Electronic Supplementary Material (ESI) for CrystEngComm.
This journal is © The Royal Society of Chemistry 2024

Electronic Supplementary Information

The C–I···O halogen bonding in crown ether chemistry

Weizhou Wang* and Baoming Ji*

College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang 471934, P. R. China

E-mail: wzw@lynu.edu.cn; lyhxxjbm@126.com

Contents

S1. Methods

S1.1. Cocrystal synthesis

S1.2. Single-crystal X-ray diffraction

S1.3. Computational details

S2. The crystallographic data and structure refinement parameters for the thirteen cocrystals

S3. The I···O distance, ratio of the I···O distance to the sum of the van der Waals radii of I and O, and C–I···O angle for the thirteen cocrystals

S4. The unit cells for the thirteen cocrystals

S5. The bond paths and bond critical points for the dimers in Fig. 4

S6. References

S7. The CheckCIF reports for the thirteen cocrystals

S1. Methods

S1.1. Cocrystal synthesis

The 12-crown-4 (purity ≥98%), 15-crown-5 (purity ≥98%), 18-crown-6 (purity ≥98%), 1,2-diiodotetrafluorobenzene (purity ≥98%), 1,3-diiodotetrafluorobenzene (purity ≥98%), 1,4-diiodotetrafluorobenzene (purity ≥98%), 1,3,5-trifluoro-2,4,6-triiodobenzene (purity ≥98%), and the solvent *n*-heptane (analytical reagent grade) were purchased from J&K Scientific Ltd. in China. Unless otherwise stated, all of them were used as received. The synthetic procedure of each cocrystal is almost the same. Using *n*-heptane as solvent, we prepared the solutions of binary mixtures of **C4**, **C5** or **C6** with **I12**, **I13**, **I14**, and **I135**, respectively, in 1:1 molar ratio by gently stirring in the air at room temperature. After a few days, single crystals of thirteen cocrystals suitable for single-crystal X-ray diffraction analyses were successfully synthesized by slowly evaporating these solutions also in the air and at room temperature.

S1.2. Single-crystal X-ray diffraction

Single-crystal X-ray diffraction data were gathered on the Bruker D8 or Oxford Diffraction SuperNova area-detector diffractometer equipped with the Mo-Kα X-ray source (λ = 0.71073 Å). The data reduction was treated by using CrysAlisPro software.[1,2] The crystal structure was solved with the SHELXS-97/SHELXS-2014 program.[3-5] The H atoms of **C4**, **C5** and **C6** were refined at idealized positions riding on the C atoms, with isotropic displacement parameters U_{iso}(H) = 1.2U_{eq}(C) and *d*(C–H) = 0.97 Å. The H atoms of the H_2O molecules were refined at idealized positions riding on the O atoms, with isotropic displacement parameters U_{iso}(H) = 1.2U_{eq}(O) and *d*(O–H) = 0.85 Å. In the [**C4**][**I13**]$_2$, I1, I1A and I1B are disordered over three sites with occupancies 0.871:0.042:0.087. In the [**C4**][**I135**], I1 and I1A are disordered over two sites with occupancies 0.921:0.079. In the [**C5**][**I12**], C13~C16, O5 and C13'~C16', O5' are disordered over two sites with occupancies 0.648:0.352. In the [**C5**][**I14**], C1~C6, F1~F4, I1~I2 and C1A~C6A, F1A~F4A, I1A~I2A are disordered over two sites with occupancies 0.418:0.582, and C7~C12, F5~F8, I3~I4 and C7A~C12A, F5A~F8A, I3A~I4A are disordered over two sites with occupancies 0.687:0.313. In the [**C5**][**I135**]$_2$, C1~C12,

F1~F6, I1~I6 and C1A~C12A, F1A~F6A, I1A~I6A are disordered over two sites with occupancies 0.535:0.465, and C1~C6, O2~O3 and C1A~C6A, O2A~O3A are disordered over two sites with occupancies 0.982:0.108. Crystallographic data of the thirteen cocrystals were listed in the next section. The CIF files of the thirteen cocrystals (CCDC deposition numbers: 2281927-2281938, 2379639) can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html. At the same time, the CIF files of the thirteen cocrystals were also provided as the electronic supplementary materials. The checkcif files for the thirteen cocrystal structures can be found in the Supplementary Materials.

S1.3. Computational details

All the density functional theory (DFT) calculations were carried out at the PBE0-D3(BJ)/def2-TZVPP level of theory with the Gaussian 09 program.[6-10] Previous studies on the noncovalent interactions have shown that the PBE0-D3(BJ)/def2-TZVPP calculations can give comparable results with the "golden standard" coupled cluster calculations.[11,12] It is well known that the accuracy of the DFT calculations also depends on the number of points used in the numerical integration. An "ultrafine" integration grid (99 radial, 590 angular points) was used for all the DFT calculations to avoid the possible integration grid errors.

To further confirm the existence of the motifs **M1**, **M2**, **M3** and **M4**, the "atoms in molecules" (AIM) analysis has been performed with the PBE0-D3(BJ)/def2-TZVPP electron density.[13] The AIM2000 software was employed to carry out the AIM analysis.[14]

S2. The crystallographic data and structure refinement parameters for the thirteen cocrystals.

Cocrystal	**[C4][I12]$_2$**	**[C4][I13]$_2$**	**[C4][I14]$_2$**
CCDC No.	2281927	2281935	2281928
Empirical formula	$C_{20}H_{16}F_8I_4O_4$	$C_{10}H_8F_4I_2O_2$	$C_{10}H_8F_4I_2O_2$
Formula weight	979.93	489.96	489.96
Crystal size/mm^3	0.30 × 0.30 × 0.10	0.18 × 0.15 × 0.14	0.18 × 0.15 × 0.11
Crystal system	triclinic	monoclinic	monoclinic
Space group	*P*-1	$P2_1/c$	$P2_1$
a/Å	8.1008(7)	12.0376(4)	7.5395(6)
b/Å	8.2512(6)	15.4508(8)	8.6077(7)
c/Å	10.7542(8)	7.5142(3)	12.2413(10)
α/°	76.846(6)	90	94.007(7)
β/°	78.005(7)	95.120(3)	103.778(7)
γ/°	89.406(6)	90	113.576(7)
V/Å^3	684.17(9)	1391.99(10)	694.90(10)
Z	1	4	2
ρ_{calc}/g·cm^{-3}	2.378	2.338	2.342
T/K	295(2)	293(2)	293(2)
2ϑ range for data collection/°	5.800–50.980	6.796–56.608	7.440–52.494
Reflections collected	4527	8105	5112
Independent reflections [R_{int}]	2524 [0.0258]	2945 [0.0561]	2833 [0.0317]
R_1, wR_2 (I > 2σ(I))	0.0377, 0.0685	0.0439, 0.0858	0.0410, 0.0819
R_1, wR_2 (all data)	0.0534, 0.0776	0.0615, 0.0949	0.0609, 0.0966
Goodness-of-fit on F^2	1.042	1.060	1.056

Cocrystal	**[C4][I135]**	**[C5][I12]**	**[C5][W]$_2$[I13]**
CCDC No.	2281934	2281931	2281938
Empirical formula	$C_{14}H_{16}F_3I_3O_4$	$C_{16}H_{20}F_4I_2O_5$	$C_{16}H_{24}F_4I_2O_7$
Formula weight	685.97	622.12	658.15
Crystal size/mm^3	0.23 × 0.17 × 0.16	0.40 × 0.30 × 0.10	0.45 × 0.35 × 0.29
Crystal system	monoclinic	monoclinic	monoclinic
Space group	$P2_1/m$	$P2_1/c$	$P2_1/c$
a/Å	4.6368(5)	12.3775(18)	7.4810(5)
b/Å	29.139(4)	7.6176(10)	22.1452(19)
c/Å	7.3508(6)	22.623(3)	14.6860(11)
α/°	90	90	90
β/°	97.411(9)	100.287(13)	103.285(7)
γ/°	90	90	90
V/Å^3	984.9(2)	2098.8(5)	2367.9(3)
Z	2	4	4
ρ_{calc}/g·cm^{-3}	2.313	1.969	1.846
T/K	293(2)	295(2)	293(2)
2ϑ range for data collection/°	6.988–58.632	6.300–51.000	6.698–51.000
Reflections collected	4437	8868	12945
Independent reflections [R_{int}]	2308 [0.0335]	3884 [0.1102]	4376 [0.0468]
R_1, wR_2 (I > 2σ(I))	0.0531, 0.0957	0.0878, 0.2460	0.0594, 0.1128
R_1, wR_2 (all data)	0.0800, 0.1101	0.1262, 0.3317	0.0865, 0.1279
Goodness-of-fit on F^2	1.078	1.128	1.024

Cocrystal	**[C5][I14]**	**[C5][I135]$_2$**	**[C6][W]$_4$[I12]$_2$**
CCDC No.	2281933	2281936	2281929
Empirical formula	$C_{32}H_{40}F_8I_4O_{10}$	$C_{22}H_{19}F_6I_6O_5$	$C_{24}H_{32}F_8I_4O_{10}$
Formula weight	1244.24	1238.77	1140.10
Crystal size/mm^3	0.21 × 0.17 × 0.12	0.33 × 0.27 × 0.25	0.40 × 0.40 × 0.20
Crystal system	triclinic	monoclinic	triclinic
Space group	*P*-1	*C*c	*P*-1
a/Å	9.7355(6)	16.1779(9)	7.6327(5)
b/Å	12.4217(7)	8.4631(4)	8.0627(6)
c/Å	18.1787(11)	25.3518(15)	15.0373(12)
α/°	88.582(5)	90	88.107(6)
β/°	80.631(5)	105.819(6)	77.223(6)
γ/°	78.136(5)	90	89.966(6)
V/Å^3	2122.6(2)	3339.6(3)	901.97(12)
Z	2	4	1
ρ_{calc}/g·cm^{-3}	1.947	2.464	2.099
T/K	296(2)	295(2)	295(2)
2*ϑ* range for data collection/°	6.992–52.208	6.682–49.994	6.660–51.000
Reflections collected	17211	9499	5889
Independent reflections [R_{int}]	7872 [0.0383]	5483 [0.0272]	3348 [0.0312]
R_1, wR_2 (I > 2σ(I))	0.0607, 0.1542	0.0388, 0.0902	0.0419, 0.0885
R_1, wR_2 (all data)	0.0930, 0.1778	0.0432, 0.0949	0.0601, 0.0957
Goodness-of-fit on F^2	1.033	1.050	1.110

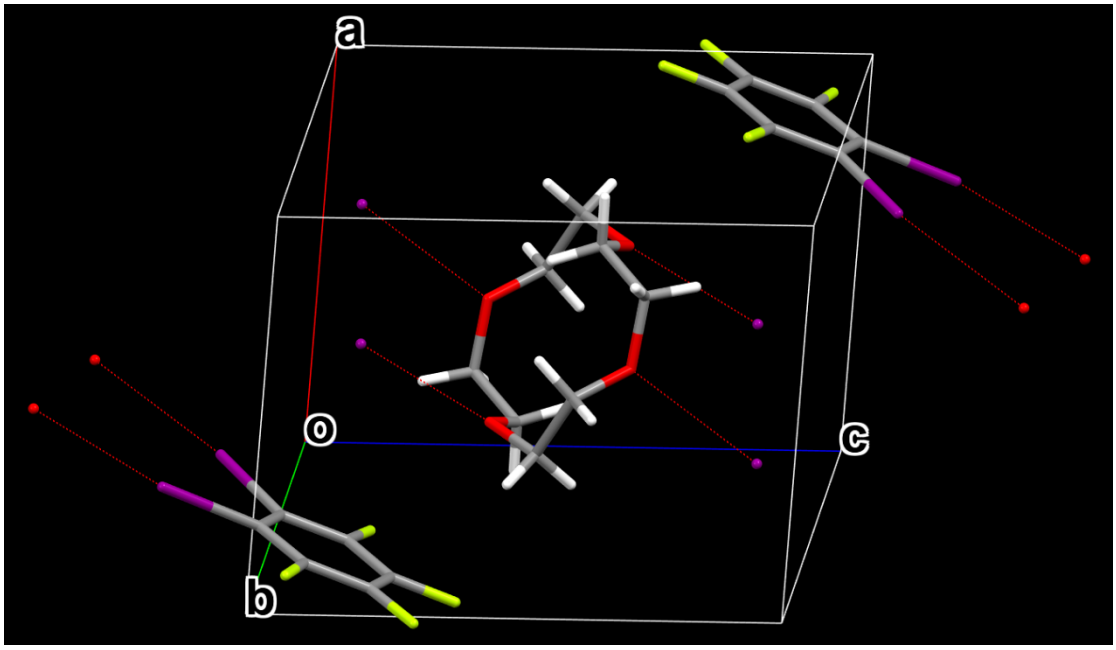
Cocrystal	**[C6][I13]$_2$**	**[C6][W]$_3$[I14]**	**[C6][I14]**	**[C6][I135]$_2$**
CCDC No.	2281932	2281930	2379639	2281937
Empirical formula	$C_{12}H_{12}F_4I_2O_3$	$C_{18}H_{30}F_4I_2O_9$	$C_{18}H_{24}F_4I_2O_6$	$C_{24}H_{24}F_6I_6O_6$
Formula weight	534.02	720.22	666.17	1283.87
Crystal size/mm^3	0.41 × 0.35 × 0.24	0.23 × 0.15 × 0.11	0.35 × 0.30 × 0.21	0.35 × 0.29 × 0.21
Crystal system	orthorhombic	triclinic	monoclinic	monoclinic
Space group	*P*bca	*P*-1	*I*2/a	$P2_1$/c
a/Å	7.8540(6)	7.5332(5)	14.2455(5)	8.5263(6)
b/Å	15.3678(14)	13.1800(8)	10.6548(5)	25.6556(16)
c/Å	27.2763(16)	14.1357(8)	15.5033(5)	8.8944(5)
α/°	90	105.855(5)	90	90
β/°	90	91.408(5)	94.012(3)	115.244(8)
γ/°	90	103.061(5)	90	90
V/Å^3	3292.2(4)	1309.58(14)	2347.37(16)	1759.8(2)
Z	8	2	4	2
ρ_{calc}/g·cm^{-3}	2.155	1.826	1.885	2.423
T/K	293(2)	295(2)	296(2)	293(2)
2ϑ range for data collection/°	6.940–51.000	6.520–144.720	6.964–58.464	6.380–51.000
Reflections collected	9490	9667	12761	8707
Independent reflections [R_{int}]	3056 [0.0343]	5033 [0.0511]	2841 [0.0321]	3266 [0.0435]
R_1, wR_2 (I > 2σ(I))	0.0490, 0.0987	0.0572, 0.1499	0.0268, 0.0524	0.0629, 0.1510
R_1, wR_2 (all data)	0.0665, 0.1059	0.0677, 0.1642	0.0317, 0.0544	0.0815, 0.1642
Goodness-of-fit on F^2	1.133	1.026	1.059	1.078

S3. The I···O distance (Å), ratio of the I···O distance to the sum of the van der Waals radii of I and O, and C–I···O angle (°) for the thirteen cocrystals.

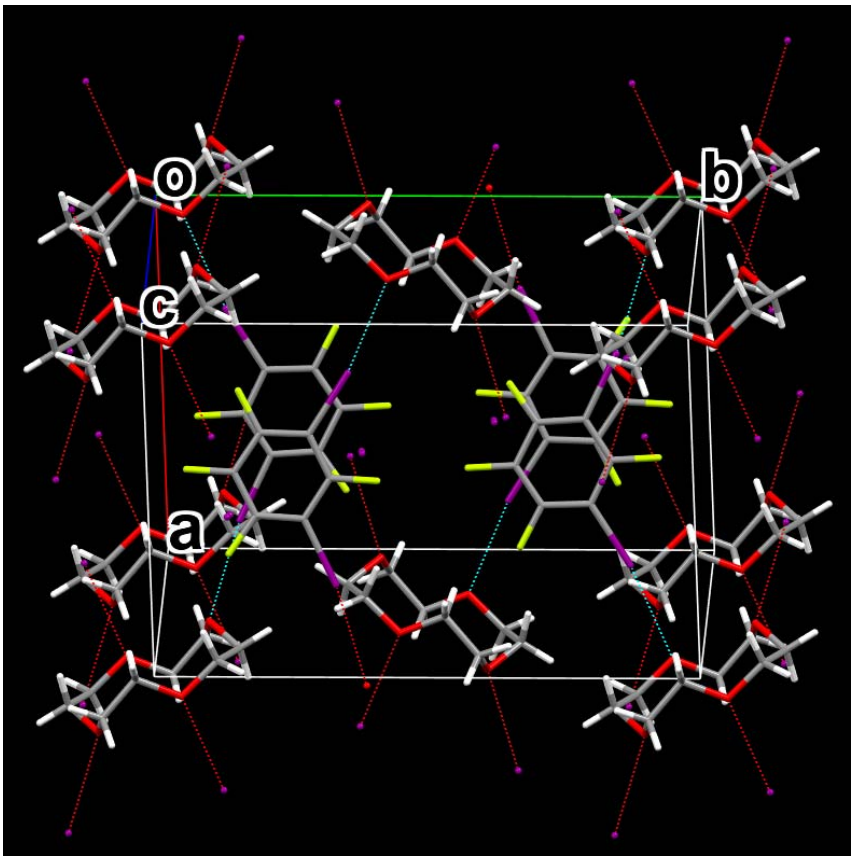
Cocrystal	I···O distance	Ratio	C–I···O angle
[C4][I12]$_2$	**M1**: 3.013(4)	0.86	171.5(2)
	M1: 3.130(4)	0.89	159.0(2)
[C4][I13]$_2$	**M1:** 2.992(5)	0.85	169.2(2)
	M1: 3.001(5)	0.86	169.2(2)
[C4][I14]$_2$	**M1:** 2.987(4)	0.85	167.8(2)
	M1: 3.001(4)	0.86	172.6(2)
[C4][I135]	**M1:** 2.926(6)	0.84	175.2(3)
[**C5**][**I12**]	**M1:** 2.909(9)	0.83	172.0(4)
	M1: 2.918(1)	0.83	178.9(4)
[**C5**][**W**]$_2$[**I13]**	**M4:** 2.843(6)	0.81	176.0(3)
	M4: 2.885(7)	0.82	174.9(3)
[**C5**][**I14]**	**M1:** 2.955(7)	0.84	171.2(3)
	M1: 3.036(1)	0.87	168.2(3)
	M2: 3.188(7)	0.91	158.1(3)
	M2: 3.308(7)	0.95	150.8(3)
	M3: 2.919(7)	0.83	175.0(3)
[C5][I135]$_2$	**M1:** 2.878(1)	0.82	175.7(4)
	M1: 3.024(2)	0.86	175.8(4)
	M3: 3.127(1)	0.89	170.3(4)
[**C6**][**W**]$_4$[**I12**]$_2$	**M4:** 2.897(4)	0.83	174.0(2)
	M4: 3.266(5)	0.93	154.1(2)
[C6][I13]$_2$	**M1:** 2.940(4)	0.84	173.0(2)
	M1: 3.141(4)	0.90	174.5(2)
[**C6**][**I14]**	**M1:** 3.062(2)	0.87	169.3(7)
[**C6**][**W**]$_3$[**I14]**	**M1:** 2.964(6)	0.85	177.4(2)
	M4: 2.897(5)	0.83	176.9(2)
[C6][I135]$_2$	**M1:** 3.000(7)	0.85	175.2(3)
	M3: 3.003(7)	0.86	168.5(3)

S4. The unit cells for the thirteen cocrystals. Color code: H, white; C, gray; O, red; F, yellow green; I, purple.

