-H(41) | 120.0 | C(50)-C(55)-H(55) | 120.0 |
| F(42)-C(46)-F(41) | 105.7(7) | F(50)-C(56)-F(51) | 107.5(4) |
| F(42)-C(46)-F(40) | 105.7(8) | F(50)-C(56)-F(52) | 105.9(4) |
| F(41)-C(46)-F(40) | 106.1(8) | F(51)-C(56)-F(52) | 103.9(4) |
| F(42)-C(46)-C(43) | 113.6(7) | F(50)-C(56)-C(53) | 113.4(4) |
| F(41)-C(46)-C(43) | 114.5(7) | F(51)-C(56)-C(53) | 113.0(4) |
| F(40)-C(46)-C(43) | 110.7(8) | F(52)-C(56)-C(53) | 112.5(4) |
| F(41A)-C(46A)-F(42A) | 107.3(10) | N(11)-S(11)-S(12) | 94.27(15) |
| F(41A)-C(46A)-F(40A) | 106.8(11) | N(12)-S(12)-S(11) | 95.15(14) |
| F(42A)-C(46A)-F(40A) | 105.3(10) | C(6)-N(11)-S(11) | 114.7(3) |
| F(41A)-C(46A)-C(43) | 110.2(10) | C(6)-N(12)-S(12) | 113.6(3) |
| F(42A)-C(46A)-C(43) | 112.6(10) | N(12)-C(6)-N(11) | 122.2(4) |
| F(40A)-C(46A)-C(43) | 114.2(13) | N(12)-C(6)-C(60) | 120.8(4) |
| N(9)-S(9)-S(10) | 94.02(15) | N(11)-C(6)-C(60) | 116.9(4) |
| N(10)-S(10)-S(9) | 94.60(15) | C(64)-C(63)-C(62) | 118.6(4) |
| C(5)-N(9)-S(9) | 114.5(3) | C(64)-C(63)-C(66) | 123.2(4) |
| C(5)-N(10)-S(10) | 114.0(3) | C(62)-C(63)-C(66) | 118.1(4) |
| N(10)-C(5)-N(9) | 122.8(4) | C(65)-C(60)-C(61) | 119.5(4) |
| N(10)-C(5)-C(50) | 119.0(4) | C(65)-C(60)-C(6) | 122.0(4) |
| N(9)-C(5)-C(50) | 118.2(4) | C(61)-C(60)-C(6) | 118.6(4) |
| C(50)-C(51)-C(52) | 120.5(5) | C(62)-C(61)-C(60) | 120.3(4) |
| C(50)-C(51)-H(51) | 119.8 | C(62)-C(61)-H(61) | 119.9 |
| C(52)-C(51)-H(51) | 119.8 | C(60)-C(61)-H(61) | 119.9 |
| C(53)-C(52)-C(51) | 119.6(5) | C(61)-C(62)-C(63) | 120.7(5) |
| C(53)-C(52)-H(52) | 120.2 | C(61)-C(62)-H(62) | 119.7 |
| C(51)-C(52)-H(52) | 120.2 | C(63)-C(62)-H(62) | 119.7 |
| C(51)-C(50)-C(55) | 119.9(4) | C(65)-C(64)-C(63) | 121.1(5) |
| C(51)-C(50)-C(5) | 119.7(4) | C(65)-C(64)-H(64) | 119.5 |
| C(55)-C(50)-C(5) | 120.4(4) | C(63)-C(64)-H(64) | 119.5 |
| C(52)-C(53)-C(54) | 120.2(4) | F(61)-C(66)-F(60) | 107.0(4) |
| C(52)-C(53)-C(56) | 120.9(5) | F(61)-C(66)-F(62) | 107.3(4) |
| C(54)-C(53)-C(56) | 118.8(4) | F(60)-C(66)-F(62) | 105.1(4) |
| C(55)-C(54)-C(53) | 119.9(4) | F(61)-C(66)-C(63) | 113.5(4) |

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| F(60)-C(66)-C(63) | 111.4(4) | F(71)-C(76)-C(73) | 112.1(4) |
| F(62)-C(66)-C(63) | 112.0(4) | F(70)-C(76)-C(73) | 111.9(4) |
| C(64)-C(65)-C(60) | 119.9(4) | N(15)-S(15)-S(16) | 94.81(14) |
| C(64)-C(65)-H(65) | 120.0 | N(16)-S(16)-S(15) | 94.24(15) |
| C(60)-C(65)-H(65) | 120.0 | C(8)-N(15)-S(15) | 113.5(3) |
| N(13)-S(13)-S(14) | 95.20(15) | C(8)-N(16)-S(16) | 115.1(3) |
| N(14)-S(14)-S(13) | 94.61(15) | N(15)-C(8)-N(16) | 122.3(4) |
| C(7)-N(13)-S(13) | 113.1(3) | N(15)-C(8)-C(80) | 120.4(4) |
| C(7)-N(14)-S(14) | 114.0(3) | N(16)-C(8)-C(80) | 117.3(4) |
| N(14)-C(7)-N(13) | 123.0(4) | C(81)-C(80)-C(85) | 119.6(4) |
| N(14)-C(7)-C(70) | 118.8(4) | C(81)-C(80)-C(8) | 121.1(4) |
| N(13)-C(7)-C(70) | 118.2(4) | C(85)-C(80)-C(8) | 119.2(4) |
| C(71)-C(70)-C(75) | 119.0(4) | C(84)-C(83)-C(82) | 120.7(4) |
| C(71)-C(70)-C(7) | 120.4(4) | C(84)-C(83)-C(86) | 118.9(4) |
| C(75)-C(70)-C(7) | 120.7(4) | C(82)-C(83)-C(86) | 120.4(4) |
| C(72)-C(73)-C(74) | 120.1(4) | C(82)-C(81)-C(80) | 120.3(4) |
| C(72)-C(73)-C(76) | 117.9(4) | C(82)-C(81)-H(81) | 119.9 |
| C(74)-C(73)-C(76) | 122.0(4) | C(80)-C(81)-H(81) | 119.9 |
| C(72)-C(71)-C(70) | 119.7(4) | C(83)-C(84)-C(85) | 120.2(4) |
| C(72)-C(71)-H(71) | 120.1 | C(83)-C(84)-H(84) | 119.9 |
| C(70)-C(71)-H(71) | 120.1 | C(85)-C(84)-H(84) | 119.9 |
| C(75)-C(74)-C(73) | 119.4(4) | C(84)-C(85)-C(80) | 119.7(4) |
| C(75)-C(74)-H(74) | 120.3 | C(84)-C(85)-H(85) | 120.2 |
| C(73)-C(74)-H(74) | 120.3 | C(80)-C(85)-H(85) | 120.2 |
| C(71)-C(72)-C(73) | 120.6(4) | F(82)-C(86)-F(80) | 106.5(4) |
| C(71)-C(72)-H(72) | 119.7 | F(82)-C(86)-F(81) | 107.2(4) |
| C(73)-C(72)-H(72) | 119.7 | F(80)-C(86)-F(81) | 104.3(4) |
| C(74)-C(75)-C(70) | 121.2(4) | F(82)-C(86)-C(83) | 113.6(4) |
| C(74)-C(75)-H(75) | 119.4 | F(80)-C(86)-C(83) | 112.2(4) |
| C(70)-C(75)-H(75) | 119.4 | F(81)-C(86)-C(83) | 112.4(4) |
| F(72)-C(76)-F(71) | 106.2(4) | C(81)-C(82)-C(83) | 119.5(4) |
| F(72)-C(76)-F(70) | 108.6(4) | C(81)-C(82)-H(82) | 120.3 |
| F(71)-C(76)-F(70) | 105.6(4) | C(83)-C(82)-H(82) | 120.3 |
| F(72)-C(76)-C(73) | 112.1(4) | | |

