Single crystal X-ray diffraction analysis of inclusion complexes of triflate-functionalized pillar[5]arenes with 1,4-dibromobutane and n-hexane guests

Mickey Vinodh, Shaima G. Alshammari and Talal F. Al-Azemi*

Table of contents

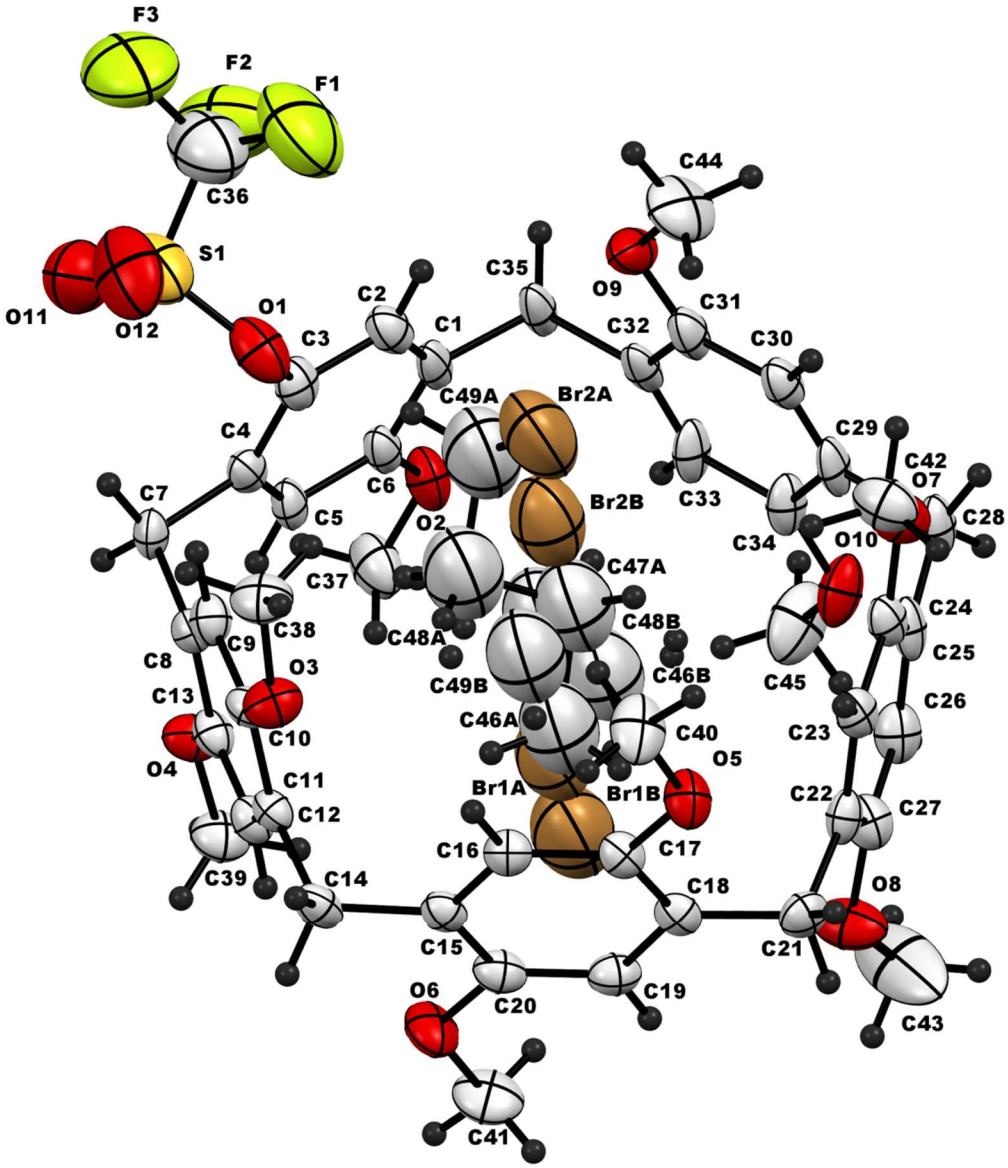

Figure S1: Thermal Ellipsoid representation (30%probability) of **Pil_TF1_ButBr2**

