Supporting Information for

"Selective Adsorption of Chlorinated Volatile Organic Compounds Vapors by Microcrystalline 1D Coordination Polymers"

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CheckCIF files CCDC **2·TCM^{SC}**: 1497288; **3·TCM^{SC}**: 1497289; **1·TCE^{SC}**: 1497290; **1·MeCN^{SC}** 1507642**; 2·MeCN^{SC}** 1507643**; 3·MeCN^{SC}** 1507644**; 3·MeCN^{SC} (100K)** 1507645.

Material and Methods

All the chemicals were purchased from Sigma-Aldrich and used without further purification. X-ray powder diffraction measurements were carried out using a D8-Advance Bruker diffractometer using the reflection mode at room temperature and Bruker D2-Phaser diffractometer also using reflection mode. Single crystal X-ray diffraction experiments were carried out using a Bruker X8 Prospector APEX-II/CCD diffractometer equipped with a microfocusing mirror (Cu-*Kα* radiation, λ = 1.54178 Å). The low and high temperature experiments were carried out using the Oxford Cryosystems 700 Series Cryostream Cooler device. Single crystals of **1-3·TCM^{SC}** glued in a glass fiber were introduced in a sealed jar containing vapours of MeCN for single-crystal-to-single-crystal experiments overnight.

Solid liquid interface reactions: Microcrystalline solids of **1-3^{Pwd}** were obtained by preparing a suspension of **L** in MeCN and left stirring vigorously for 5 minutes. Then a methanol solution of ZnX_2 (where X = Cl, Br, and I) was added in the suspension and left stirring for 5 minutes (Figure S1). Yield is calculated by considering the formula [(**Y**-ZnX_2)·MeCN] (Y = **1-3**).

Preparation of **1^{Pwd}**: 100 mg of **L** (0.2249 mmol) were suspended in 4 mL of MeCN and left stirring vigorously. Then 30.6 mg of $ZnCl_2$ (1 eq.) dissolved in 1 mL of MeOH were added to the suspension and left for 5 minutes. From the reaction, 133 mg of white solid was filtered and collected.

Preparation of **2^{Pwd}**: 100 mg of **L** (0.2249 mmol) were suspended in 4 mL of MeCN and left stirring vigorously. Then 50.6 mg of $ZnBr_2$ (1 eq.) dissolved in 1 mL of MeOH were added to the suspension and left for 5 minutes. From the reaction, 150 mg of white solid was filtered and collected.

Preparation of **3^{Pwd}**: 100 mg of **L** (0.2249 mmol) were suspended in 4 mL of MeCN and left stirring vigorously. Then 71.8 mg of ZnI_2 (1 eq.) dissolved in 1 mL of MeOH were added to the suspension and left for 5 minutes. From the reaction, 174 mg of white solid was filtered and collected.

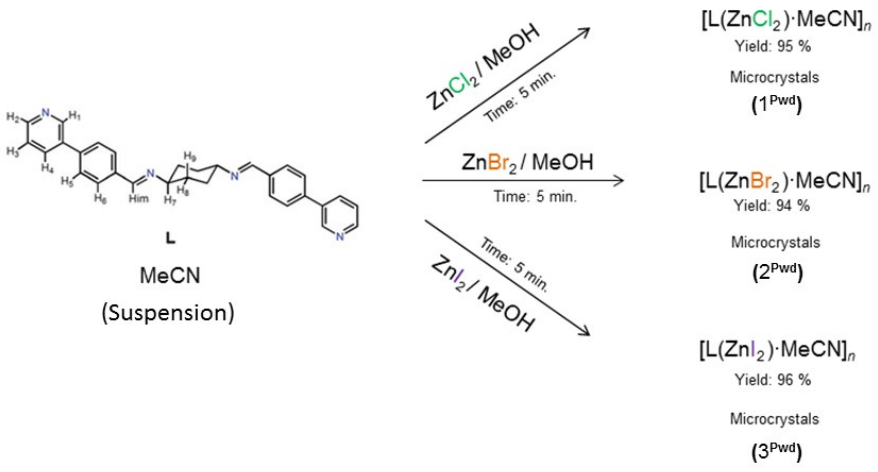


Figure S1. Solid-liquid interface synthesis of **1-3^{Pwd}**.

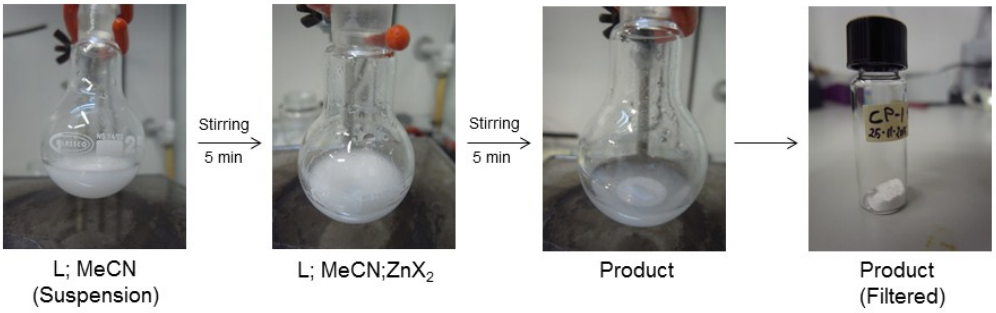


Figure S2. Several pictures taken during the small-scale synthesis of **1^{Pwd}** coordination polymer using the solid-liquid interface reaction method. Where X in ZnX_2 is Cl, Br or I. In this case X = Cl.

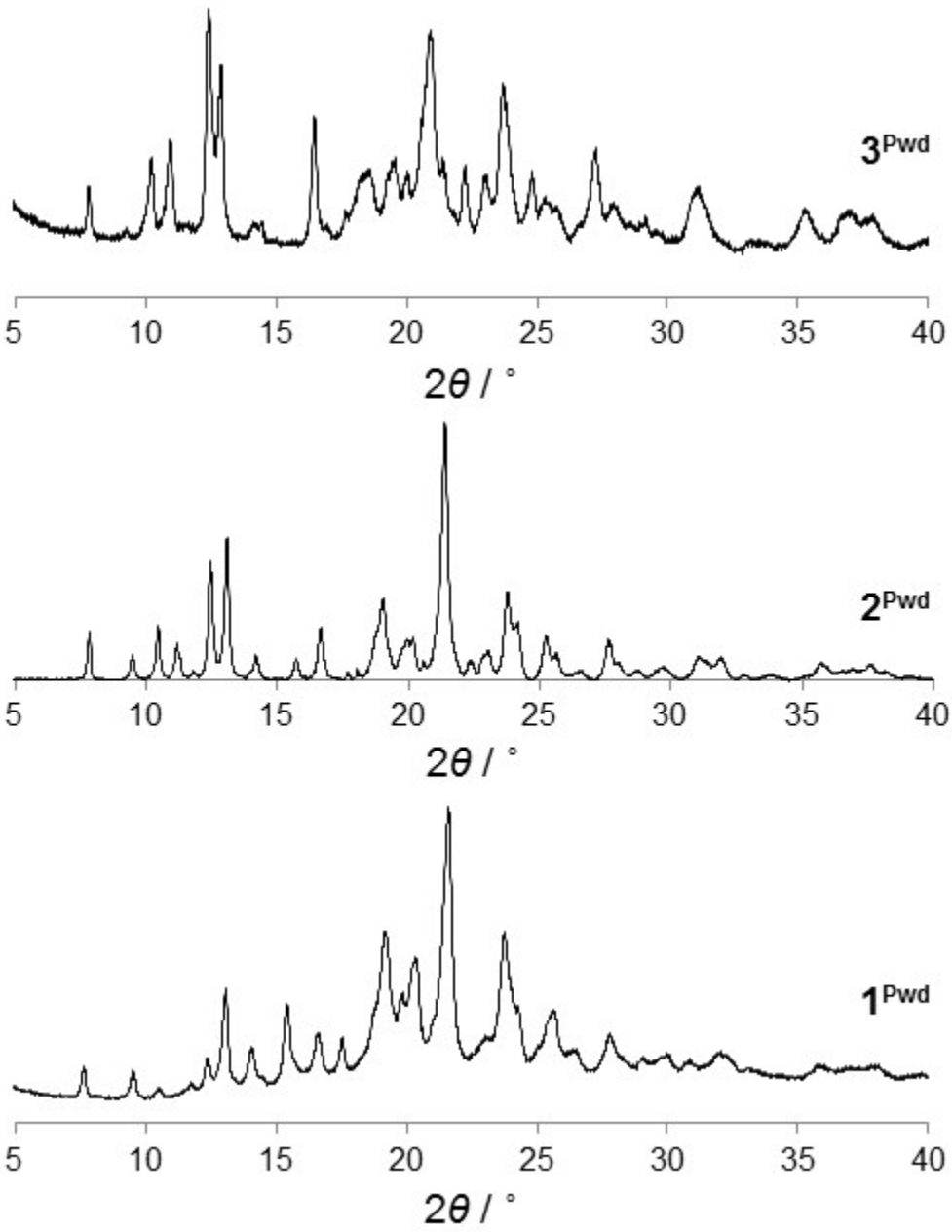


Figure S3. Experimental XRPD obtained after adding a MeOH solution of ZnX_2 (where X = Cl, Br and I) into a suspension of **L** in MeCN.

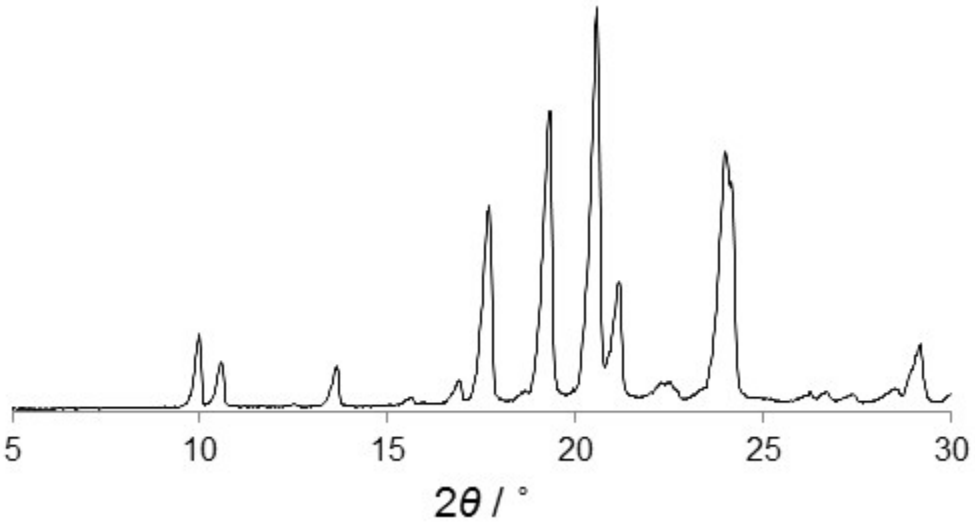


Figure S4. Experimental XRPD corresponding to **L**.

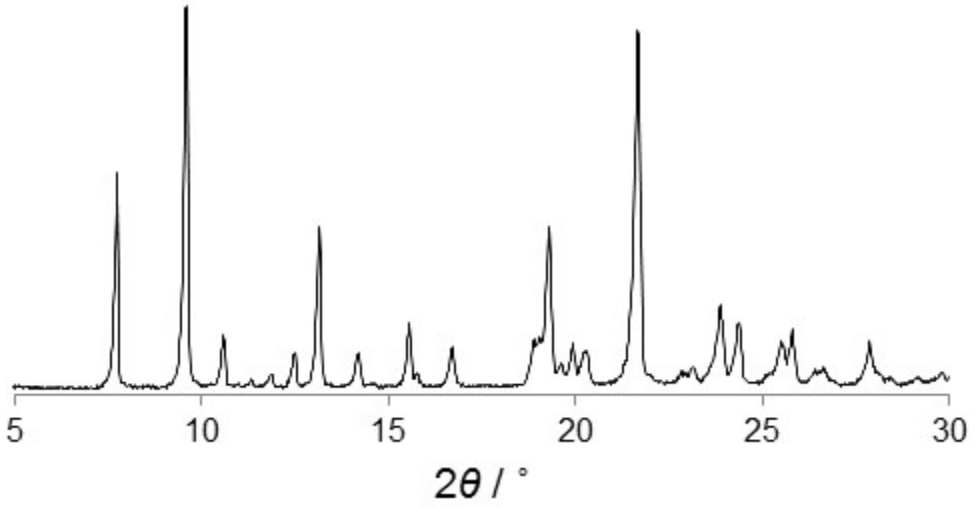


Figure S5. Experimental XRPD corresponding to **1·TCM** obtained by fast precipitation (*i.e.*, instant synthesis) and measured at 300 K. See *Chem. Commun*., 2015, **51**, 12357.

Since the solid liquid interface intrinsically gives microcrystalline materials (*i.e.*, the starting materials are powders already) single crystal X-ray diffraction cannot be carried out. However, one way to know if the product obtained is an isostructural coordination polymer to **1-3·TCM^{SC}**, we set up gas solid reactions in order to include MeCN in single crystals of **1-3·TCM^{SC}** following a SC-to-SC guest exchange process where the original TCM guest molecules are replaced by MeCN as shown in Figure S6).