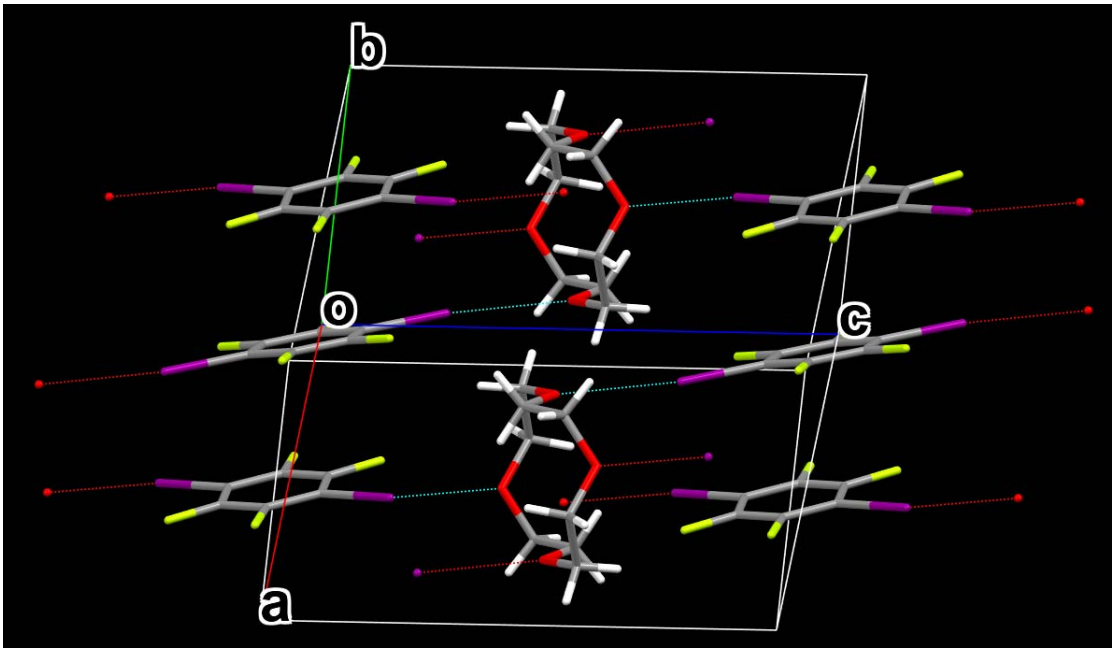
$[C4][I12]_2$



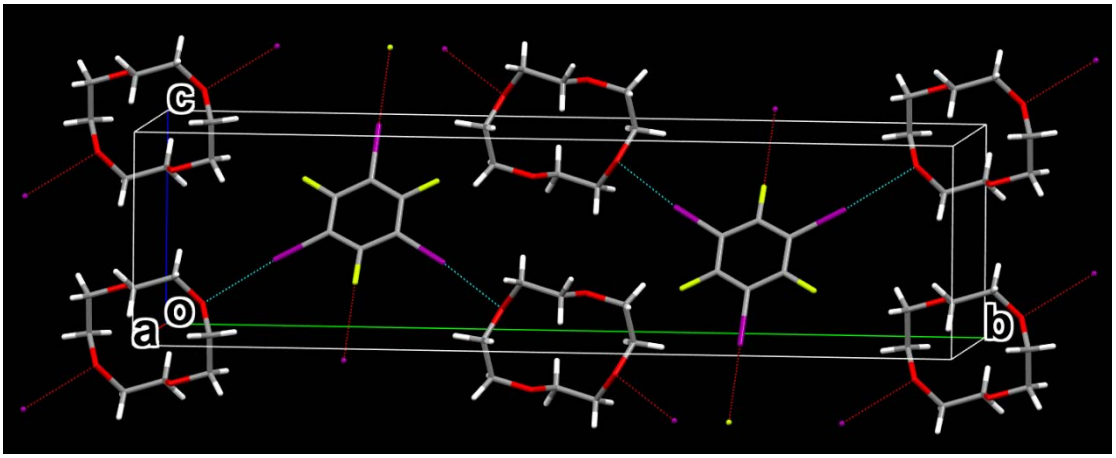
$[C4][I13]_2$



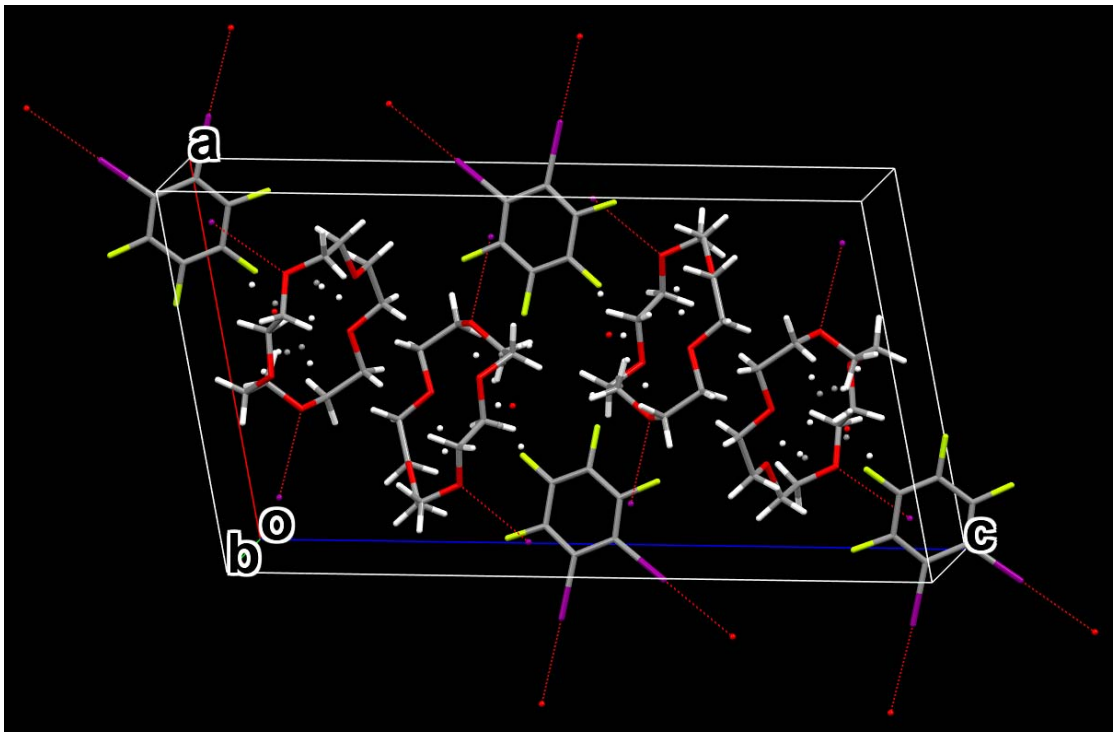
[C4][I14]$_2$



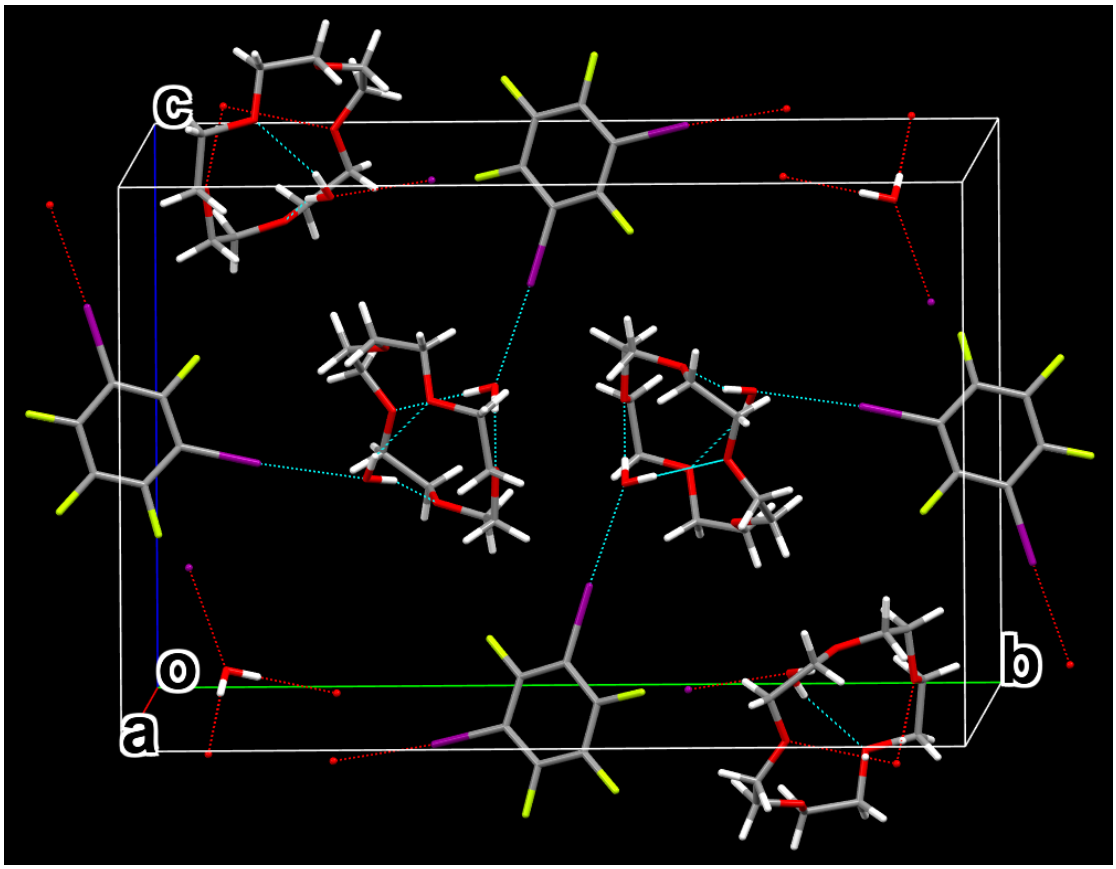
[C4][I135]



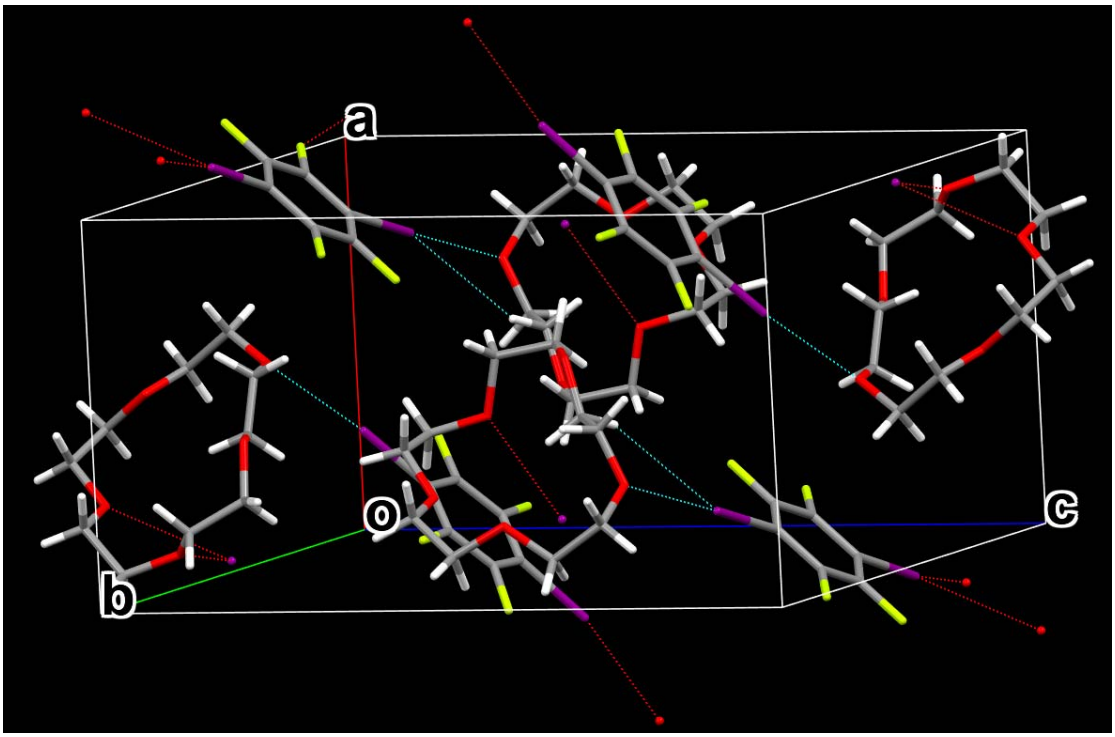
[C5][I12]



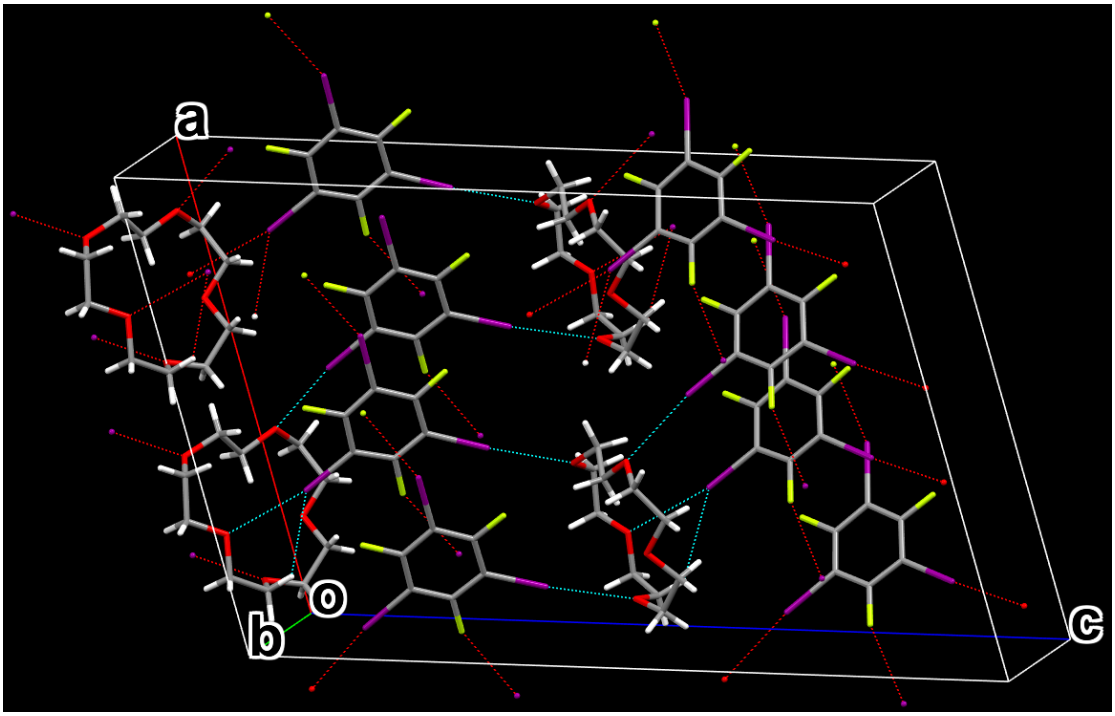
[C5][W]$_2$[I13]



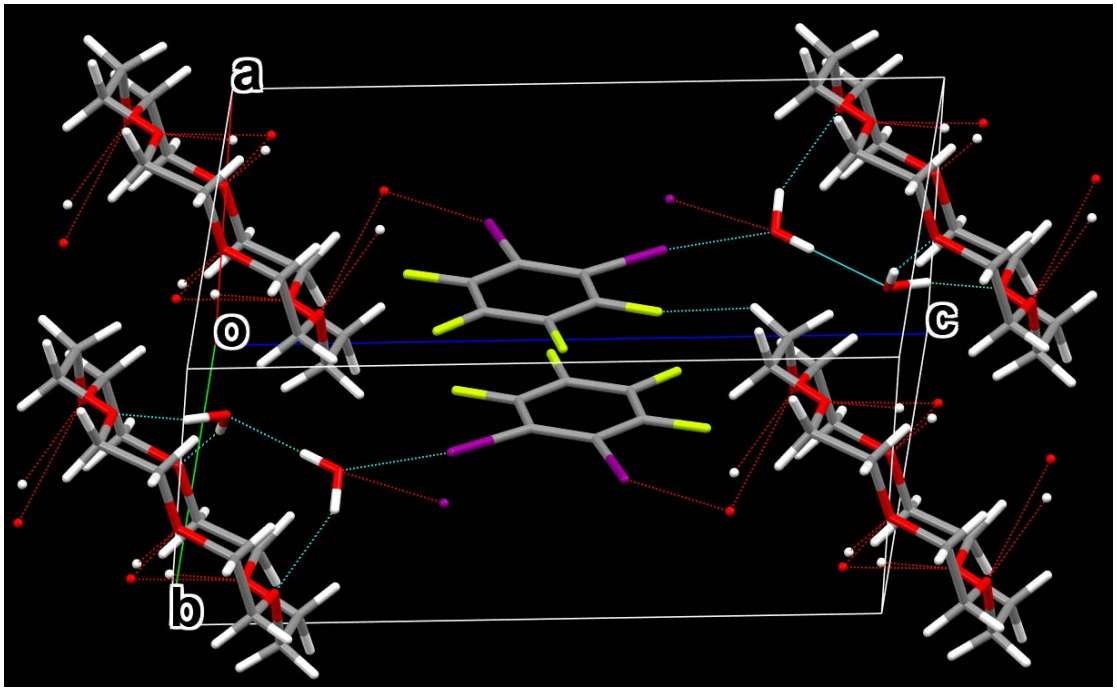
[C5][I14]