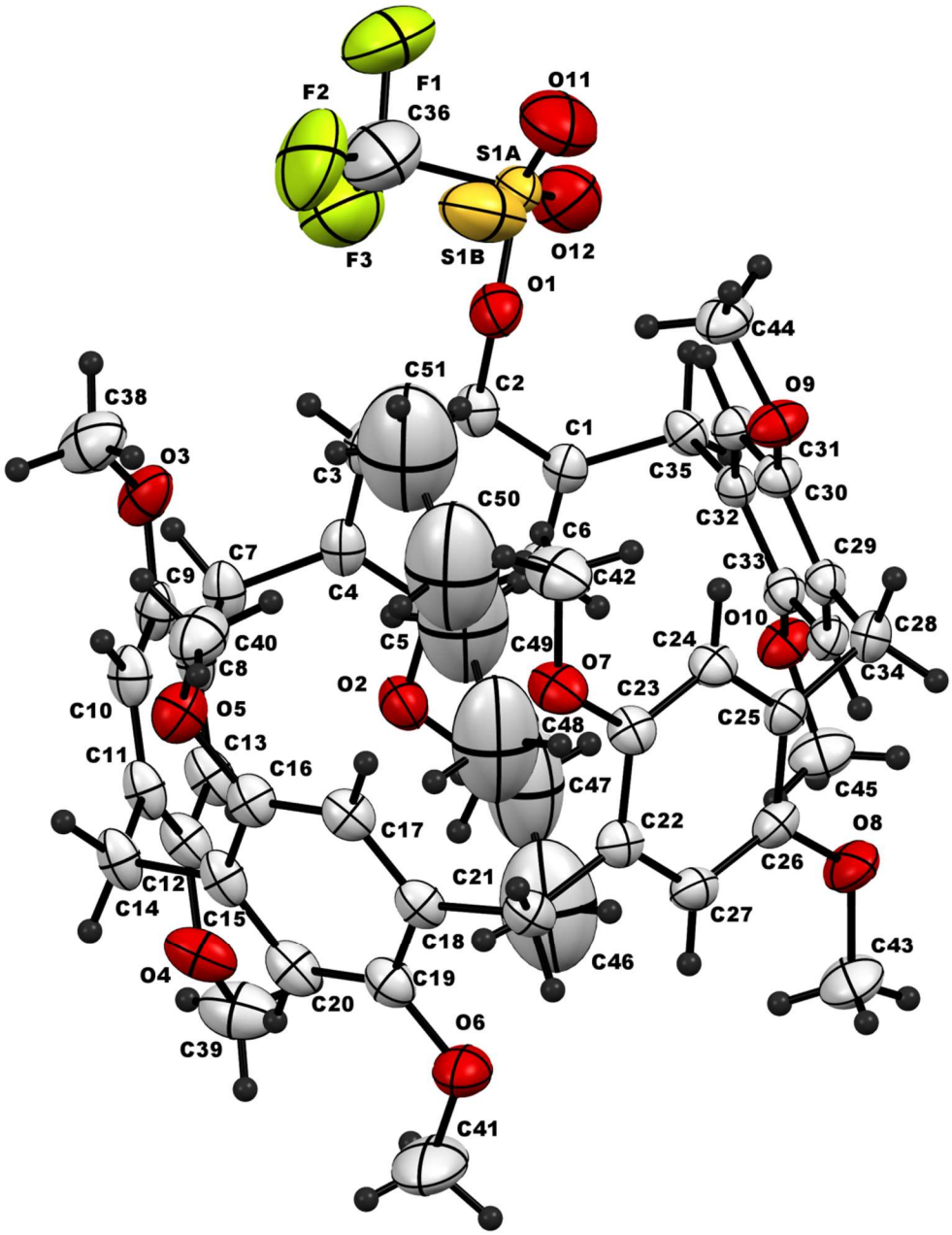

Figure S2: Thermal Ellipsoid representation (30%probability) of **Pil_TF1_Hex**

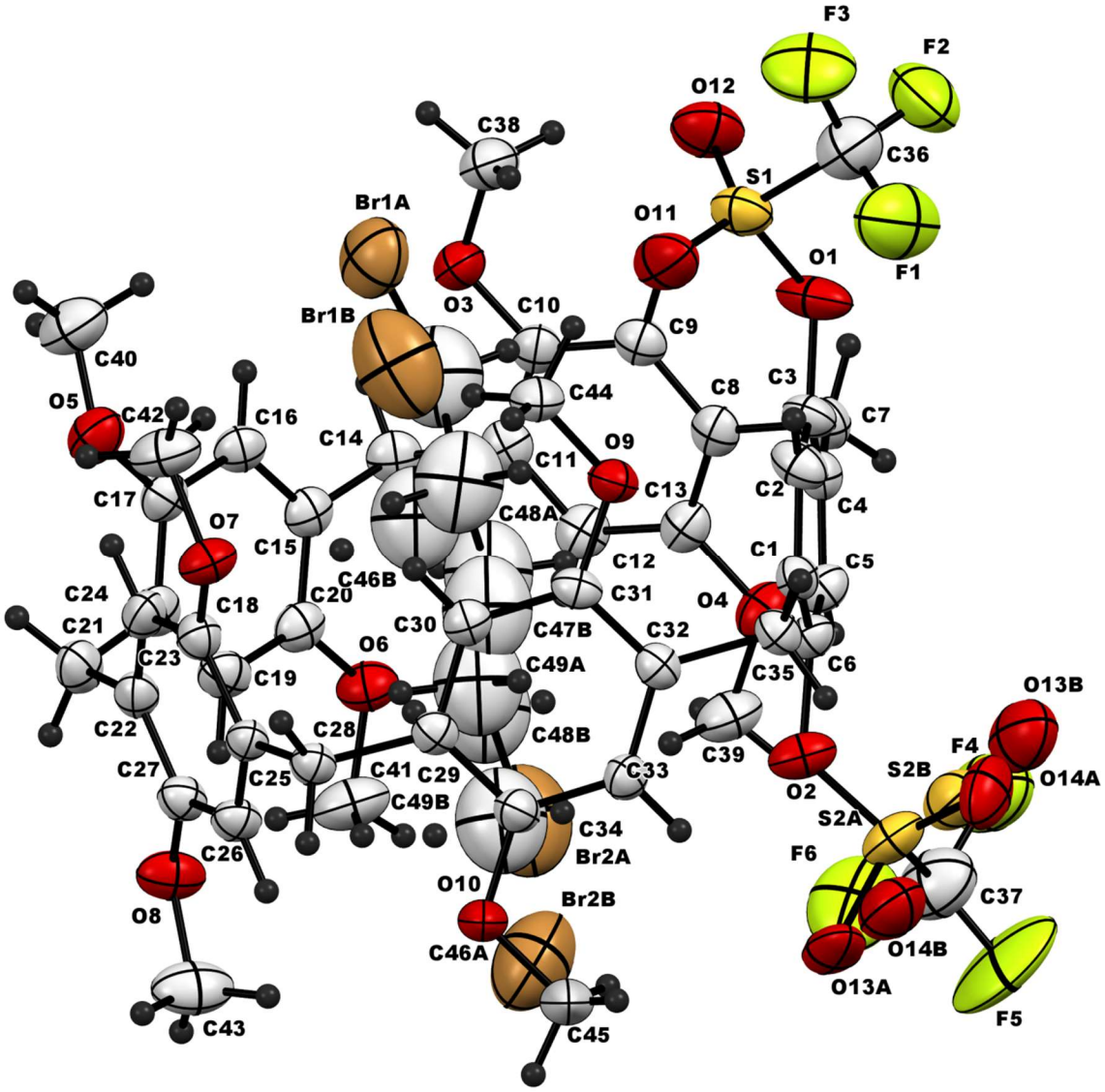

Figure S3: Thermal Ellipsoid representation (30%probability) of **Pil_TF2_ButBr2**