**Table S5D.** Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **1**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| S(1) | 24(1) | 18(1) | 13(1) | -1(1) | 2(1) | 10(1) |
| S(2) | 17(1) | 15(1) | 15(1) | 1(1) | 2(1) | 6(1) |
| N(1) | 22(2) | 14(2) | 16(2) | 0(2) | 4(2) | 5(2) |
| N(2) | 17(2) | 14(2) | 13(2) | 2(2) | 4(2) | 5(2) |
| C(1) | 8(2) | 14(2) | 18(2) | 2(2) | 1(2) | 1(2) |
| C(10) | 12(2) | 14(2) | 12(2) | 0(2) | 3(2) | 4(2) |
| C(11) | 16(3) | 16(2) | 18(2) | 1(2) | 2(2) | 4(2) |
| C(12) | 13(2) | 15(2) | 20(2) | 4(2) | 5(2) | 4(2) |
| C(13) | 13(2) | 17(2) | 15(2) | 4(2) | 3(2) | 5(2) |
| C(14) | 18(3) | 14(2) | 17(2) | 0(2) | 4(2) | 5(2) |
| C(15) | 17(3) | 12(2) | 17(2) | 3(2) | 5(2) | 3(2) |
| C(16) | 24(3) | 19(3) | 20(2) | 3(2) | 3(2) | 5(2) |
| F(11) | 24(2) | 57(2) | 22(2) | 9(2) | -6(1) | -3(2) |
| F(10) | 39(2) | 33(2) | 22(2) | 10(1) | 3(1) | -6(2) |
| F(12) | 99(3) | 51(2) | 16(2) | 8(2) | 19(2) | 48(2) |
| S(3) | 21(1) | 27(1) | 14(1) | 0(1) | 3(1) | 12(1) |
| S(4) | 20(1) | 20(1) | 13(1) | 4(1) | 2(1) | 6(1) |
| N(3) | 18(2) | 24(2) | 16(2) | 4(2) | 4(2) | 10(2) |
| N(4) | 19(2) | 13(2) | 13(2) | 2(2) | -1(2) | 0(2) |
| C(2) | 14(2) | 19(2) | 16(2) | 3(2) | 0(2) | 5(2) |
| C(20) | 10(2) | 16(2) | 14(2) | -1(2) | 0(2) | 4(2) |
| C(21) | 15(2) | 11(2) | 17(2) | -2(2) | 0(2) | 5(2) |
| C(22) | 16(2) | 14(2) | 13(2) | 4(2) | 1(2) | 4(2) |
| C(24) | 27(3) | 13(2) | 17(2) | -2(2) | 4(2) | 4(2) |
| C(23) | 15(2) | 20(2) | 17(2) | 1(2) | 2(2) | 2(2) |
| C(25) | 15(3) | 14(2) | 24(2) | 1(2) | 1(2) | 5(2) |
| C(26) | 28(3) | 25(3) | 14(2) | -1(2) | -1(2) | 5(2) |
| F(20) | 56(2) | 42(2) | 15(2) | 2(1) | 11(1) | 26(2) |
| F(21) | 55(2) | 38(2) | 16(2) | 5(1) | -1(1) | 27(2) |
| F(22) | 51(2) | 63(2) | 17(2) | -3(2) | -5(2) | -26(2) |
| S(5) | 21(1) | 16(1) | 14(1) | -3(1) | -1(1) | 7(1) |
| S(6) | 22(1) | 12(1) | 17(1) | 2(1) | 3(1) | 7(1) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N(5) | 19(2) | 14(2) | 17(2) | 0(2) | 0(2) | 8(2) |
| N(6) | 19(2) | 13(2) | 14(2) | 2(2) | 4(2) | 5(2) |
| C(3) | 14(2) | 12(2) | 16(2) | -3(2) | -2(2) | 2(2) |
| C(30) | 11(2) | 12(2) | 16(2) | -4(2) | 0(2) | 2(2) |
| C(34) | 19(3) | 19(3) | 18(3) | 0(2) | -4(2) | 4(2) |
| C(32) | 23(3) | 19(3) | 22(3) | -2(2) | 8(2) | 9(2) |
| C(33) | 13(2) | 14(2) | 25(3) | -2(2) | -1(2) | 4(2) |
| C(31) | 24(3) | 13(2) | 18(2) | 1(2) | 7(2) | 8(2) |
| C(35) | 17(3) | 16(2) | 17(2) | -1(2) | -1(2) | 1(2) |
| C(36) | 24(2) | 18(2) | 31(2) | 0(2) | 3(2) | 8(1) |
| F(30) | 34(3) | 23(3) | 76(4) | -12(3) | 3(3) | 10(2) |
| F(31) | 28(3) | 30(3) | 67(4) | 4(2) | 21(2) | 16(2) |
| F(32) | 104(6) | 64(4) | 32(3) | 9(3) | 4(3) | 72(4) |
| C(36A) | 24(2) | 18(2) | 31(2) | 0(2) | 3(2) | 8(1) |
| F(30A) | 70(10) | 32(7) | 34(5) | 2(4) | 2(4) | 39(6) |
| F(31A) | 16(4) | 15(5) | 72(8) | -5(5) | 4(4) | 9(3) |
| F(32A) | 12(5) | 18(5) | 59(6) | 13(4) | 9(5) | 11(4) |
| S(7) | 24(1) | 16(1) | 15(1) | 2(1) | 5(1) | 9(1) |
| S(8) | 25(1) | 16(1) | 12(1) | 2(1) | 0(1) | 8(1) |
| N(7) | 24(2) | 12(2) | 15(2) | 0(2) | 2(2) | 4(2) |
| N(8) | 25(2) | 11(2) | 13(2) | 2(2) | 2(2) | 5(2) |
| C(4) | 15(2) | 11(2) | 14(2) | -2(2) | 1(2) | -3(2) |
| C(43) | 18(3) | 17(2) | 24(2) | 0(2) | 3(2) | 5(2) |
| C(40) | 16(2) | 9(2) | 13(2) | -2(2) | 2(2) | 0(2) |
| C(45) | 27(3) | 17(2) | 16(2) | 0(2) | 4(2) | 6(2) |
| C(42) | 19(3) | 18(2) | 14(2) | 4(2) | 2(2) | 3(2) |
| C(44) | 31(3) | 18(2) | 16(2) | -4(2) | 4(2) | 5(2) |
| C(41) | 20(3) | 17(2) | 15(2) | -1(2) | 4(2) | 3(2) |
| C(46) | 24(2) | 18(2) | 31(2) | 0(2) | 3(2) | 8(1) |
| F(40) | 46(6) | 29(5) | 30(3) | -6(3) | 0(3) | 29(4) |
| F(41) | 39(4) | 34(4) | 37(3) | 2(3) | 1(3) | 25(3) |
| F(42) | 44(4) | 19(3) | 100(8) | 14(3) | 38(4) | 16(3) |
| C(46A) | 24(2) | 18(2) | 31(2) | 0(2) | 3(2) | 8(1) |
| F(40A) | 25(6) | 43(8) | 40(5) | 2(5) | 8(5) | 17(5) |
| F(41A) | 45(7) | 39(7) | 32(4) | 9(4) | 5(4) | 26(6) |
| F(42A) | 26(4) | 18(4) | 67(8) | -3(4) | -1(4) | 3(3) |
| S(9) | 23(1) | 16(1) | 19(1) | 4(1) | 7(1) | 9(1) |

| | | | | | | |
|---|---|---|---|---|---|---|
| S(10) | 22(1) | 14(1) | 19(1) | -2(1) | 3(1) | 7(1) |
| N(9) | 19(2) | 19(2) | 19(2) | 5(2) | 5(2) | 5(2) |
| N(10) | 17(2) | 14(2) | 17(2) | -2(2) | 0(2) | 3(2) |
| C(5) | 12(2) | 12(2) | 14(2) | -4(2) | 3(2) | -1(2) |
| C(51) | 26(3) | 19(3) | 17(2) | 0(2) | -2(2) | 9(2) |
| C(52) | 28(3) | 21(3) | 19(3) | -6(2) | 0(2) | 8(2) |
| C(50) | 14(2) | 12(2) | 18(2) | -1(2) | 3(2) | -1(2) |
| C(53) | 16(2) | 11(2) | 25(3) | 0(2) | 7(2) | 1(2) |
| C(54) | 17(3) | 18(3) | 21(3) | 3(2) | 0(2) | 5(2) |
| C(55) | 18(3) | 18(3) | 15(2) | -1(2) | 5(2) | 5(2) |
| C(56) | 26(3) | 21(3) | 27(3) | 2(2) | 4(2) | 12(2) |
| F(51) | 33(2) | 18(2) | 88(3) | 14(2) | 15(2) | 10(1) |
| F(50) | 123(3) | 62(3) | 31(2) | 6(2) | 17(2) | 75(2) |
| F(52) | 25(2) | 35(2) | 71(2) | -7(2) | -6(2) | 15(2) |
| S(11) | 26(1) | 17(1) | 13(1) | 1(1) | -1(1) | 9(1) |
| S(12) | 24(1) | 14(1) | 14(1) | 1(1) | 5(1) | 8(1) |
| N(11) | 24(2) | 18(2) | 14(2) | 1(2) | 4(2) | 8(2) |
| N(12) | 18(2) | 17(2) | 13(2) | 3(2) | 3(2) | 9(2) |
| C(6) | 11(2) | 17(2) | 15(2) | -1(2) | 5(2) | 2(2) |
| C(63) | 12(2) | 12(2) | 22(2) | 1(2) | 4(2) | 1(2) |
| C(60) | 10(2) | 12(2) | 14(2) | 1(2) | 6(2) | 1(2) |
| C(61) | 39(3) | 21(3) | 14(2) | 4(2) | 3(2) | 14(2) |
| C(62) | 34(3) | 18(3) | 17(3) | -2(2) | 4(2) | 10(2) |
| C(64) | 27(3) | 20(3) | 17(3) | 4(2) | 0(2) | 8(2) |
| C(66) | 23(3) | 16(2) | 21(3) | 1(2) | 4(2) | 7(2) |
| C(65) | 22(3) | 15(2) | 12(2) | -2(2) | 0(2) | 4(2) |
| F(60) | 38(2) | 30(2) | 34(2) | 4(1) | 16(1) | 21(2) |
| F(61) | 49(2) | 28(2) | 26(2) | 6(1) | 1(1) | 23(2) |
| F(62) | 32(2) | 14(2) | 68(2) | -11(2) | 1(2) | 5(1) |
| S(13) | 21(1) | 22(1) | 12(1) | 3(1) | 1(1) | 6(1) |
| S(14) | 21(1) | 28(1) | 11(1) | -2(1) | 1(1) | 12(1) |
| N(13) | 21(2) | 19(2) | 11(2) | 0(2) | 1(2) | 8(2) |
| N(14) | 23(2) | 21(2) | 11(2) | 1(2) | 1(2) | 10(2) |
| C(7) | 8(2) | 17(2) | 13(2) | -3(2) | -1(2) | 2(2) |
| C(70) | 8(2) | 17(2) | 14(2) | 2(2) | 1(2) | 2(2) |
| C(73) | 9(2) | 25(3) | 13(2) | 2(2) | 0(2) | 1(2) |
| C(71) | 21(3) | 14(2) | 13(2) | 0(2) | -1(2) | 6(2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(74) | 13(2) | 22(3) | 16(2) | 7(2) | 2(2) | 6(2) |
| C(72) | 21(3) | 15(2) | 15(2) | 0(2) | 2(2) | 6(2) |
| C(75) | 12(2) | 17(2) | 18(2) | -2(2) | 1(2) | 4(2) |
| C(76) | 25(3) | 28(3) | 17(2) | 5(2) | 3(2) | -2(2) |
| F(70) | 49(2) | 44(2) | 16(2) | 4(1) | 0(1) | 22(2) |
| F(71) | 45(2) | 33(2) | 23(2) | 3(1) | 18(1) | 14(2) |
| F(72) | 60(2) | 47(2) | 14(2) | 0(1) | -6(2) | -28(2) |
| S(15) | 20(1) | 16(1) | 12(1) | 2(1) | 2(1) | 9(1) |
| S(16) | 26(1) | 15(1) | 14(1) | 0(1) | 2(1) | 8(1) |
| N(15) | 14(2) | 18(2) | 15(2) | 3(2) | 0(2) | 6(2) |
| N(16) | 22(2) | 15(2) | 13(2) | 2(2) | 0(2) | 5(2) |
| C(8) | 11(2) | 16(2) | 19(2) | 1(2) | 4(2) | 3(2) |
| C(80) | 9(2) | 17(2) | 15(2) | 2(2) | 1(2) | 6(2) |
| C(83) | 8(2) | 21(2) | 16(2) | 4(2) | 2(2) | 4(2) |
| C(81) | 13(2) | 17(2) | 14(2) | 4(2) | 2(2) | 4(2) |
| C(84) | 14(2) | 15(2) | 23(2) | 7(2) | 6(2) | 1(2) |
| C(85) | 16(2) | 13(2) | 15(2) | -1(2) | 3(2) | 5(2) |
| C(86) | 19(2) | 21(3) | 22(3) | 5(2) | 4(2) | 6(2) |
| C(82) | 12(2) | 17(2) | 16(2) | -2(2) | 0(2) | 5(2) |
| F(80) | 26(2) | 32(2) | 20(2) | 7(1) | -4(1) | 3(1) |
| F(82) | 68(2) | 45(2) | 20(2) | 7(2) | 16(2) | 36(2) |
| F(81) | 35(2) | 35(2) | 19(2) | 12(1) | 5(1) | -8(1) |