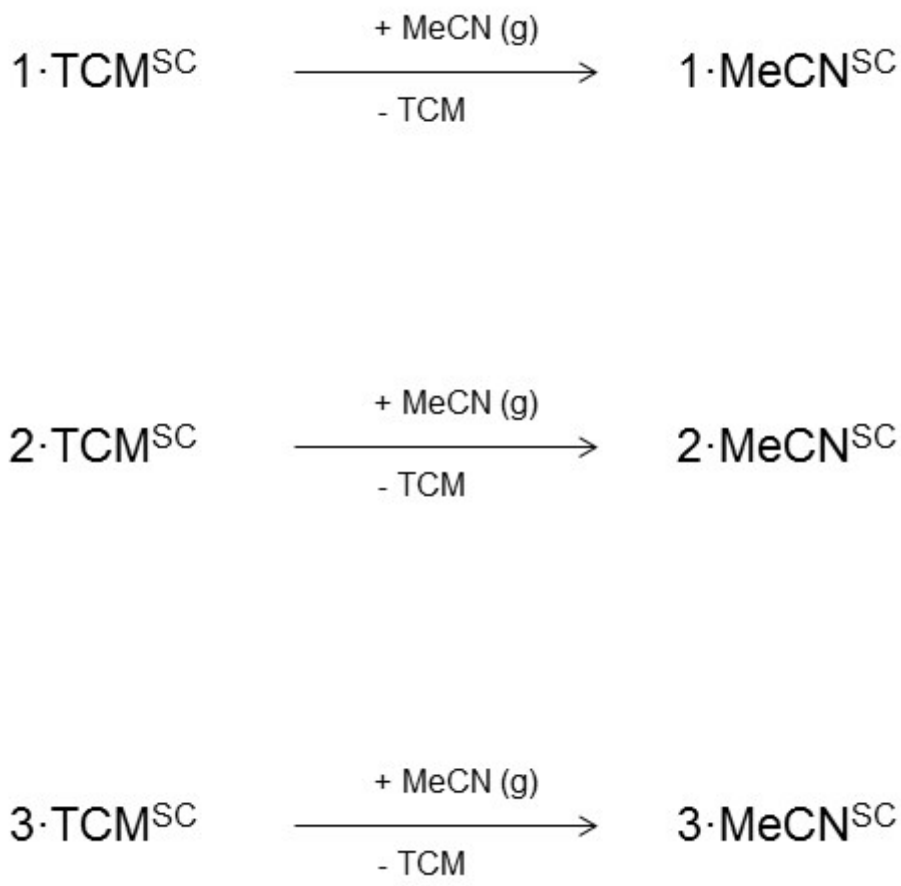
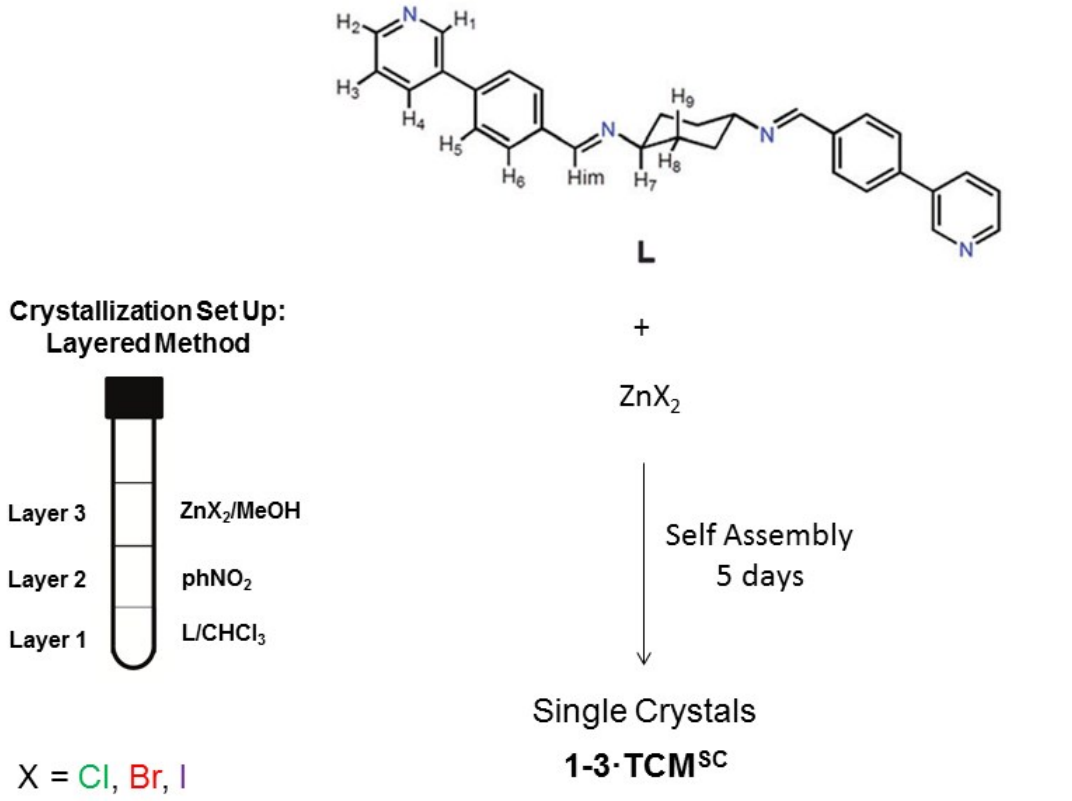


Figure S6. SC-to-SC guest exchange process where the original TCM guest molecules are replaced by MeCN.

In order to perform the experiments shown in Figure S6, single crystals of the bromide and iodide which are isostructural to **1·TCM^{SC}** were set up for crystallization following the triple layer method described in *Chem. Commun*., 2015, **51**, 12357.



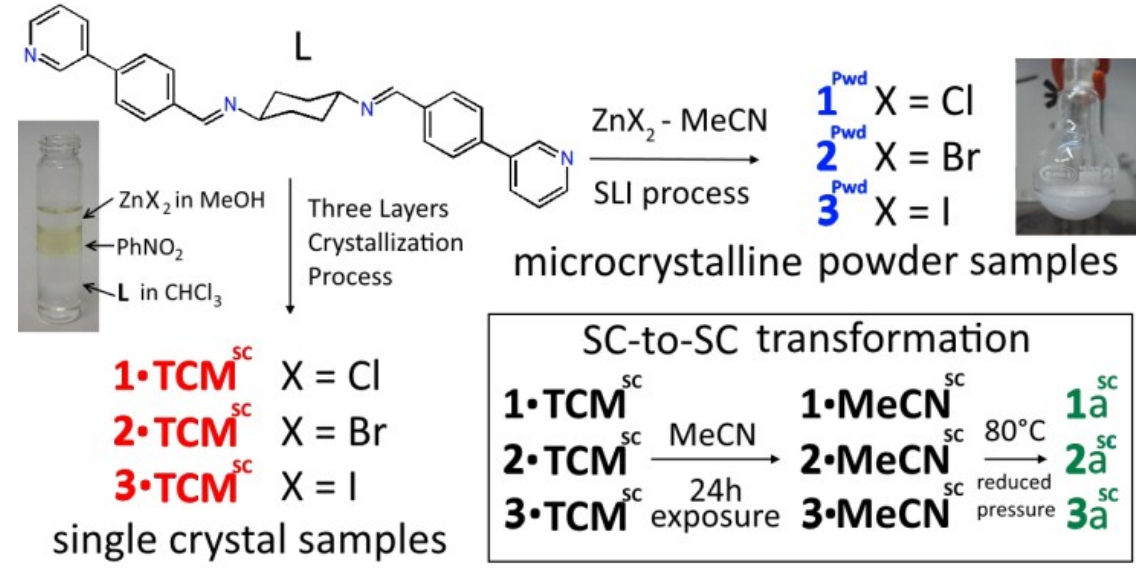


Figure S7. (Top) Single crystal preparation of **1-3·TCM^{SC}**. (Bottom) Summary explaining the two methods (i.e., slow crystallization (layering) and fast crystallization (solid liquid interface reaction) carried out in this work. Inset showing the SC-to-SC guest exchange reactions done.

Synthesis of crystals 2·TCM with ligand L.

Single crystals of **2·TCM^{SC}** were prepared out using the triple layering method by treating **L** (40 mg) with $ZnBr_2$ (20.27 mg) following the procedure used to crystallize **1·TCM^{SC}**. The bottom layer was a chloroform solution of **L** onto which a layer of nitrobenzene (middle layer) was deposited dropwise. The upper layer consists on a methanol solution of $ZnBr_2$. Prism-like single crystals were obtained after one week. The crystal structure of **2·TCM^{SC}** has been solved using single crystal X-ray diffraction. The coordination polymer crystallizes in the orthorhombic system in the *Pnna* space group and have similar lattice parameters (Table S1), and therefore are isostructural to the chloride polymer **1·TCM^{SC}**. The crystal structure of **2·TCM^{SC}** is shown in Figure S8.

Table S1. Crystallographic data regarding the lattice parameters of **2·TCM^{SC}**.

Crystal data for	**2·TCM^{SC}**
Empirical formula	$C_{30.75}\,H_{28.75}\,N_4\,Br_2Cl_2Zn_1$
Formula weight	750.42 g/mol
Temperature	100 K
Crystal system	Orthorhombic
Space group	*Pnna*
Unit cell dimensions	a = 14.6557(10) Å α = 90.00 °
	b = 22.6968(16) Å β = 90.00 °
	c = 10.1625(8)Å γ = 90.00 °
Volume	3380.4(4)Å^3
Z	4
R- Factor (%)	6.19
Density (calculated)	1.474 g/cm^3

The CCDC number for **2·TCM^{SC}: 1497288**.

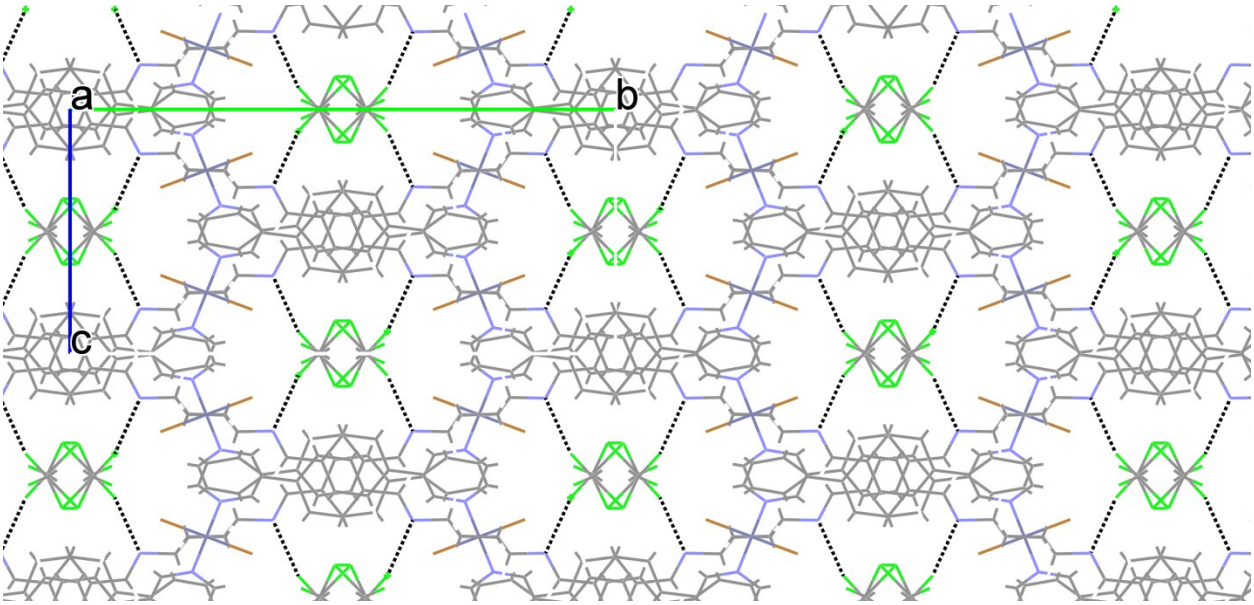


Figure S8. Single crystal data of **2·TCM**^{SC} measured at 100K.

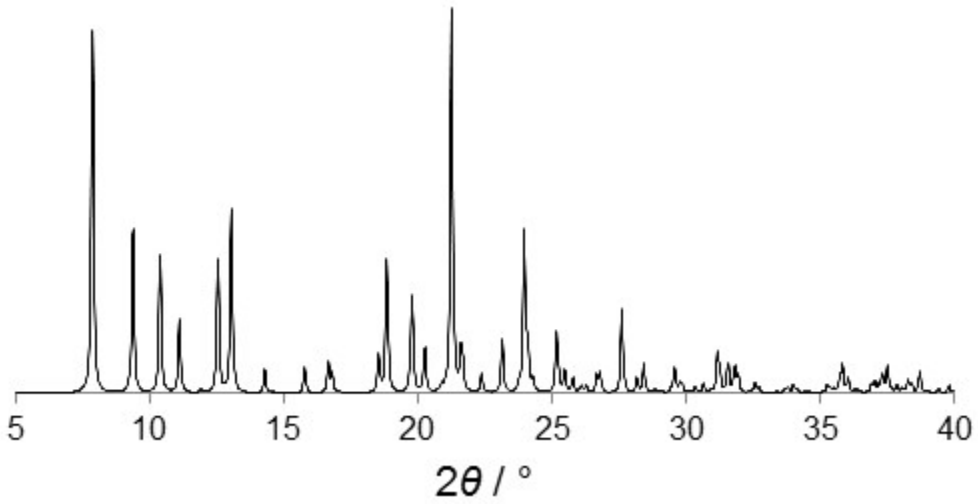


Figure S9. Simulated XRDP obtained from the single crystal **2·TCM**^{SC}.

Synthesis of crystals 3·TCM^{SC} with ligand L.

Single crystals of **3·TCM^{SC}** were prepared out using the triple layering method by treating **L** (40 mg) with ZnI_2 (26.56 mg) following the procedure used to crystallize **1·TCM^{SC}**. The bottom layer was a chloroform solution of **L** onto which a layer of nitrobenzene (middle layer) was deposited dropwise. The upper layer was a methanol solution of ZnI_2. Prism-like single crystals were obtained after one week. The crystal structure of **3·TCM^{SC}** has been solved using single crystal X-ray diffraction. The coordination polymer crystallizes in the orthorhombic system in the *Pnna* space group and have similar lattice parameters (Table S2), and therefore are isostructural to the chloride polymers **1·TCM^{SC}** and **2·TCM^{SC}**. The crystal structure of **3·TCM^{SC}** is shown in Figure S10.

Table S2. Crystallographic data regarding the lattice parameters of **3·TCM^{SC}**.

Crystal data for	**3·TCM^{SC}**	
Empirical formula	$C_{30.76}H_{28.76}N_4I_2Cl_{2.5}Zn_1$	
Formula weight	862.12 g/mol	
Temperature	100 K	
Crystal system	Orthorhombic	
Space group	*Pnna*	
Unit cell dimensions	a = 14.4863(13) Å	α = 90.00 °
	b = 22.1482(19)Å	β = 90 °
	c = 11.0890(11)Å	γ = 90.00 °
Volume	3557.9(6) Å^3	
Z	4	
R- Factor (%)	5.58	
Density (calculated)	1.609 g/cm^3	

The CCDC number for **3·TCM^{SC}: 1497289**.

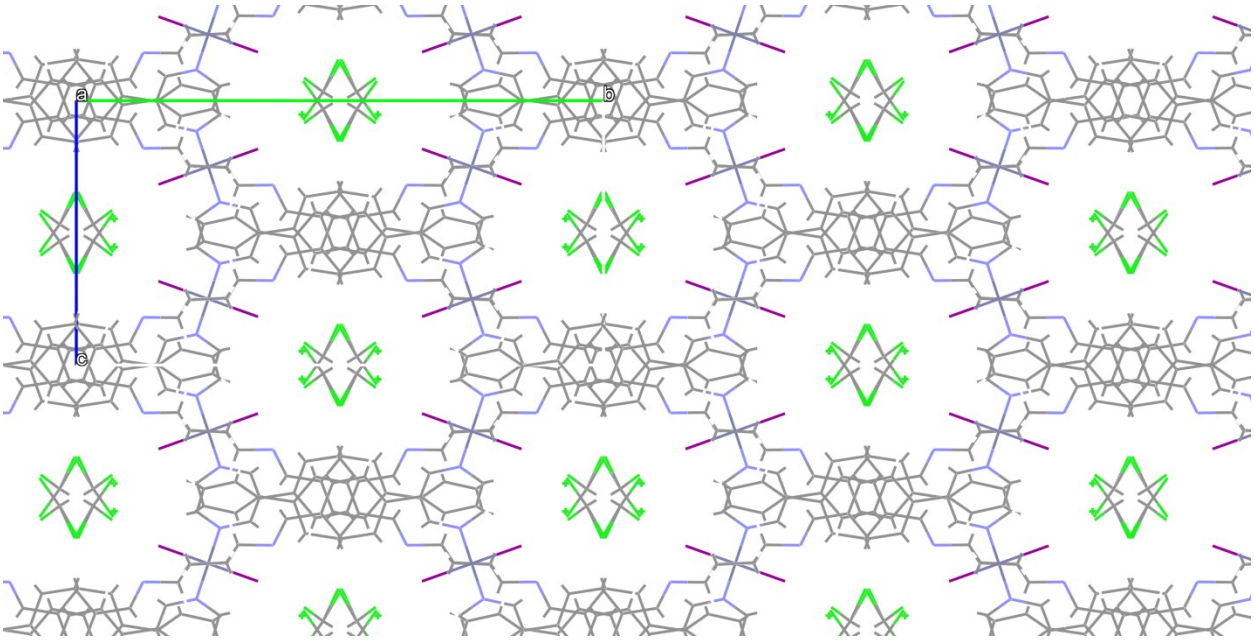


Figure S10. Single crystal data of **3·TCM^{SC}** viewed along the *a*-axis.