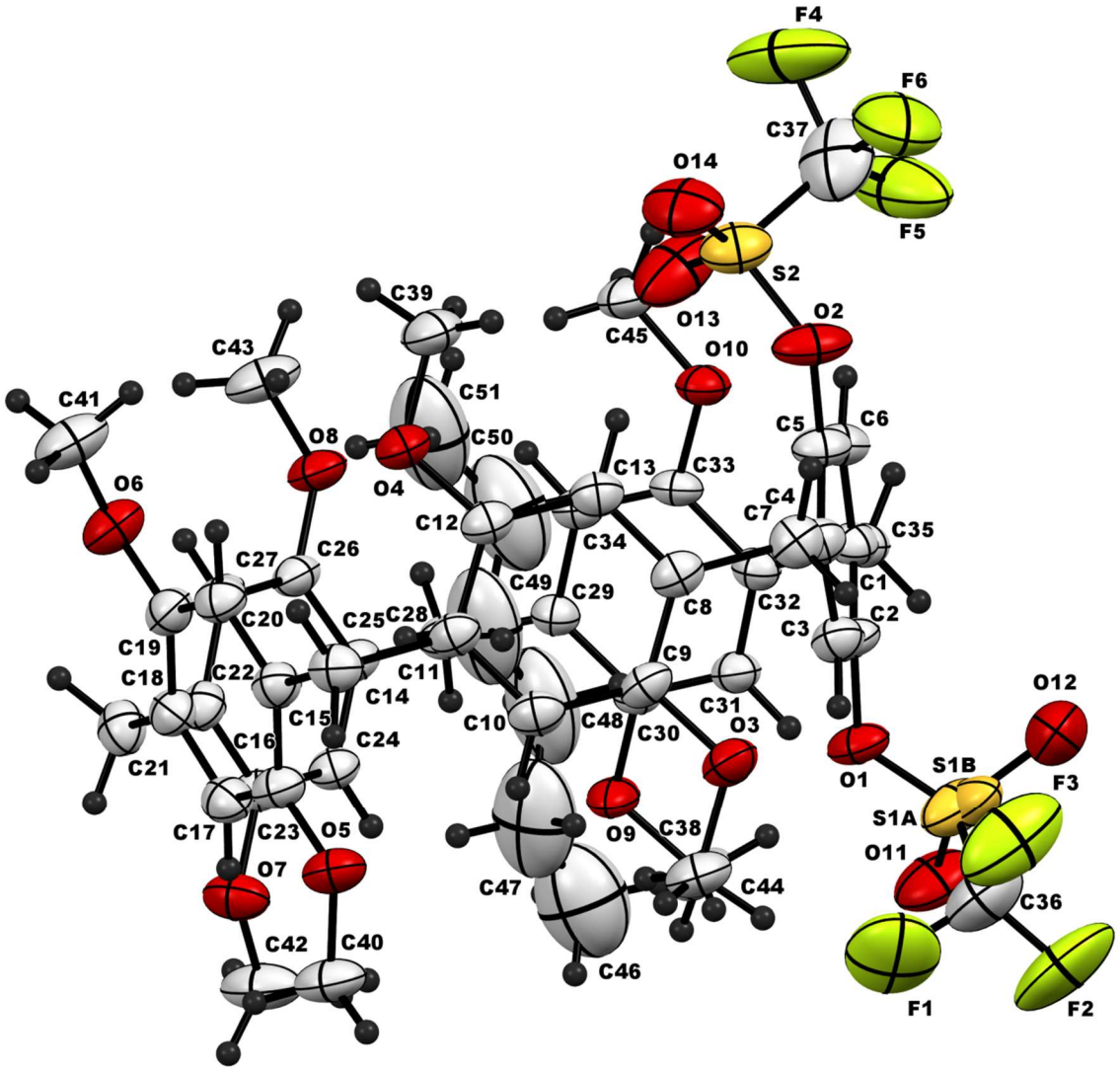

Figure S4: Thermal Ellipsoid representation (30%probability) of **Pil_TF2_Hex**

Table S1: Non-bonding interactions between Pillararene host and dibromobutane guest in the **Pil_TF2_ButBr2** crystals (Å, º).

A-B...C	A-B	B...C	B....C	A-B...C
C46A-H46A…O3	0.97	2.946	3.39(2)	109
C46A-H46B…O3	0.97	2.970	3.39(2)	108
C47A-H47A…O7	0.97	3.417	4.36(2)	164
C47A-H47B…O9	0.97	2.940	3.82(2)	151
C48A-H48A…*Cg*3	0.97	2.967	3.898	160.97
C48A-H48B…*Cg*2	0.97	3.402	3.654	121.00
C49A-H49A…*Cg*4	0.97	2.802	3.756	168.40
C49A-H49B…*Cg*5	0.97	2.743	3.648	155.59

*Cg*2 - *Cg*5 are the centroid of the pillararene phenyl rings constitute C8-C13, C15-C20, C22-27 and C29-C34 respectively

Table S2: Non-bonding interactions between Pillararene host and n-Hehane guest in the **Pil_TF2_Hex** crystals (Å, º).

A-B...C	A-B	B...C	B....C	A-B...C
C46-H46B…O9	0.96	3.201	3.91(2)	132
C46-H46C…O1	0.96	2.985	3.85(2)	150
C47-H47A…O5	0.97	3.455	4.10(2)	126
C47-H47B…O3	0.97	3.573	4.50(2)	162
C48-H48A…*Cg*4	0.97	3.209	3.981	137.73
C48-H48B…O1	0.97	2.959	3.84(2)	151.2
C49-H49A…*Cg*3	0.97	2.889	3.646	135.63
C49-H49B…*Cg*2	0.97	2.969	3.903	161.77
C50-H50A…O10	0.97	3.193	3.84(2)	125
C50-H50B… *Cg*1	0.97	3.163	4.049	152.59
C51-H51A…O13	0.96	2.898	3.55(2)	126.3
C51-H51B…O6	0.96	3.128	3.98(2)	149.0
C51-H51C…O4	0.96	2.925	3.88(2)	174.0

*Cg*1 - *Cg*4 are the centroid of the pillararene phenyl rings constitute C1-C6, C8-C13, C15-C20 and C22-27 respectively

Table S3: Non-bonding interactions between Pillararene host and dibromobutane guest in the **Pil_TF1_ButBr2** crystals (Å, º).

A-B...C	A-B	B...C	B....C	A-B...C
C46A-H46A…*Cg*2	0.97	3.212	4.076	149.30
C46A-H46B… *Cg*3	0.97	2.762	3.298	115.51
C47A-H47A… *Cg*5	0.97	2.865	3.743	151.38
C47A-H47B… *Cg*4	0.97	3.325	3.936	122.64
C48A-H48A…O3	0.97	3.012	3.88(4)	149
C48A-H48B… *Cg*1	0.97	2.957	3.656	129.71
C49A-H49A… O1	0.97	2.102	3.05(3)	164
C49A-H49B… O9	0.97	2.674	3.58(5)	156

*Cg*1 - *Cg*5 are the centroid of the pillararene phenyl rings constitute C1-C6, C8-C13, C15-C20, C22-27 and C29-C34 respectively

Table S4: Non-bonding interactions between Pillararene host and n-Hehane guest in the **Pil_TF2_Hex** crystals (Å, º).

A-B...C	A-B	B...C	B....C	A-B...C
C46-H46B…O8	0.96	3.263	4.18(3)	160
C46-H46C…O6	0.96	3.222	3.89(2)	128
C47-H47A…O10	0.97	3.116	4.07(2)	170
C47-H47B…O2	0.97	3.192	4.00(2)	142
C48-H48A…*Cg*3	0.97	2.815	3.711	154.17
C48-H48B… *Cg*4	0.97	2.781	3.730	165.59
C49-H49A…*Cg*5	0.97	3.201	4.031	144.55
C49-H49B…*Cg*2	0.97	3.092	3.959	149.63
C50-H50A…O9	0.97	3.465	4.26(3)	141
C50-H50B… O5	0.97	3.153	4.09(2)	162
C51-H51B…O1	0.96	2.895	3.73(2)	145
C51-H51C…O3	0.96	3.049	3.99(3)	167

*Cg*2- *Cg*5 are the centroid of the pillararene phenyl rings constitute C8-C13, C15-C20, C22-27 and C29-C34respectively.

Table S5: Intermolecular non-bonding interactions (shorter than the sum of van der Walls radii) in the **Pil_TF1_ButBr2** crystals (Å, º).

A-B...C	**A-B**	**B...C**	**B....C**	**A-B...C**
C36-F3...H43C^{i}	1.37(3)	2.62	3.48	119.0
C36-F3...H37B^{ii}	1.37(3)	2.73	3.52	114.0
C43-43C...F3^{iii}	0.96	2.62	3.37(4)	135.0
C37-H37B...F3^{iv}	0.96	2.73	3.08(2)	102.4

Symmetry code: (i) x, 1+y, 1+z; (ii) x, y, 1+z; (iii) x, -1+y, -1+z; (iv) x, y, -1+z

Table S6: Intermolecular non-bonding interactions (shorter than the sum of van der Walls radii) in the **Pil_TF1_Hexane** crystals (Å, º).