**Table S5E**. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for **1**.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(11) | 5943 | 3034 | 2884 | 20 |
| H(12) | 5775 | 3138 | 1838 | 19 |
| H(14) | 5595 | 908 | 1609 | 19 |
| H(15) | 5763 | 798 | 2654 | 18 |
| H(21) | 2095 | 1915 | 2606 | 17 |
| H(22) | 1597 | 1832 | 1546 | 17 |
| H(24) | 1899 | -358 | 1545 | 23 |
| H(25) | 2430 | -258 | 2598 | 21 |
| H(34) | 1205 | 7314 | 4569 | 23 |
| H(32) | 1831 | 7835 | 2856 | 24 |
| H(31) | 700 | 8825 | 2939 | 21 |
| H(35) | 96 | 8333 | 4660 | 21 |
| H(45) | 7129 | 7573 | 2862 | 24 |
| H(42) | 7420 | 6178 | 4582 | 21 |
| H(44) | 7921 | 6446 | 2821 | 26 |
| H(41) | 6599 | 7294 | 4623 | 21 |
| H(51) | 5302 | 6558 | 2188 | 25 |
| H(52) | 4139 | 7564 | 2199 | 27 |
| H(54) | 3445 | 7412 | 363 | 22 |
| H(55) | 4620 | 6414 | 356 | 20 |
| H(61) | 9058 | 7703 | 2245 | 28 |
| H(62) | 8294 | 8827 | 2314 | 27 |
| H(64) | 7324 | 8744 | 492 | 25 |
| H(65) | 8051 | 7610 | 420 | 20 |
| H(71) | 6775 | 4587 | 7674 | 19 |
| H(74) | 6466 | 6722 | 6585 | 20 |
| H(72) | 6332 | 4486 | 6622 | 20 |
| H(75) | 6847 | 6821 | 7633 | 19 |
| H(81) | 301 | 5705 | 7625 | 17 |
| H(84) | 507 | 8062 | 6821 | 21 |
| H(85) | 693 | 7964 | 7866 | 17 |
| H(82) | 157 | 5815 | 6581 | 18 |

**Table S5F.** Torsion angles [°] for **1**.

| | | | |
|---|---|---|---|
| S(2)-S(1)-N(1)-C(1) | 1.3(3) | S(1)-S(2)-N(2)-C(1) | 2.3(3) |
| S(1)-N(1)-C(1)-N(2) | 0.1(5) | | |
| S(1)-N(1)-C(1)-C(10) | 178.0(3) | | |
| S(2)-N(2)-C(1)-N(1) | -2.0(5) | | |
| S(2)-N(2)-C(1)-C(10) | -179.8(3) | | |
| N(1)-C(1)-C(10)-C(11) | 17.2(6) | | |
| N(2)-C(1)-C(10)-C(11) | -164.9(4) | | |
| N(1)-C(1)-C(10)-C(15) | -161.7(4) | | |
| N(2)-C(1)-C(10)-C(15) | 16.2(6) | | |
| C(15)-C(10)-C(11)-C(12) | 2.3(6) | | |
| C(1)-C(10)-C(11)-C(12) | -176.7(4) | | |
| C(10)-C(11)-C(12)-C(13) | -0.5(7) | | |
| C(11)-C(12)-C(13)-C(14) | -1.3(7) | | |
| C(11)-C(12)-C(13)-C(16) | 176.9(4) | | |
| C(12)-C(13)-C(14)-C(15) | 1.3(7) | | |
| C(16)-C(13)-C(14)-C(15) | -176.9(4) | | |
| C(13)-C(14)-C(15)-C(10) | 0.5(7) | | |
| C(11)-C(10)-C(15)-C(14) | -2.3(6) | | |
| C(1)-C(10)-C(15)-C(14) | 176.7(4) | | |
| C(14)-C(13)-C(16)-F(12) | -29.7(6) | | |
| C(12)-C(13)-C(16)-F(12) | 152.1(4) | | |
| C(14)-C(13)-C(16)-F(11) | 91.8(5) | | |
| C(12)-C(13)-C(16)-F(11) | -86.4(5) | | |
| C(14)-C(13)-C(16)-F(10) | -150.3(4) | | |
| C(12)-C(13)-C(16)-F(10) | 31.4(6) | | |
| S(4)-S(3)-N(3)-C(2) | -0.6(3) | | |
| S(3)-S(4)-N(4)-C(2) | -2.4(3) | | |
| S(4)-N(4)-C(2)-N(3) | 2.6(6) | | |
| S(4)-N(4)-C(2)-C(20) | -175.8(3) | | |
| S(3)-N(3)-C(2)-N(4) | -1.1(6) | | |
| S(3)-N(3)-C(2)-C(20) | 177.3(3) | | |
| N(4)-C(2)-C(20)-C(25) | 13.5(6) | | |
| N(3)-C(2)-C(20)-C(25) | -164.9(4) | | |
| N(4)-C(2)-C(20)-C(21) | -168.4(4) | | |
| N(3)-C(2)-C(20)-C(21) | 13.1(6) | | |
| C(25)-C(20)-C(21)-C(22) | 2.7(7) | | |
| C(2)-C(20)-C(21)-C(22) | -175.3(4) | | |
| C(20)-C(21)-C(22)-C(23) | -0.4(7) | | |
| C(21)-C(22)-C(23)-C(24) | -1.4(7) | | |
| C(21)-C(22)-C(23)-C(26) | 175.5(4) | | |
| C(25)-C(24)-C(23)-C(22) | 0.8(7) | | |
| C(25)-C(24)-C(23)-C(26) | -176.1(4) | | |
| C(23)-C(24)-C(25)-C(20) | 1.5(7) | | |
| C(21)-C(20)-C(25)-C(24) | -3.3(7) | | |
| C(2)-C(20)-C(25)-C(24) | 174.8(4) | | |
| C(22)-C(23)-C(26)-F(21) | 1.6(7) | | |
| C(24)-C(23)-C(26)-F(21) | 178.5(4) | | |
| C(22)-C(23)-C(26)-F(22) | -118.7(5) | | |
| C(24)-C(23)-C(26)-F(22) | 58.2(6) | | |
| C(22)-C(23)-C(26)-F(20) | 122.3(5) | | |
| C(24)-C(23)-C(26)-F(20) | -60.8(6) | | |
| S(6)-S(5)-N(5)-C(3) | 3.6(3) | | |
| S(5)-S(6)-N(6)-C(3) | 2.7(3) | | |
| S(5)-N(5)-C(3)-N(6) | -2.6(6) | | |
| S(5)-N(5)-C(3)-C(30) | 176.8(3) | | |
| S(6)-N(6)-C(3)-N(5) | -0.7(6) | | |
| S(6)-N(6)-C(3)-C(30) | 179.9(3) | | |
| N(5)-C(3)-C(30)-C(35) | 13.3(6) | | |
| N(6)-C(3)-C(30)-C(35) | -167.3(4) | | |
| N(5)-C(3)-C(30)-C(31) | -167.5(4) | | |
| N(6)-C(3)-C(30)-C(31) | 11.9(6) | | |
| C(35)-C(34)-C(33)-C(32) | -1.1(7) | | |
| C(35)-C(34)-C(33)-C(36A) | -176.9(7) | | |
| C(35)-C(34)-C(33)-C(36) | -178.2(5) | | |
| C(31)-C(32)-C(33)-C(34) | 0.4(7) | | |
| C(31)-C(32)-C(33)-C(36A) | 175.8(8) | | |
| C(31)-C(32)-C(33)-C(36) | 177.7(5) | | |
| C(33)-C(32)-C(31)-C(30) | -0.3(7) | | |
| C(35)-C(30)-C(31)-C(32) | 0.7(7) | | |
| C(3)-C(30)-C(31)-C(32) | -178.5(4) | | |
| C(31)-C(30)-C(35)-C(34) | -1.4(6) | | |