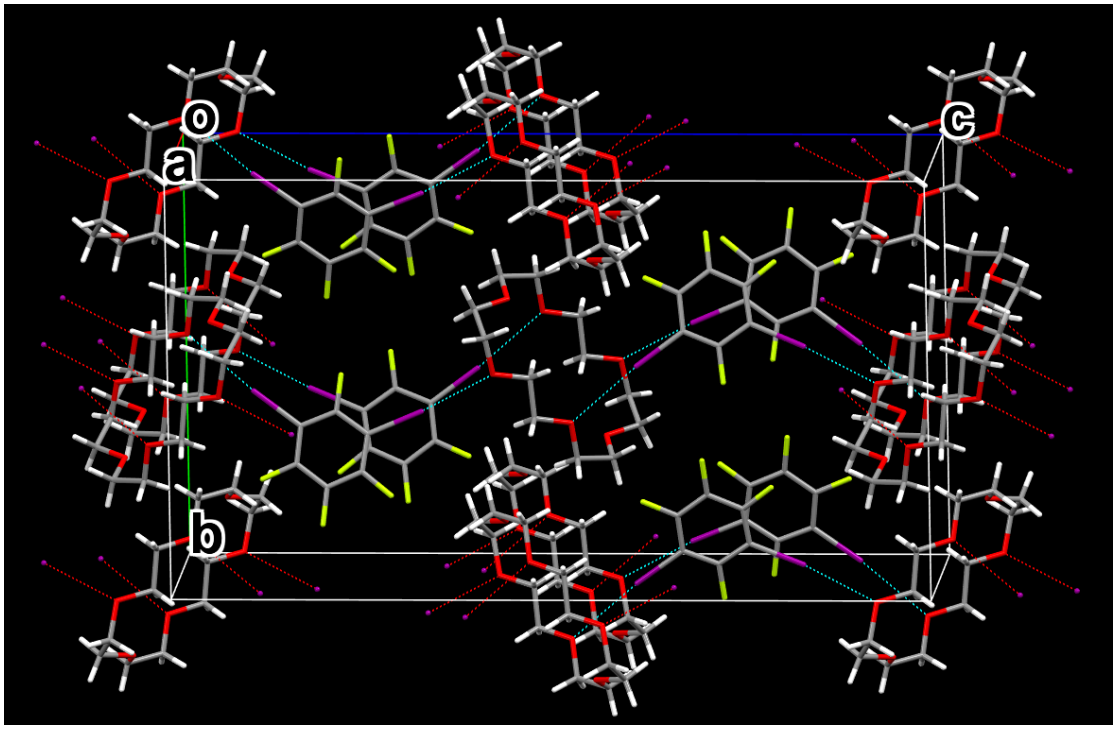
[C5][I135]$_2$



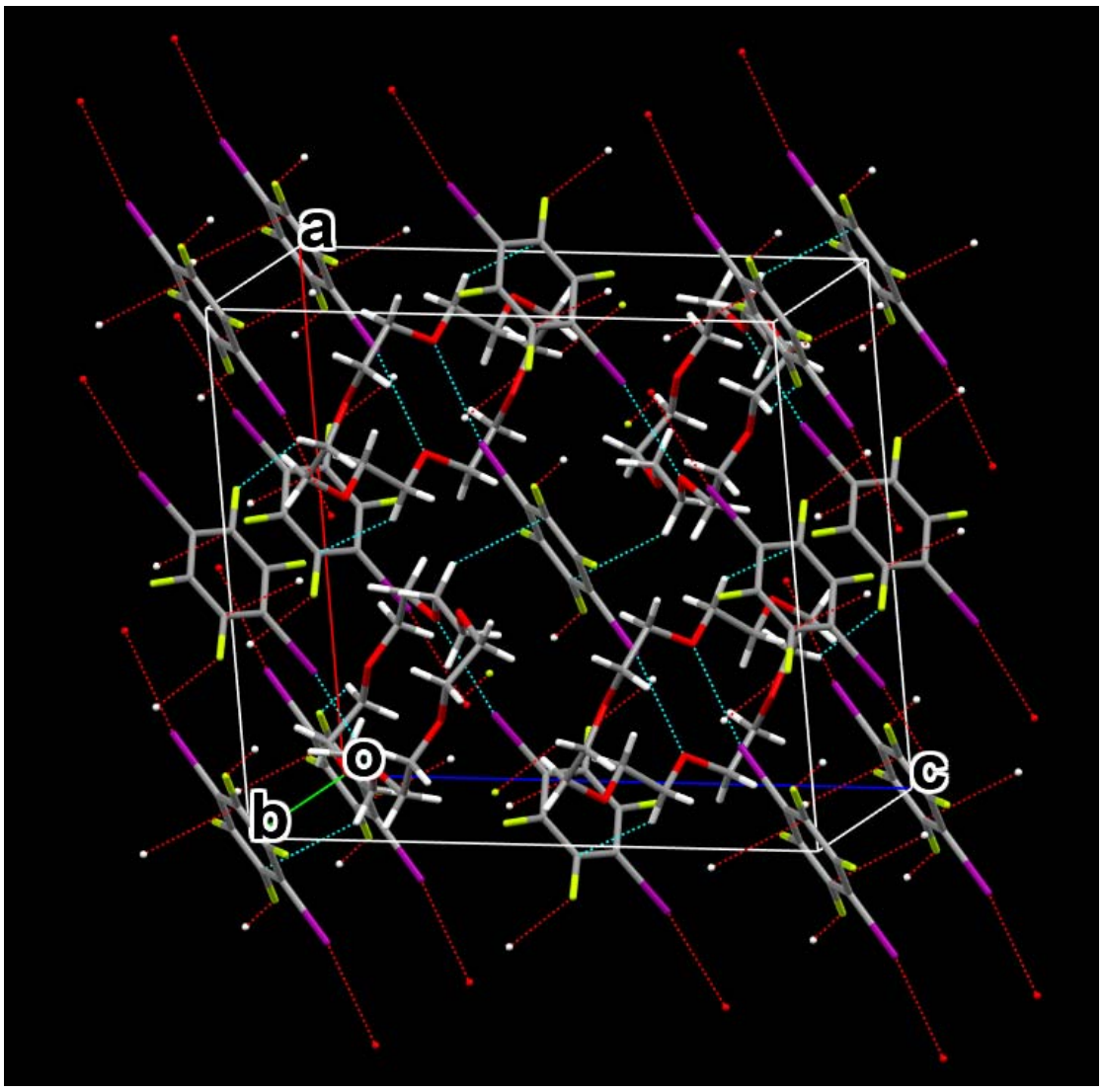
[C6][W]$_4$[I12]$_2$



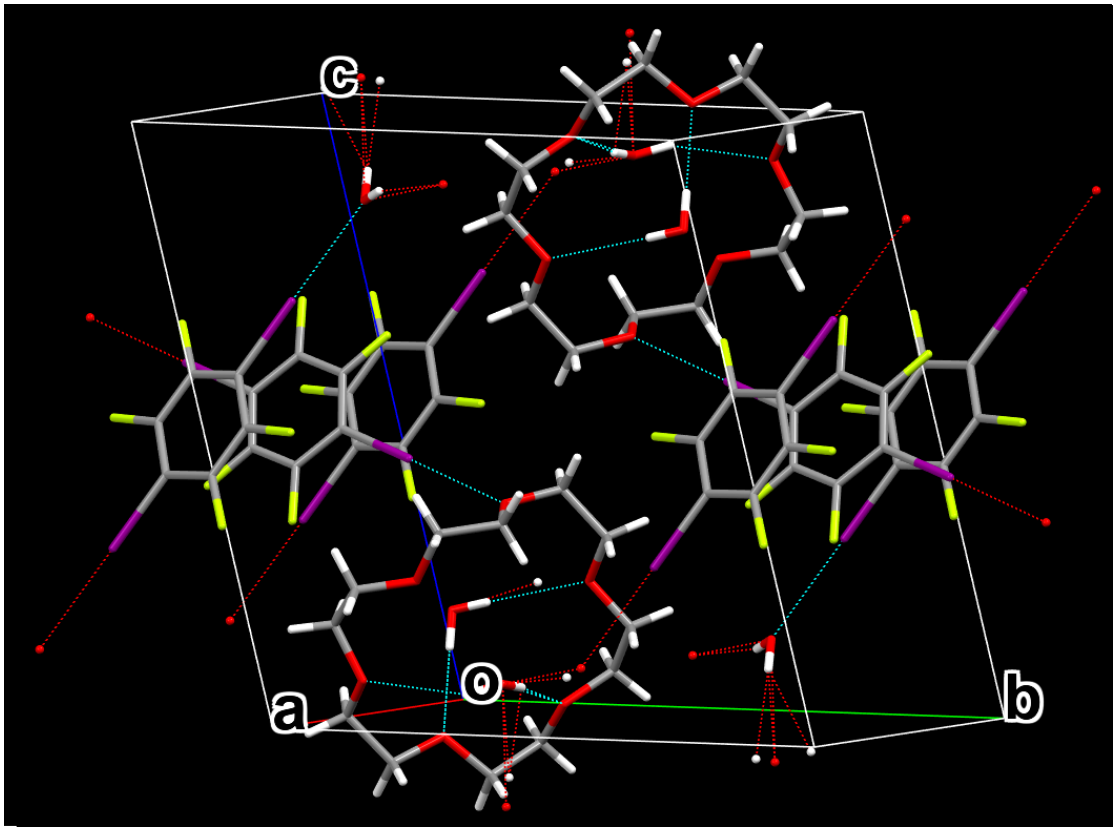
[C6][I13]$_2$



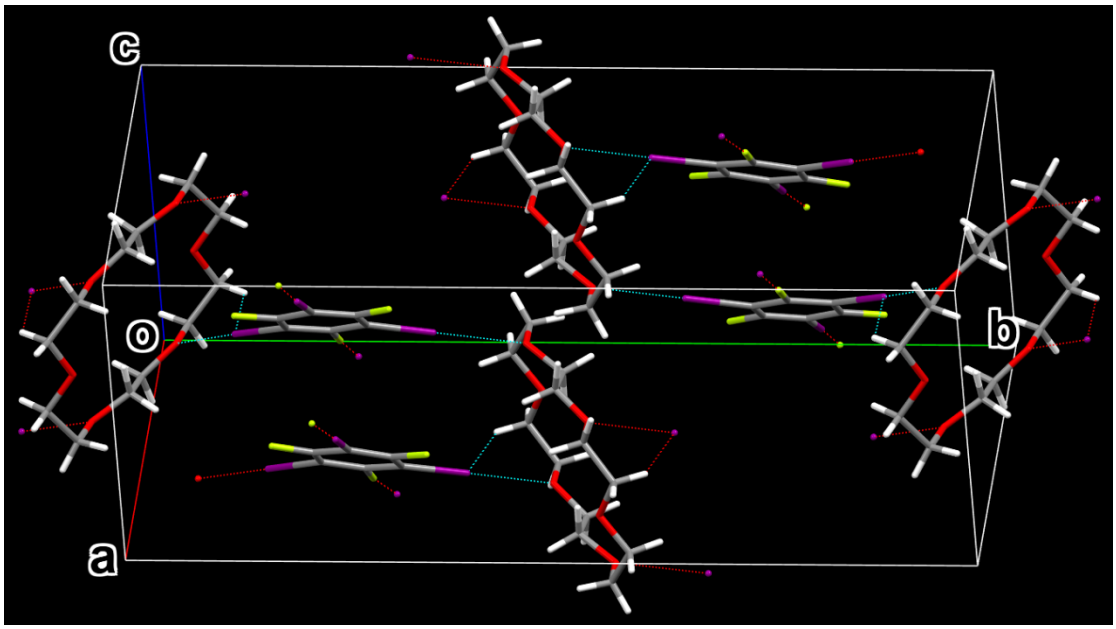
[C6][I14]



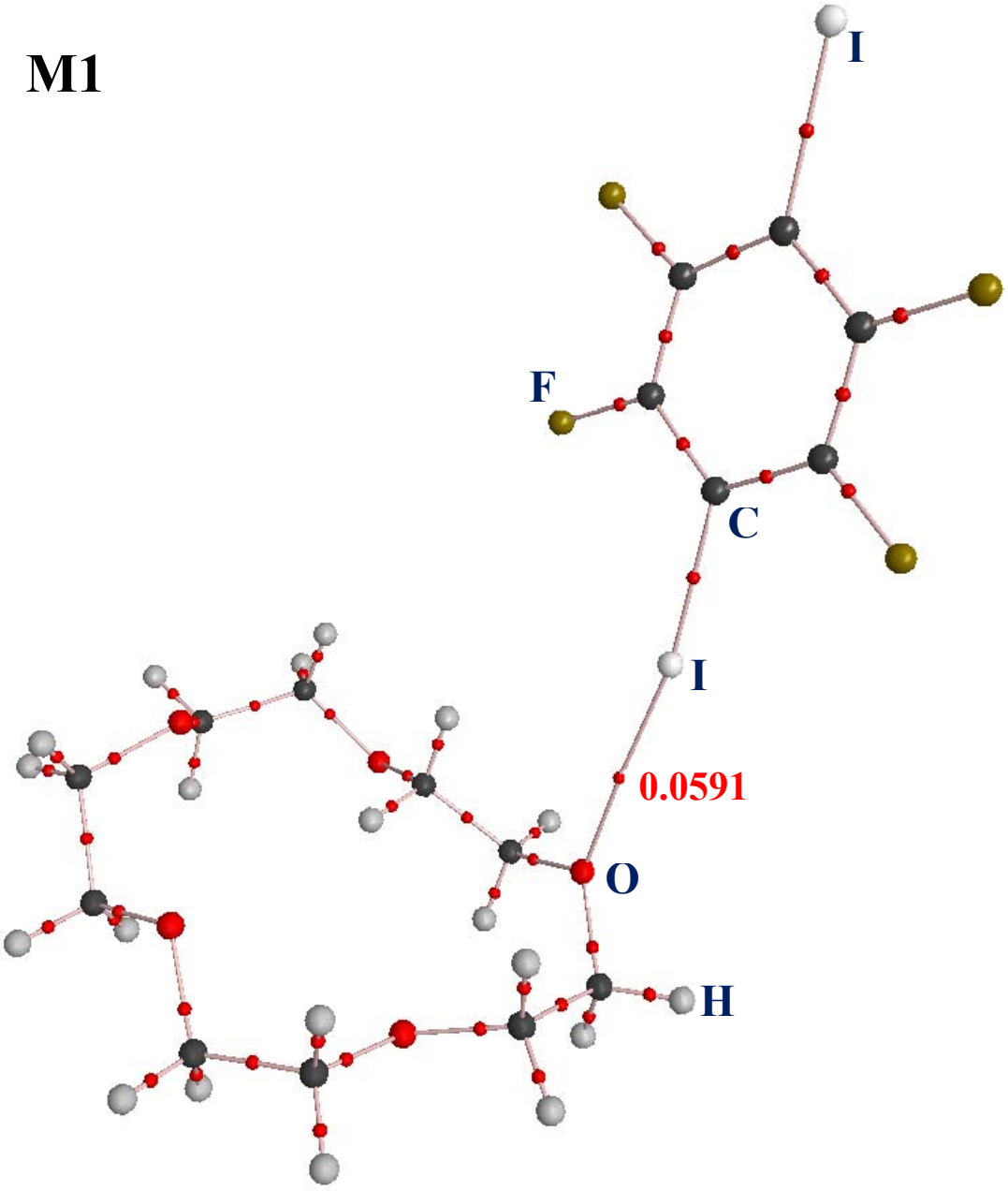
[C6][W]$_3$[I14]