A-B...C	**A-B**	**B...C**	**B....C**	**A-B...C**
C37-H37C...F1^{i}	0.96	2.682	3.01(1)	100.4
C36-F1...H37C^{ii}	1.46(2)	2.682	3.53	114.0

Symmetry code: (i) x, y, -1+z; (ii) x, y, 1+z.

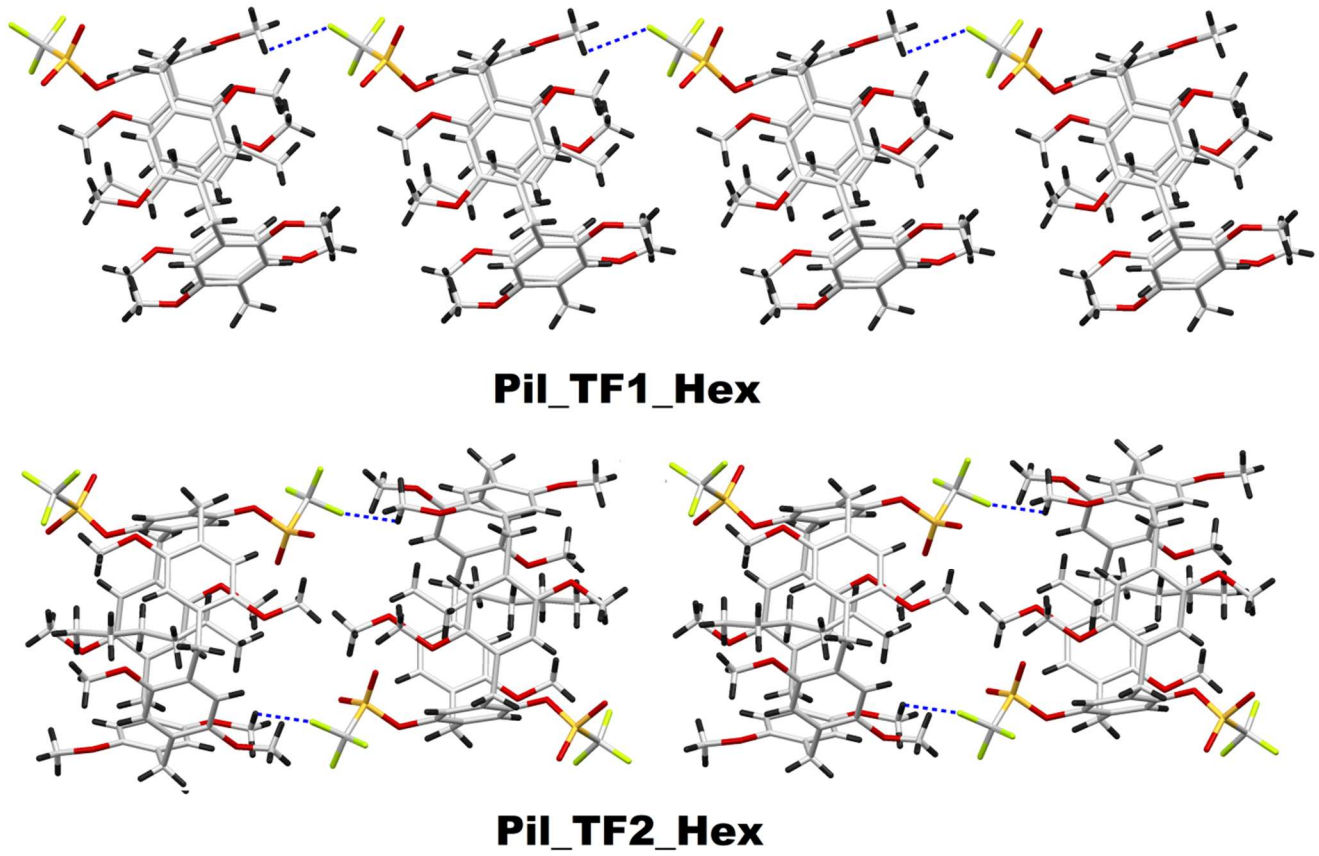

Figure S5. Crystal network showing linear pillararne chain of **Pil_TF1_Hex** as well as linear array constituting cofacial dimmeres of **Pil_TF2_Hex .** Both these arrangements are enabled by F...H interactions.

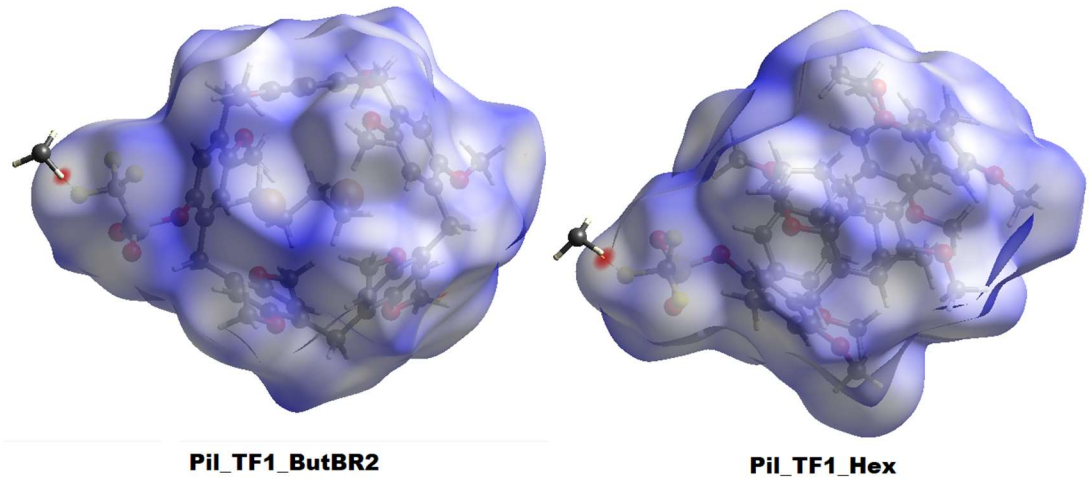

Figure S6. Hirshfeld surfaces (mapped with dnorm) of the **Pil_TF1_ButBr2** and **Pil_TF1_Hex** crystals.