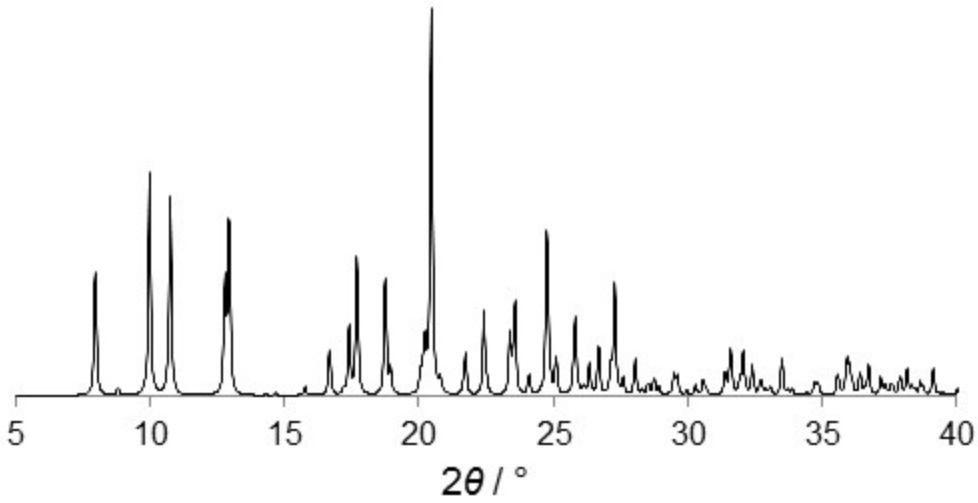


Figure S11. Simulated XRPD of **3·TCM^{SC}.**

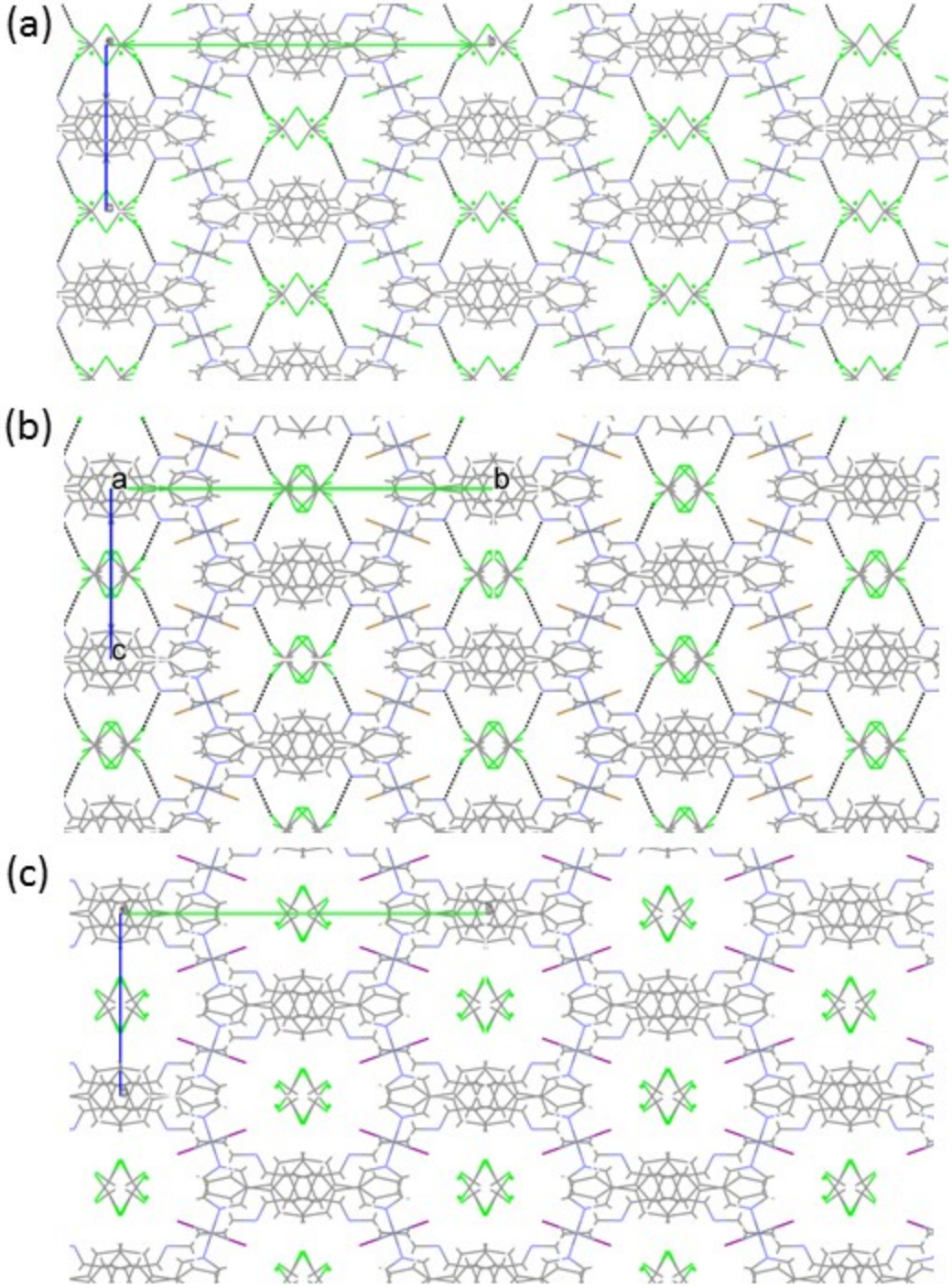


Figure S12. Crystal packing of **1-3·TCM**^{SC} showing the hydrogen bonding interactions between TCM and the iminic N atoms as dashed black lines.

Single crystal structure of 1·MeCN^{SC}.

Single crystal structure of **1·MeCN^{SC}** after the guest exchange reaction in **1·TCM^{SC}**, where TCM is replaced by MeCN to give **1·TCM^{SC}**, the lattice parameters are: a = 14.7485(14) Å; b = 22.317(2) Å; c = 10.4570(11) Å, $\alpha = \beta = \gamma = 90.00^{\circ}$ V = 3441.8 (6) Å^3. The Bravais lattice remains Orthorhombic and the space group is maintained to *Pnna*. The data collection was carried out at 300 K. For further crystallographic information see the attached checkCIF files at the end of document and the CIF files. The CCDC number for **1·MeCN^{SC}: 1507642**.

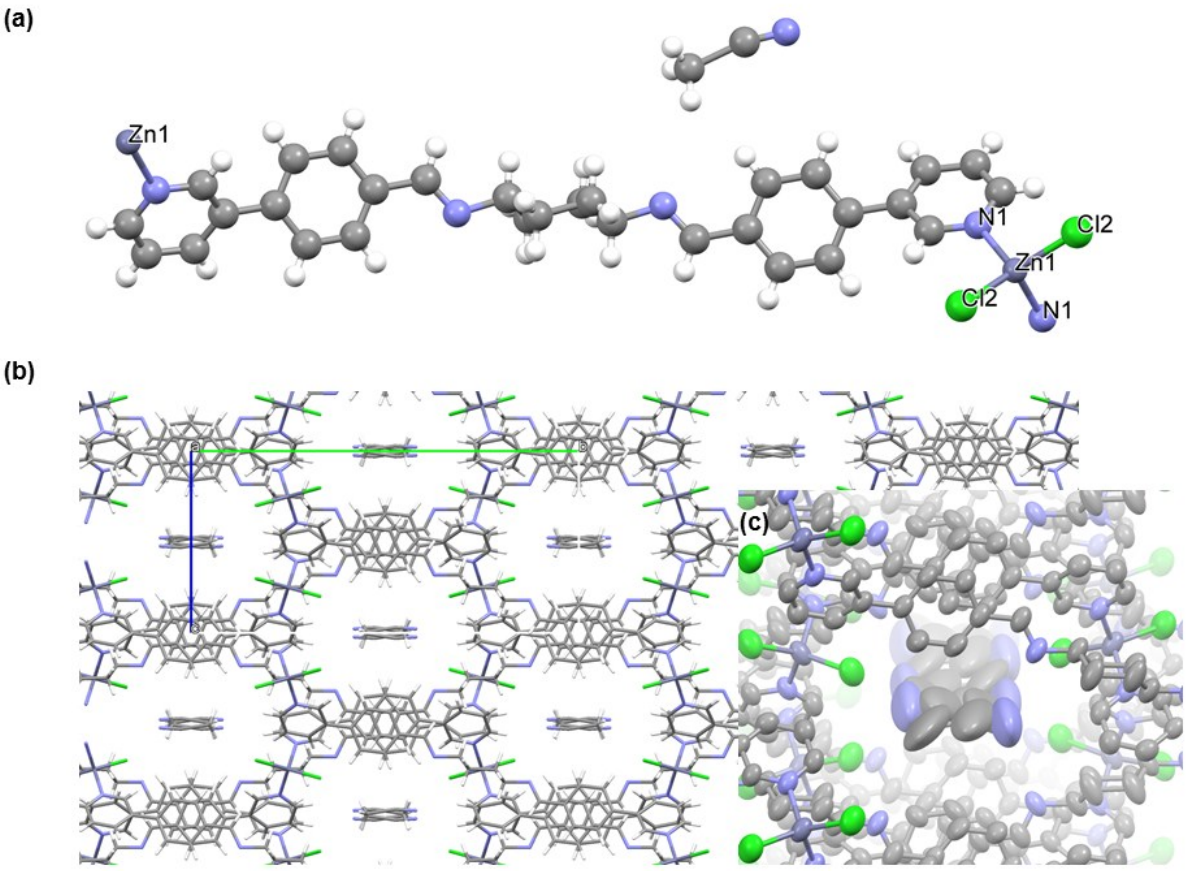


Figure S13. Crystal structure of **1·MeCN^{SC}** obtained after SC-to-SC according to Figure S6. Inset: view of the MeCN guest molecule with the displacement ellipsoids showing that the vibration is along the channel direction.

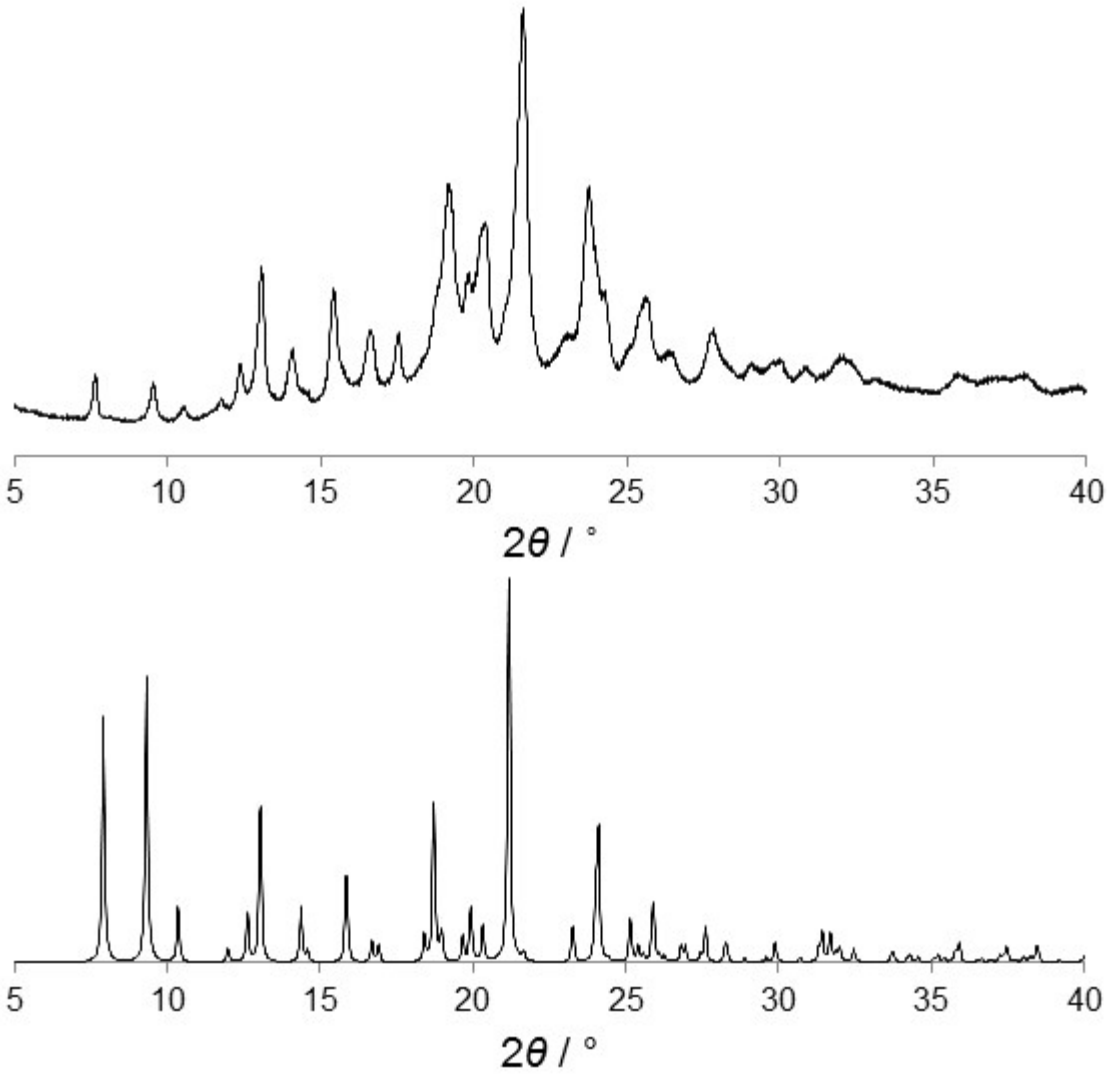


Figure S14. Experimental SLI of $ZnCl_2$ in MeCN. Simulated after guest exchange of **1·TCM**^{SC} for MeCN to give **1·MeCN**^{SC}.

Single crystal structure of 2·MeCN^{SC}.

Single crystal structure of **2·MeCN^{SC}** after the guest exchange reaction in **2·TCM^{SC}**, where TCM is replaced by MeCN to give **2·TCM^{SC}**, the lattice parameters are: a = 14.8955(11) Å; b = 22.5127(17) Å; c = 10.2599(7) Å, $\alpha = \beta = \gamma$ = 90.00 ° V = 3440.5 (4) Å^3. The Bravais lattice remains Orthorhombic and the space group is maintained to *Pnna*. The data collection was carried out at 300 K. For further crystallographic information see the attached checkCIF files at the end of document and the CIF files. The CCDC number for **2·MeCN^{SC}: 1507643**

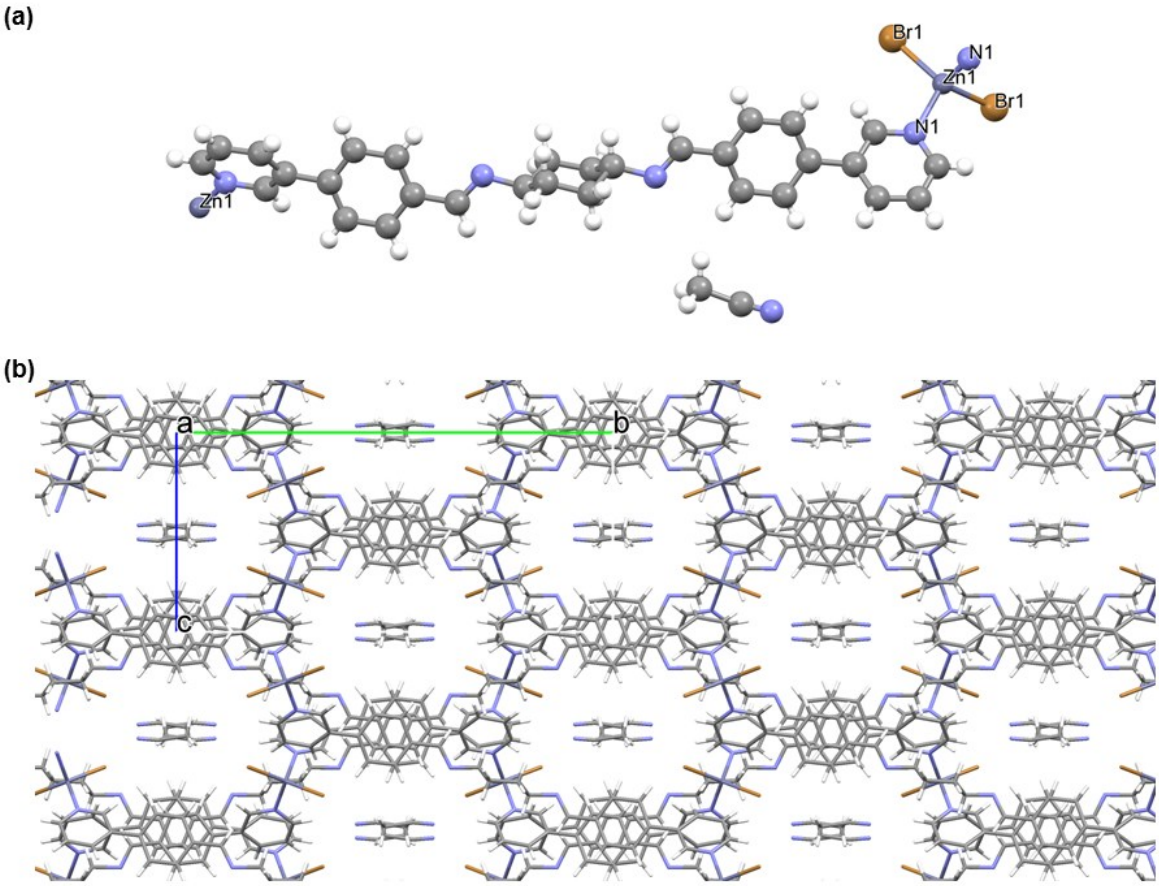


Figure S15. Crystal structure of **2·MeCN^{SC}** obtained after SC-to-SC according to Figure S6.