| | |
|---|---|
| C(3)-C(30)-C(35)-C(34) | 177.9(4) |
| C(33)-C(34)-C(35)-C(30) | 1.6(7) |
| C(34)-C(33)-C(36)-F(32) | -21.5(9) |
| C(32)-C(33)-C(36)-F(32) | 161.4(6) |
| C(34)-C(33)-C(36)-F(30) | 98.6(7) |
| C(32)-C(33)-C(36)-F(30) | -78.5(7) |
| C(34)-C(33)-C(36)-F(31) | -144.0(6) |
| C(32)-C(33)-C(36)-F(31) | 38.8(8) |
| C(34)-C(33)-C(36A)-F(32A) | 31.7(13) |
| C(32)-C(33)-C(36A)-F(32A) | -143.9(9) |
| C(34)-C(33)-C(36A)-F(30A) | 153.7(9) |
| C(32)-C(33)-C(36A)-F(30A) | -21.8(14) |
| C(34)-C(33)-C(36A)-F(31A) | -89.4(10) |
| C(32)-C(33)-C(36A)-F(31A) | 95.1(10) |
| S(8)-S(7)-N(7)-C(4) | -0.4(3) |
| S(7)-S(8)-N(8)-C(4) | -0.3(3) |
| S(7)-N(7)-C(4)-N(8) | 0.4(6) |
| S(7)-N(7)-C(4)-C(40) | -178.2(3) |
| S(8)-N(8)-C(4)-N(7) | 0.0(6) |
| S(8)-N(8)-C(4)-C(40) | 178.5(3) |
| N(7)-C(4)-C(40)-C(45) | -172.9(4) |
| N(8)-C(4)-C(40)-C(45) | 8.5(6) |
| N(7)-C(4)-C(40)-C(41) | 7.3(6) |
| N(8)-C(4)-C(40)-C(41) | -171.3(4) |
| C(41)-C(40)-C(45)-C(44) | 2.3(7) |
| C(4)-C(40)-C(45)-C(44) | -177.5(4) |
| C(44)-C(43)-C(42)-C(41) | 0.9(7) |
| C(46)-C(43)-C(42)-C(41) | -178.8(5) |
| C(46A)-C(43)-C(42)-C(41) | -173.7(7) |
| C(42)-C(43)-C(44)-C(45) | -0.5(7) |
| C(46)-C(43)-C(44)-C(45) | 179.3(6) |
| C(46A)-C(43)-C(44)-C(45) | 174.6(7) |
| C(40)-C(45)-C(44)-C(43) | -1.1(7) |
| C(43)-C(42)-C(41)-C(40) | 0.2(7) |
| C(45)-C(40)-C(41)-C(42) | -1.9(7) |
| C(4)-C(40)-C(41)-C(42) | 178.0(4) |
| C(44)-C(43)-C(46)-F(42) | -119.1(8) |
| C(42)-C(43)-C(46)-F(42) | 60.6(9) |
| C(44)-C(43)-C(46)-F(41) | 119.4(9) |
| C(42)-C(43)-C(46)-F(41) | -60.8(10) |
| C(44)-C(43)-C(46)-F(40) | -0.5(11) |
| C(42)-C(43)-C(46)-F(40) | 179.3(7) |
| C(44)-C(43)-C(46A)-F(41A) | 156.0(10) |
| C(42)-C(43)-C(46A)-F(41A) | -29.1(14) |
| C(44)-C(43)-C(46A)-F(42A) | -84.3(11) |
| C(42)-C(43)-C(46A)-F(42A) | 90.7(11) |
| C(44)-C(43)-C(46A)-F(40A) | 35.8(14) |
| C(42)-C(43)-C(46A)-F(40A) | -149.3(10) |
| S(10)-S(9)-N(9)-C(5) | -0.1(3) |
| S(9)-S(10)-N(10)-C(5) | -1.6(3) |
| S(10)-N(10)-C(5)-N(9) | 2.0(6) |
| S(10)-N(10)-C(5)-C(50) | -178.2(3) |
| S(9)-N(9)-C(5)-N(10) | -1.1(6) |
| S(9)-N(9)-C(5)-C(50) | 179.1(3) |
| C(50)-C(51)-C(52)-C(53) | 0.1(7) |
| C(52)-C(51)-C(50)-C(55) | -0.8(7) |
| C(52)-C(51)-C(50)-C(5) | 179.9(4) |
| N(10)-C(5)-C(50)-C(51) | -179.7(4) |
| N(9)-C(5)-C(50)-C(51) | 0.0(6) |
| N(10)-C(5)-C(50)-C(55) | 1.0(6) |
| N(9)-C(5)-C(50)-C(55) | -179.3(4) |
| C(51)-C(52)-C(53)-C(54) | 0.8(7) |
| C(51)-C(52)-C(53)-C(56) | -178.6(4) |
| C(52)-C(53)-C(54)-C(55) | -1.1(7) |
| C(56)-C(53)-C(54)-C(55) | 178.4(4) |
| C(53)-C(54)-C(55)-C(50) | 0.4(7) |
| C(51)-C(50)-C(55)-C(54) | 0.6(7) |
| C(5)-C(50)-C(55)-C(54) | 179.9(4) |
| C(52)-C(53)-C(56)-F(50) | -7.7(7) |
| C(54)-C(53)-C(56)-F(50) | 172.8(4) |
| C(52)-C(53)-C(56)-F(51) | 114.9(5) |
| C(54)-C(53)-C(56)-F(51) | -64.6(6) |
| C(52)-C(53)-C(56)-F(52) | -127.9(5) |
| C(54)-C(53)-C(56)-F(52) | 52.6(6) |

| | |
|---|---|
| S(12)-S(11)-N(11)-C(6) | 0.2(3) |
| S(11)-S(12)-N(12)-C(6) | 0.6(3) |
| S(12)-N(12)-C(6)-N(11) | -0.6(6) |
| S(12)-N(12)-C(6)-C(60) | 177.5(3) |
| S(11)-N(11)-C(6)-N(12) | 0.2(6) |
| S(11)-N(11)-C(6)-C(60) | -178.0(3) |
| N(12)-C(6)-C(60)-C(65) | 3.9(7) |
| N(11)-C(6)-C(60)-C(65) | -177.9(4) |
| N(12)-C(6)-C(60)-C(61) | -174.6(4) |
| N(11)-C(6)-C(60)-C(61) | 3.6(6) |
| C(65)-C(60)-C(61)-C(62) | 0.5(7) |
| C(6)-C(60)-C(61)-C(62) | 179.0(4) |
| C(60)-C(61)-C(62)-C(63) | 0.1(7) |
| C(64)-C(63)-C(62)-C(61) | -1.2(7) |
| C(66)-C(63)-C(62)-C(61) | -179.1(4) |
| C(62)-C(63)-C(64)-C(65) | 1.7(7) |
| C(66)-C(63)-C(64)-C(65) | 179.5(4) |
| C(64)-C(63)-C(66)-F(61) | 6.5(7) |
| C(62)-C(63)-C(66)-F(61) | -175.6(4) |
| C(64)-C(63)-C(66)-F(60) | 127.3(5) |
| C(62)-C(63)-C(66)-F(60) | -54.8(6) |
| C(64)-C(63)-C(66)-F(62) | -115.2(5) |
| C(62)-C(63)-C(66)-F(62) | 62.6(6) |
| C(63)-C(64)-C(65)-C(60) | -1.2(7) |
| C(61)-C(60)-C(65)-C(64) | 0.1(7) |
| C(6)-C(60)-C(65)-C(64) | -178.4(4) |
| S(14)-S(13)-N(13)-C(7) | -1.3(3) |
| S(13)-S(14)-N(14)-C(7) | -1.2(3) |
| S(14)-N(14)-C(7)-N(13) | 0.6(6) |
| S(14)-N(14)-C(7)-C(70) | 178.5(3) |
| S(13)-N(13)-C(7)-N(14) | 0.7(6) |
| S(13)-N(13)-C(7)-C(70) | -177.3(3) |
| N(14)-C(7)-C(70)-C(71) | -160.8(4) |
| N(13)-C(7)-C(70)-C(71) | 17.2(6) |
| N(14)-C(7)-C(70)-C(75) | 18.9(6) |
| N(13)-C(7)-C(70)-C(75) | -163.1(4) |
| C(75)-C(70)-C(71)-C(72) | -1.0(7) |
| C(7)-C(70)-C(71)-C(72) | 178.7(4) |
| C(72)-C(73)-C(74)-C(75) | -0.2(7) |
| C(76)-C(73)-C(74)-C(75) | -179.3(4) |
| C(70)-C(71)-C(72)-C(73) | 1.3(7) |
| C(74)-C(73)-C(72)-C(71) | -0.7(7) |
| C(76)-C(73)-C(72)-C(71) | 178.5(4) |
| C(73)-C(74)-C(75)-C(70) | 0.5(7) |
| C(71)-C(70)-C(75)-C(74) | 0.1(7) |
| C(7)-C(70)-C(75)-C(74) | -179.6(4) |
| C(72)-C(73)-C(76)-F(72) | 42.8(6) |
| C(74)-C(73)-C(76)-F(72) | -138.1(5) |
| C(72)-C(73)-C(76)-F(71) | -76.6(5) |
| C(74)-C(73)-C(76)-F(71) | 102.6(5) |
| C(72)-C(73)-C(76)-F(70) | 165.0(4) |
| C(74)-C(73)-C(76)-F(70) | -15.9(6) |
| S(16)-S(15)-N(15)-C(8) | 1.7(3) |
| S(15)-S(16)-N(16)-C(8) | 1.5(3) |
| S(15)-N(15)-C(8)-N(16) | -1.0(6) |
| S(15)-N(15)-C(8)-C(80) | 179.2(3) |
| S(16)-N(16)-C(8)-N(15) | -0.7(6) |
| S(16)-N(16)-C(8)-C(80) | 179.1(3) |
| N(15)-C(8)-C(80)-C(81) | 20.1(6) |
| N(16)-C(8)-C(80)-C(81) | -159.7(4) |
| N(15)-C(8)-C(80)-C(85) | -161.7(4) |
| N(16)-C(8)-C(80)-C(85) | 18.5(6) |
| C(85)-C(80)-C(81)-C(82) | -0.9(6) |
| C(8)-C(80)-C(81)-C(82) | 177.3(4) |
| C(82)-C(83)-C(84)-C(85) | -0.1(7) |
| C(86)-C(83)-C(84)-C(85) | 177.0(4) |
| C(83)-C(84)-C(85)-C(80) | -1.0(7) |
| C(81)-C(80)-C(85)-C(84) | 1.5(6) |
| C(8)-C(80)-C(85)-C(84) | -176.8(4) |
| C(84)-C(83)-C(86)-F(82) | 156.9(4) |
| C(82)-C(83)-C(86)-F(82) | -26.0(6) |
| C(84)-C(83)-C(86)-F(80) | -82.2(5) |
| C(82)-C(83)-C(86)-F(80) | 94.9(5) |
| C(84)-C(83)-C(86)-F(81) | 35.0(6) |

| | | | |
|---|---|---|---|
| C(82)-C(83)-C(86)-F(81) | -147.9(4) | C(84)-C(83)-C(82)-C(81) | 0.7(7) |
| C(80)-C(81)-C(82)-C(83) | -0.2(6) | C(86)-C(83)-C(82)-C(81) | -176.3(4) |

**Table S5G.** Least-squares planes (x,y,z in crystal coordinates) and deviations from them in **1**

8.3112 (0.0058) x + 4.6772 (0.0221) y + 0.0375 (0.0292) z = 3.6256 (0.0300)