CheckCIF of Pil_TF1_ButBr2

checkCIF (basic structural check) running

Checking for embedded fcf data in CIF ...
Found embedded fcf data in CIF. Extracting fcf data from uploaded CIF, please wait **. . . . .**

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) Pil_TF1_ButBr2

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No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report
Structure factor report

Datablock: Pil_TF1_ButBr2

```
Bond precision:      C-C = 0.0104 A              Wavelength=1.54178
Cell:          a=36.345(4)    b=12.1749(13)  c=11.4616(13)
               alpha=90       beta=90        gamma=90
Temperature:150 K
                    Calculated                      Reported
Volume              5071.7(10)                      5071.7(10)
Space group         P n a 21                        P n a 21
Hall group          P 2c -2n                        P 2c -2n
Moiety formula      C45 H47 F3 O12 S, C4 H8 Br2     ?
Sum formula         C49 H55 Br2 F3 O12 S            C49 H55 Br2 F3 O12 S
Mr                  1084.79                         1084.81
Dx,g cm-3           1.421                           1.421
Z                   4                               4
Mu (mm-1)           3.009                           3.009
F000                2232.0                          2232.0
F000'               2234.50
h,k,lmax            43,14,13                        42,14,13
Nref                9011[ 4753]                     7434
Tmin,Tmax           0.542,0.582                     0.570,0.630
```

```
Tmin'              0.491
Correction method= # Reported T Limits: Tmin=0.570
Tmax=0.630 AbsCorr = MULTI-SCAN
Data completeness= 1.56/0.82    Theta(max)= 66.841

R(reflections)= 0.1162( 6869)                wR2(reflections)=
                                             0.3285( 7434)

S = 1.512             Npar= 660
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

STRVA01_ALERT_4_C Flack test results are ambiguous.
From the CIF: _refine_ls_abs_structure_Flack 0.330
From the CIF: _refine_ls_abs_structure_Flack_su 0.080
PLAT082_ALERT_2_C High R1 Value ..... 0.12 Report
PLAT084_ALERT_3_C High wR2 Value (i.e. > 0.25) ..... 0.33 Report
PLAT090_ALERT_3_C Poor Data / Parameter Ratio (Zmax > 18) ..... 7.15 Note
PLAT213_ALERT_2_C Atom C43 has ADP max/min Ratio ..... 3.7 prolat
PLAT220_ALERT_2_C NonSolvent Resd 1 C Ueq(max)/Ueq(min) Range 5.1 Ratio
PLAT222_ALERT_3_C NonSolvent Resd 1 H Uiso(max)/Uiso(min) Range 5.7 Ratio
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of O8 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including Br1A 0.205 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including Br1B 0.206 Check
PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.0104 Ang.
PLAT601_ALERT_2_C Unit Cell Contains Solvent Accessible VOIDS of . 31 Ang**3
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.596 30 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 2 Check
PLAT977_ALERT_2_C Check Negative Difference Density on H49D . -0.39 eA-3
PLAT992_ALERT_5_C Repd & Actual _reflns_number_gt Values Differ by 23 Check

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Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 15 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 20 Report
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 3 Report
PLAT173_ALERT_4_G The CIF-Embedded .res File Contains DANG Records 3 Report
PLAT177_ALERT_4_G The CIF-Embedded .res File Contains DELU Records 2 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records 2 Report
PLAT188_ALERT_3_G A Non-default SIMU Restraint Value has been used 0.0100 Report

PLAT188_ALERT_3_G A Non-default SIMU Restraint Value has been used 0.0100 Report
PLAT242_ALERT_2_G Low 'MainMol' Ueq as Compared to Neighbors of C36 Check
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 2) 100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 3) 100% Note
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 2) 7.42 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 3) 6.58 Check
PLAT413_ALERT_2_G Short Inter XH3 .. XHn H45B ..H46C . 1.72 Ang.
x,y,z = 1_555 Check
PLAT413_ALERT_2_G Short Inter XH3 .. XHn H45B ..H46D . 2.13 Ang.
x,y,z = 1_555 Check
PLAT432_ALERT_2_G Short Inter X...Y Contact C2 ..C49A . 3.14 Ang.
x,y,z = 1_555 Check
And 6 other PLAT432 Alerts
More ...
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 244 Note
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still 68% Note
PLAT915_ALERT_3_G No Flack x Check Done: Low Friedel Pair Coverage 64 %
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File 2 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 2 Info

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0 **ALERT level A** = Most likely a serious problem - resolve or explain
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16 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
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1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
22 ALERT type 2 Indicator that the structure model may be wrong or deficient
11 ALERT type 3 Indicator that the structure quality may be low
9 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of*

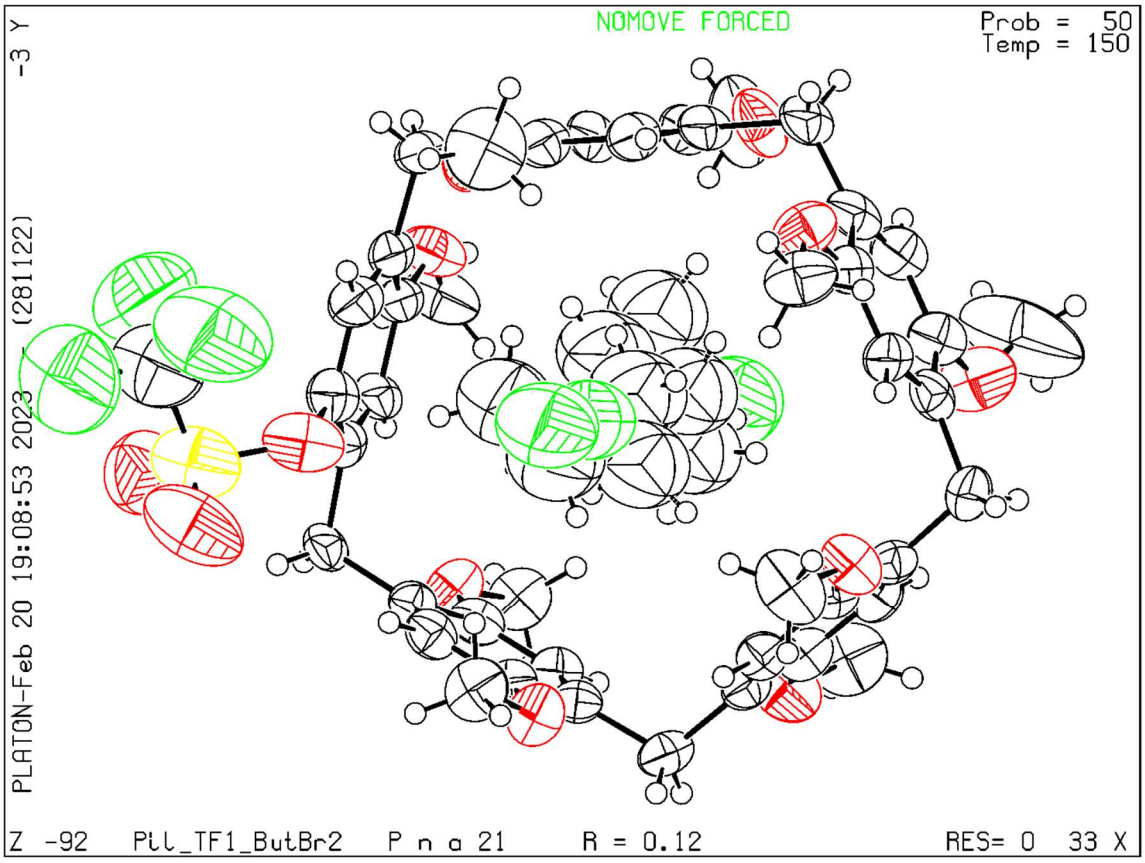
Synchrotron Radiation); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 28/11/2022; check.def file version of 28/11/2022

Datablock Pil_TF1_ButBr2 - ellipsoid plot



CheckCIF of Pil_TF1_Hex

checkCIF (basic structural check) running

Checking for embedded fcf data in CIF ...
Found embedded fcf data in CIF. Extracting fcf data from uploaded CIF, please wait **. . . . .**

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) Pil_TF1_Hex

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No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report
Structure factor report

Datablock: Pil_TF1_Hex