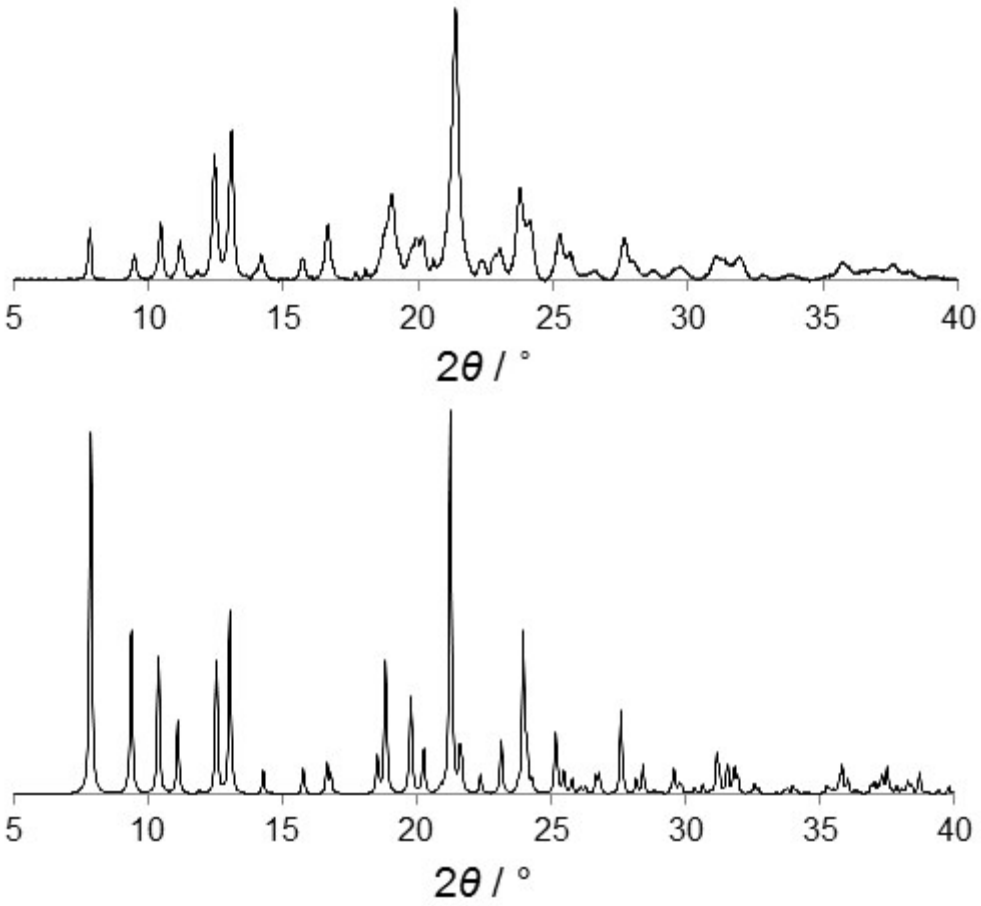


Figure S16. Experimental SLI of $ZnBr_2$ in MeCN. Simulated after guest exchange of **2·TCM^{SC}** for MeCN to give **2·MeCN^{SC}**.

Single crystal structure of 3·MeCN^{SC}.

Single crystal structure of **3·MeCN^{SC}** after the guest exchange reaction in **3·TCM^{SC}**, where TCM is replaced by MeCN to give **3·TCM^{SC}**, the lattice parameters are: a = 15.077(3) Å; b = 22.570(4) Å; c = 10.412(2) Å, $\alpha = \beta = \gamma = 90.00$ ° V = 3543.2(11) Å^3 . The Bravais lattice remains Orthorhombic and the space group is maintained to *Pnna*. The data collection was carried out at 300 K. . For further crystallographic information see the attached checkCIF files at the end of document and the CIF files. The CCDC number for **3·MeCN^{SC}: 1507644**

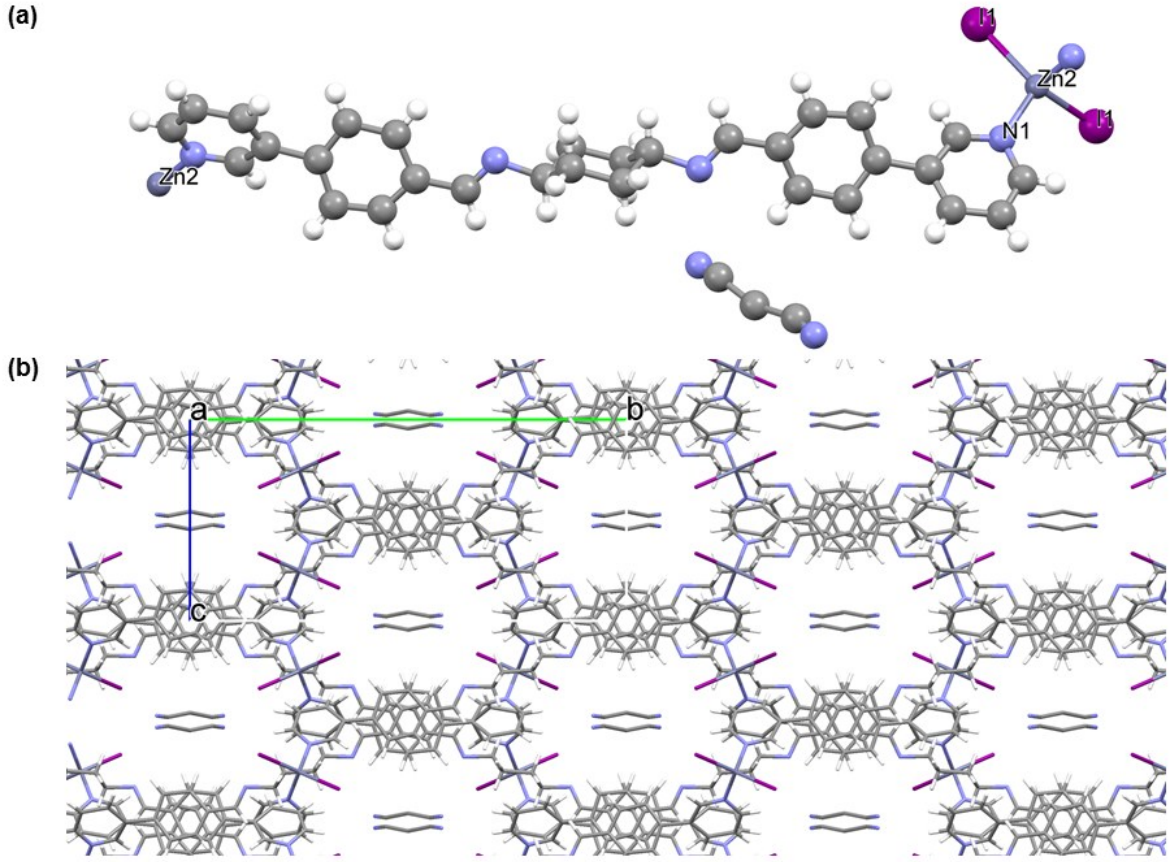


Figure S17. Crystal structure of **3·MeCN**^{SC} obtained after SC-to-SC according to Figure S6.

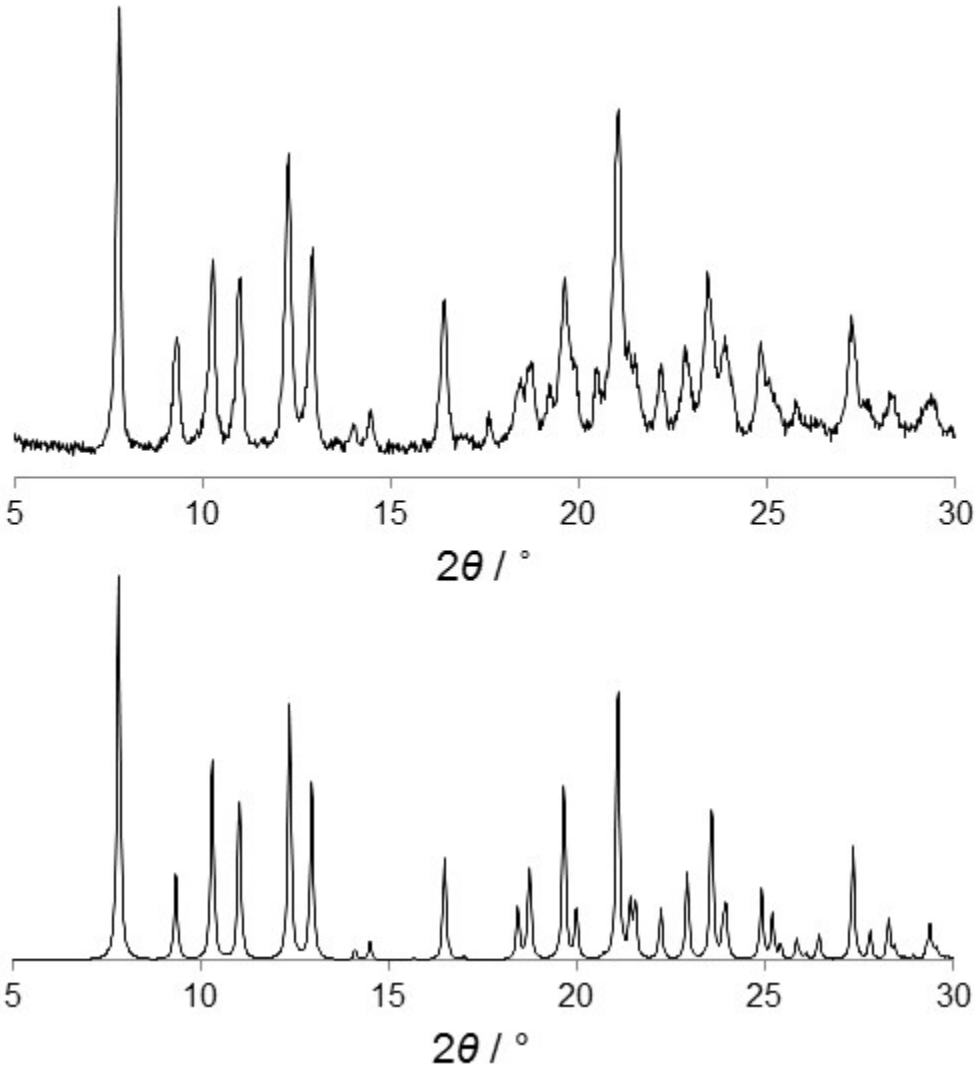


Figure S18. (Top) Experimental XRPD pattern obtained using the solid liquid interface method by adding ZnI_2 in MeOH to a suspension of **L** in MeCN vigorously stirred. (Bottom) Simulated XRPD pattern obtained after a SC-to-SC guest exchange of **3·TCM**^{SC} for MeCN to give **3·MeCN**^{SC}. The good match between both XRPD patterns confirms that MeCN replaced the original TCM guest molecule.

Gas solid experiments using microcrystalline sample of 3·MeCN^{Pwd} obtained by the solid liquid interface method.

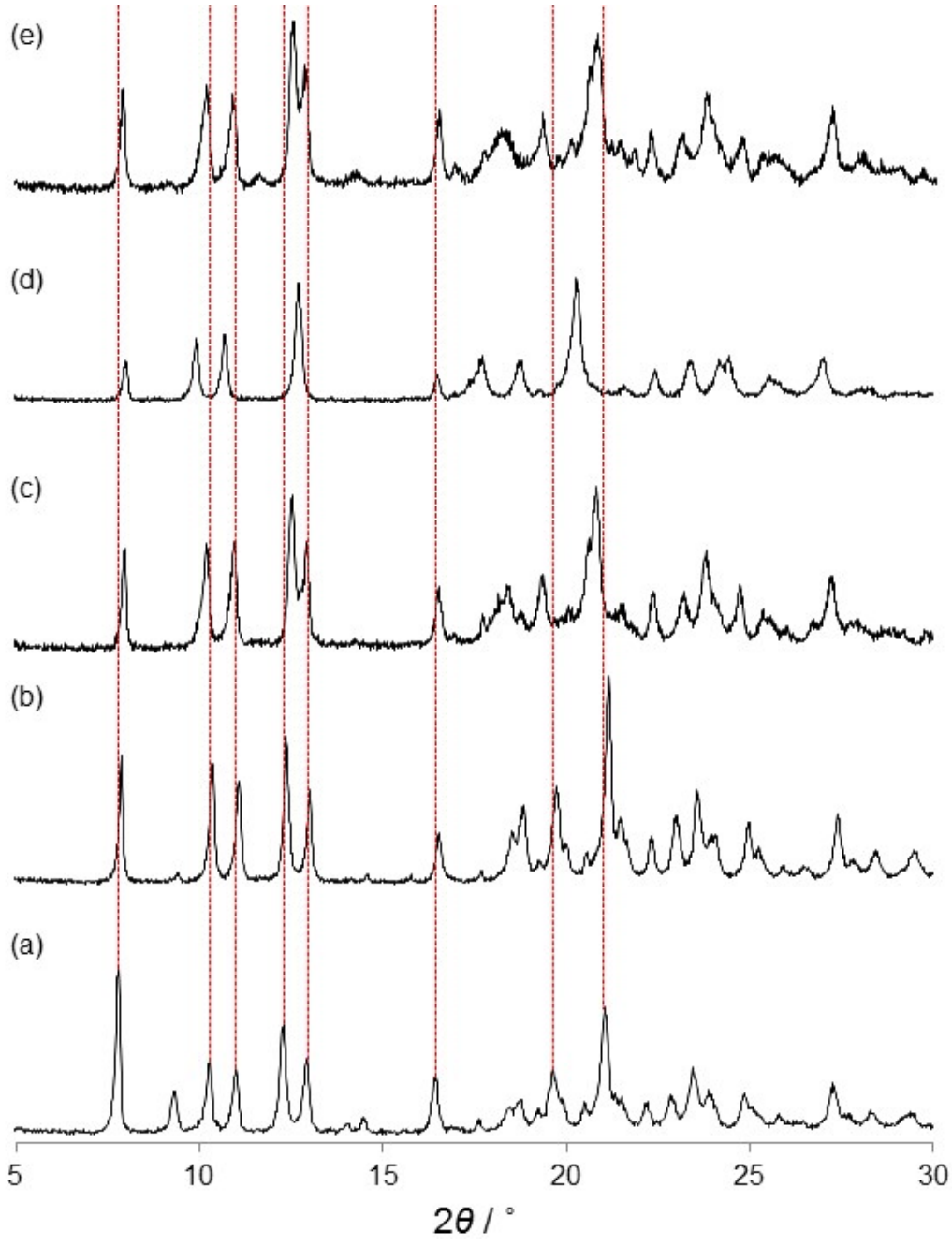


Figure S19. Experimental XRPD diffraction patterns measured at room temperature of **3·MeCN^{Pwd}** (a) exposed to DCE to give **3·DCE^{Pwd}**(b), TCE to give **3·TCE^{Pwd}** (c), TCM to give **3·TCM^{Pwd}** (d) and DCM to give **3·DCM^{Pwd}** (e). It is clear that there is an anisotropic peak shift corroborating the structural adaptation to the different guest molecules and that the structure is maintained after the guest exchange.