* 0.0008 (0.0027) C8
* -0.0113 (0.0023) N15
* 0.0102 (0.0023) N16
* 0.0122 (0.0014) S15
* -0.0120 (0.0015) S16

Rms deviation of fitted atoms = 0.0102

8.2937 (0.0060) x + 5.1137 (0.0225) y - 2.7637 (0.0287) z = 6.5003
(0.0280) Angle to previous plane (with approximate esd) = 7.348 ( 0.157 )

* -0.0002 (0.0027) C7
* 0.0085 (0.0023) N13
* -0.0082 (0.0023) N14
* -0.0095 (0.0015) S13
* 0.0094 (0.0015) S14

Rms deviation of fitted atoms = 0.0080

7.8677 (0.0071) x + 6.5943 (0.0225) y - 3.1754 (0.0280) z = 11.2893 (0.0076)
Angle to previous plane (with approximate esd) = 5.061 ( 0.174 )

* 0.0018 (0.0027) C6
* 0.0014 (0.0023) N11
* -0.0039 (0.0022) N12
* -0.0026 (0.0015) S11
* 0.0034 (0.0015) S12

Rms deviation of fitted atoms = 0.0028

7.3324 (0.0080) x + 8.1602 (0.0215) y - 3.2936 (0.0278) z = 8.5612
(0.0066) Angle to previous plane (with approximate esd) = 5.422 ( 0.192 )

* -0.0078 (0.0027) C5
* -0.0001 (0.0023) N9
* 0.0110 (0.0022) N10
* 0.0047 (0.0015) S9
* -0.0077 (0.0015) S10

Rms deviation of fitted atoms = 0.0073

7.7091 (0.0074) x + 6.7552 (0.0226) y + 0.2073 (0.0282) z = 10.4044
(0.0188) Angle to previous plane (with approximate esd) = 10.269 ( 0.185 )

* -0.0010 (0.0027) C4
* 0.0029 (0.0023) N7
* -0.0016 (0.0023) N8
* -0.0028 (0.0014) S7
* 0.0024 (0.0015) S8

Rms deviation of fitted atoms = 0.0023

7.2549 (0.0081) x + 8.2058 (0.0215) y - 0.5861 (0.0280) z = 7.0504
(0.0253) Angle to previous plane (with approximate esd) = 5.367 ( 0.196 )
* 0.0049 (0.0027) C3
* -0.0243 (0.0023) N5
* 0.0177 (0.0023) N6
* 0.0244 (0.0015) S5
* -0.0227 (0.0015) S6
Rms deviation of fitted atoms = 0.0202

8.2070 (0.0061) x + 5.3673 (0.0220) y - 1.6812 (0.0301) z = 1.9215
(0.0126) Angle to previous plane (with approximate esd) = 10.086 ( 0.176 )
* -0.0093 (0.0028) C2
* -0.0033 (0.0023) N3
* 0.0163 (0.0023) N4
* 0.0092 (0.0015) S3
* -0.0129 (0.0015) S4
Rms deviation of fitted atoms = 0.0111

8.2744 (0.0058) x + 4.6863 (0.0220) y + 0.5628 (0.0289) z = 5.8958
(0.0115) Angle to previous plane (with approximate esd) = 6.150 ( 0.160 )
* 0.0054 (0.0026) C1
* 0.0085 (0.0022) N1
* -0.0161 (0.0022) N2
* -0.0127 (0.0015) S1
* 0.0149 (0.0015) S2
Rms deviation of fitted atoms = 0.0122

**Details of the Crystal Structure Refinement of 2**

Both $CF_3$ groups in the structure of **2** display rotational disorder. This disorder could be described adequately a two-part disorder model but to get reasonable geometries (employing SADI restraints) it was found necessary to include the $CF_3$ carbon atoms in the model. Because of the relatively high thermal motion in this structure, possibly a consequence of collecting data at only 263(2) K, the newly developed RIGU restraints in ShelXL-2014 have been applied globally to this structure. However, in addition, a strong tendency for the fluorine atoms to go oblate or NPD was solved by applying ISOR restraints. Refined occupancies for both groups came very close to 50%, so the occupancies were frozen at 50:50 in the final cycles of refinement.

The SADI restraints on 1,2 C–F and 1,3 F–F distances permitted the two $CF_3$ groups to rotate apart without requiring the two rotamers to be staggered. The combination of this approach provided a successful disorder model which is depicted in Figure S10. In the final difference Fourier map, the highest peak of 0.66 $e/Å^3$ at (0.2723 0.9619 0.4493) and the deepest hole of -0.29 $e/Å^3$ at (0.3184 0.9178 0.4642) associated with the heaviest atoms in the unit cell, namely antimony, and are *not* associated with the $CF_3$ groups. This is considered a positive indication of the adequacy of the disorder models applied to the trifluoromethyl groups.

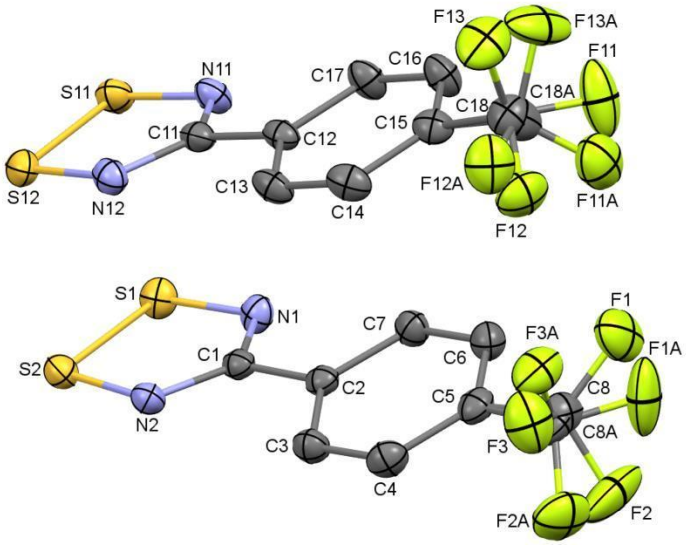


**Figure S10.** Depiction of the two-component disorder models developed for the $CF_3$ groups of the dimer in **2**. Displacement ellipsoids drawn at 20%. The refined occupancies are 48:52 for C8/C8A and 51:49 for C18/C18A.

**Table S6**. Crystal structure report for **2**

**Table S6A**. Crystal data and structure refinement for [CF3Phdtda]2.SbPh3 (**2**) in P-1.

| | | |
|---|---|---|
| Empirical formula | C34 H23 F6 N4 S4 Sb | |
| Formula weight | 851.55 | |
| Temperature | 263(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Triclinic | |
| Space group | P-1 | |
| Unit cell dimensions | a = 11.4543(10) Å | α= 73.3640(10)°. |
| | b = 11.7399(10) Å | β= 73.2470(10)°. |
| | c = 13.9480(12) Å | γ = 82.5790(10)°. |
| Volume | 1718.6(3) $Å^3$ | |
| Z | 2 | |
| Density (calculated) | 1.646 $Mg/m^3$ | |
| Absorption coefficient | 1.110 $mm^{-1}$ | |
| F(000) | 848 | |
| Crystal size | 0.460 x 0.420 x 0.280 $mm^3$ | |
| Theta range for data collection | 1.813 to 28.578°. | |
| Index ranges | -15<=h<=15, -15<=k<=15, -18<=l<=18 | |
| Reflections collected | 19773 | |
| Independent reflections | 8041 [R(int) = 0.0195] | |
| Completeness to theta = 25.250° | 99.6 % | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 8041 / 594 / 516 | |
| Goodness-of-fit on $F^2$ | 1.054 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0313, wR2 = 0.0789 | |
| R indices (all data) | R1 = 0.0404, wR2 = 0.0864 | |
| Extinction coefficient | n/a | |
| Largest diff. peak and hole | 0.664 and -0.590 $e.Å^{-3}$ | |

**Table S6B**. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters (Å$^2$x $10^3$) for **2**. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 4731(1) | 3841(1) | 4461(1) | 59(1) |
| S(2) | 5263(1) | 2693(1) | 3496(1) | 56(1) |
| N(1) | 3712(2) | 4590(2) | 3885(2) | 54(1) |
| N(2) | 4284(2) | 3284(2) | 2822(2) | 51(1) |
| C(1) | 3610(2) | 4204(2) | 3101(2) | 46(1) |
| C(2) | 2732(2) | 4841(2) | 2507(2) | 47(1) |
| C(3) | 2450(3) | 4361(2) | 1803(2) | 57(1) |
| C(4) | 1668(3) | 4969(3) | 1221(2) | 64(1) |
| C(5) | 1160(2) | 6074(3) | 1329(2) | 58(1) |
| C(6) | 1429(3) | 6559(3) | 2022(2) | 64(1) |
| C(7) | 2205(2) | 5949(2) | 2613(2) | 57(1) |
| C(8) | 329(12) | 6808(11) | 698(10) | 78(3) |
| F(1) | 310(7) | 7942(5) | 596(7) | 124(3) |
| F(2) | -749(6) | 6417(9) | 1061(7) | 157(4) |
| F(3) | 673(9) | 6713(8) | -265(6) | 117(3) |
| C(8A) | 328(11) | 6674(10) | 663(9) | 73(3) |
| F(1A) | -392(7) | 7517(9) | 1000(6) | 141(3) |
| F(2A) | -357(8) | 5980(7) | 489(7) | 127(2) |
| F(3A) | 939(7) | 7216(7) | -279(5) | 98(2) |
| S(11) | 6673(1) | 5563(1) | 3050(1) | 62(1) |
| S(12) | 7171(1) | 4483(1) | 2016(1) | 61(1) |
| N(11) | 5768(2) | 6462(2) | 2431(2) | 58(1) |
| N(12) | 6319(2) | 5253(2) | 1277(2) | 57(1) |
| C(11) | 5709(2) | 6195(2) | 1576(2) | 50(1) |
| C(12) | 4962(2) | 7002(2) | 922(2) | 49(1) |
| C(13) | 4760(3) | 6690(2) | 96(2) | 60(1) |
| C(14) | 4099(3) | 7449(3) | -529(2) | 64(1) |
| C(15) | 3645(3) | 8528(3) | -350(2) | 60(1) |
| C(16) | 3834(3) | 8845(3) | 472(2) | 69(1) |
| C(17) | 4487(3) | 8083(2) | 1108(2) | 63(1) |
| C(18) | 3050(10) | 9376(11) | -1103(9) | 73(3) |
| F(11) | 2242(8) | 10124(7) | -703(5) | 136(3) |