```
Bond precision:      C-C = 0.0088 A              Wavelength=1.54178
Cell:          a=36.5154(14)  b=12.1508(5)   c=11.4520(4)
               alpha=90       beta=90        gamma=90
Temperature:150 K
                    Calculated                    Reported
Volume              5081.2(3)                     5081.2(3)
Space group         P n a 21                      P n a 21
Hall group          P 2c -2n                      P 2c -2n
Moiety formula      C45 H47 F3 O12 S, C6 H14      ?
Sum formula         C51 H61 F3 O12 S              C51 H61 F3 O12 S
Mr                  955.06                        955.05
Dx,g cm-3           1.248                         1.248
Z                   4                             4
Mu (mm-1)           1.160                         1.160
F000                2024.0                        2024.0
F000'               2032.05
h,k,lmax            43,14,13                      43,14,13
Nref                8982[ 4739]                   7710
```

```
Tmin,Tmax          0.789,0.870                    0.790,0.840
Tmin'              0.784
Correction method= # Reported T Limits: Tmin=0.790
Tmax=0.840 AbsCorr = MULTI-SCAN
Data completeness= 1.63/0.86    Theta(max)= 66.617
R(reflections)= 0.0731( 7322)                  wR2(reflections)=
                                               0.2155( 7710)
S = 0.999             Npar= 615
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT220_ALERT_2_C NonSolvent Resd 1 C Ueq(max)/Ueq(min) Range 4.0 Ratio
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of O11 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of O1 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of O6 Check
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor .... 3.3 Note
PLAT260_ALERT_2_C Large Average Ueq of Residue Including C46 0.280 Check
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00882 Ang.
PLAT601_ALERT_2_C Unit Cell Contains Solvent Accessible VOIDS of . 42 Ang**3
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.595 7 Report

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Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 11 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 15 Report
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 1 Report
PLAT173_ALERT_4_G The CIF-Embedded .res File Contains DANG Records 1 Report
PLAT176_ALERT_4_G The CIF-Embedded .res File Contains SADI Records 3 Report
PLAT177_ALERT_4_G The CIF-Embedded .res File Contains DELU Records 3 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records 3 Report
PLAT188_ALERT_3_G A Non-default SIMU Restraint Value has been used 0.0100 Report
And 2 other PLAT188 Alerts
More ...
PLAT192_ALERT_3_G A Non-default DELU Restraint Value for First Par 0.0050 Report
PLAT230_ALERT_2_G Hirshfeld Test Diff for S1A --O1 . 6.7 s.u.
And 5 other PLAT230 Alerts
More ...
PLAT301_ALERT_3_G Main Residue Disorder .....(Resd 1) 2% Note

PLAT395_ALERT_2_G Deviating X-O-Y Angle From 120 for O1 . 22.3 Degree
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 127 Note
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still 86% Note
PLAT915_ALERT_3_G No Flack x Check Done: Low Friedel Pair Coverage 70 %
PLAT965_ALERT_2_G The SHELXL WEIGHT Optimisation has not Converged Please Check
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 2 Info

---

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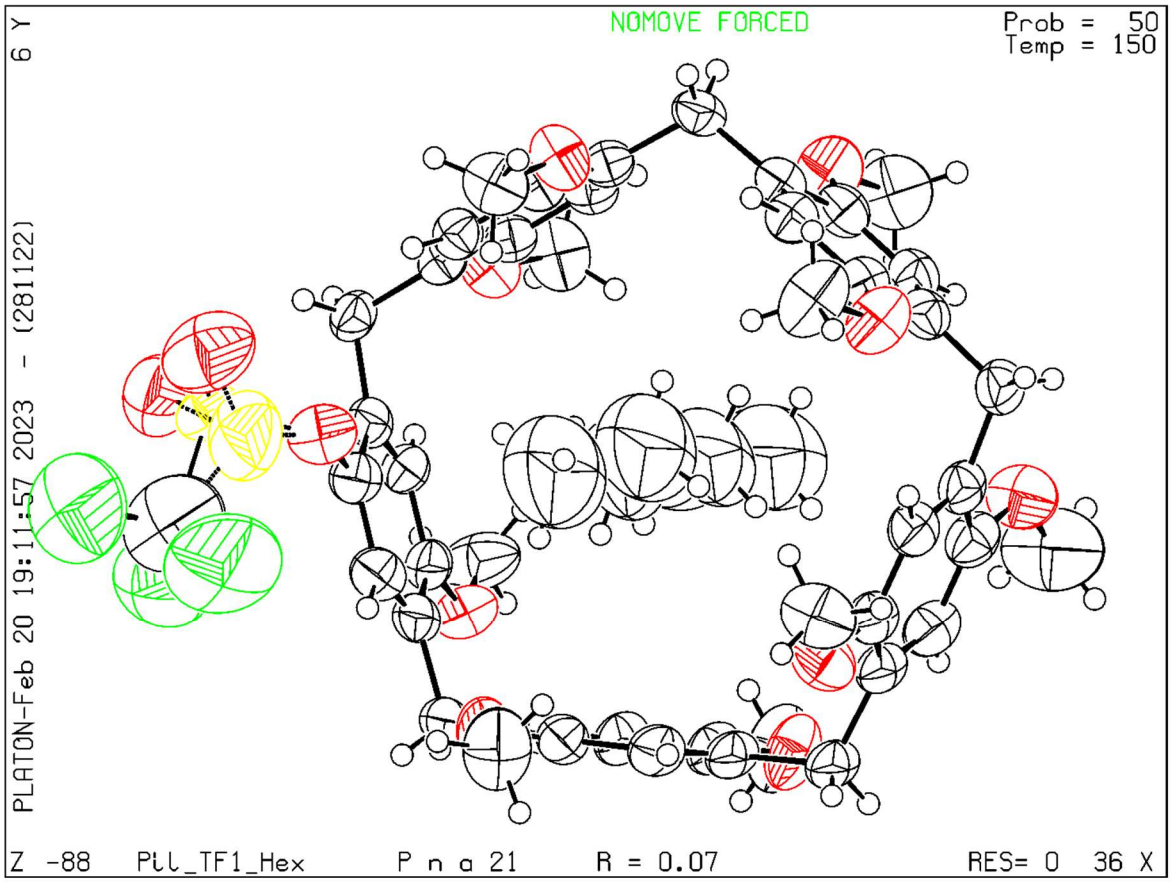
PLATON version of 28/11/2022; check.def file version of 28/11/2022