Single-crystal-to-single-crystal reaction to include TCE in single crystals of 1·TCM^{SC}.

The crystal structure corresponding to the SCSC guest exchange reaction by exposing a single crystal of **1·TCM^{SC}** to vapors of 1,1,2-trichloroethylene (TCE) to give the **1·TCE^{SC}** coordination polymer.

Table S3. Crystallographic data regarding the lattice parameters of **1·TCE^{SC}**.

Crystal data for	**1·TCE^{SC}**	
Empirical formula	C_{32} H_{29} $N_4Cl_5Zn_1$	
Formula weight	712.22 g/mol	
Temperature	200 K	
Crystal system	Orthorhombic	
Space group	*Pnna*	
Unit cell dimensions	a = 14.7372(11) Å	α = 90.00 °
	b = 22.7121(16) Å	β = 90 °
	c = 9.9658(8) Å	γ = 90.00 °
Volume	3335.7(4)Å^3	
Z	4	
R- Factor (%)	8.39	
Density (calculated)	1.418 g/cm3	

The CCDC number for **1·TCE^{SC}**: **1497290**.

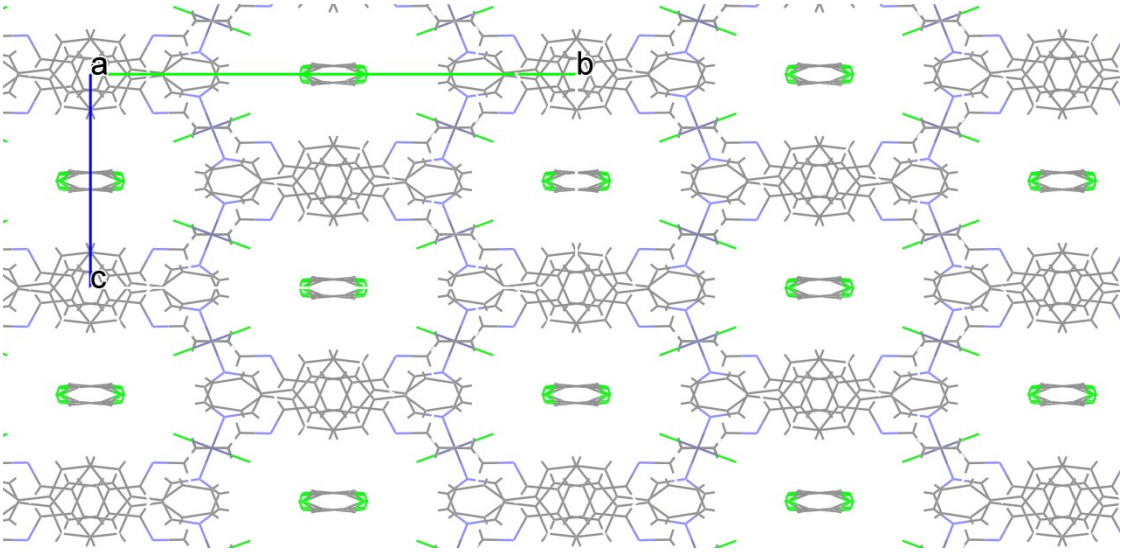


Figure S20. Crystal packing of **1·TCE^{SC}** showing the inclusion of TCE guest molecules in the 1D channels.

NMR analysis

A certain weighted amount of the powder samples (ca. 3-5 mg) is introduced into a NMR tube. 500 μL of DMSO-d6 and 5μL of a 4.6mM solution of nitromethane in DMSO-d_6 are added. The sample is then heated till complete dissolution to ensure complete solvent release from the coordination polymer. Spectra were recorded at 305 K on a Bruker Avance 400 (400 MHz, SW= 14 ppm, NS = 16). In order to check the effect of heating on the resulting amount of volatile solvents detected, a control experiment was also performed avoiding the heating step. No significant difference was spotted, thus indicating that DMSO molecules can disrupt the CP structure and release all the solvent guest molecules at r.t.

Equation for the determination of the *R* factor by ^{1}H-NMR analysis. *R* is defined as the molar ratio between the solvent guest molecules and **L**-ZnX_2 units (X = Cl, Br or I).

$$R_{solvent\ guest} = \frac{moles\ of\ solvent\ guest}{moles\ of\ L \cdot ZnX_2\ units} =$$

$$= \frac{Volume \cdot [solvent\ guest]_{NMR}}{(mg_{tot} - MW_{solvent\ guest} \cdot Volume \cdot [solvent\ guest]_{NMR})/MF_{L \cdot ZnX_2}}$$

Volume =; volume of the DMSO-d6 used (500 μL + 5 μL of internal standard)
$[\text{solvent guest}]_{NMR}$ = concentration of the guest solvent as measured by 1H-NMR by comparison of the integral value of an internal standard (Nitromethane)
mg_{tot} = total amount of material weighted
$MW_{\text{solvent guest}}$ = molecular weight of the solvent guest
$MF_{\text{L-ZnX2 units}}$ = formula weight of the L-ZnX_2 unit

Equation for the determination of the *S* factor. *S* is defined as the ration between the *R* factors of two given solvents.

$$S_{solvent1/solvent2} = \frac{R_{solvent1}}{R_{solvent2}}$$

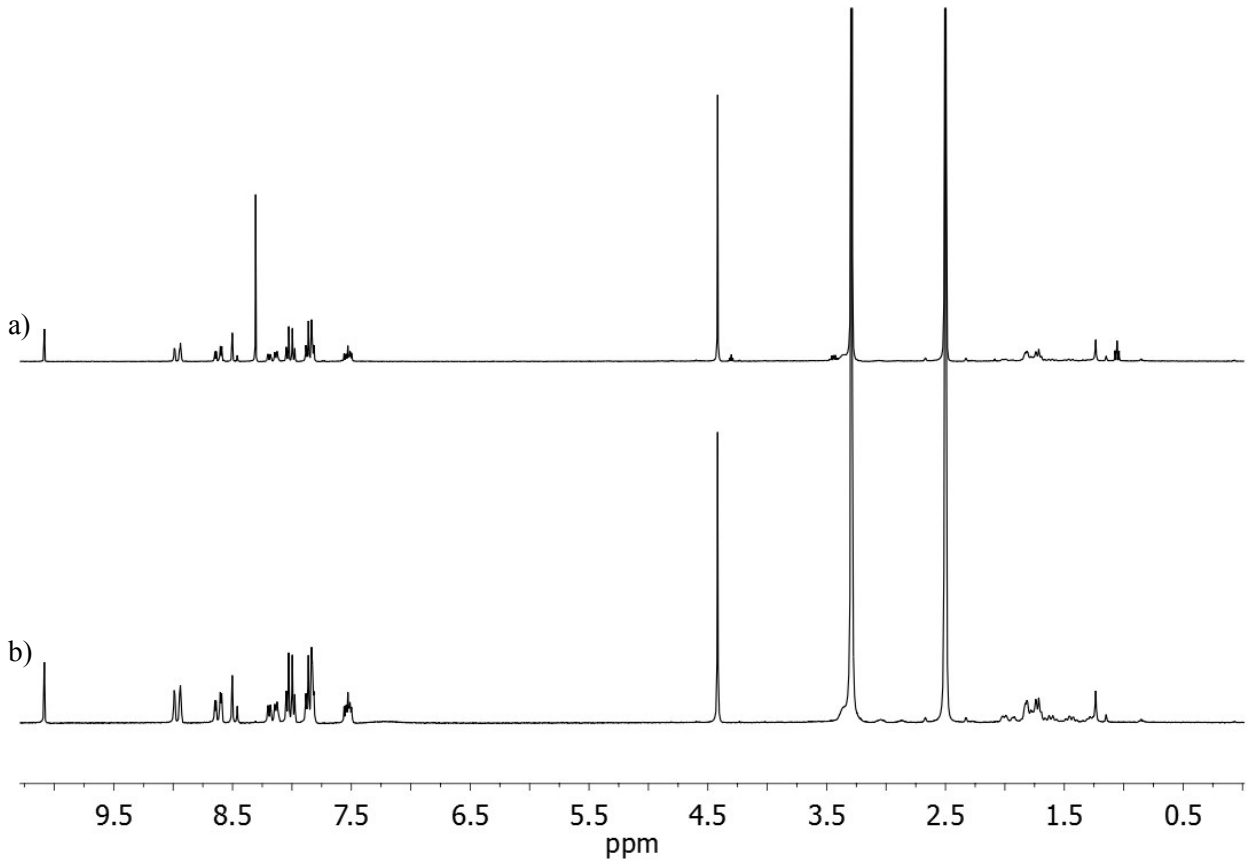


Figure S21. ^{1}H-NMR characterization in DMSO-d_6 of sample **3^{Pwd}** exposed to TCM vapors for 24h a) and b) after thermal treatment under reduced pressure (80°C for 10 h).

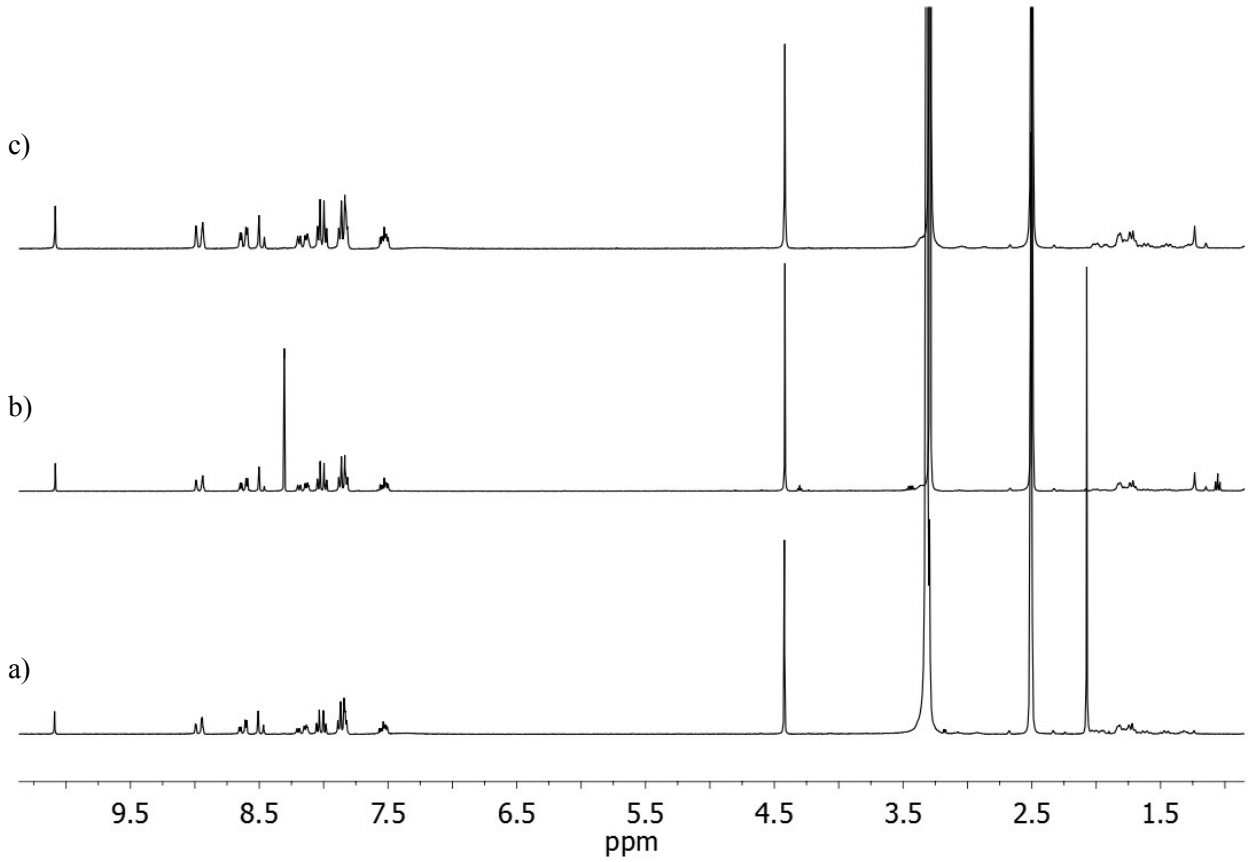


Figure S22. a) ^{1}H-NMR characterization in DMSO-d6 of **3^{Pwd}**; b) after exposure for 24h to TCM vapors and c) after thermal treatment under reduced pressure (80°C for 10 h).

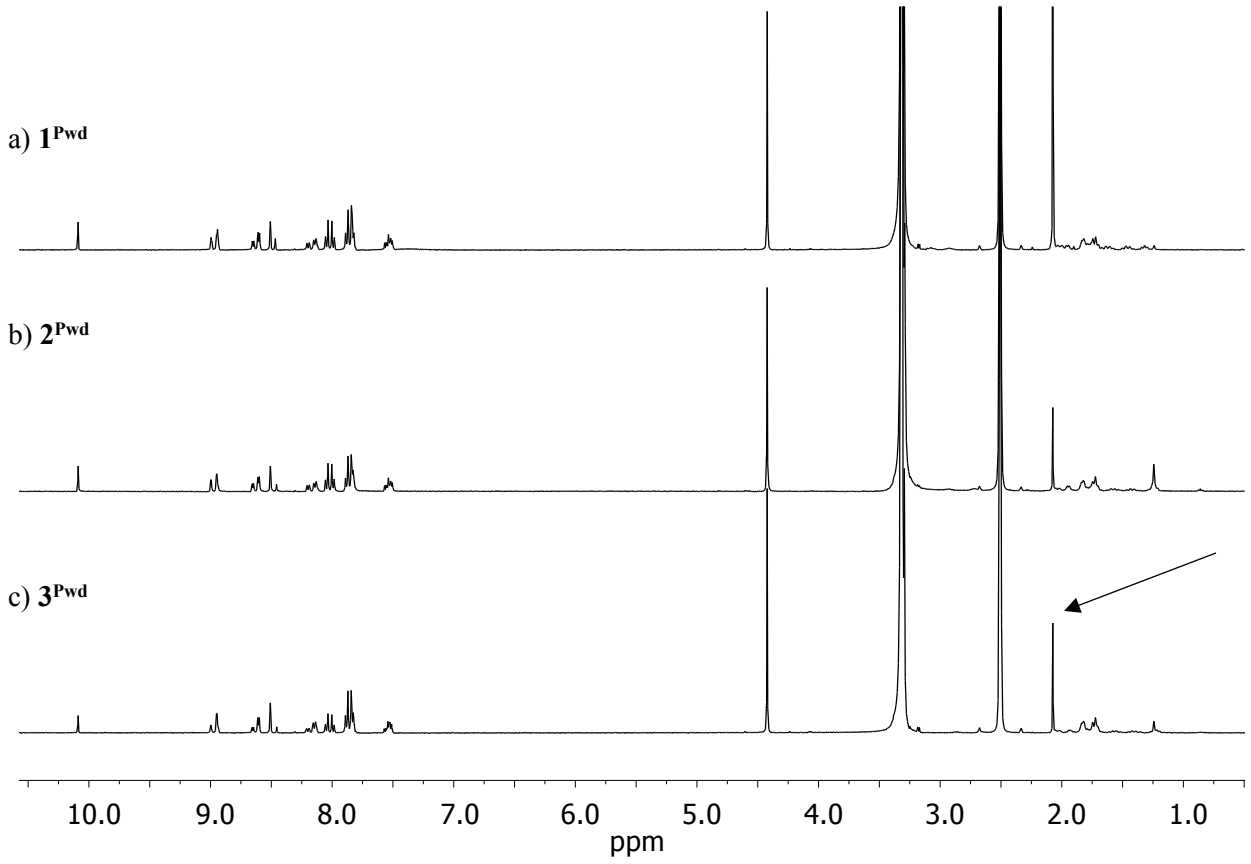


Figure S23. ^{1}H-NMR characterization in DMSO-d_6 of materials **1-3^{Pwd}**, as synthesized, showing the presence of MeCN.