| | | | | |
|---|---|---|---|---|
| F(12) | 2420(6) | 8910(9) | -1547(7) | 101(2) |
| F(13) | 3864(6) | 9984(7) | -1901(4) | 114(2) |
| C(18A) | 2942(13) | 9399(12) | -1037(10) | 88(3) |
| F(11A) | 1805(8) | 9552(7) | -580(7) | 120(2) |
| F(12A) | 2964(7) | 8987(9) | -1844(6) | 105(2) |
| F(13A) | 3414(8) | 10439(5) | -1414(8) | 150(3) |
| Sb(1) | 6584(1) | 301(1) | 5808(1) | 53(1) |
| C(20) | 7487(2) | -974(2) | 4966(2) | 46(1) |
| C(21) | 8361(2) | -1831(2) | 5274(2) | 55(1) |
| C(22) | 8864(3) | -2670(3) | 4723(2) | 63(1) |
| C(23) | 8505(3) | -2662(3) | 3856(2) | 62(1) |
| C(24) | 7644(3) | -1827(3) | 3546(2) | 62(1) |
| C(25) | 7137(2) | -993(2) | 4093(2) | 56(1) |
| C(30) | 7609(2) | -147(2) | 6953(2) | 47(1) |
| C(31) | 8667(2) | 394(2) | 6833(2) | 55(1) |
| C(32) | 9284(3) | 66(3) | 7598(2) | 61(1) |
| C(33) | 8863(3) | -817(3) | 8486(2) | 65(1) |
| C(34) | 7818(3) | -1372(3) | 8617(2) | 64(1) |
| C(35) | 7188(3) | -1033(2) | 7862(2) | 55(1) |
| C(40) | 7636(2) | 1798(2) | 4804(2) | 47(1) |
| C(41) | 8515(2) | 1743(2) | 3888(2) | 55(1) |
| C(42) | 9207(3) | 2708(2) | 3299(2) | 60(1) |
| C(43) | 9036(3) | 3745(2) | 3619(2) | 60(1) |
| C(44) | 8159(3) | 3815(2) | 4524(2) | 57(1) |
| C(45) | 7457(2) | 2856(2) | 5100(2) | 51(1) |

**Table S6C**. Bond lengths [Å] and angles [°] for **2**

| | | | |
|---|---|---|---|
| S(1)-N(1) | 1.629(2) | C(15)-C(16) | 1.382(4) |
| S(1)-S(2) | 2.0865(9) | C(15)-C(18) | 1.483(16) |
| S(2)-N(2) | 1.629(2) | C(15)-C(18A) | 1.510(17) |
| N(1)-C(1) | 1.336(3) | C(16)-C(17) | 1.379(4) |
| N(2)-C(1) | 1.330(3) | C(16)-H(16) | 0.9300 |
| C(1)-C(2) | 1.479(3) | C(17)-H(17) | 0.9300 |
| C(2)-C(3) | 1.390(3) | C(18)-F(11) | 1.308(10) |
| C(2)-C(7) | 1.391(4) | C(18)-F(13) | 1.318(10) |
| C(3)-C(4) | 1.376(4) | C(18)-F(12) | 1.325(11) |
| C(3)-H(3) | 0.9300 | C(18A)-F(11A) | 1.289(11) |
| C(4)-C(5) | 1.381(4) | C(18A)-F(13A) | 1.303(11) |
| C(4)-H(4) | 0.9300 | C(18A)-F(12A) | 1.339(11) |
| C(5)-C(6) | 1.373(4) | Sb(1)-C(20) | 2.145(2) |
| C(5)-C(8A) | 1.494(15) | Sb(1)-C(30) | 2.155(2) |
| C(5)-C(8) | 1.504(16) | Sb(1)-C(40) | 2.160(3) |
| C(6)-C(7) | 1.379(4) | C(20)-C(21) | 1.392(3) |
| C(6)-H(6) | 0.9300 | C(20)-C(25) | 1.393(3) |
| C(7)-H(7) | 0.9300 | C(21)-C(22) | 1.386(4) |
| C(8)-F(2) | 1.282(11) | C(21)-H(21) | 0.9300 |
| C(8)-F(1) | 1.295(11) | C(22)-C(23) | 1.382(4) |
| C(8)-F(3) | 1.320(11) | C(22)-H(22) | 0.9300 |
| C(8A)-F(1A) | 1.309(11) | C(23)-C(24) | 1.369(4) |
| C(8A)-F(2A) | 1.313(11) | C(23)-H(23) | 0.9300 |
| C(8A)-F(3A) | 1.314(10) | C(24)-C(25) | 1.380(4) |
| S(11)-N(11) | 1.626(2) | C(24)-H(24) | 0.9300 |
| S(11)-S(12) | 2.0972(10) | C(25)-H(25) | 0.9300 |
| S(12)-N(12) | 1.629(2) | C(30)-C(31) | 1.386(3) |
| N(11)-C(11) | 1.339(3) | C(30)-C(35) | 1.394(3) |
| N(12)-C(11) | 1.334(3) | C(31)-C(32) | 1.385(4) |
| C(11)-C(12) | 1.485(4) | C(31)-H(31) | 0.9300 |
| C(12)-C(17) | 1.380(4) | C(32)-C(33) | 1.375(4) |
| C(12)-C(13) | 1.390(3) | C(32)-H(32) | 0.9300 |
| C(13)-C(14) | 1.375(4) | C(33)-C(34) | 1.377(5) |
| C(13)-H(13) | 0.9300 | C(33)-H(33) | 0.9300 |
| C(14)-C(15) | 1.368(4) | C(34)-C(35) | 1.383(4) |
| C(14)-H(14) | 0.9300 | C(34)-H(34) | 0.9300 |

| | |
|---|---|
| C(35)-H(35) | 0.9300 |
| C(40)-C(45) | 1.391(3) |
| C(40)-C(41) | 1.392(3) |
| C(41)-C(42) | 1.382(4) |
| C(41)-H(41) | 0.9300 |
| C(42)-C(43) | 1.385(4) |
| C(42)-H(42) | 0.9300 |
| C(43)-C(44) | 1.384(4) |
| C(43)-H(43) | 0.9300 |
| C(44)-C(45) | 1.380(4) |
| C(44)-H(44) | 0.9300 |
| C(45)-H(45) | 0.9300 |

| | |
|---|---|
| N(1)-S(1)-S(2) | 94.40(8) |
| N(2)-S(2)-S(1) | 94.42(8) |
| C(1)-N(1)-S(1) | 114.27(18) |
| C(1)-N(2)-S(2) | 114.39(17) |
| N(2)-C(1)-N(1) | 122.5(2) |
| N(2)-C(1)-C(2) | 118.7(2) |
| N(1)-C(1)-C(2) | 118.7(2) |
| C(3)-C(2)-C(7) | 118.6(2) |
| C(3)-C(2)-C(1) | 120.5(2) |
| C(7)-C(2)-C(1) | 120.9(2) |
| C(4)-C(3)-C(2) | 120.8(3) |
| C(4)-C(3)-H(3) | 119.6 |
| C(2)-C(3)-H(3) | 119.6 |
| C(3)-C(4)-C(5) | 119.9(3) |
| C(3)-C(4)-H(4) | 120.1 |
| C(5)-C(4)-H(4) | 120.1 |
| C(6)-C(5)-C(4) | 120.0(3) |
| C(6)-C(5)-C(8A) | 123.7(5) |
| C(4)-C(5)-C(8A) | 116.3(5) |
| C(6)-C(5)-C(8) | 117.0(5) |
| C(4)-C(5)-C(8) | 123.0(5) |
| C(5)-C(6)-C(7) | 120.3(3) |
| C(5)-C(6)-H(6) | 119.8 |
| C(7)-C(6)-H(6) | 119.8 |
| C(6)-C(7)-C(2) | 120.4(3) |
| C(6)-C(7)-H(7) | 119.8 |
| C(2)-C(7)-H(7) | 119.8 |
| F(2)-C(8)-F(1) | 111.5(11) |
| F(2)-C(8)-F(3) | 104.5(11) |
| F(1)-C(8)-F(3) | 104.0(10) |
| F(2)-C(8)-C(5) | 110.3(9) |
| F(1)-C(8)-C(5) | 114.3(10) |
| F(3)-C(8)-C(5) | 111.7(10) |
| F(1A)-C(8A)-F(2A) | 107.4(10) |
| F(1A)-C(8A)-F(3A) | 104.2(10) |
| F(2A)-C(8A)-F(3A) | 102.7(10) |
| F(1A)-C(8A)-C(5) | 113.3(9) |
| F(2A)-C(8A)-C(5) | 116.4(9) |
| F(3A)-C(8A)-C(5) | 111.7(9) |
| N(11)-S(11)-S(12) | 94.56(8) |
| N(12)-S(12)-S(11) | 94.12(8) |
| C(11)-N(11)-S(11) | 114.21(19) |
| C(11)-N(12)-S(12) | 114.57(19) |
| N(12)-C(11)-N(11) | 122.5(2) |
| N(12)-C(11)-C(12) | 119.1(2) |
| N(11)-C(11)-C(12) | 118.4(2) |
| C(17)-C(12)-C(13) | 119.1(3) |
| C(17)-C(12)-C(11) | 120.6(2) |
| C(13)-C(12)-C(11) | 120.3(2) |
| C(14)-C(13)-C(12) | 120.5(2) |
| C(14)-C(13)-H(13) | 119.7 |
| C(12)-C(13)-H(13) | 119.7 |
| C(15)-C(14)-C(13) | 120.1(3) |
| C(15)-C(14)-H(14) | 119.9 |
| C(13)-C(14)-H(14) | 119.9 |
| C(14)-C(15)-C(16) | 119.9(3) |
| C(14)-C(15)-C(18) | 118.7(5) |
| C(16)-C(15)-C(18) | 121.2(5) |
| C(14)-C(15)-C(18A) | 121.7(5) |
| C(16)-C(15)-C(18A) | 118.4(5) |
| C(17)-C(16)-C(15) | 120.3(3) |