Datablock Pil_TF1_Hex - ellipsoid plot


NOMOVE FORCED
Prob = 50
Temp = 150
6 Y
PLATON-Feb 20 19:11:57 2023 - (281122)
Z -88 Pll_TF1_Hex P n a 21 R = 0.07 RES= 0 36 X

CheckCIF of Pil_TF2_ButBr2

checkCIF (basic structural check) running

Checking for embedded fcf data in CIF ...
Found embedded fcf data in CIF. Extracting fcf data from uploaded CIF, please wait **. . . .**

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) Pil_TF2_ButBr2

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No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report
Structure factor report

Datablock: Pil_TF2_ButBr2

```
Bond precision:      C-C = 0.0075 A              Wavelength=0.71075
Cell:          a=18.4426(10)  b=18.580(1)    c=15.8047(7)
               alpha=90       beta=98.962(7) gamma=90
Temperature:150 K
                    Calculated                      Reported
Volume              5349.6(5)                       5349.6(5)
Space group         P 21/c                          P 21/c
Hall group          -P 2ybc                         -P 2ybc
Moiety formula      C45 H44 F6 O14 S2, C4 H8 Br2    ?
Sum formula         C49 H52 Br2 F6 O14 S2           C49 H52 Br2 F6 O14 S2
Mr                  1202.83                         1202.84
Dx,g cm-3           1.493                           1.493
Z                   4                               4
Mu (mm-1)           1.677                           1.677
F000                2456.0                          2456.0
F000'               2456.15
h,k,lmax            21,22,18                        21,22,18
Nref                9456                            9339
Tmin,Tmax           0.722,0.765                     0.720,0.790
```

```
Tmin'              0.708
Correction method= # Reported T Limits: Tmin=0.720
Tmax=0.790 AbsCorr = MULTI-SCAN
Data completeness= 0.988       Theta(max)= 25.040
R(reflections)= 0.0947( 5489)                wR2(reflections)=
                                             0.3210( 9339)
S = 1.040            Npar= 741
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT910_ALERT_3_B Missing # of FCF Reflection(s) Below Theta(Min). 18 Note

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Alert level C

PLAT084_ALERT_3_C High wR2 Value (i.e. > 0.25) ..... 0.32 Report
PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density .... 2.16 Report
PLAT213_ALERT_2_C Atom F5 has ADP max/min Ratio ..... 3.2 oblate
PLAT220_ALERT_2_C NonSolvent Resd 1 C Ueq(max)/Ueq(min) Range 3.5 Ratio
PLAT230_ALERT_2_C Hirshfeld Test Diff for S1 --C36 . 5.3 s.u.
PLAT234_ALERT_4_C Large Hirshfeld Difference S2A --C37 . 0.16 Ang.
PLAT234_ALERT_4_C Large Hirshfeld Difference F5 --C37 . 0.19 Ang.
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of F4 Check
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of O1 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of S1 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of O10 Check
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor .... 2.2 Note
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor .... 2.1 Note
PLAT260_ALERT_2_C Large Average Ueq of Residue Including Br1A 0.191 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including Br1B 0.206 Check
PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.0075 Ang.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 7.601 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 2.129 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.595 98 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 2 Check

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Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 18 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 21 Report
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 3 Report
PLAT173_ALERT_4_G The CIF-Embedded .res File Contains DANG Records 2 Report

PLAT176_ALERT_4_G The CIF-Embedded .res File Contains SADI Records 2 Report
PLAT177_ALERT_4_G The CIF-Embedded .res File Contains DELU Records 2 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records 2 Report
PLAT188_ALERT_3_G A Non-default SIMU Restraint Value has been used 0.0100 Report
PLAT188_ALERT_3_G A Non-default SIMU Restraint Value has been used 0.0100 Report
PLAT230_ALERT_2_G Hirshfeld Test Diff for S2A --O2 . 6.3 s.u.
And 2 other PLAT230 Alerts
More ...
PLAT242_ALERT_2_G Low 'MainMol' Ueq as Compared to Neighbors of C36 Check
PLAT242_ALERT_2_G Low 'MainMol' Ueq as Compared to Neighbors of C37 Check
PLAT301_ALERT_3_G Main Residue Disorder .....(Resd 1) 4% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 2) 100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 3) 100% Note
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 2) 8.50 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 3) 5.50 Check
PLAT395_ALERT_2_G Deviating X-O-Y Angle From 120 for O2 . 27.9 Degree
PLAT395_ALERT_2_G Deviating X-O-Y Angle From 120 for O13B . 92.0 Degree
PLAT434_ALERT_2_G Short Inter HL..HL Contact F2 ..F5 . 2.75 Ang.
-x,1-y,1-z = 3_566 Check
PLAT721_ALERT_1_G Bond Calc 0.96000, Rep 0.97000 Dev... 0.01 Ang.
C48B -H48C 1_555 1_555 ..... # 27 Check
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle(s) in CIF ... 38.50 Deg.
O13B -S2B -F4 1_555 1_555 1_555 ..... # 89 Check
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle(s) in CIF ... 44.30 Deg.
C37 -S2B -F4 1_555 1_555 1_555 ..... # 91 Check
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 287 Note
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..... 4.0 Low
PLAT965_ALERT_2_G The SHELXL WEIGHT Optimisation has not Converged Please Check
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 3 Info

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0 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **ALERT level B** = A potentially serious problem, consider carefully
20 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
30 **ALERT level G** = General information/check it is not something unexpected

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
24 ALERT type 2 Indicator that the structure model may be wrong or deficient
12 ALERT type 3 Indicator that the structure quality may be low
13 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

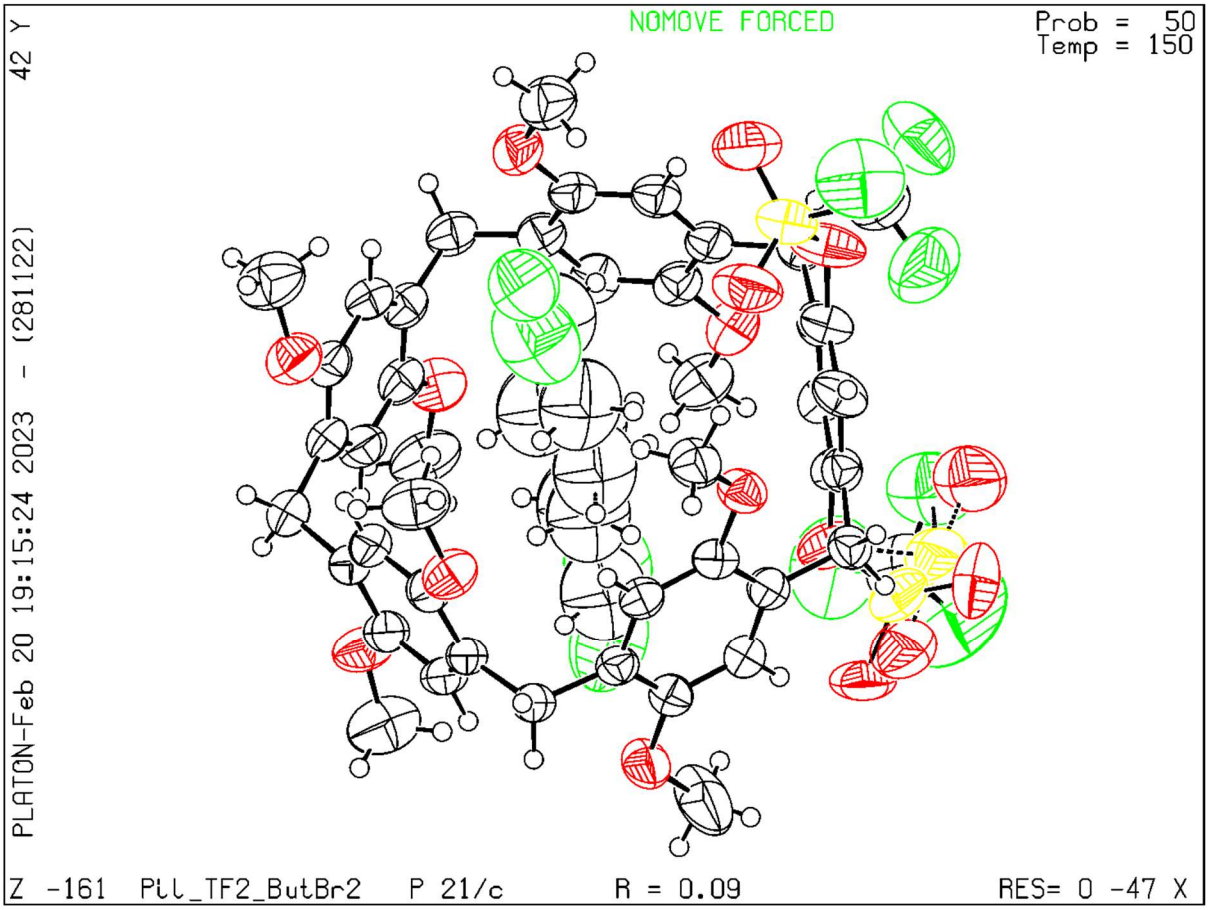
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 28/11/2022; check.def file version of 28/11/2022

Datablock Pil_TF2_ButBr2 - ellipsoid plot


NOMOVE FORCED
Prob = 50
Temp = 150
PLATON-Feb 20 19:15:24 2023 - (281122)
42 Y
Z -161 Pll_TF2_ButBr2 P 21/c R = 0.09
RES= 0 -47 X

CheckCIF of Pil_TF2_Hex

checkCIF (basic structural check) running

Checking for embedded fcf data in CIF ...
Found embedded fcf data in CIF. Extracting fcf data from uploaded CIF, please wait **. . . .**

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) Pil_TF2_Hex

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No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report
Structure factor report

Datablock: Pil_TF2_Hex