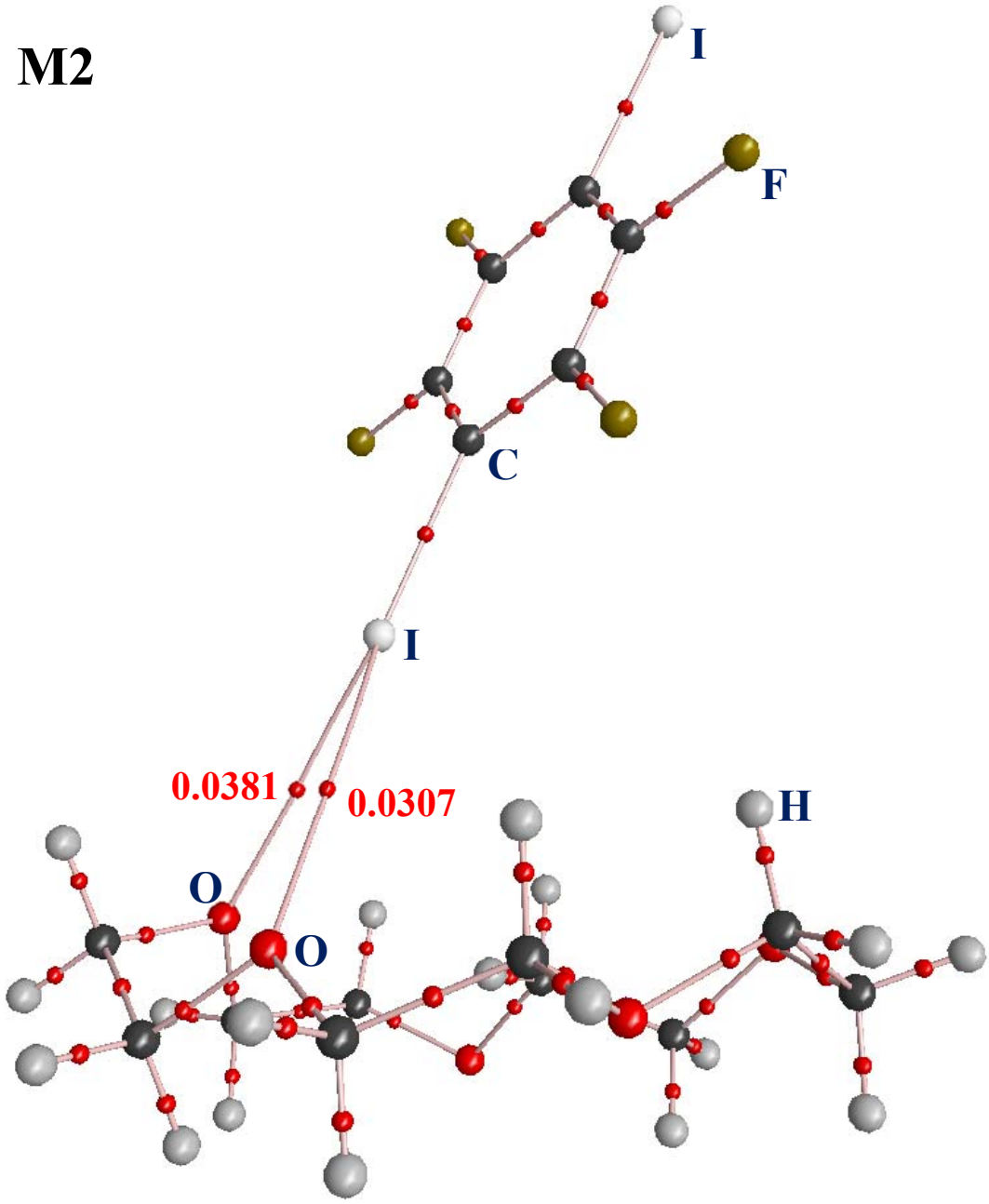
[C6][I135]$_2$



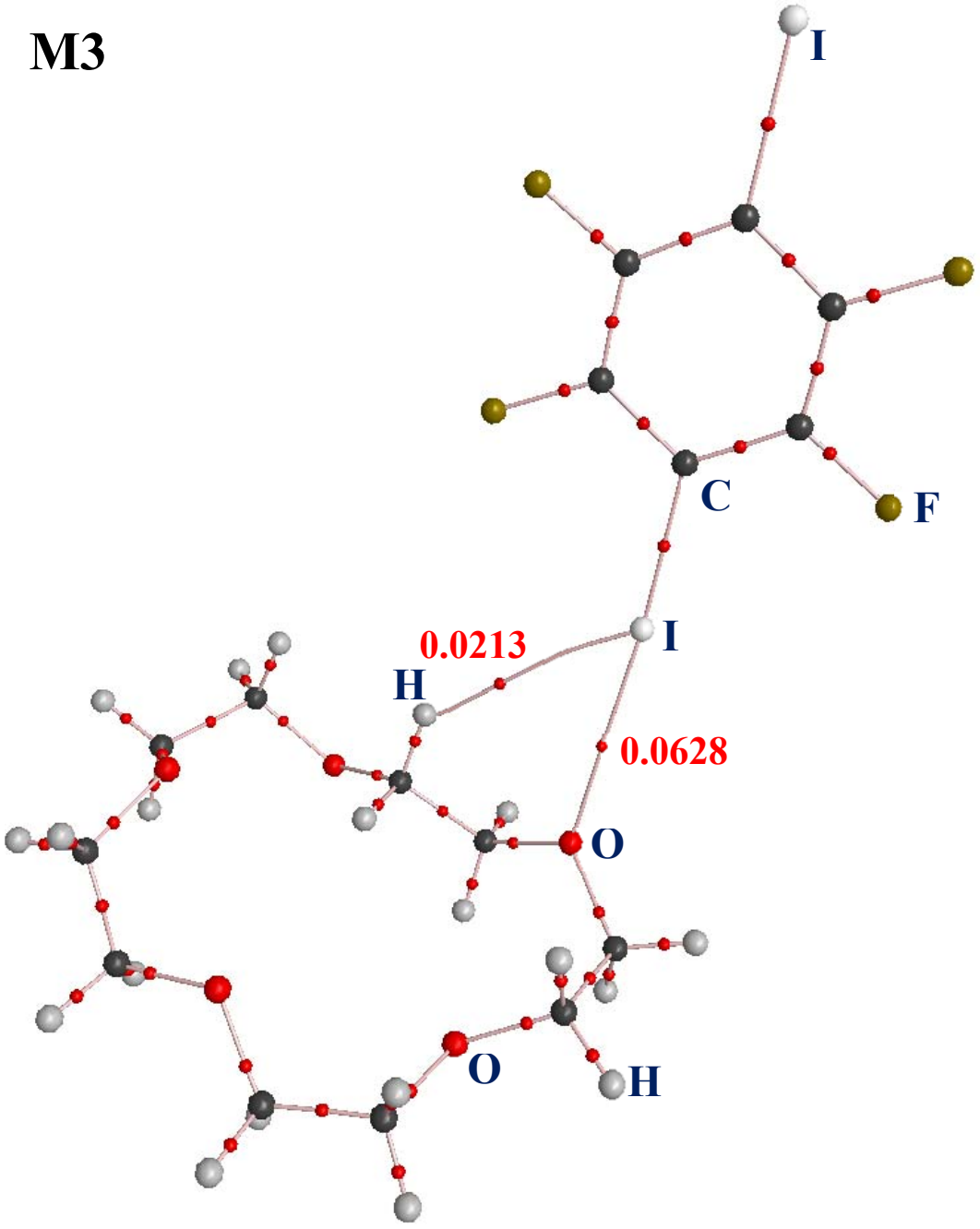
S5. The bond paths and bond critical points (small red dots) for the dimers in Fig. 4. For clarity, the ring critical points and cage critical points are omitted. The red numbers are the values of the electron density Laplacians (au).



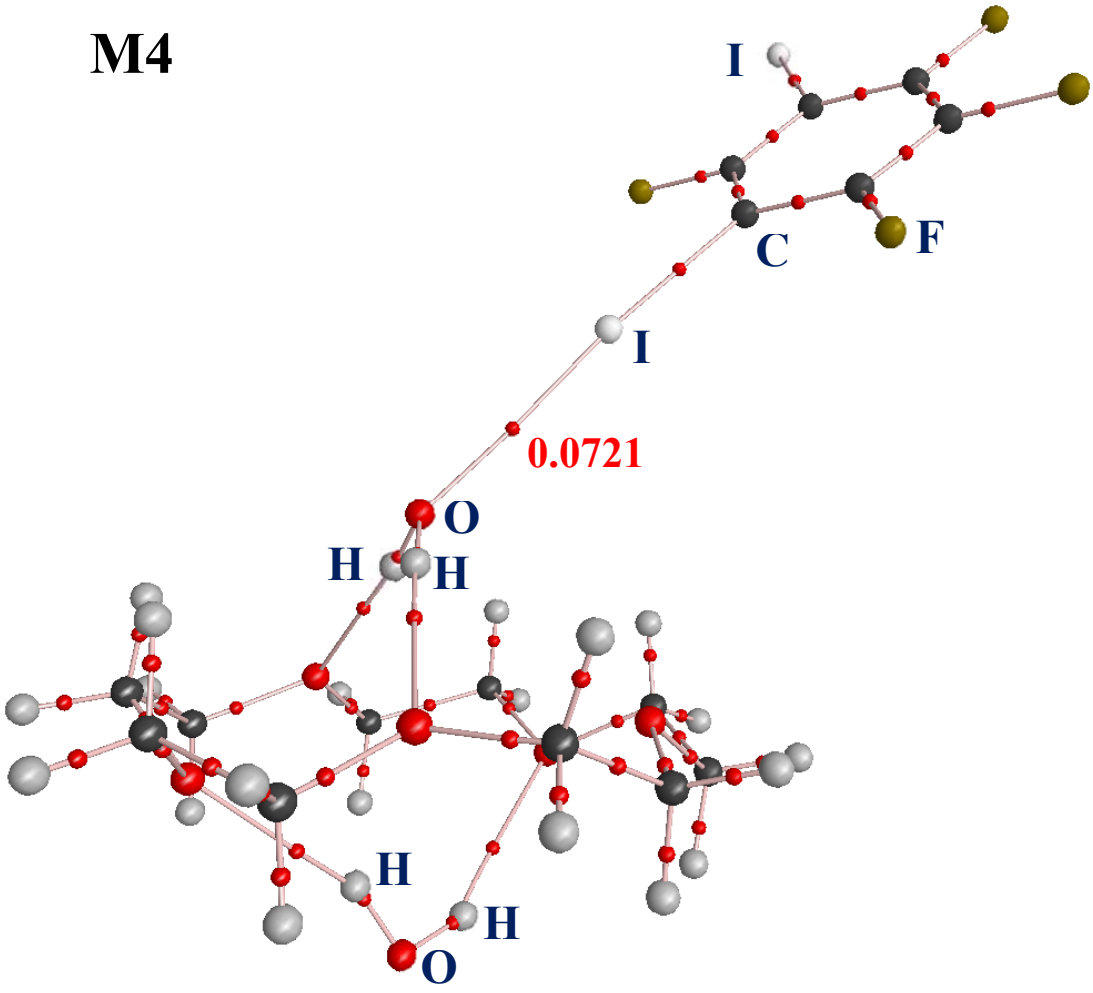
M2



M3



M4



S6. Notes and references

1 CrysAlisPro, Rigaku Oxford Diffraction, Version 1.171.36.32, England, 2013.

2 CrysAlisPro, Rigaku Oxford Diffraction, Version 1.171.38.41r, England, 2015.

3 G. M. Sheldrick, *SHELXS-97: Program for X-ray Crystal Structure Solution*, University of Gottingen: Gottingen, Germany, 1997.

4 G. M. Sheldrick, *SHELXS-97: Program for X-ray Crystal Structure Refinement*, University of Gottingen: Gottingen, Germany, 1997.

5 G. M. Sheldrick, *Acta Cryst.*, 2008, **A64**, 112–122.

6 C. Adamo and V. Barone, *J. Chem. Phys.*, 1999, **110**, 6158–6169.

7 S. Grimme, J. Antony, S. Ehrlich and H. Krieg, *J. Chem. Phys.*, 2010, **132**, 154104.

8 S. Grimme, S. Ehrlich and L. Goerigk, *J. Comput. Chem.*, 2011, **32**, 1456–1465.

9 F. Weigend and R. Ahlrichs, *Phys. Chem. Chem. Phys.*, 2005, **7**, 3297–3305.

10 Gaussian 09, Revision D.01, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, T. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2013.

11 W. Wang, T. Sun, Y. Zhang and Y. B. Wang, *J. Chem. Phys.*, 2015, **143**, 114312.

12 W. Wang, Y. Zhang and Y. B. Wang, *Int. J. Quantum Chem.*, 2017, **117**, e25345.

13 R. F. W. Bader, *Atoms in Molecules, A Quantum Theory*, Clarendon Press, Oxford, 1990.

14 F. Biegler-König, J. Schönbohm and D. Bayles, *J. Comput. Chem.*, 2001, **22**, 545–559.

S7. The CheckCIF reports for the thirteen cocrystals.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C4-I12

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C4-I12

Bond precision: C-C = 0.0089 A Wavelength=0.71073

Cell: a=8.1008(7) b=8.2512(6) c=10.7542(8)
alpha=76.846(6) beta=78.005(7) gamma=89.406(6)
Temperature: 295 K

	Calculated	Reported
Volume	684.17(10)	684.17(9)
Space group	P -1	P-1
Hall group	-P 1	?
Moiety formula	C6 F4 I2, 0.5(C8 H16 O4)	?
Sum formula	C10 H8 F4 I2 O2	C20 H16 F8 I4 O4
Mr	489.96	979.93
Dx,g cm-3	2.378	2.378
Z	2	1
Mu (mm-1)	4.634	4.634
F000	452.0	452.0
F000'	450.39	
h,k,lmax	9,9,13	9,9,13
Nref	2527	2524
Tmin,Tmax	0.271,0.629	0.337,0.654
Tmin'	0.228	

Correction method= # Reported T Limits: Tmin=0.337 Tmax=0.654
AbsCorr = MULTI-SCAN

Data completeness= 0.999 Theta(max)= 25.490

R(reflections)= 0.0377(1992) wR2(reflections)= 0.0776(2524)

S = 1.042 Npar= 163

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

```
PLAT334_ALERT_2_C Small <C-C> Benzene Dist.   C5       -C10      .       1.37 Ang.
PLAT342_ALERT_3_C Low Bond Precision on  C-C Bonds ..............    0.00887 Ang.
```

Alert level G

```
PLAT005_ALERT_5_G No Embedded Refinement Details Found  in the CIF     Please Do !
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ...          2 Check
PLAT431_ALERT_2_G Short Inter HL..A Contact  I1       ..O3       .       3.01 Ang.
                                              x,1+y,-1+z  =      1_564 Check
PLAT431_ALERT_2_G Short Inter HL..A Contact  I2       ..O2       .       3.13 Ang.
                                                x,y,-1+z  =      1_554 Check
PLAT899_ALERT_4_G SHELXL-97  is Deprecated and Succeeded by SHELXL     2019/3 Note
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).          4 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          1 Info
PLAT992_ALERT_5_G Repd & Actual _reflns_number_gt Values Differ by          6 Check
```

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

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
4 ALERT type 2 Indicator that the structure model may be wrong or deficient
2 ALERT type 3 Indicator that the structure quality may be low
1 ALERT type 4 Improvement, methodology, query or suggestion
2 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

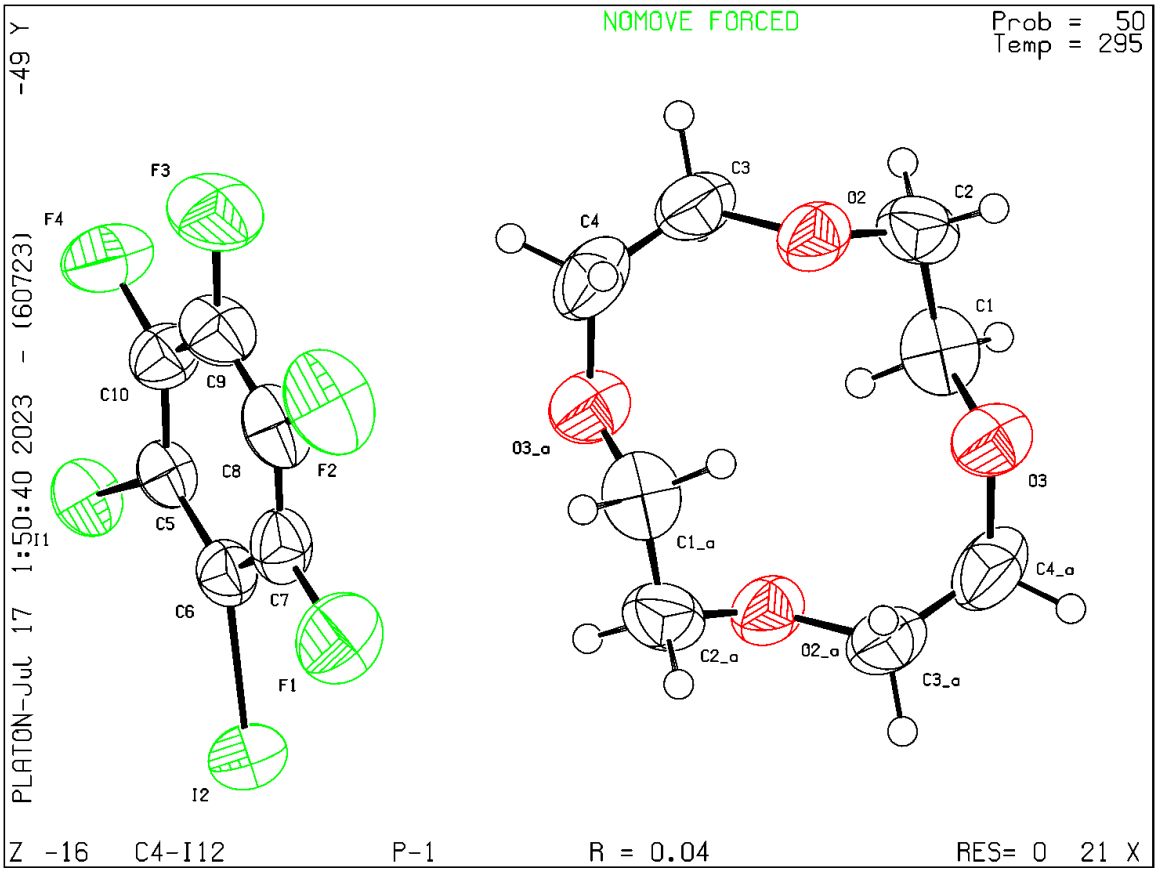
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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 06/07/2023; check.def file version of 30/06/2023

Datablock C4-I12 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C4-I13

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C4-I13

```
Bond precision:   C-C = 0.0081 A          Wavelength=0.71073

Cell:             a=12.0376(4)     b=15.4508(8)        c=7.5142(3)
                  alpha=90         beta=95.120(3)      gamma=90
Temperature:      293 K
```

	Calculated	Reported
Volume	1391.99(10)	1391.99(10)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C6 F4 I2, 0.5(C8 H16 O4)	C6 F4 I2, C4 H8 O2
Sum formula	C10 H8 F4 I2 O2	C10 H8 F4 I2 O2
Mr	489.96	489.96
Dx,g cm-3	2.338	2.338
Z	4	4
Mu (mm-1)	4.555	4.555
F000	904.0	904.0
F000'	900.77	
h,k,lmax	16,20,10	15,19,9
Nref	3455	2945
Tmin,Tmax	0.445,0.529	0.298,1.000
Tmin'	0.436	

```
Correction method= # Reported T Limits: Tmin=0.298 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.852          Theta(max)= 28.304

R(reflections)= 0.0439( 2262)                wR2(reflections)=
                                             0.0949( 2945)
S = 1.060                 Npar= 173
```

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

```
PLAT042_ALERT_1_C Calc. and Reported MoietyFormula Strings  Differ     Please Check
PLAT342_ALERT_3_C Low Bond Precision on  C-C Bonds ..............    0.00812 Ang.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      6.704 Check
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).          6 Note
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600          5 Report
```

Alert level G

```
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ...          3 Report
PLAT012_ALERT_1_G N.O.K.   _shelx_res_checksum Found in CIF ......     Please Check
PLAT013_ALERT_1_G N.O.K.   _shelx_hkl_checksum Found in CIF ......     Please Check
PLAT171_ALERT_4_G The CIF-Embedded .res File Contains EADP Records          1 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records          1 Report
PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records          1 Report
PLAT188_ALERT_3_G A Non-default SIMU Restraint Value has been used     0.0100 Report
PLAT190_ALERT_3_G A Non-default RIGU Restraint Value for First Par     0.0100 Report
PLAT190_ALERT_3_G A Non-default RIGU Restraint Value for SecondPar     0.0200 Report
PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K)        293 Check
PLAT200_ALERT_1_G Reported   _diffrn_ambient_temperature ..... (K)        293 Check
PLAT301_ALERT_3_G Main Residue  Disorder ..............(Resd  1  )         8% Note
PLAT431_ALERT_2_G Short Inter HL..A Contact  I2       ..O2       .       3.00 Ang.
                                              -1+x,y,-1+z  =      1_454 Check
PLAT434_ALERT_2_G Short Inter HL..HL Contact F1       ..F1       .       2.78 Ang.
                                               1-x,1-y,-z  =      3_665 Check
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............         25 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        479 Note
PLAT913_ALERT_3_G Missing # of Very Strong Reflections in FCF ....          2 Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File          7 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........        2.8 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          0 Info
```

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

   5 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   5 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
   4 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