| | | | |
|---|---|---|---|
| C(17)-C(16)-H(16) | 119.9 | C(20)-C(25)-H(25) | 119.4 |
| C(15)-C(16)-H(16) | 119.9 | C(31)-C(30)-C(35) | 118.2(2) |
| C(16)-C(17)-C(12) | 120.1(3) | C(31)-C(30)-Sb(1) | 123.56(19) |
| C(16)-C(17)-H(17) | 120.0 | C(35)-C(30)-Sb(1) | 118.21(18) |
| C(12)-C(17)-H(17) | 120.0 | C(32)-C(31)-C(30) | 120.8(3) |
| F(11)-C(18)-F(13) | 108.8(10) | C(32)-C(31)-H(31) | 119.6 |
| F(11)-C(18)-F(12) | 102.3(11) | C(30)-C(31)-H(31) | 119.6 |
| F(13)-C(18)-F(12) | 103.0(9) | C(33)-C(32)-C(31) | 120.3(3) |
| F(11)-C(18)-C(15) | 113.9(9) | C(33)-C(32)-H(32) | 119.9 |
| F(13)-C(18)-C(15) | 111.2(9) | C(31)-C(32)-H(32) | 119.9 |
| F(12)-C(18)-C(15) | 116.6(10) | C(32)-C(33)-C(34) | 119.8(3) |
| F(11A)-C(18A)-F(13A) | 108.1(11) | C(32)-C(33)-H(33) | 120.1 |
| F(11A)-C(18A)-F(12A) | 105.9(11) | C(34)-C(33)-H(33) | 120.1 |
| F(13A)-C(18A)-F(12A) | 107.1(11) | C(33)-C(34)-C(35) | 120.1(3) |
| F(11A)-C(18A)-C(15) | 113.6(10) | C(33)-C(34)-H(34) | 120.0 |
| F(13A)-C(18A)-C(15) | 112.3(10) | C(35)-C(34)-H(34) | 120.0 |
| F(12A)-C(18A)-C(15) | 109.5(10) | C(34)-C(35)-C(30) | 120.8(3) |
| C(20)-Sb(1)-C(30) | 97.46(9) | C(34)-C(35)-H(35) | 119.6 |
| C(20)-Sb(1)-C(40) | 96.88(9) | C(30)-C(35)-H(35) | 119.6 |
| C(30)-Sb(1)-C(40) | 95.76(9) | C(45)-C(40)-C(41) | 118.3(2) |
| C(21)-C(20)-C(25) | 117.8(2) | C(45)-C(40)-Sb(1) | 118.64(18) |
| C(21)-C(20)-Sb(1) | 123.60(18) | C(41)-C(40)-Sb(1) | 123.04(18) |
| C(25)-C(20)-Sb(1) | 118.48(18) | C(42)-C(41)-C(40) | 120.8(2) |
| C(22)-C(21)-C(20) | 120.7(2) | C(42)-C(41)-H(41) | 119.6 |
| C(22)-C(21)-H(21) | 119.6 | C(40)-C(41)-H(41) | 119.6 |
| C(20)-C(21)-H(21) | 119.6 | C(41)-C(42)-C(43) | 120.1(2) |
| C(23)-C(22)-C(21) | 120.2(3) | C(41)-C(42)-H(42) | 119.9 |
| C(23)-C(22)-H(22) | 119.9 | C(43)-C(42)-H(42) | 119.9 |
| C(21)-C(22)-H(22) | 119.9 | C(44)-C(43)-C(42) | 119.7(3) |
| C(24)-C(23)-C(22) | 119.8(3) | C(44)-C(43)-H(43) | 120.2 |
| C(24)-C(23)-H(23) | 120.1 | C(42)-C(43)-H(43) | 120.2 |
| C(22)-C(23)-H(23) | 120.1 | C(45)-C(44)-C(43) | 120.0(2) |
| C(23)-C(24)-C(25) | 120.3(3) | C(45)-C(44)-H(44) | 120.0 |
| C(23)-C(24)-H(24) | 119.8 | C(43)-C(44)-H(44) | 120.0 |
| C(25)-C(24)-H(24) | 119.8 | C(44)-C(45)-C(40) | 121.1(2) |
| C(24)-C(25)-C(20) | 121.2(2) | C(44)-C(45)-H(45) | 119.4 |
| C(24)-C(25)-H(25) | 119.4 | C(40)-C(45)-H(45) | 119.4 |

**Table S6D**. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **2**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| S(1) | 63(1) | 71(1) | 45(1) | -19(1) | -14(1) | 1(1) |
| S(2) | 62(1) | 46(1) | 62(1) | -15(1) | -17(1) | 0(1) |
| N(1) | 51(1) | 66(1) | 49(1) | -26(1) | -10(1) | 3(1) |
| N(2) | 53(1) | 48(1) | 57(1) | -20(1) | -15(1) | -3(1) |
| C(1) | 43(1) | 48(1) | 46(1) | -16(1) | -3(1) | -8(1) |
| C(2) | 42(1) | 50(1) | 46(1) | -14(1) | -4(1) | -9(1) |
| C(3) | 63(2) | 54(1) | 60(2) | -22(1) | -18(1) | -4(1) |
| C(4) | 67(2) | 75(2) | 58(2) | -22(1) | -22(1) | -9(1) |
| C(5) | 47(1) | 67(2) | 53(1) | -6(1) | -7(1) | -8(1) |
| C(6) | 59(2) | 61(2) | 69(2) | -20(1) | -15(1) | 4(1) |
| C(7) | 57(2) | 60(2) | 62(2) | -28(1) | -15(1) | 2(1) |
| C(8) | 80(6) | 78(5) | 75(6) | -4(4) | -33(4) | -8(4) |
| F(1) | 140(6) | 84(3) | 163(7) | -10(3) | -94(5) | 17(3) |
| F(2) | 65(3) | 212(9) | 148(7) | 59(6) | -48(3) | -42(4) |
| F(3) | 154(8) | 128(7) | 85(4) | -37(4) | -64(4) | 33(5) |
| C(8A) | 51(4) | 91(5) | 63(5) | -1(3) | -10(3) | -10(3) |
| F(1A) | 128(6) | 196(7) | 123(6) | -84(6) | -78(5) | 102(5) |
| F(2A) | 112(5) | 142(5) | 147(6) | -13(5) | -82(5) | -25(4) |
| F(3A) | 86(3) | 117(6) | 73(3) | 17(3) | -28(2) | -19(4) |
| S(11) | 67(1) | 55(1) | 74(1) | -23(1) | -30(1) | -5(1) |
| S(12) | 52(1) | 59(1) | 69(1) | -21(1) | -6(1) | 0(1) |
| N(11) | 75(2) | 47(1) | 62(1) | -22(1) | -25(1) | -2(1) |
| N(12) | 61(1) | 53(1) | 54(1) | -20(1) | -5(1) | -1(1) |
| C(11) | 55(1) | 44(1) | 50(1) | -15(1) | -6(1) | -11(1) |
| C(12) | 59(1) | 43(1) | 42(1) | -12(1) | -4(1) | -12(1) |
| C(13) | 86(2) | 46(1) | 49(1) | -19(1) | -11(1) | -9(1) |
| C(14) | 89(2) | 63(2) | 45(1) | -17(1) | -14(1) | -15(1) |
| C(15) | 69(2) | 59(2) | 47(1) | -8(1) | -11(1) | -12(1) |
| C(16) | 93(2) | 54(2) | 66(2) | -25(1) | -27(2) | 9(1) |
| C(17) | 88(2) | 55(2) | 57(2) | -27(1) | -26(1) | 4(1) |
| C(18) | 74(4) | 82(5) | 57(4) | -10(3) | -14(3) | -7(4) |
| F(11) | 183(7) | 147(7) | 88(4) | -56(5) | -75(5) | 98(6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| F(12) | 90(4) | 126(4) | 97(5) | -16(4) | -46(4) | -17(4) |
| F(13) | 112(4) | 126(5) | 71(3) | 29(3) | -25(3) | -18(4) |
| C(18A) | 120(6) | 73(5) | 78(6) | -6(4) | -50(5) | -9(4) |
| F(11A) | 128(4) | 114(5) | 135(5) | -33(4) | -69(4) | 19(4) |
| F(12A) | 126(6) | 133(5) | 65(3) | -28(4) | -45(4) | 17(5) |
| F(13A) | 189(8) | 71(3) | 203(9) | 49(4) | -137(6) | -50(4) |
| Sb(1) | 39(1) | 61(1) | 57(1) | -15(1) | -13(1) | 0(1) |
| C(20) | 43(1) | 46(1) | 50(1) | -8(1) | -13(1) | -10(1) |
| C(21) | 53(1) | 62(2) | 58(1) | -15(1) | -27(1) | 2(1) |
| C(22) | 61(2) | 59(2) | 73(2) | -19(1) | -26(1) | 6(1) |
| C(23) | 69(2) | 55(2) | 66(2) | -21(1) | -12(1) | -12(1) |
| C(24) | 73(2) | 63(2) | 58(2) | -16(1) | -26(1) | -14(1) |
| C(25) | 59(2) | 55(1) | 60(2) | -9(1) | -31(1) | -4(1) |
| C(30) | 47(1) | 45(1) | 49(1) | -15(1) | -10(1) | 1(1) |
| C(31) | 49(1) | 56(1) | 58(1) | -9(1) | -14(1) | -5(1) |
| C(32) | 56(2) | 62(2) | 75(2) | -27(1) | -26(1) | 4(1) |
| C(33) | 81(2) | 64(2) | 60(2) | -26(1) | -33(1) | 18(1) |
| C(34) | 89(2) | 52(2) | 47(1) | -10(1) | -18(1) | 1(1) |
| C(35) | 63(2) | 49(1) | 52(1) | -16(1) | -10(1) | -8(1) |
| C(40) | 46(1) | 47(1) | 47(1) | -12(1) | -15(1) | 9(1) |
| C(41) | 56(1) | 49(1) | 58(1) | -19(1) | -10(1) | 6(1) |
| C(42) | 61(2) | 56(2) | 54(1) | -14(1) | -4(1) | 5(1) |
| C(43) | 65(2) | 48(1) | 59(2) | -7(1) | -14(1) | 2(1) |
| C(44) | 68(2) | 45(1) | 57(1) | -15(1) | -20(1) | 8(1) |
| C(45) | 52(1) | 54(1) | 46(1) | -15(1) | -16(1) | 12(1) |