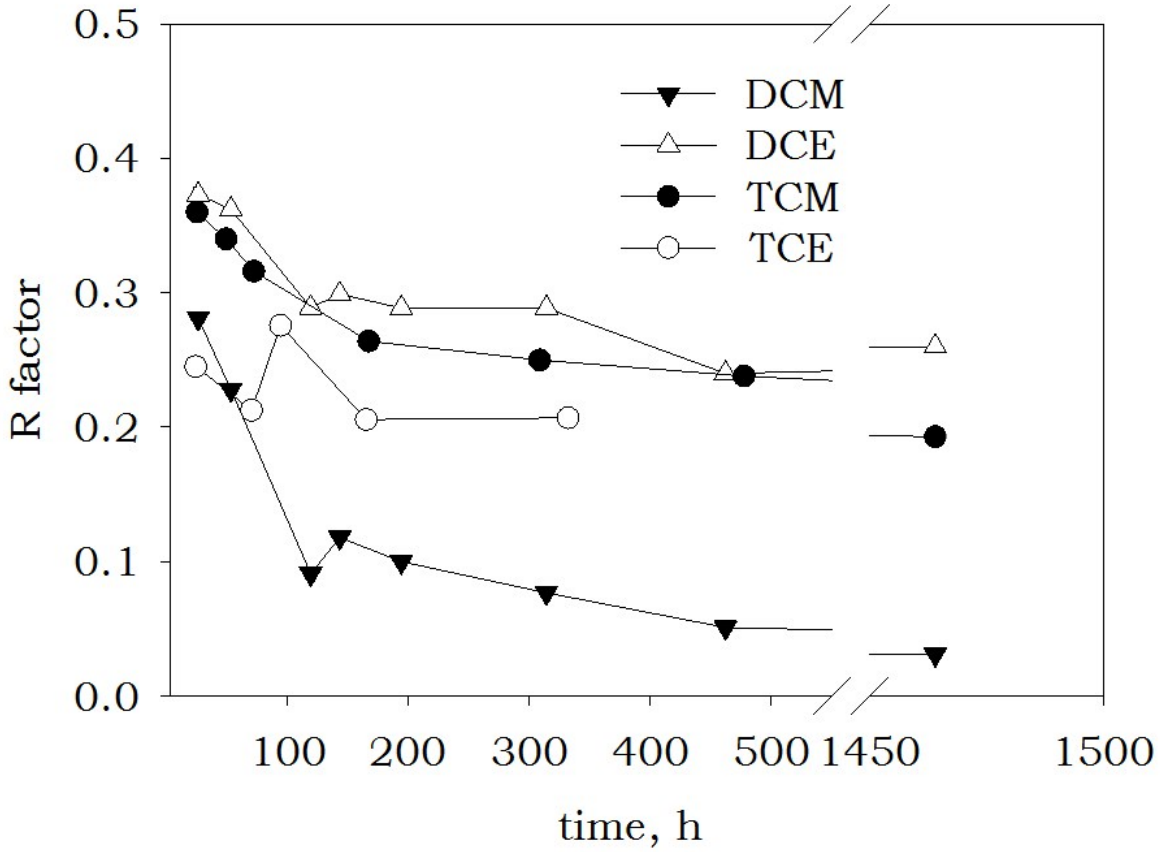


Figure S24. Plot of the *R* factor over time for various solvents in samples of **1**$^{\mathbf{Pwd}}$ as measured by ^{1}H-NMR. Samples were exposed to each solvent for 24 h and then left in ambient condition. *R* factors were calculated by using nitromethane as internal standard.

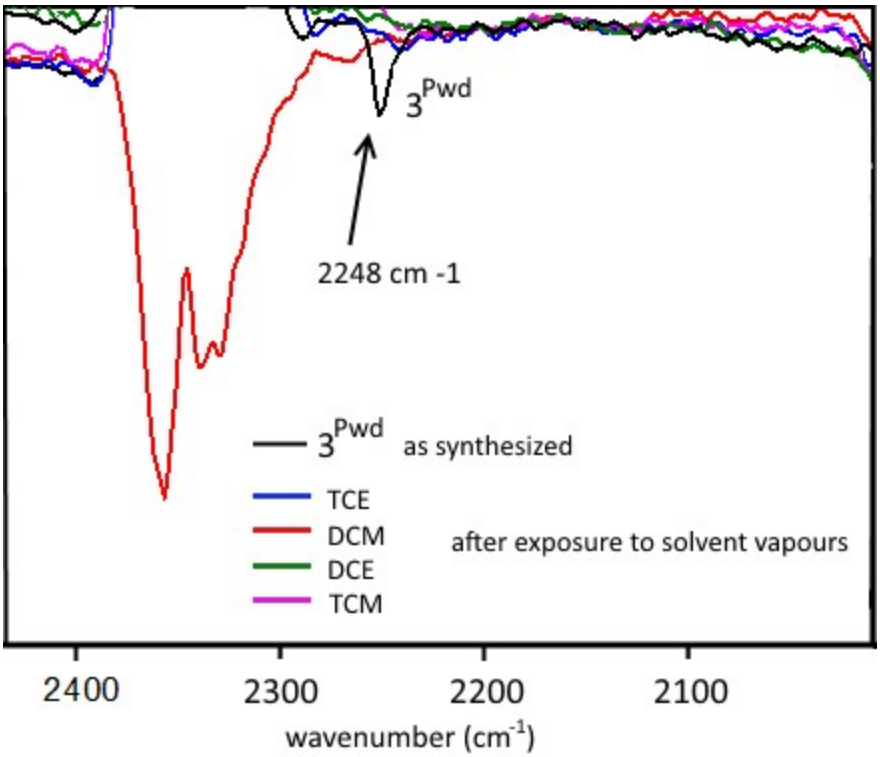


Figure S25. Plot of the ATR-FT-IR spectra in the 2450-2000 cm^{-1} region of a) **3^{Pwd}** as synthesized and of the material exposed to vapours of TCE, DCE, DCM and TCM. The absorption peak at 2248 cm^{-1} corresponds to included MeCN molecules.

checkCIF (basic structural check) running

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

Structure factors have been supplied for datablock(s) 2TCM_100K

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
Please wait while processing .... Interpreting this report
Structure factor report

Datablock: 2TCM_100K

Bond precision: C-C = 0.0098 A Wavelength=1.54178

Cell: a=14.6557(10) b=22.6968(16) c=10.1625(8)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	3380.4(4)	3380.4(4)
Space group	P n n a	P n n a
Hall group	-P 2a 2bc	-P 2a 2bc
Moiety formula	C30 H28 Br2 N4 Zn, 0.68(C H Cl3)	?
Sum formula	C30.68 H28.68 Br2 Cl2.04 N4 Zn	C30.75 H28.75 Br2 Cl2 N4 Zn
Mr	750.92	750.42
Dx,g cm-3	1.475	1.474

Z	4	4
Mu (mm-1)	5.454	5.425
F000	1501.8	1501.0
F000'	1495.68	
h,k,lmax	17,26,12	17,26,12
Nref	2938	2841
Tmin,Tmax	0.624,0.722	0.623,0.753
Tmin'	0.554	

Correction method= # Reported T Limits: Tmin=0.623 Tmax=0.753 AbsCorr = MULTI-SCAN

Data completeness= 0.967 Theta(max)= 65.954

R(reflections)= 0.0619(2205) wR2(reflections)= 0.1980(2841)

S = 1.091 Npar= 204

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

PLAT934_ALERT_3_B Number of (Iobs-Icalc)/SigmaW > 10 Outliers .... 2 Check

Alert level C

PLAT041_ALERT_1_C Calc. and Reported SumFormula Strings Differ Please Check
PLAT077_ALERT_4_C Unitcell contains non-integer number of atoms .. Please Check
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor .... 2.1 Note
PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00975 Ang.
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.592 97 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 1 Check
PLAT971_ALERT_2_C Check Calcd Residual Density 0.92A From Cl1S 1.57 eA-3
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density 0 Note

Alert level G

FORMU01_ALERT_2_G There is a discrepancy between the atom counts in the
_chemical_formula_sum and the formula from the _atom_site* data.
Atom count from _chemical_formula_sum:C30.75 H28.75 Br2 Cl2 N4 Zn1
Atom count from the _atom_site data: C30.68 H28.68 Br2 Cl2.04 N4 Zn
CELLZ01_ALERT_1_G Difference between formula and atom_site contents detected.
CELLZ01_ALERT_1_G WARNING: H atoms missing from atom site list. Is this intentional?
From the CIF: _cell_formula_units_Z 4
From the CIF: _chemical_formula_sum C30.75 H28.75 Br2 Cl2 N4 Zn
TEST: Compare cell contents of formula and atom_site data

atom	Z*formula	cif sites	diff
C	123.00	122.72	0.28
H	115.00	114.72	0.28
Br	8.00	8.00	0.00
Cl	8.00	8.16	-0.16
N	16.00	16.00	0.00
Zn	4.00	4.00	0.00

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 4 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 4 Report
PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 1 Info
PLAT068_ALERT_1_G Reported F000 Differs from Calcd (or Missing)... Please Check
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large 0.13 Report
PLAT176_ALERT_4_G The CIF-Embedded .res File Contains SADI Records 1 Report
PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records 1 Report
PLAT300_ALERT_4_G Atom Site Occupancy of <Cl1S is Constrained at 0.34 Check
And 4 other PLAT300 Alerts
More ...
PLAT302_ALERT_4_G Anion/Solvent Disorder ..... Percentage = 100 Note
PLAT789_ALERT_4_G Atoms with Negative _atom_site_disorder_group # 5 Check
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 21 Note
PLAT909_ALERT_3_G Percentage of Observed Data at Theta(Max) Still 71 %

---

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

4 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
6 ALERT type 3 Indicator that the structure quality may be low
10 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

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Publication of your CIF in IUCr journals

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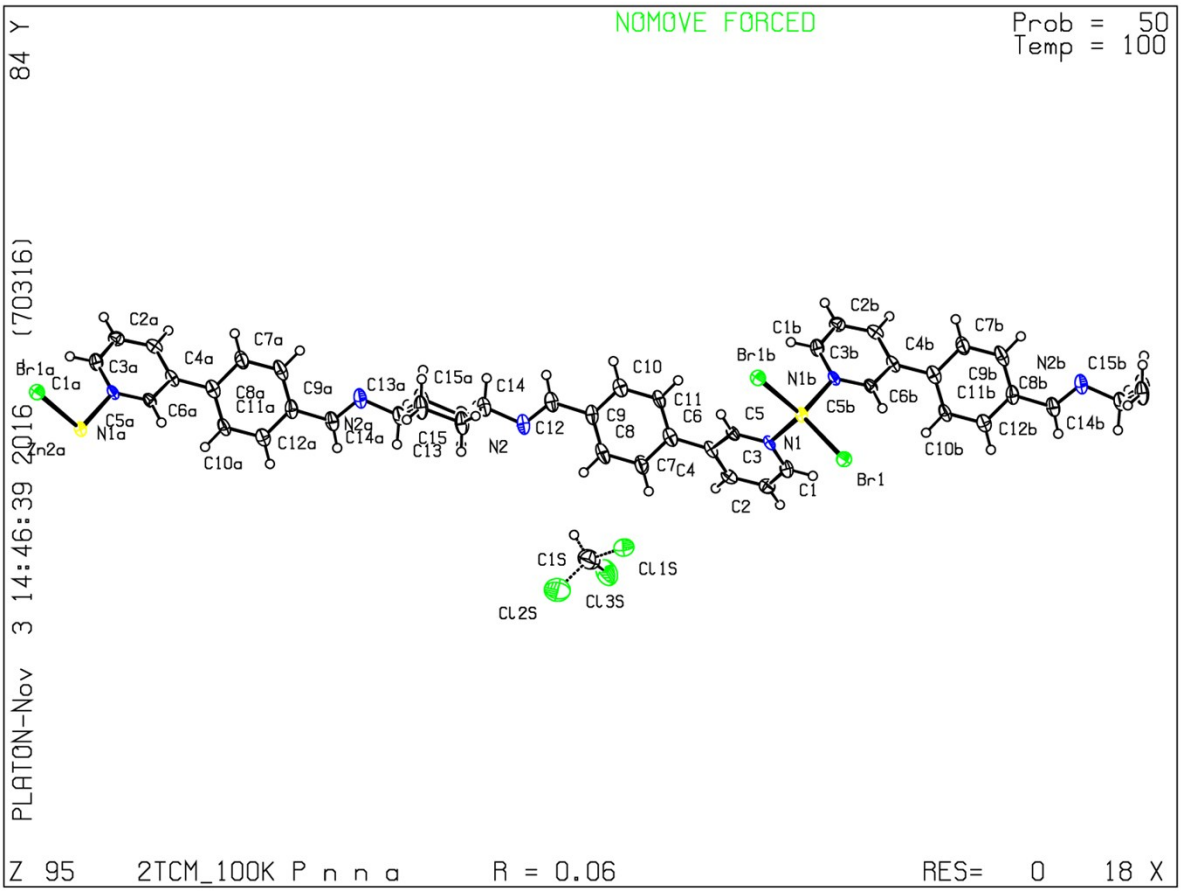
C or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

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 11/08/2016; check.def file version of 04/08/2016

Datablock 2TCM_100K - ellipsoid plot



Download CIF editor (publCIF) from the IUCr
Download CIF editor (enCIFer) from the CCDC
Test a new CIF entry

checkCIF (basic structural check) running

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

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) 3TCM_100K

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

Datablock: 3TCM_100K

Bond precision: C-C = 0.0125 A Wavelength=1.54178

Cell: a=14.4863(13) b=22.1482(19) c=11.0890(11)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	3557.9(6)	3557.9(6)
Space group	P n n a	P n n a
Hall group	-P 2a 2bc	-P 2a 2bc
Moiety formula	C30 H28 I2 N4 Zn, 0.8(C H Cl3), 2(Cl0.10)	?
Sum formula	C30.80 H28.80 Cl2.60 I2 N4 Zn	C15.38 H14.38 Cl1.25 I N2 Zn0.50
Mr	866.34	431.06

Dx,g cm-3	1.617	1.609
Z	4	8
Mu (mm-1)	16.571	16.500
F000	1687.2	1679.0
F000'	1685.97	
h,k,lmax	17,26,13	17,26,13
Nref	3105	3038
Tmin,Tmax	0.351,0.372	0.458,0.753
Tmin'	0.167	

Correction method= # Reported T Limits: Tmin=0.458
Tmax=0.753 AbsCorr = MULTI-SCAN

Data completeness= 0.978 Theta(max)= 66.019

R(reflections)= 0.0558(1936) wR2(reflections)= 0.1766(3038)

S = 1.111 Npar= 208

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

SHFSU01_ALERT_2_C The absolute value of parameter shift to su ratio > 0.05
Absolute value of the parameter shift to su ratio given 0.090
Additional refinement cycles may be required.
PLAT041_ALERT_1_C Calc. and Reported SumFormula Strings Differ Please Check
PLAT043_ALERT_1_C Calculated and Reported Mol. Weight Differ by .. 4.22 Check
PLAT068_ALERT_1_C Reported F000 Differs from Calcd (or Missing)... Please Check
PLAT077_ALERT_4_C Unitcell contains non-integer number of atoms .. Please Check
PLAT080_ALERT_2_C Maximum Shift/Error ..... 0.09 Why ?
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor .... 2.6 Note
PLAT342_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.0125 Ang.
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd. # 1 Note
C30 H28 I2 N4 Zn
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.593 66 Report
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density 0 Note

---

Alert level G

FORMU01_ALERT_2_G There is a discrepancy between the atom counts in the
_chemical_formula_sum and the formula from the _atom_site* data.
Atom count from _chemical_formula_sum:C15.38 H14.38 Cl1.25 I1 N2 Zn0
Atom count from the _atom_site data: C15.4 H14.4 Cl1.3 I1 N2 Zn0.5
CELLZ01_ALERT_1_G Difference between formula and atom_site contents detected.