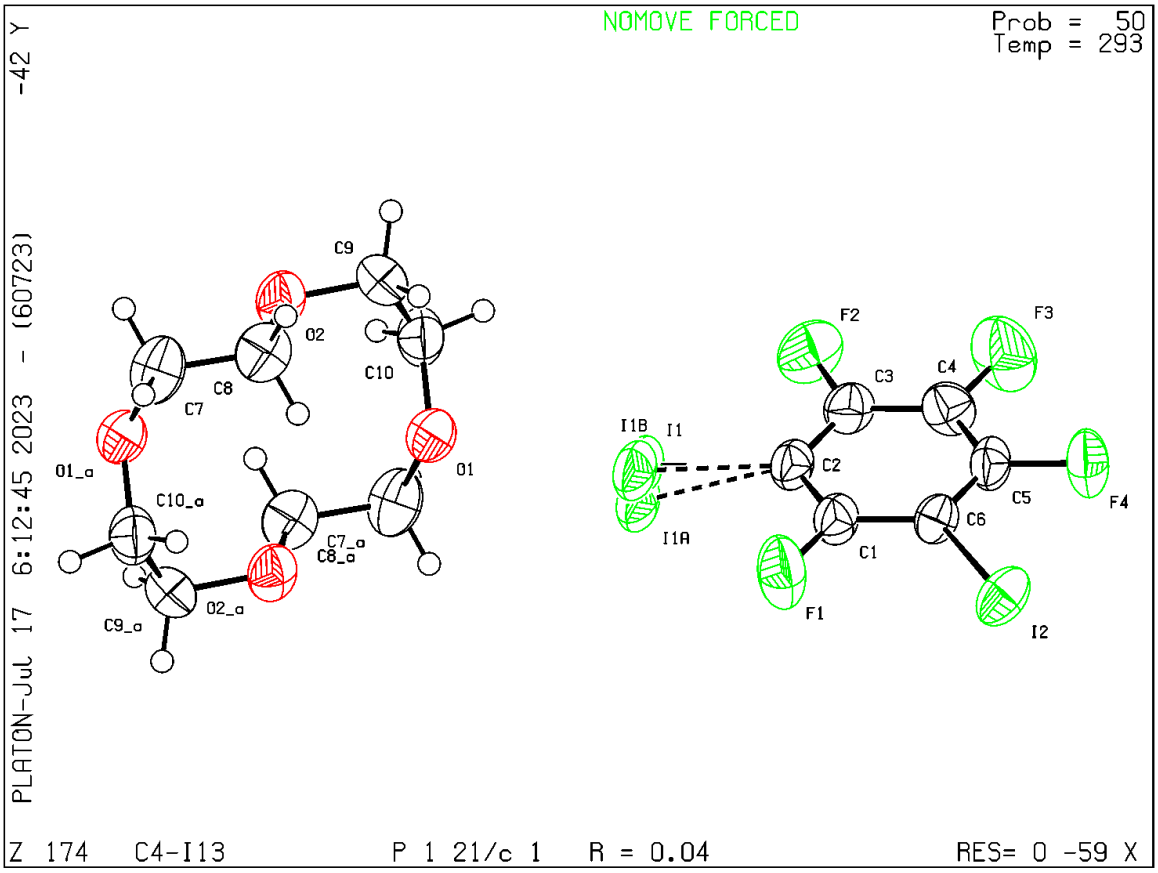
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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 06/07/2023; check.def file version of 30/06/2023

Datablock C4-I13 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C4-I14

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C4-I14

Bond precision: C-C = 0.0089 A Wavelength=0.71073

Cell: a=7.5395(6) b=8.6077(7) c=12.2413(10)
alpha=94.007(7) beta=103.778(7) gamma=113.576(7)
Temperature: 293 K

	Calculated	Reported
Volume	694.90(11)	694.90(10)
Space group	P -1	P-1
Hall group	-P 1	?
Moiety formula	C6 F4 I2, 0.5(C8 H16 O4)	?
Sum formula	C10 H8 F4 I2 O2	C10 H8 F4 I2 O2
Mr	489.96	489.96
Dx,g cm-3	2.342	2.342
Z	2	2
Mu (mm-1)	4.562	4.562
F000	452.0	452.0
F000'	450.39	
h,k,lmax	9,10,15	9,10,15
Nref	2841	2833
Tmin,Tmax	0.444,0.605	0.494,0.634
Tmin'	0.436	

Correction method= # Reported T Limits: Tmin=0.494 Tmax=0.634
AbsCorr = MULTI-SCAN

Data completeness= 0.997 Theta(max)= 26.370

R(reflections)= 0.0410(2117) wR2(reflections)= 0.0966(2833)

S = 1.056 Npar= 164

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 A

```
PLAT431_ALERT_2_A Short Inter HL..A Contact  I2       ..O1       .       2.99 Ang.
                                          1-x,1-y,1-z  =      2_666 Check
```

Author Response: The Checkcif software has a bug and cannot identify the halogen bond.

Alert level C

```
PLAT199_ALERT_1_C Reported _cell_measurement_temperature ..... (K)        293 Check
PLAT200_ALERT_1_C Reported   _diffrn_ambient_temperature ..... (K)        293 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C6 Check
PLAT334_ALERT_2_C Small <C-C> Benzene Dist.   C4       -C6_b     .       1.37 Ang.
PLAT342_ALERT_3_C Low Bond Precision on  C-C Bonds ..............    0.00888 Ang.
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).          7 Note
```

Alert level G

```
PLAT005_ALERT_5_G No Embedded Refinement Details Found  in the CIF     Please Do !
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)      0.007 Degree
PLAT431_ALERT_2_G Short Inter HL..A Contact  I1       ..O2       .       3.00 Ang.
                                          x,y,z  =      1_555 Check
```

Author Response: The Checkcif software has a bug and cannot identify the halogen bond.

```
PLAT764_ALERT_4_G Overcomplete CIF Bond List Detected (Rep/Expd) .       1.17 Ratio
PLAT899_ALERT_4_G SHELXL-97  is Deprecated and Succeeded by SHELXL     2019/3 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600          1 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          0 Info
```

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

   3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   5 ALERT type 2 Indicator that the structure model may be wrong or deficient
   2 ALERT type 3 Indicator that the structure quality may be low
   3 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

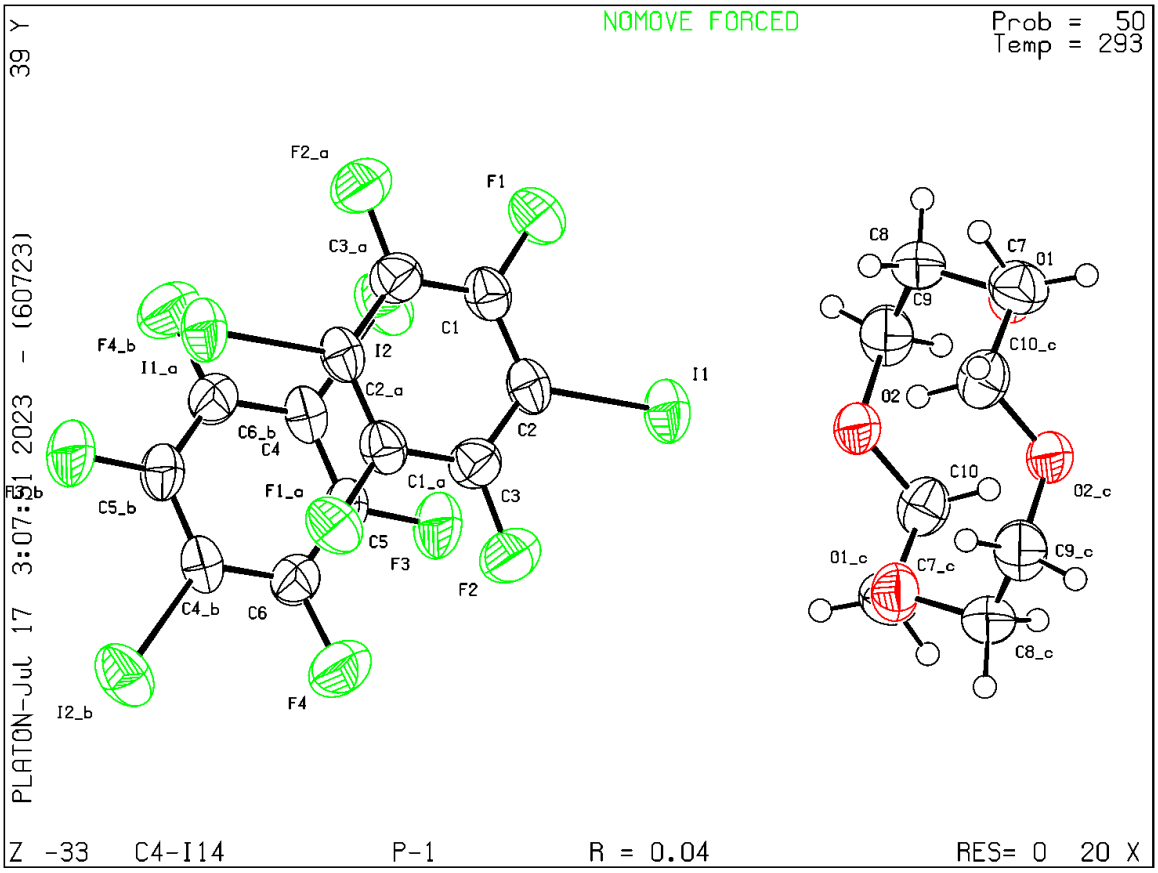
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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 06/07/2023; check.def file version of 30/06/2023

Datablock C4-I14 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C4-I135

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C4-I135

```
Bond precision:   C-C = 0.0088 A          Wavelength=0.71073

Cell:              a=4.6368(5)     b=29.139(4)          c=7.3508(6)
                   alpha=90        beta=97.411(9)       gamma=90
Temperature:       293 K

                 Calculated                 Reported
Volume           984.88(19)                 984.9(2)
Space group      P 21/m                     P 1 21/m 1
Hall group       -P 2yb                     -P 2yb
Moiety formula   C6 F3 I3, C8 H16 O4        C6 F3 I3, C8 H16 O4
Sum formula      C14 H16 F3 I3 O4           C14 H16 F3 I3 O4
Mr               685.97                     685.97
Dx,g cm-3        2.313                      2.313
Z                2                          2
Mu (mm-1)        4.802                      4.802
F000             636.0                      636.0
F000'            633.50
h,k,lmax         6,40,10                    6,39,9
Nref             2755                       2308
Tmin,Tmax        0.390,0.464                0.402,1.000
Tmin'            0.319

Correction method= # Reported T Limits: Tmin=0.402 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.838           Theta(max)= 29.316

R(reflections)= 0.0531( 1665)                wR2(reflections)=
                                             0.1101( 2308)
S = 1.078                  Npar= 119
```

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

```
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of        O2 Check
PLAT342_ALERT_3_C Low Bond Precision on  C-C Bonds ..............   0.00883 Ang.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......    11.472 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......     2.685 Check
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).        5 Note
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  0.88Ang From O2      .      0.49 eA-3
```

Alert level G

```
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ...        4 Report
PLAT012_ALERT_1_G N.O.K.   _shelx_res_checksum Found in CIF ......    Please Check
PLAT013_ALERT_1_G N.O.K.   _shelx_hkl_checksum Found in CIF ......    Please Check
PLAT171_ALERT_4_G The CIF-Embedded .res File Contains EADP Records        1 Report
PLAT177_ALERT_4_G The CIF-Embedded .res File Contains DELU Records        1 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records        1 Report
PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records        1 Report
PLAT188_ALERT_3_G A Non-default SIMU Restraint Value has been used    0.0100 Report
PLAT190_ALERT_3_G A Non-default RIGU Restraint Value for First Par    0.0100 Report
PLAT190_ALERT_3_G A Non-default RIGU Restraint Value for SecondPar    0.0200 Report
PLAT192_ALERT_3_G A Non-default DELU Restraint Value for First Par    0.0010 Report
PLAT192_ALERT_3_G A Non-default DELU Restraint Value for SecondPar    0.0020 Report
PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K)       293 Check
PLAT200_ALERT_1_G Reported   _diffrn_ambient_temperature ..... (K)       293 Check
PLAT301_ALERT_3_G Main Residue  Disorder .............(Resd  1  )       17% Note
PLAT434_ALERT_2_G Short Inter HL..HL Contact I2       ..F2       .      3.04 Ang.
                                                -1+x,y,1+z  =     1_456 Check
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............       11 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600      430 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........      1.9 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.        0 Info
```

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

   4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   5 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
   5 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

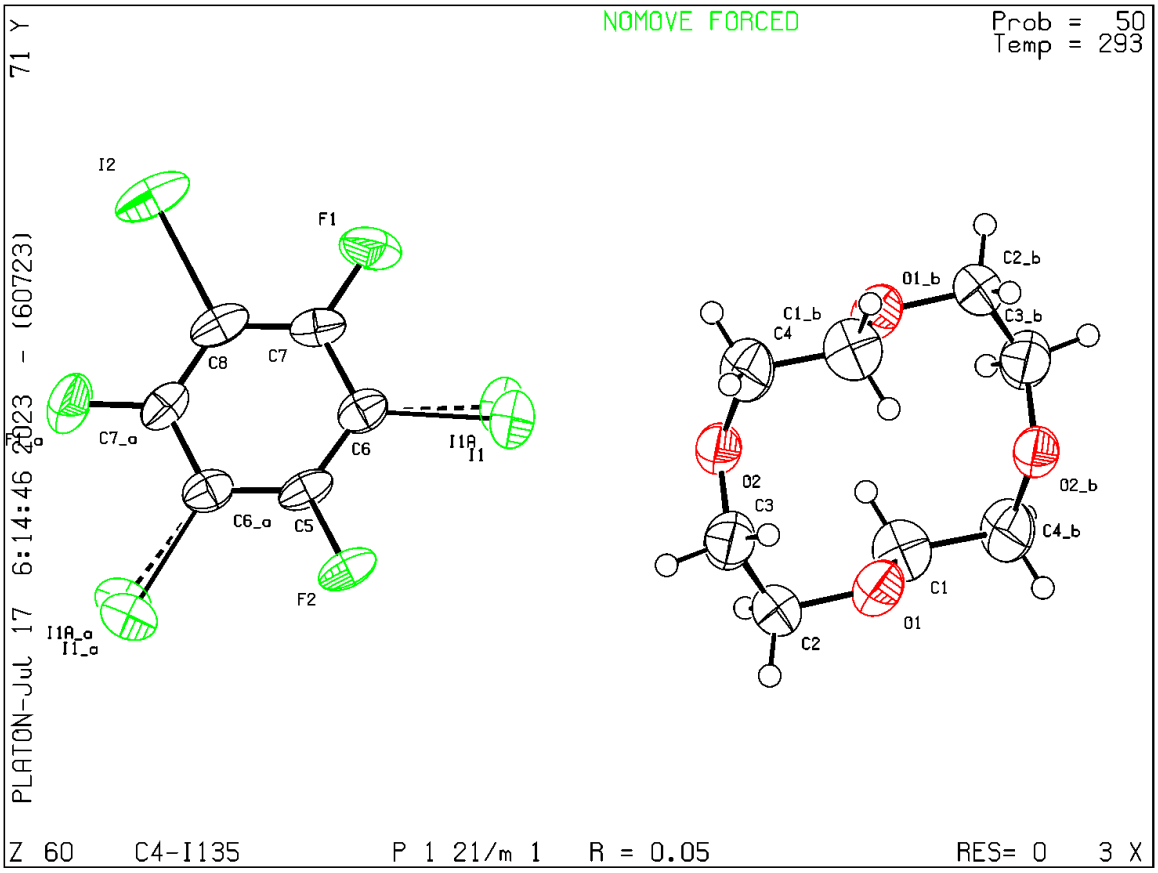
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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 06/07/2023; check.def file version of 30/06/2023

Datablock C4-I135 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C5-I12

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C5-I12

```
Bond precision:   C-C = 0.0196 A          Wavelength=0.71073

Cell:             a=12.3775(18)     b=7.6176(10)          c=22.623(3)
                  alpha=90          beta=100.287(13)      gamma=90
Temperature:      295 K

                 Calculated                 Reported
Volume           2098.8(5)                  2098.8(5)
Space group      P 21/c                     P2(1)/c
Hall group       -P 2ybc                    ?
Moiety formula   C10 H20 O5, C6 F4 I2       ?
Sum formula      C16 H20 F4 I2 O5           C16 H20 F4 I2 O5
Mr               622.12                     622.12
Dx,g cm-3        1.969                      1.969
Z                4                          4
Mu (mm-1)        3.054                      3.054
F000             1192.0                     1192.0
F000'            1188.92
h,k,lmax         14,9,27                    14,9,27
Nref             3900                       3884
Tmin,Tmax        0.346,0.737                0.375,0.750
Tmin'            0.283

Correction method= # Reported T Limits: Tmin=0.375 Tmax=0.750
AbsCorr = MULTI-SCAN

Data completeness= 0.996           Theta(max)= 25.500

R(reflections)= 0.0878( 2728)                wR2(reflections)=
                                             0.3317( 3884)
S = 1.128                  Npar= 238
```

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 A

```
PLAT431_ALERT_2_A Short Inter HL..A Contact  I1       ..O4       .       2.92 Ang.
                                          2-x,1-y,1-z  =      3_766 Check
```

Author Response: The Checkcif software has a bug and cannot identify the halogen bond.

```
PLAT431_ALERT_2_A Short Inter HL..A Contact  I2       ..O1       .       2.91 Ang.
                                          1+x,y,z  =      1_655 Check
```

Author Response: The Checkcif software has a bug and cannot identify the halogen bond.