```
Bond precision:      C-C = 0.0093 A              Wavelength=1.54178
Cell:          a=18.1057(13)  b=18.7444(14)  c=15.8021(11)
               alpha=90       beta=99.305(4) gamma=90
Temperature: 150 K
                    Calculated                      Reported
Volume              5292.4(7)                       5292.4(7)
Space group         P 21/c                          P 21/c
Hall group          -P 2ybc                         -P 2ybc
Moiety formula      C45 H44 F6 O14 S2, C6 H14       ?
Sum formula         C51 H58 F6 O14 S2               C51 H58 F6 O14 S2
Mr                  1073.09                         1073.09
Dx,g cm-3           1.347                           1.347
Z                   4                               4
Mu (mm-1)           1.648                           1.648
F000                2248.0                          2248.0
F000'               2258.65
h,k,lmax            21,22,18                        21,22,18
Nref                9354                            8995
Tmin,Tmax           0.743,0.756                     0.710,0.780
```

```
Tmin'              0.674
Correction method= # Reported T Limits: Tmin=0.710
Tmax=0.780 AbsCorr = MULTI-SCAN
Data completeness= 0.962       Theta(max)= 66.588
R(reflections)= 0.0985( 4711)                wR2(reflections)=
                                             0.3482( 8995)
S = 1.037            Npar= 676
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT029_ALERT_3_C _diffrn_measured_fraction_theta_full value Low . 0.962 Why?
PLAT084_ALERT_3_C High wR2 Value (i.e. > 0.25) ..... 0.35 Report
PLAT230_ALERT_2_C Hirshfeld Test Diff for S2 --C37 . 5.4 s.u.
PLAT234_ALERT_4_C Large Hirshfeld Difference S1B --C36 . 0.16 Ang.
And 4 other PLAT234 Alerts
More ...
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of O2 Check
And 2 other PLAT241 Alerts
More ...
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of S2 Check
And 2 other PLAT242 Alerts
More ...
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor .... 3.1 Note
PLAT260_ALERT_2_C Large Average Ueq of Residue Including C46 0.233 Check
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00927 Ang.
PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C47 - C48 . 1.43 Ang.
PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C49 - C50 . 1.42 Ang.
PLAT905_ALERT_3_C Negative K value in the Analysis of Variance ... -7.785 Report
PLAT905_ALERT_3_C Negative K value in the Analysis of Variance ... -0.052 Report
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.595 358
Report

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 10 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 10 Report
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large 5.18
Why ?
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 1
Report
PLAT173_ALERT_4_G The CIF-Embedded .res File Contains DANG Records 1
Report
PLAT176_ALERT_4_G The CIF-Embedded .res File Contains SADI Records 1
Report
PLAT177_ALERT_4_G The CIF-Embedded .res File Contains DELU Records 2
Report

PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records 2 Report
PLAT188_ALERT_3_G A Non-default SIMU Restraint Value has been used 0.0200 Report
PLAT188_ALERT_3_G A Non-default SIMU Restraint Value has been used 0.0100 Report
PLAT230_ALERT_2_G Hirshfeld Test Diff for S1B --O11 . 15.4 s.u.
And 2 other PLAT230 Alerts
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PLAT242_ALERT_2_G Low 'MainMol' Ueq as Compared to Neighbors of C37 Check
PLAT301_ALERT_3_G Main Residue Disorder .....(Resd 1) 1% Note
PLAT395_ALERT_2_G Deviating X-O-Y Angle From 120 for O1 . 6.5 Degree
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 89 Note
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 1 Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File 3 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..... 4.5 Low
PLAT965_ALERT_2_G The SHELXL WEIGHT Optimisation has not Converged Please Check
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 2 Info

---

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PLATON version of 28/11/2022; check.def file version of 28/11/2022

Datablock Pil_TF2_Hex - ellipsoid plot