CELLZ01_ALERT_1_G ALERT: Large difference may be due to a
symmetry error - see SYMMG tests
From the CIF: _cell_formula_units_Z 8
From the CIF: _chemical_formula_sum C15.38 H14.38 Cl1.25 I N2 Zn0.50
TEST: Compare cell contents of formula and atom_site data

atom	Z*formula	cif sites	diff
C	123.04	123.20	-0.16
H	115.04	115.20	-0.16
Cl	10.00	10.40	-0.40
I	8.00	8.00	0.00
N	16.00	16.00	0.00
Zn	4.00	4.00	0.00

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 4 Note
PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 1 Info
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ... 0.50 Check
PLAT176_ALERT_4_G The CIF-Embedded .res File Contains SADI Records 1 Report
PLAT300_ALERT_4_G Atom Site Occupancy of <Cl1 is Constrained at 0.4 Check
And 5 other PLAT300 Alerts
More ...
PLAT302_ALERT_4_G Anion/Solvent Disorder ..... Percentage = 100 Note
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..... 1 Note
PLAT789_ALERT_4_G Atoms with Negative _atom_site_disorder_group # 6 Check
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 3 Note
PLAT909_ALERT_3_G Percentage of Observed Data at Theta(Max) Still 34 %
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min) 2 Note
PLAT933_ALERT_2_G Number of OMIT records in Embedded RES ..... 5 Note

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0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
11 **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

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
5 ALERT type 3 Indicator that the structure quality may be low
12 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.

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Publication of your CIF in other journals

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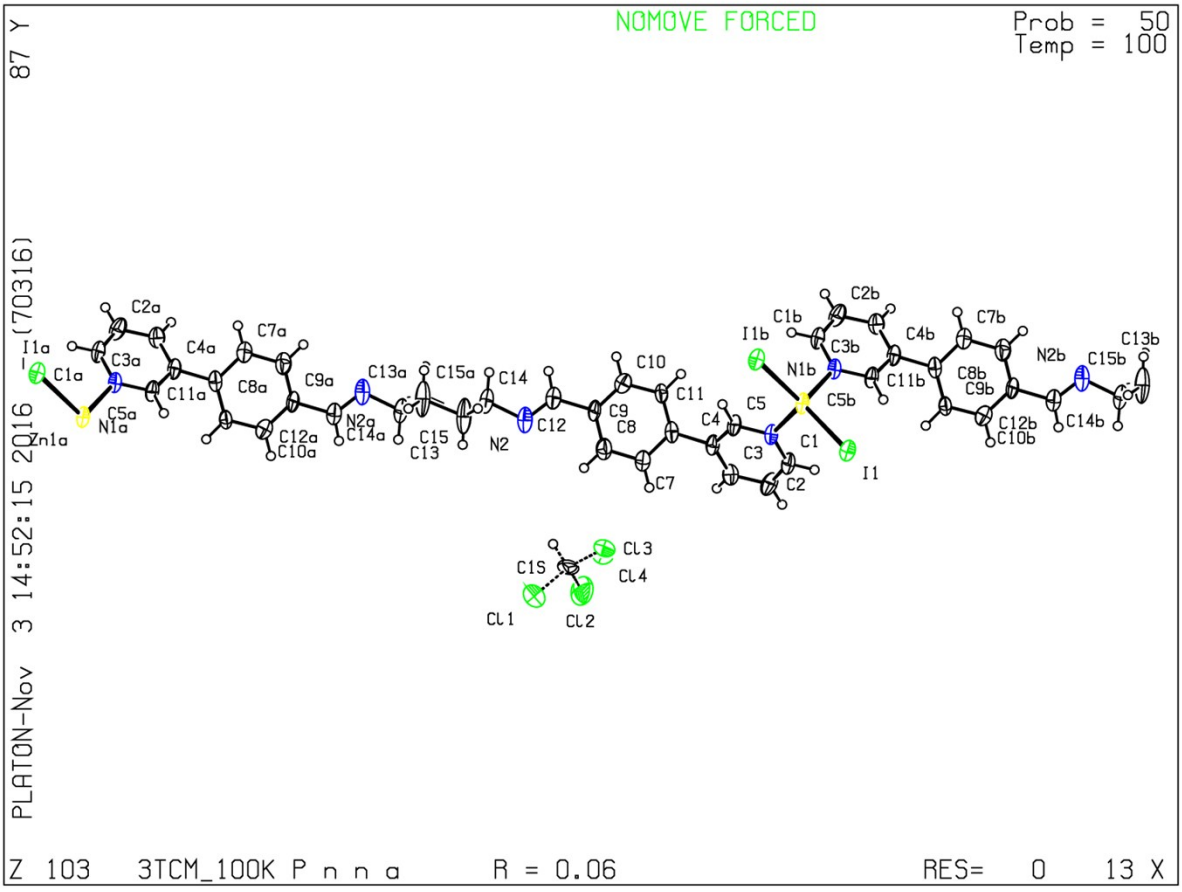
PLATON version of 11/08/2016; check.def file version of 04/08/2016

Datablock 3TCM_100K - ellipsoid plot



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checkCIF (basic structural check) running

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

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) 1TCE200K

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

Datablock: 1TCE200K

Bond precision: C-C = 0.0065 A Wavelength=1.54178

Cell: a=14.7372(11) b=22.7121(16) c=9.9658(8)

alpha=90 beta=90 gamma=90

Temperature: 200 K

	Calculated	Reported
Volume	3335.7(4)	3335.7(4)
Space group	P n n a	P n n a
Hall group	-P 2a 2bc	-P 2a 2bc
Moiety formula	C30 H28 Cl2 N4 Zn, C2 H Cl3	?
Sum formula	C32 H29 Cl5 N4 Zn	C16 H14.50 Cl2.50 N2 Zn0.50
Mr	712.23	356.11
Dx,g cm-3	1.418	1.418

```
Z                   4                                   8

Mu (mm-1)           4.926                               4.926

F000                1456.0                              1456.0

F000'               1459.77

h,k,lmax            17,26,11                            17,25,11

Nref                2904                                2828

Tmin,Tmax           0.049,0.052                         0.531,0.753

Tmin'               0.008

Correction method= # Reported T Limits: Tmin=0.531
Tmax=0.753 AbsCorr = MULTI-SCAN

Data completeness= 0.974        Theta(max)= 65.886

R(reflections)= 0.0839( 2207)      wR2(reflections)= 0.2599( 2828)

S = 1.040             Npar= 213
```

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

RINTA01_ALERT_3_C The value of Rint is greater than 0.12
Rint given 0.141
PLAT020_ALERT_3_C The value of Rint is greater than 0.12 ..... 0.141 Report
PLAT084_ALERT_3_C High wR2 Value (i.e. > 0.25) ..... 0.26 Report
PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.0065 Ang.
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd. # 1 Note
C30 H28 Cl2 N4 Zn
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.592 65 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 2 Check

---

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 5 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 5 Report
PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 1 Info
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ... 0.50 Check
PLAT063_ALERT_4_G Crystal Size Likely too Large for Beam Size .... 0.80 mm
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large 0.19 Report
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 1 Report
PLAT176_ALERT_4_G The CIF-Embedded .res File Contains SADI Records 1 Report
PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records 1 Report
PLAT300_ALERT_4_G Atom Site Occupancy of *Cl2 is Constrained at 0.5 Check
And 5 other PLAT300 Alerts
More ...

PLAT302_ALERT_4_G Anion/Solvent Disorder ..... Percentage = 100 Note
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..... 3 Note
PLAT789_ALERT_4_G Atoms with Negative _atom_site_disorder_group # 6 Check
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 28 Note
PLAT909_ALERT_3_G Percentage of Observed Data at Theta(Max) Still 54 %
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density 1 Note

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain
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1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
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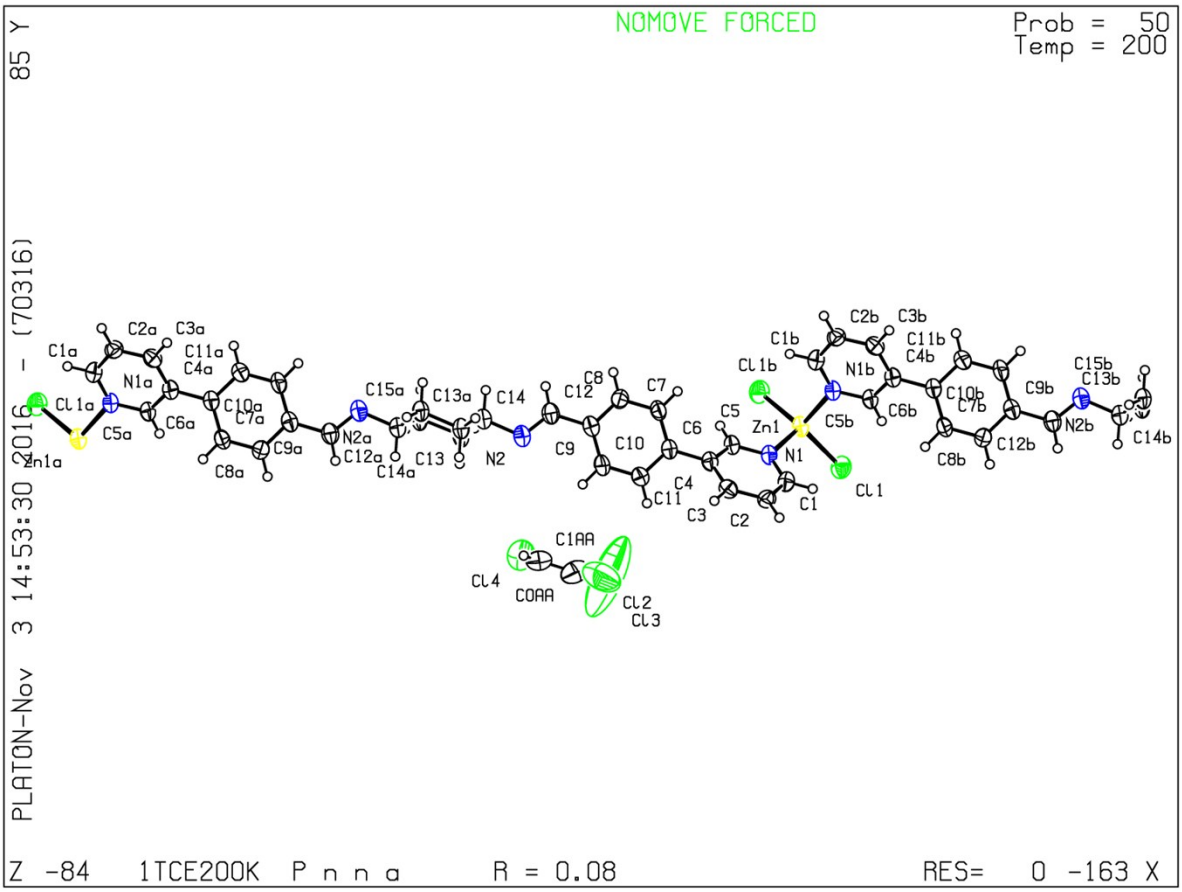
PLATON version of 11/08/2016; check.def file version of 04/08/2016

Datablock 1TCE200K - ellipsoid plot



checkCIF (basic structural check) running

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

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) 1MeCN

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

Datablock: 1MeCN

Bond precision: C-C = 0.0055 A Wavelength=1.54178

Cell: a=14.7485(14) b=22.317(2) c=10.4570(11)

alpha=90 beta=90 gamma=90

Temperature: 296 K

	Calculated	Reported
Volume	3441.8(6)	3441.8(6)
Space group	P n n a	P n n a
Hall group	-P 2a 2bc	-P 2a 2bc
Moiety formula	C30 H28 Cl2 N4 Zn, 2(C2 H3 N)	?
Sum formula	C34 H34 Cl2 N6 Zn	C17 H17 Cl N3 Zn0.50
Mr	662.96	331.47
Dx,g cm-3	1.279	1.279
Z	4	8
Mu (mm-1)	2.660	2.660
F000	1376.0	1376.0
F000'	1375.66	
h,k,lmax	17,26,12	0,0,0
Nref	3020	2947
Tmin,Tmax	0.555,0.587	0.480,0.616
Tmin'	0.329	

Correction method= # Reported T Limits: Tmin=0.480
Tmax=0.616 AbsCorr = REFDELF

Data completeness= 0.976 Theta(max)= 66.160

R(reflections)= 0.0384(2252) wR2(reflections)= 0.1111(2947)

S = 1.017 Npar= 195

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

PLAT390_ALERT_3_B Deviating Methyl C3S X-C-H Bond Angle ..... 100 Degree

Alert level C

PLAT243_ALERT_4_C High 'Solvent' Ueq as Compared to Neighbors of C1S Check
PLAT390_ALERT_3_C Deviating Methyl C3S X-C-H Bond Angle ..... 117 Degree
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd. # 1 Note
C30 H28 Cl2 N4 Zn
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.593 73 Report
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density 0 Note

Alert level G

PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 1 Info
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ... 0.50 Check
PLAT344_ALERT_2_G Unusual sp? Angle Range in Solvent/Ion for . C1S Check
PLAT367_ALERT_2_G Long? C(sp?)-C(sp?) Bond C1S - C3S .. 1.68 Ang.
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..... 3 Note
PLAT909_ALERT_3_G Percentage of Observed Data at Theta(Max) Still 41 %

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **ALERT level B** = A potentially serious problem, consider carefully
5 **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
3 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
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

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Publication of your CIF in other journals