Alert level C

```
PLAT084_ALERT_3_C High wR2 Value (i.e. > 0.25) ...................       0.33 Report
PLAT214_ALERT_2_C Atom C6        (Anion/Solvent) ADP max/min Ratio        4.2 oblate
PLAT234_ALERT_4_C Large Hirshfeld Difference C7       --C8       .       0.23 Ang.
PLAT342_ALERT_3_C Low Bond Precision on  C-C Bonds ...............    0.01956 Ang.
PLAT360_ALERT_2_C Short  C(sp3)-C(sp3) Bond  C11      - C12      .       1.43 Ang.
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).          5 Note
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600         11 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) .          1 Check
PLAT971_ALERT_2_C Check Calcd Resid. Dens.  0.16Ang From I2              2.49 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens.  0.16Ang From I1              1.78 eA-3
PLAT972_ALERT_2_C Check Calcd Resid. Dens.  0.66Ang From I2             -1.92 eA-3
PLAT972_ALERT_2_C Check Calcd Resid. Dens.  0.68Ang From I2             -1.82 eA-3
PLAT972_ALERT_2_C Check Calcd Resid. Dens.  0.80Ang From I1             -1.82 eA-3
PLAT972_ALERT_2_C Check Calcd Resid. Dens.  1.65Ang From I1             -1.52 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H7A       .      -0.47 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H10B      .      -0.63 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H12B      .      -0.32 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H14A      .      -0.32 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H15A      .      -0.35 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H15B      .      -0.41 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H14C      .      -0.38 eA-3
```

Alert level G

```
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ...          5 Report
PLAT005_ALERT_5_G No Embedded Refinement Details Found  in the CIF     Please Do !
PLAT072_ALERT_2_G SHELXL First  Parameter in WGHT  Unusually Large        0.20 Report
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT  Unusually Large        6.31 Why ?
PLAT301_ALERT_3_G Main Residue  Disorder ..............(Resd  1  )         33% Note
PLAT398_ALERT_2_G Deviating  C-O-C   Angle From 120 for O5       .      109.3 Degree
PLAT410_ALERT_2_G Short Intra H...H Contact  H12B     ..H13A     .       1.96 Ang.
                                                    x,y,z  =      1_555 Check
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle(s) in CIF ...        5.37 Deg.
             C16  -O1   -C16'    1_555   1_555   1_555 ......     #  107 Check
```

```
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle(s) in CIF ...       1.20 Deg.
              C13  -O4   -C13'    1_555   1_555   1_555 ......     #  110 Check
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............         37 Note
PLAT899_ALERT_4_G SHELXL-97  is Deprecated and Succeeded by SHELXL     2019/3 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          0 Info
PLAT992_ALERT_5_G Repd & Actual _reflns_number_gt Values Differ by          2 Check
```

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

   0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
  23 ALERT type 2 Indicator that the structure model may be wrong or deficient
   7 ALERT type 3 Indicator that the structure quality may be low
   4 ALERT type 4 Improvement, methodology, query or suggestion
   2 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

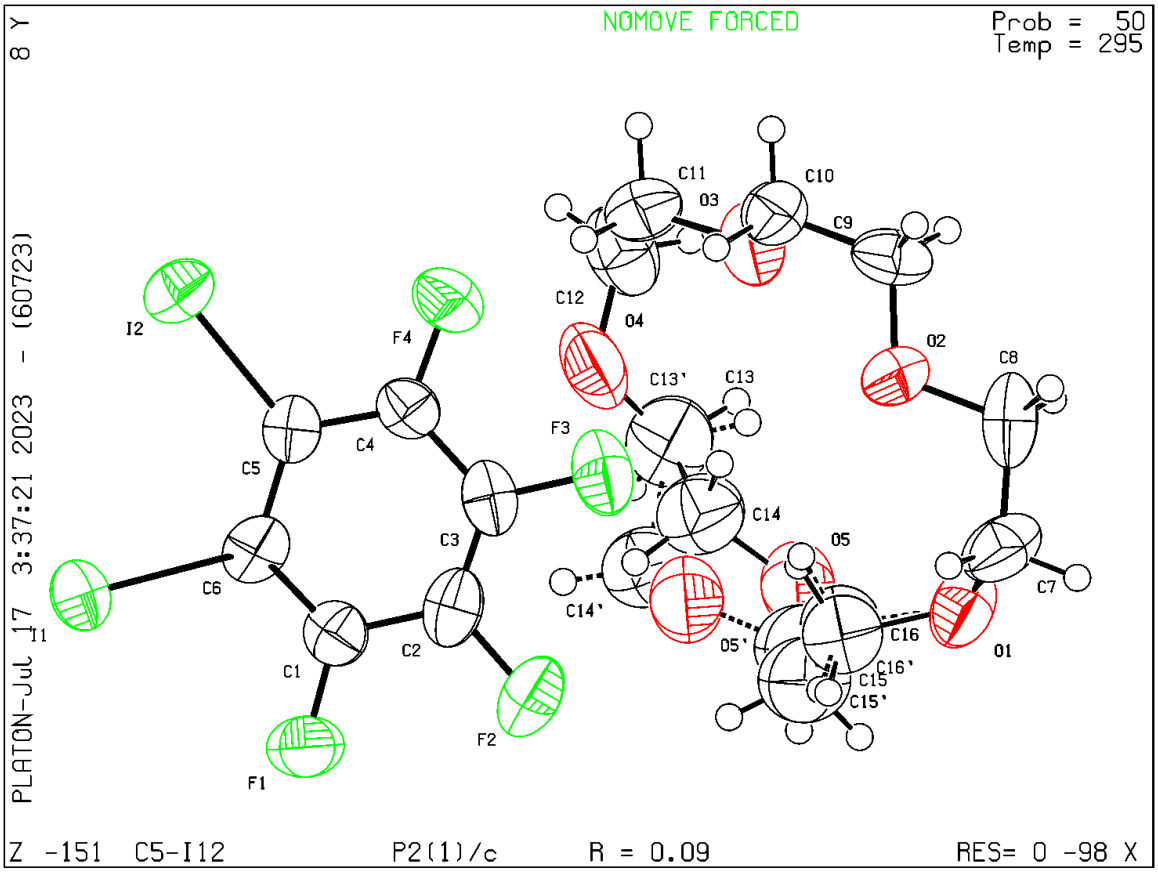
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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 06/07/2023; check.def file version of 30/06/2023

Datablock C5-I12 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C5-I13-W

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C5-I13-W

Bond precision: C-C = 0.0154 A Wavelength=0.71073

Cell: a=7.4810(5) b=22.1452(19) c=14.6860(11)
alpha=90 beta=103.285(7) gamma=90
Temperature: 293 K

	Calculated	Reported
Volume	2367.9(3)	2367.9(3)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C10 H20 O5, C6 F4 I2, 2(H2 O)	C6 F4 I2, 2(H2 O), C10 H20 O5
Sum formula	C16 H24 F4 I2 O7	C16 H24 F4 I2 O7
Mr	658.15	658.15
Dx,g cm-3	1.846	1.846
Z	4	4
Mu (mm-1)	2.718	2.718
F000	1272.0	1272.0
F000'	1269.00	
h,k,lmax	9,26,17	9,26,17
Nref	4397	4376
Tmin,Tmax	0.332,0.455	0.399,1.000
Tmin'	0.283	

Correction method= # Reported T Limits: Tmin=0.399 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.995 Theta(max)= 25.500

R(reflections)= 0.0594(3201) wR2(reflections)= 0.1279(4376)

S = 1.024 Npar= 262

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

PLAT042_ALERT_1_C	Calc. and Reported MoietyFormula Strings Differ		Please Check
PLAT234_ALERT_4_C	Large Hirshfeld Difference O3	--C9 .	0.17 Ang.
PLAT234_ALERT_4_C	Large Hirshfeld Difference O4	--C11 .	0.18 Ang.
PLAT234_ALERT_4_C	Large Hirshfeld Difference O5	--C13 .	0.19 Ang.
PLAT241_ALERT_2_C	High 'MainMol' Ueq as Compared to Neighbors of		O6 Check
PLAT241_ALERT_2_C	High 'MainMol' Ueq as Compared to Neighbors of		C16 Check
PLAT242_ALERT_2_C	Low 'MainMol' Ueq as Compared to Neighbors of		O4 Check
PLAT242_ALERT_2_C	Low 'MainMol' Ueq as Compared to Neighbors of		O5 Check
PLAT242_ALERT_2_C	Low 'MainMol' Ueq as Compared to Neighbors of		C15 Check
PLAT260_ALERT_2_C	Large Average Ueq of Residue Including	O3	0.189 Check
PLAT342_ALERT_3_C	Low Bond Precision on C-C Bonds		0.01536 Ang.
PLAT906_ALERT_3_C	Large K Value in the Analysis of Variance		11.114 Check
PLAT906_ALERT_3_C	Large K Value in the Analysis of Variance		2.428 Check
PLAT910_ALERT_3_C	Missing # of FCF Reflection(s) Below Theta(Min).		9 Note
PLAT976_ALERT_2_C	Check Calcd Resid. Dens. 1.08Ang From O6	.	-0.47 eA-3

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 ...		15 Report
PLAT007_ALERT_5_G	Number of Unrefined Donor-H Atoms		4 Report
PLAT012_ALERT_1_G	No _shelx_res_checksum Found in CIF		Please Check
PLAT083_ALERT_2_G	SHELXL Second Parameter in WGHT Unusually Large		7.91 Why ?
PLAT172_ALERT_4_G	The CIF-Embedded .res File Contains DFIX Records		3 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		1 Report
PLAT178_ALERT_4_G	The CIF-Embedded .res File Contains SIMU Records		1 Report
PLAT199_ALERT_1_G	Reported _cell_measurement_temperature ..... (K)		293 Check
PLAT200_ALERT_1_G	Reported _diffrn_ambient_temperature ..... (K)		293 Check
PLAT398_ALERT_2_G	Deviating C-O-C Angle From 120 for O5	.	107.8 Degree
PLAT860_ALERT_3_G	Number of Least-Squares Restraints		165 Note
PLAT883_ALERT_1_G	No Info/Value for _atom_sites_solution_primary .		Please Do !
PLAT912_ALERT_4_G	Missing # of FCF Reflections Above STh/L= 0.600		11 Note
PLAT941_ALERT_3_G	Average HKL Measurement Multiplicity		3.0 Low
PLAT967_ALERT_5_G	Note: Two-Theta Cutoff Value in Embedded .res ..		51.0 Degree
PLAT978_ALERT_2_G	Number C-C Bonds with Positive Residual Density.		0 Info

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

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

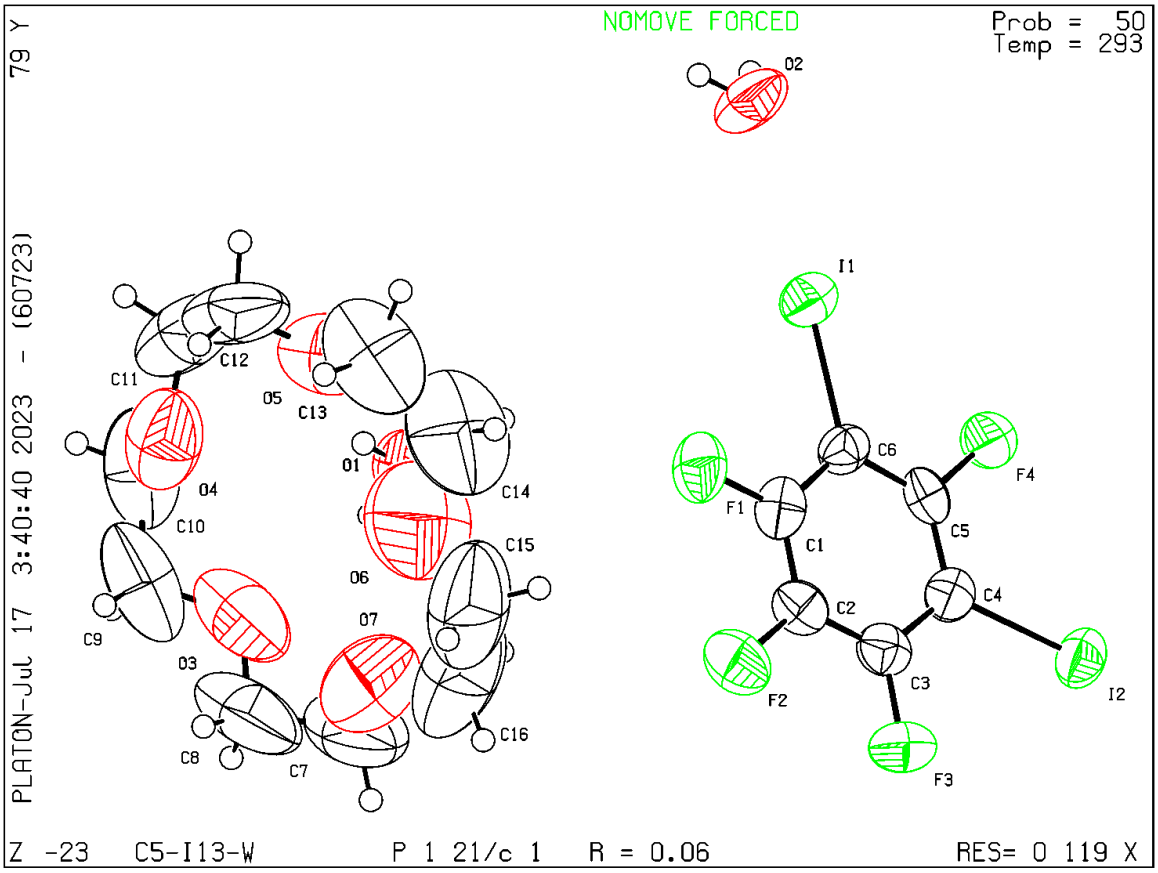
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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 06/07/2023; check.def file version of 30/06/2023

Datablock C5-I13-W - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C5-I14

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C5-I14

```
Bond precision:   C-C = 0.0121 A           Wavelength=0.71073

Cell:           a=9.7355(6)        b=12.4217(7)      c=18.1787(11)
                alpha=88.582(5)    beta=80.631(5)    gamma=78.136(5)
Temperature:    296 K

                 Calculated                 Reported
Volume           2122.6(2)                  2122.6(2)
Space group      P -1                       P -1
Hall group       -P 1                       -P 1
Moiety formula   C10 H20 O5, C6 F4 I2       C10 H20 O5, C6 F4 I2
Sum formula      C16 H20 F4 I2 O5           C16 H20 F4 I2 O5
Mr               622.12                     622.12
Dx,g cm-3        1.947                      1.947
Z                4                          4
Mu (mm-1)        3.020                      3.020
F000             1192.0                     1192.0
F000'            1188.92
h,k,lmax         12,15,22                   12,15,22
Nref             8676                       8657
Tmin,Tmax        0.545,0.696                0.570,0.713
Tmin'            0.525

Correction method= # Reported T Limits: Tmin=0.570 Tmax=0.713
AbsCorr = MULTI-SCAN

Data completeness= 0.998            Theta(max)= 26.372

R(reflections)= 0.0452( 5478)                    wR2(reflections)=
                                                 0.1042( 8657)
S = 0.997                  Npar= 688
```

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).        17 Note
                1  0  0,  -1  1  0,   0  1  0,   1  1  0,  -1 -1  1,   0 -1  1,
                1 -1  1,  -1  0  1,   0  0  1,   1  0  1,   0  1  1,   1  1  1,
                0 -1  2,   0  0  2,   1  0  2,   0  1  2,   1  1  2,
```

Author Response: The missing FCF reflections below theta(min) were caused by the high beamstop theta(min) limit set.

Alert level C

```
PLAT234_ALERT_4_C Large Hirshfeld Difference O8       --C27      .       0.21 Ang.
PLAT234_ALERT_4_C Large Hirshfeld Difference C23      --C24      .       0.18 Ang.
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of        C19 Check
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of        C25 Check
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of        C26 Check
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of        C27 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         O4 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         O7 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         O8 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         O9 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including        O6      0.125 Check
PLAT334_ALERT_2_C Small <C-C> Benzene Dist.   C7       -C12      .       1.37 Ang.
PLAT342_ALERT_3_C Low Bond Precision on  C-C Bonds ..............     0.0121 Ang.
PLAT360_ALERT_2_C Short  C(sp3)-C(sp3) Bond  C13      - C14      .       1.39 Ang.
PLAT360_ALERT_2_C Short  C(sp3)-C(sp3) Bond  C27      - C28      .       1.37 Ang.
PLAT360_ALERT_2_C Short  C(sp3)-C(sp3) Bond  C31      - C32      .       1.42 Ang.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      2.016 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600          2 Report
                8 -4  5,   9 -5  7,
PLAT977_ALERT_2_C Check Negative Difference Density on H14B      .      -0.43 eA-3
```

Alert level G

```
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite         48 Note
PLAT003_ALERT_2_G Number of Uiso or U(i,j) Restrained non-H Atoms          48 Report
PLAT012_ALERT_1_G N.O.K.   _shelx_res_checksum Found in CIF ......     Please Check
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)      0.005 Degree
PLAT168_ALERT_4_G The CIF-Embedded .res File Contains EXYZ Records          2 Report
PLAT171_ALERT_4_G The CIF-Embedded .res File Contains EADP Records          2 Report
PLAT175_ALERT_4_G The CIF-Embedded .res File Contains SAME 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
PLAT189_ALERT_3_G A Non-default SAME Restraint Value for First Par     0.0100 Report
PLAT189_ALERT_3_G A Non-default SAME Restraint Value for SecondPar     0.0200 Report
PLAT189_ALERT_3_G A Non-default SAME Restraint Value for First Par     0.0100 Report
```

```
PLAT189_ALERT_3_G A Non-default SAME Restraint Value for SecondPar     0.0200 Report
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd    3)       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd    4)       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd    5)       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd    6)       100% Note
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd    3)      6.98 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd    4)      8.24 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd    5)      5.02 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd    6)      3.76 Check
PLAT811_ALERT_5_G No ADDSYM Analysis: Too Many Excluded Atoms ....         ! Info
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............       684 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........       2.1 Low
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........     2.194 Note
             Predicted wR2: Based on SigI**2  4.75 or SHELX Weight 10.46
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.         4 Info
```

---

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
27 **ALERT level G** = General information/check it is not something unexpected

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
18 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
14 ALERT type 4 Improvement, methodology, query or suggestion
2 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

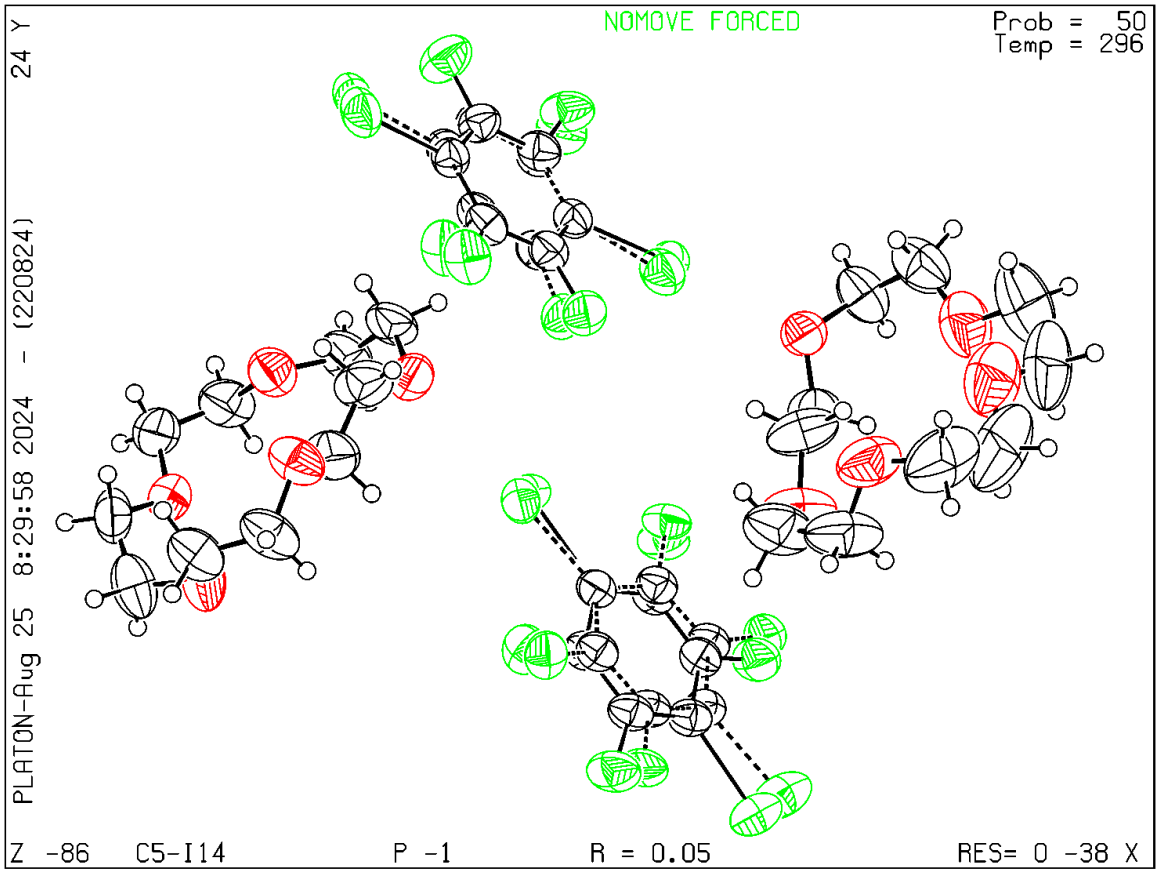
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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 22/08/2024; check.def file version of 21/08/2024

Datablock C5-I14 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C5-I135

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C5-I135