**Table S6E**. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for **2**.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(3) | 2794 | 3620 | 1725 | 68 |
| H(4) | 1481 | 4638 | 756 | 77 |
| H(6) | 1085 | 7302 | 2094 | 76 |
| H(7) | 2377 | 6282 | 3084 | 69 |
| H(13) | 5075 | 5962 | -34 | 72 |
| H(14) | 3960 | 7229 | -1075 | 77 |
| H(16) | 3520 | 9576 | 597 | 83 |
| H(17) | 4607 | 8298 | 1663 | 75 |
| H(21) | 8610 | -1841 | 5855 | 67 |
| H(22) | 9445 | -3240 | 4938 | 76 |
| H(23) | 8847 | -3222 | 3485 | 75 |
| H(24) | 7400 | -1822 | 2964 | 74 |
| H(25) | 6551 | -432 | 3875 | 67 |
| H(31) | 8966 | 985 | 6231 | 66 |
| H(32) | 9987 | 444 | 7511 | 73 |
| H(33) | 9283 | -1039 | 8996 | 78 |
| H(34) | 7537 | -1975 | 9213 | 77 |
| H(35) | 6474 | -1400 | 7962 | 66 |
| H(41) | 8639 | 1048 | 3671 | 66 |
| H(42) | 9787 | 2662 | 2687 | 72 |
| H(43) | 9509 | 4391 | 3228 | 72 |
| H(44) | 8043 | 4508 | 4743 | 68 |
| H(45) | 6853 | 2919 | 5696 | 61 |

**Table S6F**. Torsion angles [°] for **2**.

| | | | |
|---|---|---|---|
| S(2)-S(1)-N(1)-C(1) | 0.81(19) | S(11)-S(12)-N(12)-C(11) | 1.07(19) |
| S(1)-S(2)-N(2)-C(1) | 1.58(18) | S(12)-N(12)-C(11)-N(11) | -1.9(3) |
| S(2)-N(2)-C(1)-N(1) | -1.4(3) | S(12)-N(12)-C(11)-C(12) | 176.35(17) |
| S(2)-N(2)-C(1)-C(2) | 177.22(16) | S(11)-N(11)-C(11)-N(12) | 1.6(3) |
| S(1)-N(1)-C(1)-N(2) | 0.2(3) | S(11)-N(11)-C(11)-C(12) | -176.61(18) |
| S(1)-N(1)-C(1)-C(2) | -178.47(17) | N(12)-C(11)-C(12)-C(17) | -169.1(3) |
| N(2)-C(1)-C(2)-C(3) | 11.9(3) | N(11)-C(11)-C(12)-C(17) | 9.2(4) |
| N(1)-C(1)-C(2)-C(3) | -169.4(2) | N(12)-C(11)-C(12)-C(13) | 9.1(4) |
| N(2)-C(1)-C(2)-C(7) | -165.8(2) | N(11)-C(11)-C(12)-C(13) | -172.6(2) |
| N(1)-C(1)-C(2)-C(7) | 12.9(3) | C(17)-C(12)-C(13)-C(14) | 0.2(4) |
| C(7)-C(2)-C(3)-C(4) | 0.1(4) | C(11)-C(12)-C(13)-C(14) | -178.1(2) |
| C(1)-C(2)-C(3)-C(4) | -177.6(2) | C(12)-C(13)-C(14)-C(15) | 0.7(4) |
| C(2)-C(3)-C(4)-C(5) | 0.4(4) | C(13)-C(14)-C(15)-C(16) | -1.1(5) |
| C(3)-C(4)-C(5)-C(6) | -0.5(4) | C(13)-C(14)-C(15)-C(18) | 173.6(5) |
| C(3)-C(4)-C(5)-C(8A) | 179.7(5) | C(13)-C(14)-C(15)-C(18A) | 178.4(6) |
| C(3)-C(4)-C(5)-C(8) | 178.1(6) | C(14)-C(15)-C(16)-C(17) | 0.5(5) |
| C(4)-C(5)-C(6)-C(7) | 0.1(4) | C(18)-C(15)-C(16)-C(17) | -174.1(5) |
| C(8A)-C(5)-C(6)-C(7) | 179.9(5) | C(18A)-C(15)-C(16)-C(17) | -179.0(6) |
| C(8)-C(5)-C(6)-C(7) | -178.6(5) | C(15)-C(16)-C(17)-C(12) | 0.5(5) |
| C(5)-C(6)-C(7)-C(2) | 0.5(4) | C(13)-C(12)-C(17)-C(16) | -0.8(4) |
| C(3)-C(2)-C(7)-C(6) | -0.6(4) | C(11)-C(12)-C(17)-C(16) | 177.5(3) |
| C(1)-C(2)-C(7)-C(6) | 177.2(2) | C(14)-C(15)-C(18)-F(11) | 153.9(7) |
| C(6)-C(5)-C(8)-F(2) | -102.9(9) | C(16)-C(15)-C(18)-F(11) | -31.5(10) |
| C(4)-C(5)-C(8)-F(2) | 78.5(10) | C(14)-C(15)-C(18)-F(13) | -82.8(8) |
| C(6)-C(5)-C(8)-F(1) | 23.6(11) | C(16)-C(15)-C(18)-F(13) | 91.9(8) |
| C(4)-C(5)-C(8)-F(1) | -155.0(7) | C(14)-C(15)-C(18)-F(12) | 34.9(9) |
| C(6)-C(5)-C(8)-F(3) | 141.4(7) | C(16)-C(15)-C(18)-F(12) | -150.4(6) |
| C(4)-C(5)-C(8)-F(3) | -37.3(11) | C(14)-C(15)-C(18A)-F(11A) | 112.3(8) |
| C(6)-C(5)-C(8A)-F(1A) | -18.9(11) | C(16)-C(15)-C(18A)-F(11A) | -68.2(10) |
| C(4)-C(5)-C(8A)-F(1A) | 160.9(7) | C(14)-C(15)-C(18A)-F(13A) | -124.7(9) |
| C(6)-C(5)-C(8A)-F(2A) | -144.1(7) | C(16)-C(15)-C(18A)-F(13A) | 54.8(11) |
| C(4)-C(5)-C(8A)-F(2A) | 35.7(9) | C(14)-C(15)-C(18A)-F(12A) | -5.8(11) |
| C(6)-C(5)-C(8A)-F(3A) | 98.4(8) | C(16)-C(15)-C(18A)-F(12A) | 173.6(6) |
| C(4)-C(5)-C(8A)-F(3A) | -81.8(9) | C(25)-C(20)-C(21)-C(22) | 0.2(4) |
| S(12)-S(11)-N(11)-C(11) | -0.6(2) | Sb(1)-C(20)-C(21)-C(22) | 176.6(2) |

| | | | |
|---|---|---|---|
| C(20)-C(21)-C(22)-C(23) | 0.3(4) | C(33)-C(34)-C(35)-C(30) | 1.3(4) |
| C(21)-C(22)-C(23)-C(24) | -0.4(4) | C(31)-C(30)-C(35)-C(34) | -0.8(4) |
| C(22)-C(23)-C(24)-C(25) | 0.2(4) | Sb(1)-C(30)-C(35)-C(34) | 178.9(2) |
| C(23)-C(24)-C(25)-C(20) | 0.2(4) | C(45)-C(40)-C(41)-C(42) | 1.1(4) |
| C(21)-C(20)-C(25)-C(24) | -0.4(4) | Sb(1)-C(40)-C(41)-C(42) | -176.8(2) |
| Sb(1)-C(20)-C(25)-C(24) | -177.1(2) | C(40)-C(41)-C(42)-C(43) | 0.4(4) |
| C(35)-C(30)-C(31)-C(32) | -0.3(4) | C(41)-C(42)-C(43)-C(44) | -0.9(4) |
| Sb(1)-C(30)-C(31)-C(32) | 180.0(2) | C(42)-C(43)-C(44)-C(45) | -0.2(4) |
| C(30)-C(31)-C(32)-C(33) | 0.9(4) | C(43)-C(44)-C(45)-C(40) | 1.9(4) |
| C(31)-C(32)-C(33)-C(34) | -0.4(4) | C(41)-C(40)-C(45)-C(44) | -2.3(4) |
| C(32)-C(33)-C(34)-C(35) | -0.6(4) | Sb(1)-C(40)-C(45)-C(44) | 175.69(19) |

Symmetry transformations used to generate equivalent atoms:

**Table S6G**. Short intermolecular contacts for **2** [Å and °].

| Atom 1 | Atom 2 | d(D...A) | s.u. |
|---|---|---|---|
| S1 | S11 | 3.0440 | (0.0011) |
| S2 | S12 | 3.0913 | (0.0010) |
| S1 | S11_$1 | 3.6072 | (0.0010) |
| S1 | S1_$1 | 3.6557 | (0.0015) |
| S11 | S1_$1 | 3.6072 | (0.0010) |
| S1 | C45 | 3.4580 | (0.0025) |
| S2 | C40 | 3.5842 | (0.0024) |
| S11 | C44 | 3.1678 | (0.0027) |
| S11 | C43 | 3.3601 | (0.0028) |
| S12 | C43 | 3.3883 | (0.0030) |
| S12 | C42 | 3.4632 | (0.0029) |
| H41 | C32_$2 | 2.9313 | |
| H22 | C43_$2 | 2.9146 | |
| H22 | C44_$2 | 2.995 | |

**Table S6H.** Least Squares Planes for **2**

Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane) 6.9407 (0.0068) x + 6.4892 (0.0075) y - 4.0782 (0.0099) z = 3.9650 (0.0072)

* 0.0039 (0.0015) C1

* 0.0053 (0.0013) N1

* -0.0108 (0.0013) N2

* -0.0082 (0.0009) S1

* 0.0098 (0.0008) S2

Rms deviation of fitted atoms = 0.0080

8.0998 (0.0065) x + 6.2630 (0.0080) y - 2.6676 (0.0108) z = 8.0750 (0.0072)

Angle to previous plane (with approximate esd) = 7.868 ( 0.131 )

* 0.0087 (0.0016) C11

* -0.0045 (0.0014) N11

* -0.0076 (0.0013) N12

* 0.0000 (0.0009) S11

* 0.0034 (0.0009) S12

Rms deviation of fitted atoms = 0.0058