```
Bond precision:   C-C = 0.0185 A          Wavelength=0.71073

Cell:             a=16.1779(9)    b=8.4631(4)         c=25.3518(15)
                  alpha=90        beta=105.819(6)     gamma=90
Temperature:      295 K

                 Calculated                  Reported
Volume           3339.6(3)                   3339.6(3)
Space group      C c                         C 1 c 1
Hall group       C -2yc                      C -2yc
Moiety formula   C10 H20 O5, 2(C6 F3 I3)     C10 H20 O5, 2(C6 F3 I3)
Sum formula      C22 H20 F6 I6 O5            C22 H20 F6 I6 O5
Mr               1239.78                     1239.78
Dx,g cm-3        2.466                       2.466
Z                4                           4
Mu (mm-1)        5.643                       5.643
F000             2256.0                      2256.0
F000'            2245.88
h,k,lmax         22,11,34                    22,10,33
Nref             8963[ 4484]                 6986
Tmin,Tmax        0.175,0.244                 0.546,1.000
Tmin'            0.143

Correction method= # Reported T Limits: Tmin=0.546 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 1.56/0.78        Theta(max)= 29.066

R(reflections)= 0.0364( 5822)                wR2(reflections)=
                                             0.0727( 6986)
S = 1.028                  Npar= 643
```

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

```
PLAT090_ALERT_3_C Poor Data / Parameter Ratio (Zmax > 18) ........       6.17 Note
PLAT342_ALERT_3_C Low Bond Precision on  C-C Bonds ...............     0.0185 Ang.
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).         7 Note
               1  1  0,   2  0  0,  -1  1  1,   1  1  1,  -2  0  2,  -1  1  2,
               0  0  2,
```

Alert level G

```
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite        68 Note
PLAT003_ALERT_2_G Number of Uiso or U(i,j) Restrained non-H Atoms         66 Report
PLAT012_ALERT_1_G N.O.K.   _shelx_res_checksum Found in CIF ......     Please Check
PLAT013_ALERT_1_G N.O.K.   _shelx_hkl_checksum Found in CIF ......     Please Check
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records         1 Report
PLAT174_ALERT_4_G The CIF-Embedded .res File Contains FLAT Records         1 Report
PLAT175_ALERT_4_G The CIF-Embedded .res File Contains SAME 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
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
PLAT189_ALERT_3_G A Non-default SAME Restraint Value for First Par     0.0100 Report
PLAT189_ALERT_3_G A Non-default SAME Restraint Value for SecondPar     0.0200 Report
PLAT189_ALERT_3_G A Non-default SAME Restraint Value for First Par     0.0100 Report
PLAT189_ALERT_3_G A Non-default SAME Restraint Value for SecondPar     0.0200 Report
PLAT189_ALERT_3_G A Non-default SAME Restraint Value for First Par     0.0100 Report
PLAT189_ALERT_3_G A Non-default SAME Restraint Value for SecondPar     0.0200 Report
PLAT301_ALERT_3_G Main Residue  Disorder ..............(Resd    1)        53% 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
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd    4)       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd    5)       100% Note
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd    2)      6.48 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd    3)      8.88 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd    4)      5.52 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd    5)      3.12 Check
PLAT398_ALERT_2_G Deviating  C-O-C   Angle From 120 for O2       .     108.3 Degree
PLAT398_ALERT_2_G Deviating  C-O-C   Angle From 120 for O2A      .     106.4 Degree
PLAT410_ALERT_2_G Short Intra H...H Contact  H7B      ..H6AB     .      2.11 Ang.
                                                    x,y,z  =      1_555 Check
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels .........        12 Note
              H1AA    H1AB    H2AA    H2AB    H3AA    H3AB    H4AA    H4AB
              H5AA    H5AB    H6AA    H6AB
PLAT721_ALERT_1_G Bond    Calc     0.96000, Rep     0.97000 Dev...      0.01 Ang.
              C2A      -H2AA            1_555   1_555 ........     #   41 Check
PLAT721_ALERT_1_G Bond    Calc     0.96000, Rep     0.97000 Dev...      0.01 Ang.
              C4A      -H4AA            1_555   1_555 ........     #   48 Check
PLAT722_ALERT_1_G Angle   Calc      110.00, Rep      108.80 Dev...      1.20 Degree
              C2A     -C1A     -H1AA       1_555  1_555  1_555     #   72 Check
PLAT811_ALERT_5_G No ADDSYM Analysis: Too Many Excluded Atoms ....         ! Info
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............       908 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600       446 Note
```

```
PLAT915_ALERT_3_G No Flack x Check Done: Low Friedel Pair Coverage         67 %
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........        2.8 Low
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........      1.618 Note
              Predicted wR2: Based on SigI**2  4.50 or SHELX Weight  7.07
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          1 Info
```

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

   5 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   6 ALERT type 2 Indicator that the structure model may be wrong or deficient
  16 ALERT type 3 Indicator that the structure quality may be low
  14 ALERT type 4 Improvement, methodology, query or suggestion
   2 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

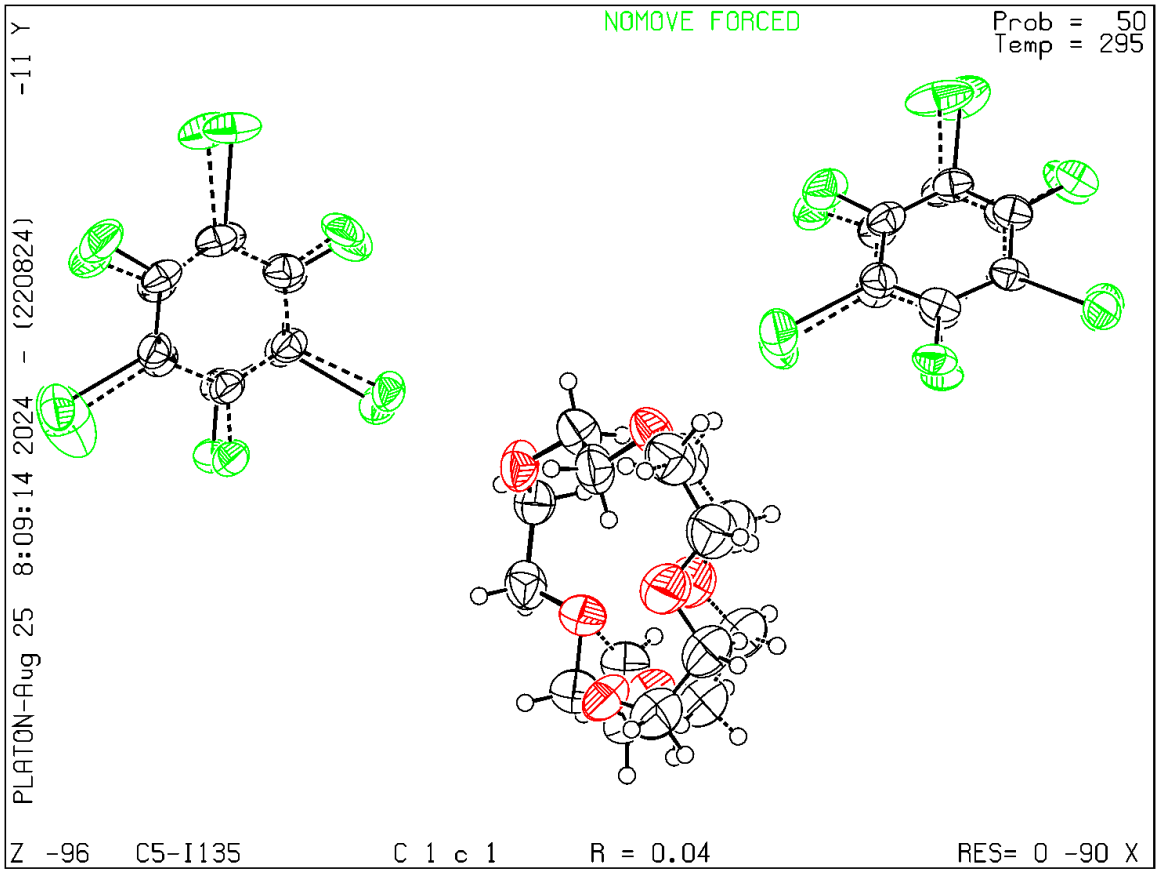
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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 22/08/2024; check.def file version of 21/08/2024

Datablock C5-I135 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C6-I12-W

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C6-I12-W

Bond precision: C-C = 0.0084 A Wavelength=0.71073

Cell: a=7.6327(5) b=8.0627(6) c=15.0373(12)
alpha=88.108(6) beta=77.223(6) gamma=89.966(6)
Temperature: 295 K

	Calculated	Reported
Volume	901.97(12)	901.97(12)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C12 H24 O6, 2(C6 F4 I2), 4(H2 O)	C12 H24 O6, 2(C6 F4 I2), 4(H2 O)
Sum formula	C24 H32 F8 I4 O10	C24 H32 F8 I4 O10
Mr	1140.10	1140.09
Dx,g cm-3	2.099	2.099
Z	1	1
Mu (mm-1)	3.543	3.543
F000	540.0	540.0
F000'	538.45	
h,k,lmax	10,10,20	10,10,20
Nref	4718	4051
Tmin,Tmax	0.264,0.492	0.331,0.538
Tmin'	0.222	

Correction method= # Reported T Limits: Tmin=0.331 Tmax=0.538
AbsCorr = MULTI-SCAN

Data completeness= 0.859 Theta(max)= 28.832

R(reflections)= 0.0435(2906) wR2(reflections)= 0.0994(4051)

S = 1.055 Npar= 209

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

```
PLAT334_ALERT_2_C Small <C-C> Benzene Dist.   C1       -C6       .       1.37 Ang.
PLAT342_ALERT_3_C Low Bond Precision on  C-C Bonds ..............    0.00844 Ang.
PLAT355_ALERT_3_C Long   O-H (X0.82,N0.98A)  O5       - H5B      .       1.05 Ang.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      3.032 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      2.116 Check
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).          7 Note
               1  0  0,   0  1  0,   0 -1  1,   0  0  1,   1  0  1,   0  1  1,
               0  0  2,
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600         12 Report
               7 -1  8,   7 -1  9,   4 -2 11,   5 -1 11,   6 -1 11,   5 -2 13,
               5 -2 14,   6  2 14,   6  3 14,   6  1 15,   6  2 15,   5 -2 16,
```

Alert level G

```
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite          3 Note
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............          4 Report
             H4A   H4B   H5A   H5B
PLAT012_ALERT_1_G N.O.K.   _shelx_res_checksum Found in CIF ......     Please Check
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)      0.006 Degree
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records          2 Report
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd.  #          4 Note
             H2 O
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............          2 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        648 Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File          8 Note
               6  4 14,   6  0 16,   8  4 14,   6  2 14,   6  3 14,   6  5 15,
               6  1 15,   5 -2 14,
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........        1.7 Low
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........      2.349 Note
             Predicted wR2: Based on SigI**2  4.23 or SHELX Weight  9.42
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          0 Info
```

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

  2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
  4 ALERT type 2 Indicator that the structure model may be wrong or deficient
  8 ALERT type 3 Indicator that the structure quality may be low
  3 ALERT type 4 Improvement, methodology, query or suggestion
  2 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

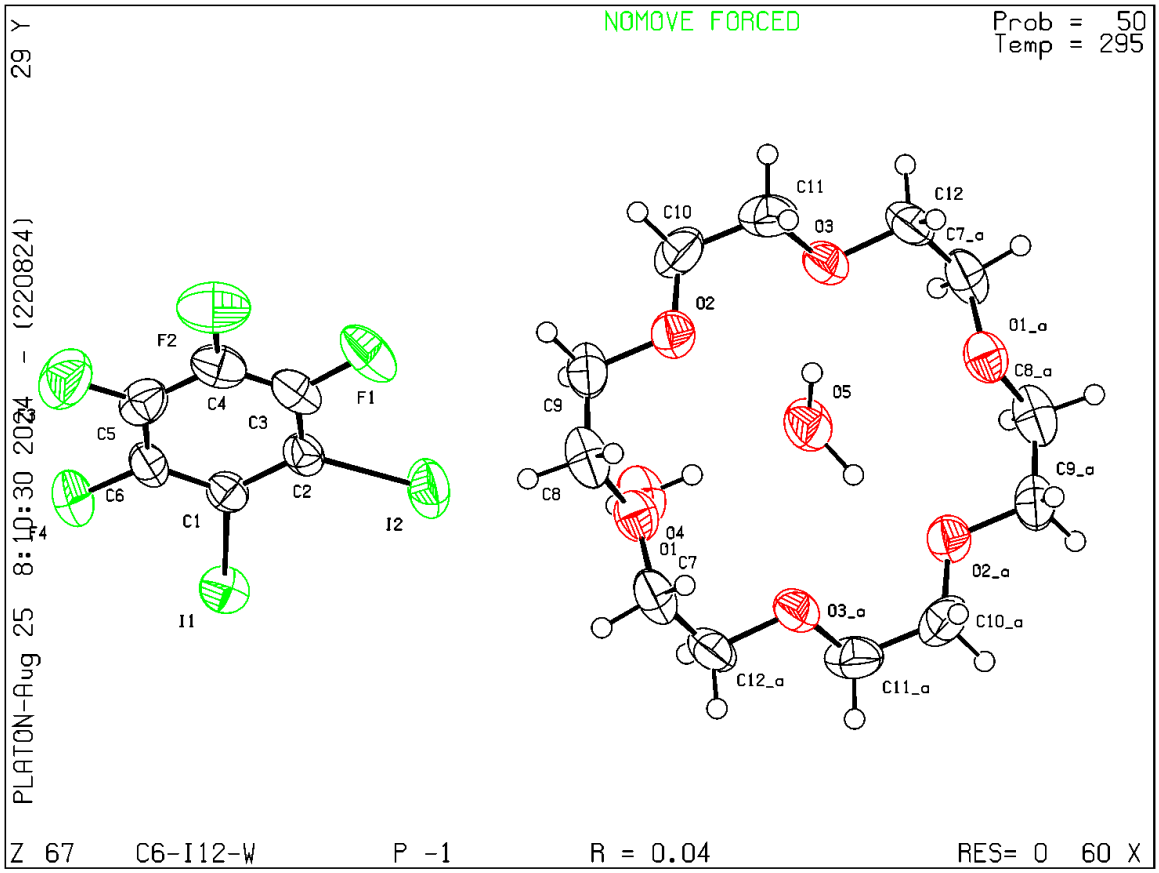
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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 22/08/2024; check.def file version of 21/08/2024

Datablock C6-I12-W - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C6-I13

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C6-I13

```
Bond precision:   C-C = 0.0092 A          Wavelength=0.71073

Cell:             a=7.8540(6)     b=15.3678(14)      c=27.2763(16)
                  alpha=90        beta=90            gamma=90
Temperature:      293 K

                 Calculated                Reported
Volume           3292.2(4)                 3292.2(4)
Space group      P b c a                   Pbca
Hall group       -P 2ac 2ab                ?
Moiety formula   C12 H24 O6, 2(C6 F4 I2)   ?
Sum formula      C24 H24 F8 I4 O6          C12 H12 F4 I2 O3
Mr               1068.03                   534.02
Dx,g cm-3        2.155                     2.155
Z                4                         8
Mu (mm-1)        3.866                     3.866
F000             2000.0                    2000.0
F000'            1993.64
h,k,lmax         9,18,33                   9,18,33
Nref             3076                      3056
Tmin,Tmax        0.223,0.395               0.300,0.457
Tmin'            0.188

Correction method= # Reported T Limits: Tmin=0.300 Tmax=0.457
AbsCorr = MULTI-SCAN

Data completeness= 0.993           Theta(max)= 25.500

R(reflections)= 0.0490( 2437)                wR2(reflections)=
                                             0.1059( 3056)
S = 1.133                Npar= 191
```

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 A

```
PLAT431_ALERT_2_A Short Inter HL..A Contact  I2       ..O1       .       2.94 Ang.
                                                    x,y,z  =      1_555 Check
```

Author Response: The Checkcif software has a bug and cannot identify the halogen bond.

Alert level C

```
PLAT199_ALERT_1_C Reported _cell_measurement_temperature ..... (K)        293 Check
PLAT200_ALERT_1_C Reported   _diffrn_ambient_temperature ..... (K)        293 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C6 Check
PLAT334_ALERT_2_C Small <C-C> Benzene Dist.   C1       -C6       .       1.37 Ang.
PLAT342_ALERT_3_C Low Bond Precision on  C-C Bonds ...............    0.00922 Ang.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......     15.372 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      2.775 Check
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).          8 Note
```

Alert level G

```
PLAT005_ALERT_5_G No Embedded Refinement Details Found  in the CIF     Please Do !
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ...      0.500 Check
PLAT431_ALERT_2_G Short Inter HL..A Contact  I1       ..O3       .       3.14 Ang.
                                          1/2+x,y,1/2-z  =      6_656 Check
```

Author Response: The Checkcif software has a bug and cannot identify the halogen bond.

```
PLAT899_ALERT_4_G SHELXL-97  is Deprecated and Succeeded by SHELXL     2019/3 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600         12 Note
PLAT955_ALERT_1_G Reported (CIF) and Actual (FCF) Lmax Differ by .          1 Units
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          1 Info
```

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

4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
5 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
2 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

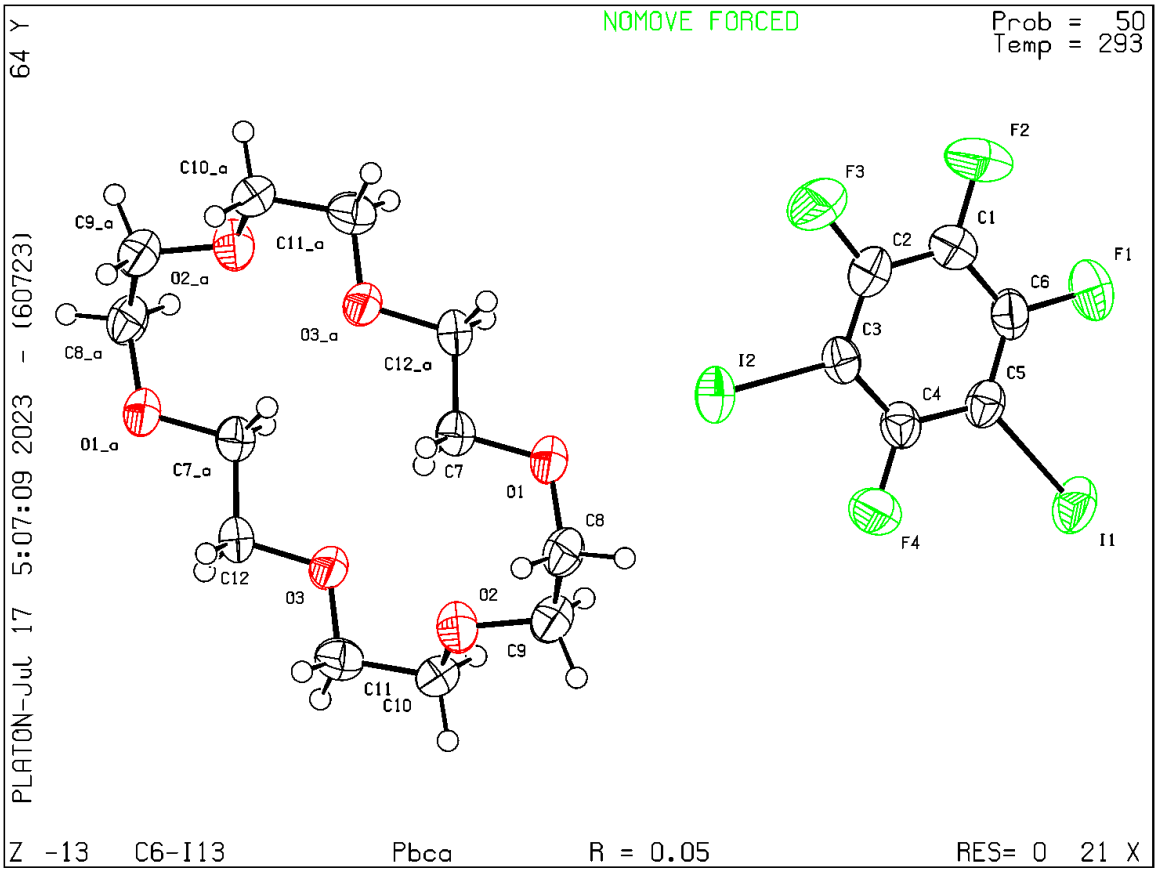
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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 06/07/2023; check.def file version of 30/06/2023

Datablock C6-I13 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C6-I14

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C6-I14

```
Bond precision:   C-C = 0.0033 A          Wavelength=0.71073

Cell:             a=14.2455(5)     b=10.6548(5)       c=15.5033(5)
                  alpha=90         beta=94.012(3)     gamma=90
Temperature:      296 K

                 Calculated                  Reported
Volume           2347.37(16)                 2347.37(16)
Space group      I 2/a                       I 1 2/a 1
Hall group       -I 2ya                      -I 2ya
Moiety formula   C12 H24 O6, C6 F4 I2        C12 H24 O6, C6 F4 I2
Sum formula      C18 H24 F4 I2 O6            C18 H24 F4 I2 O6
Mr               666.17                      666.17
Dx,g cm-3        1.885                       1.885
Z                4                           4
Mu (mm-1)        2.741                       2.741
F000             1288.0                      1288.0
F000'            1284.98
h,k,lmax         19,14,21                    19,14,20
Nref             3198                        2841
Tmin,Tmax        0.398,0.562                 0.656,1.000
Tmin'            0.368

Correction method= # Reported T Limits: Tmin=0.656 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.888           Theta(max)= 29.232

R(reflections)= 0.0268( 2552)                wR2(reflections)=
                                             0.0544( 2841)
S = 1.059                  Npar= 145
```

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

```
PLAT250_ALERT_2_C Large U3/U1 Ratio for <U(i,j)> Tensor(Resd    2)        2.1 Note
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      3.002 Check
```

Alert level G

```
PLAT012_ALERT_1_G N.O.K.   _shelx_res_checksum Found in CIF ......     Please Check
PLAT013_ALERT_1_G N.O.K.   _shelx_hkl_checksum Found in CIF ......     Please Check
PLAT164_ALERT_4_G Nr. of Refined C-H H-Atoms in Heavy-Atom Struct.          2 Note
PLAT431_ALERT_2_G Short Inter HL..A Contact  I1       ..O1       .       3.06 Ang.
                                                    x,y,z  =      1_555 Check
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).          4 Note
               1  1  0,   2  0  0,   0  1  1,   0  0  2,
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        347 Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File          1 Note
               0  2  0,
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........        4.5 Low
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........      2.066 Note
             Predicted wR2: Based on SigI**2  2.63 or SHELX Weight  5.14
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          4 Info
```

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

  2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
  4 ALERT type 2 Indicator that the structure model may be wrong or deficient
  3 ALERT type 3 Indicator that the structure quality may be low
  2 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

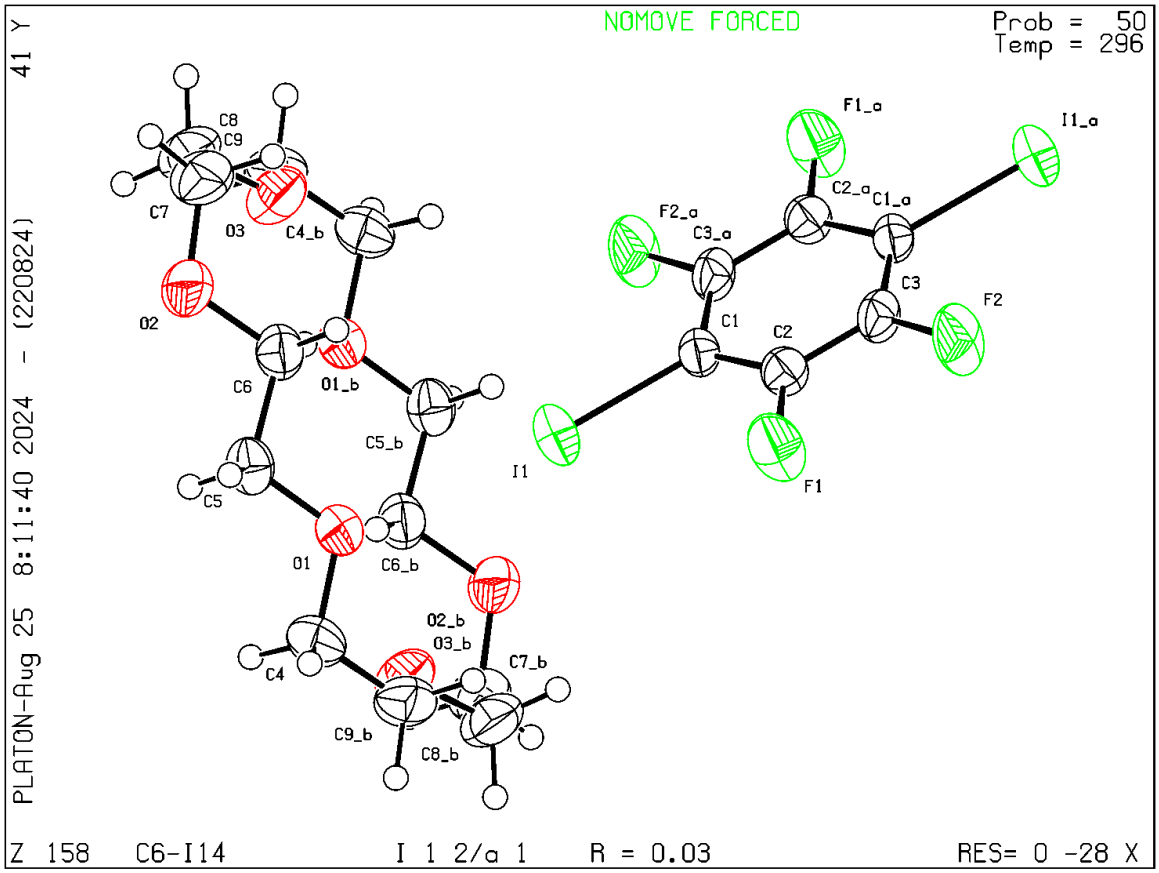
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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 22/08/2024; check.def file version of 21/08/2024

Datablock C6-I14 - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C6-I14-W

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C6-I14-W

Bond precision: C-C = 0.0113 A Wavelength=1.54184

Cell: a=7.5332(5) b=13.1800(8) c=14.1357(8)
alpha=105.855(5) beta=91.408(5) gamma=103.061(5)
Temperature: 295 K

	Calculated	Reported
Volume	1309.59(15)	1309.58(14)
Space group	P -1	P-1
Hall group	-P 1	?
Moiety formula	C12 H24 O6, C6 F4 I2, 3(H2 O)	?
Sum formula	C18 H30 F4 I2 O9	C18 H30 F4 I2 O9
Mr	720.22	720.22
Dx,g cm-3	1.827	1.826
Z	2	2
Mu (mm-1)	19.512	19.513
F000	704.0	704.0
F000'	705.35	
h,k,lmax	9,16,17	9,16,17
Nref	5195	5033
Tmin,Tmax	0.075,0.117	0.094,0.223
Tmin'	0.004	

Correction method= # Reported T Limits: Tmin=0.094 Tmax=0.223
AbsCorr = MULTI-SCAN

Data completeness= 0.969 Theta(max)= 72.360

R(reflections)= 0.0572(4062) wR2(reflections)= 0.1642(5033)

S = 1.026 Npar= 299

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 A

```
PLAT431_ALERT_2_A Short Inter HL..A Contact  I1       ..O5       .       2.96 Ang.
                                          1-x,1-y,1-z  =      2_666 Check
```

Author Response: The Checkcif software has a bug and cannot identify the halogen bond.

Alert level C

```
PLAT029_ALERT_3_C _diffrn_measured_fraction_theta_full value Low .      0.969 Why?
PLAT234_ALERT_4_C Large Hirshfeld Difference O3       --C4       .       0.20 Ang.
PLAT234_ALERT_4_C Large Hirshfeld Difference C11      --C12      .       0.16 Ang.
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of         C6 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         O2 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         O3 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         O4 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 ....        2.1 Note
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        O7      0.168 Check
PLAT334_ALERT_2_C Small <C-C> Benzene Dist.   C13      -C15_a    .       1.37 Ang.
PLAT342_ALERT_3_C Low Bond Precision on  C-C Bonds ..............    0.01125 Ang.
PLAT360_ALERT_2_C Short  C(sp3)-C(sp3) Bond  C11      - C12      .       1.43 Ang.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      2.145 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600          6 Report
PLAT971_ALERT_2_C Check Calcd Resid. Dens.  0.96Ang From I2              1.78 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens.  0.92Ang From I2              1.74 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens.  0.97Ang From I1              1.65 eA-3
PLAT972_ALERT_2_C Check Calcd Resid. Dens.  0.90Ang From I2             -2.03 eA-3
PLAT972_ALERT_2_C Check Calcd Resid. Dens.  0.93Ang From I1             -1.85 eA-3
PLAT972_ALERT_2_C Check Calcd Resid. Dens.  0.94Ang From I1             -1.77 eA-3
PLAT972_ALERT_2_C Check Calcd Resid. Dens.  0.77Ang From I2             -1.70 eA-3
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  1.05Ang From O4      .       0.43 eA-3
PLAT976_ALERT_2_C Check Calcd Resid. Dens.  0.71Ang From O7      .      -0.68 eA-3
PLAT976_ALERT_2_C Check Calcd Resid. Dens.  0.78Ang From O7      .      -0.59 eA-3
PLAT976_ALERT_2_C Check Calcd Resid. Dens.  0.72Ang From O7      .      -0.56 eA-3
```

Alert level G

```
PLAT005_ALERT_5_G No Embedded Refinement Details Found  in the CIF     Please Do !
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............          6 Report
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)      0.005 Degree
PLAT899_ALERT_4_G SHELXL-97  is Deprecated and Succeeded by SHELXL     2019/3 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        157 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          1 Info
```

1 **ALERT level A** = Most likely a serious problem - resolve or explain

```
  0 ALERT level B = A potentially serious problem, consider carefully
 28 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  6 ALERT level G = General information/check it is not something unexpected

  1 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
  4 ALERT type 3 Indicator that the structure quality may be low
  4 ALERT type 4 Improvement, methodology, query or suggestion
  2 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

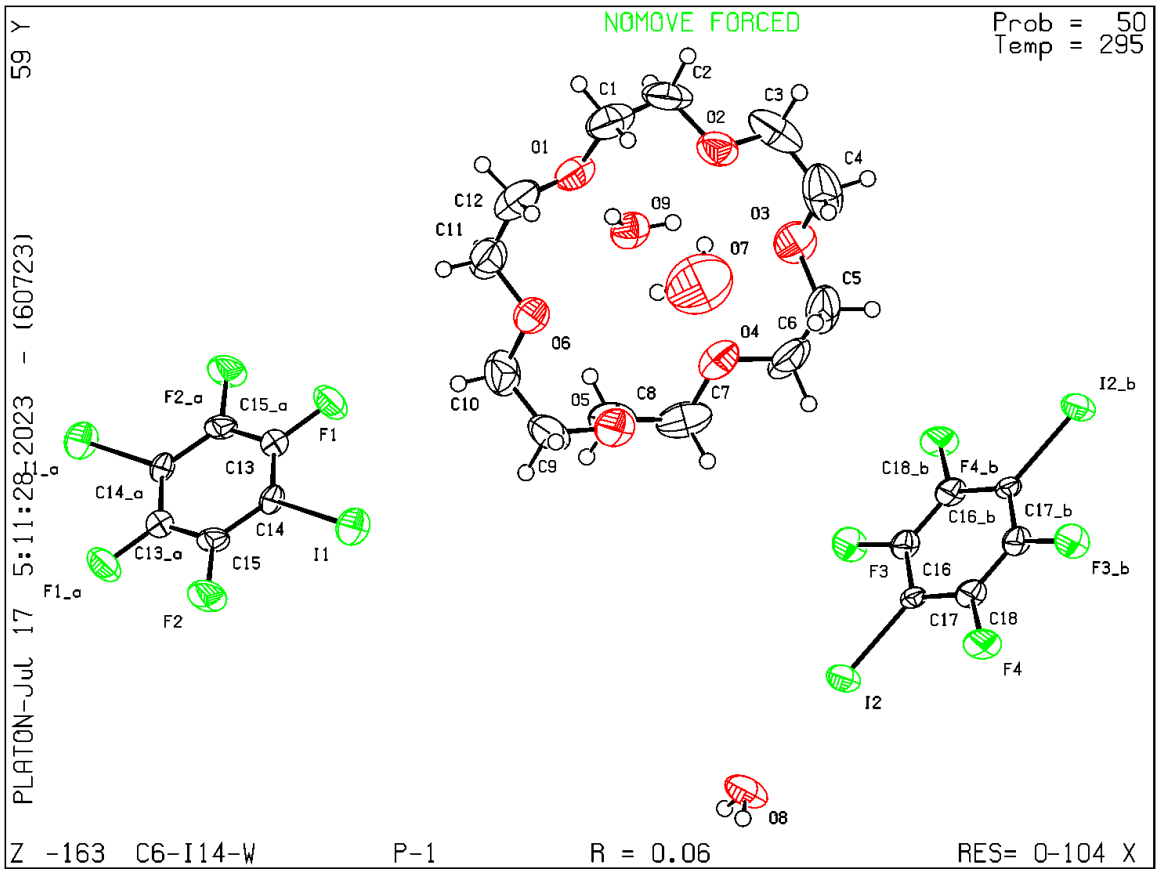
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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 06/07/2023; check.def file version of 30/06/2023

Datablock C6-I14-W - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) C6-I135

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: C6-I135

```
Bond precision:   C-C = 0.0162 A          Wavelength=0.71073

Cell:             a=8.5263(6)     b=25.6556(16)        c=8.8944(5)
                  alpha=90        beta=115.244(8)      gamma=90
Temperature:      293 K

                 Calculated                Reported
Volume           1759.8(2)                 1759.8(2)
Space group      P 21/c                    P 1 21/c 1
Hall group       -P 2ybc                   -P 2ybc
Moiety formula   C12 H24 O6, 2(C6 F3 I3)   2(C6 F3 I3), C12 H24 O6
Sum formula      C24 H24 F6 I6 O6          C24 H24 F6 I6 O6
Mr               1283.83                   1283.87
Dx,g cm-3        2.423                     2.423
Z                2                         2
Mu (mm-1)        5.361                     5.361
F000             1176.0                    1171.7
F000'            1170.96
h,k,lmax         10,31,10                  10,30,10
Nref             3277                      3266
Tmin,Tmax        0.169,0.324               0.173,1.000
Tmin'            0.141

Correction method= # Reported T Limits: Tmin=0.173 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.997           Theta(max)= 25.500

R(reflections)= 0.0629( 2510)                wR2(reflections)=
                                             0.1642( 3266)
S = 1.078                Npar= 189
```

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

```
PLAT042_ALERT_1_C Calc. and Reported MoietyFormula Strings  Differ     Please Check
PLAT342_ALERT_3_C Low Bond Precision on  C-C Bonds ..............    0.01622 Ang.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      9.320 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      2.098 Check
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).          8 Note
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600          3 Report
PLAT971_ALERT_2_C Check Calcd Resid. Dens.  0.85Ang From I3              2.07 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens.  1.02Ang From I2              1.55 eA-3
```

Alert level G

```
PLAT068_ALERT_1_G Reported F000 Differs from Calcd (or Missing)...     Please Check
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT  Unusually Large      16.74 Why ?
PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K)        293 Check
PLAT200_ALERT_1_G Reported   _diffrn_ambient_temperature ..... (K)        293 Check
PLAT431_ALERT_2_G Short Inter HL..A Contact  I1       ..O2       .       3.00 Ang.
                                          1-x,-1/2+y,1/2-z  =      2_645 Check
PLAT431_ALERT_2_G Short Inter HL..A Contact  I3       ..O1       .       3.00 Ang.
                                                x,y,z  =      1_555 Check
PLAT434_ALERT_2_G Short Inter HL..HL Contact I2       ..F3       .       3.17 Ang.
                                              1+x,y,1+z  =      1_656 Check
PLAT769_ALERT_4_G CIF Embedded Explicitly Supplied Scattering Data     Please Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........        2.3 Low
PLAT960_ALERT_3_G Number of Intensities with I  <   - 2*sig(I) ...          1 Check
PLAT967_ALERT_5_G Note: Two-Theta Cutoff Value in Embedded .res ..       51.0 Degree
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          0 Info
PLAT982_ALERT_1_G The  I-f'=   -0.4119 Deviates from  IT-value =      -0.4742 Check
PLAT983_ALERT_1_G The  I-f"=    1.8326 Deviates from  IT-Value =       1.8119 Check
```

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

   6 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   7 ALERT type 2 Indicator that the structure model may be wrong or deficient
   7 ALERT type 3 Indicator that the structure quality may be low
   1 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 06/07/2023; check.def file version of 30/06/2023

Datablock C6-I135 - ellipsoid plot