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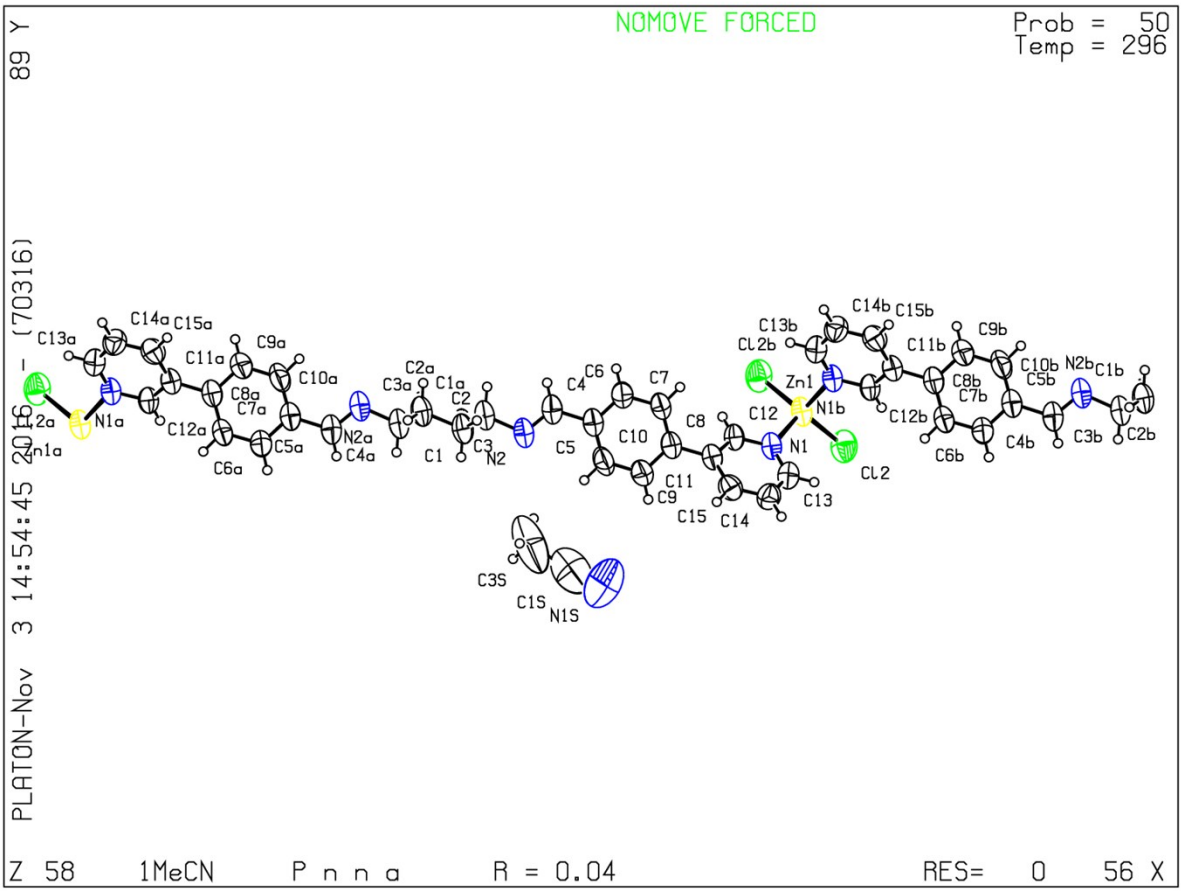
PLATON version of 11/08/2016; check.def file version of 04/08/2016

Datablock 1MeCN - ellipsoid plot


NOMOVE FORCED
Prob = 50
Temp = 296
PLATON-Nov 3 14:54:45 2016 - (70316)
89 Y
Z 58 1MeCN P n n a R = 0.04 RES= 0 56 X
Cl2a
Zn1a
N1a
C13a
C14a
C15a
C11a
C9a
C8a
C7a
C10a
C12a
C6a
C5a
N2a
C4a
C3a
C2a
C1a
C1
C2
C3
N2
C4
C6
C7
C5
C10
C8
C11
C9
C15
C14
C13
N1
C12
Cl2
Zn1
N1b
Cl2b
C13b
C14b
C15b
C11b
C9b
C8b
C7b
C10b
C5b
N2b
C1b
C12b
C6b
C4b
C3b
C2b
C3S
C1S
N1S

checkCIF (basic structural check) running

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

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) 2MeCN_2

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
Please wait while processing .... Interpreting this report
Structure factor report

Datablock: 2MeCN_2

Bond precision: C-C = 0.0151 A Wavelength=1.54178

Cell: a=14.8955(11) b=22.5127(17) c=10.2599(7)

alpha=90 beta=90 gamma=90

Temperature: 296 K

	Calculated	Reported
Volume	3440.5(4)	3440.5(4)
Space group	P n n a	P n n a
Hall group	-P 2a 2bc	-P 2a 2bc
Moiety formula	C30 H28 Br2 N4 Zn, 2(C2 H3 N)	?
Sum formula	C34 H34 Br2 N6 Zn	C17 H17 Br N3 Zn0.50
Mr	751.86	375.93

Dx,g cm-3	1.452	1.452
Z	4	8
Mu (mm-1)	3.954	3.954
F000	1520.0	1520.0
F000'	1511.31	
h,k,lmax	17,26,12	17,26,11
Nref	3020	2890
Tmin,Tmax	0.827,0.888	
Tmin'	0.701	

Correction method= Not given

Data completeness= 0.957 Theta(max)= 66.205

R(reflections)= 0.1060(1666) wR2(reflections)= 0.3070(2890)

S = 0.960 Npar= 177

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

RINTA01_ALERT_3_B The value of Rint is greater than 0.18
Rint given 0.214
SHFSU01_ALERT_2_B The absolute value of parameter shift to su ratio > 0.10
Absolute value of the parameter shift to su ratio given 0.152
Additional refinement cycles may be required.
PLAT020_ALERT_3_B The value of Rint is greater than 0.12 ..... 0.214 Report
PLAT029_ALERT_3_B _diffrn_measured_fraction_theta_full value Low . 0.957 Note
PLAT080_ALERT_2_B Maximum Shift/Error ..... 0.15 Why ?
PLAT341_ALERT_3_B Low Bond Precision on C-C Bonds ..... 0.01512 Ang.
PLAT413_ALERT_2_B Short Inter XH3 .. XHn H2S3 .. H10 .. 1.91 Ang.

Alert level C

PLAT052_ALERT_1_C Info on Absorption Correction Method Not Given Please Do !
PLAT082_ALERT_2_C High R1 Value ..... 0.11 Report
PLAT084_ALERT_3_C High wR2 Value (i.e. > 0.25) ..... 0.31 Report
PLAT202_ALERT_3_C Isotropic non-H Atoms in Anion/Solvent ..... 3 Check
PLAT243_ALERT_4_C High 'Solvent' Ueq as Compared to Neighbors of C3S Check
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd. # 1 Note
C30 H28 Br2 N4 Zn
PLAT905_ALERT_3_C Negative K value in the Analysis of Variance ... -1.226 Report
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.593 120 Report
PLAT934_ALERT_3_C Number of (Iobs-Icalc)/SigmaW > 10 Outliers .... 1 Check

PLAT972_ALERT_2_C Check Calcd Residual Density 0.93A From Br1 -1.52 eA-3
PLAT977_ALERT_2_C Check the Negative Difference Density on H2S3 -0.46 eA-3
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density 0 Note

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 3 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 3 Report
PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 1 Info
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ... 0.50 Check
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large 0.20 Report
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 2 Report
PLAT173_ALERT_4_G The CIF-Embedded .res File Contains DANG Records 1 Report
PLAT344_ALERT_2_G Unusual sp? Angle Range in Solvent/Ion for . C3S Check
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..... 3 Note
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 3 Note

0 **ALERT level A** = Most likely a serious problem - resolve or explain
7 **ALERT level B** = A potentially serious problem, consider carefully
12 **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
11 ALERT type 2 Indicator that the structure model may be wrong or deficient
10 ALERT type 3 Indicator that the structure quality may be low
5 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

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Publication of your CIF in other journals

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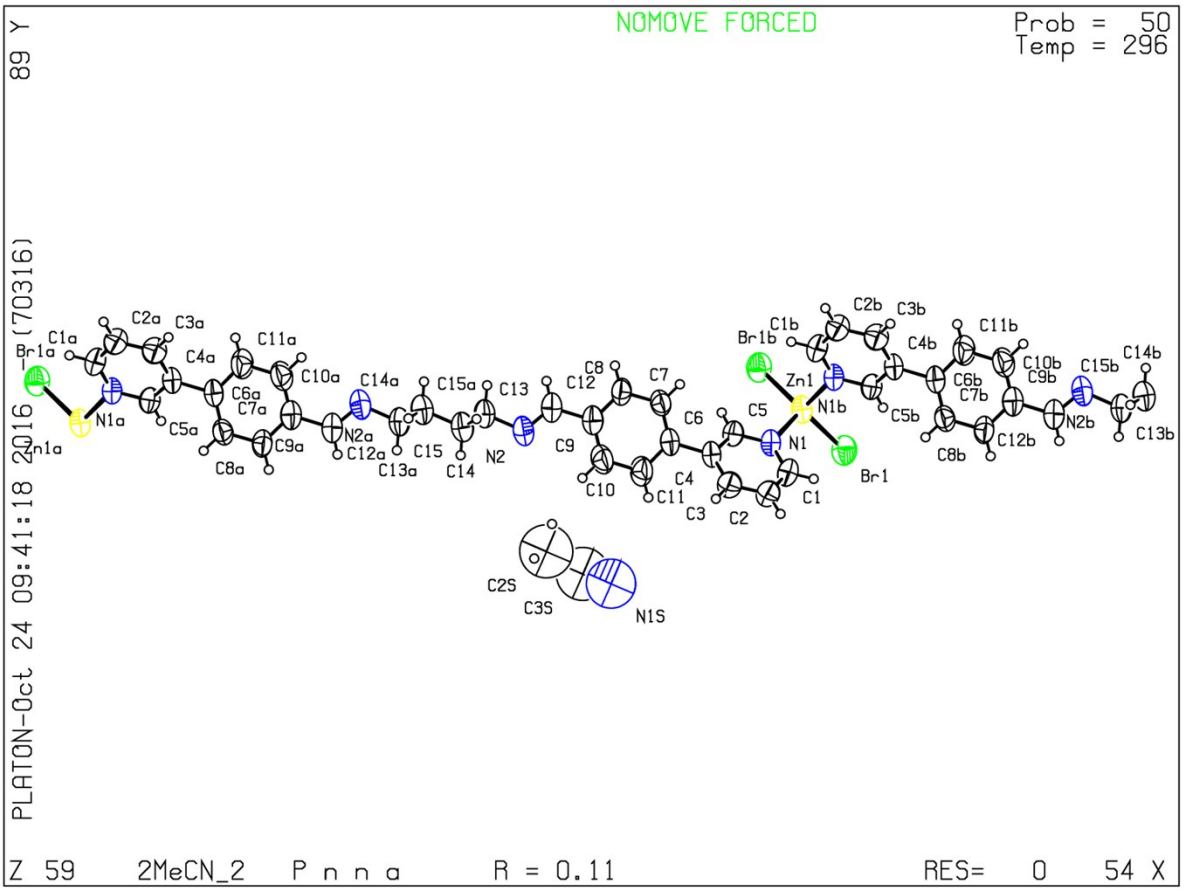
submission.

PLATON version of 11/08/2016; check.def file version of 04/08/2016

Datablock 2MeCN_2 - ellipsoid plot



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checkCIF (basic structural check) running

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

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) 3MeCN

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
Please wait while processing .... Interpreting this report
Structure factor report

Datablock: 3MeCN

Bond precision: C-C = 0.0350 A Wavelength=1.54178

Cell: a=15.077(3) b=22.570(4) c=10.412(2)

alpha=90 beta=90 gamma=90

Temperature: 296 K

	Calculated	Reported
Volume	3543.1(12)	3543.2(11)
Space group	P n n a	P n n a
Hall group	-P 2a 2bc	-P 2a 2bc
Moiety formula	C30 H28 I2 N4 Zn, C2 N	?
Sum formula	C32 H28 I2 N5 Zn	C16.50 H15.75 I N2.50 Zn0.50
Mr	801.78	408.65
Dx,g cm-3	1.503	1.532
Z	4	8
Mu (mm-1)	14.840	14.850
F000	1564.0	1602.0
F000'	1558.98	
h,k,lmax	17,26,12	17,26,12
Nref	3105	3012

```
Tmin,Tmax          0.210,0.227                    0.004,0.056

Tmin'              0.041

Correction method= # Reported T Limits: Tmin=0.004
Tmax=0.056 AbsCorr = CYLINDER

Data completeness= 0.970       Theta(max)= 66.084

R(reflections)= 0.1506( 796)       wR2(reflections)= 0.4593( 3012)

S = 1.004             Npar= 155
```

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

RINTA01_ALERT_3_A The value of Rint is greater than 0.25
Rint given 0.367
Author Response: Due to weak diffraction at high anlges as it was small crystal.

PLAT020_ALERT_3_A The value of Rint is greater than 0.12 ..... 0.367 Report
Author Response: Due to weak diffraction at high anlges as it was small crystal.

PLAT084_ALERT_3_A High wR2 Value (i.e. > 0.25) ..... 0.46 Report
Author Response: Due to weak diffraction at high anlges as it was small crystal.

Alert level B

PLAT043_ALERT_1_B Calculated and Reported Mol. Weight Differ by .. 15.52 Check
PLAT234_ALERT_4_B Large Hirshfeld Difference C6 -- C7 .. 0.28 Ang.
PLAT342_ALERT_3_B Low Bond Precision on C-C Bonds ..... 0.035 Ang.

Alert level C

CHEMW03_ALERT_2_C The ratio of given/expected molecular weight as
calculated from the _atom_site* data lies outside
the range 0.99 <> 1.01
From the CIF: _cell_formula_units_Z 8
From the CIF: _chemical_formula_weight 408.65
TEST: Calculate formula weight from _atom_site_*

atom	mass	num	sum
C	12.01	16.00	192.18
H	1.01	14.00	14.11
N	14.01	2.50	35.02
Zn	65.39	0.50	32.69
I	126.90	1.00	126.90

Calculated formula weight 400.91
PLAT041_ALERT_1_C Calc. and Reported SumFormula Strings Differ Please Check
PLAT068_ALERT_1_C Reported F000 Differs from Calcd (or Missing)... Please Check
PLAT082_ALERT_2_C High R1 Value ..... 0.15 Report

PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of C1 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C16 Check
PLAT369_ALERT_2_C Long C(sp2)-C(sp2) Bond C4 - C6 .. 1.53 Ang.
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd. # 1 Note
C30 H28 I2 N4 Zn
PLAT905_ALERT_3_C Negative K value in the Analysis of Variance ... -5.464 Report
PLAT905_ALERT_3_C Negative K value in the Analysis of Variance ... -2.072 Report
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.593 94 Report
PLAT972_ALERT_2_C Check Calcd Residual Density 0.92A From I1 -1.71 eA-3
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density 0 Note

---

Alert level G

FORMU01_ALERT_2_G There is a discrepancy between the atom counts in the
_chemical_formula_sum and the formula from the _atom_site* data.
Atom count from _chemical_formula_sum:C16.5 H15.75 I1 N2.5 Zn0.5
Atom count from the _atom_site data: C16 H14 I1 N2.5 Zn0.5
CELLZ01_ALERT_1_G Difference between formula and atom_site contents detected.
CELLZ01_ALERT_1_G ALERT: Large difference may be due to a
symmetry error - see SYMMG tests
From the CIF: _cell_formula_units_Z 8
From the CIF: _chemical_formula_sum C16.50 H15.75 I N2.50 Zn0.50
TEST: Compare cell contents of formula and atom_site data

atom	Z*formula	cif sites	diff
C	132.00	128.00	4.00
H	126.00	112.00	14.00
I	8.00	8.00	0.00
N	20.00	20.00	0.00
Zn	4.00	4.00	0.00

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 3 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 3 Report
PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 1 Info
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ... 0.50 Check
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large 0.20 Report
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 4 Report
PLAT186_ALERT_4_G The CIF-Embedded .res File Contains ISOR Records 2 Report
PLAT300_ALERT_4_G Atom Site Occupancy of *N3S is Constrained at 0.5 Check
PLAT300_ALERT_4_G Atom Site Occupancy of *C2S is Constrained at 0.5 Check
PLAT302_ALERT_4_G Anion/Solvent Disorder ..... Percentage = 67 Note
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 3 Note

---

3 **ALERT level A** = Most likely a serious problem - resolve or explain
3 **ALERT level B** = A potentially serious problem, consider carefully
13 **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
11 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
7 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

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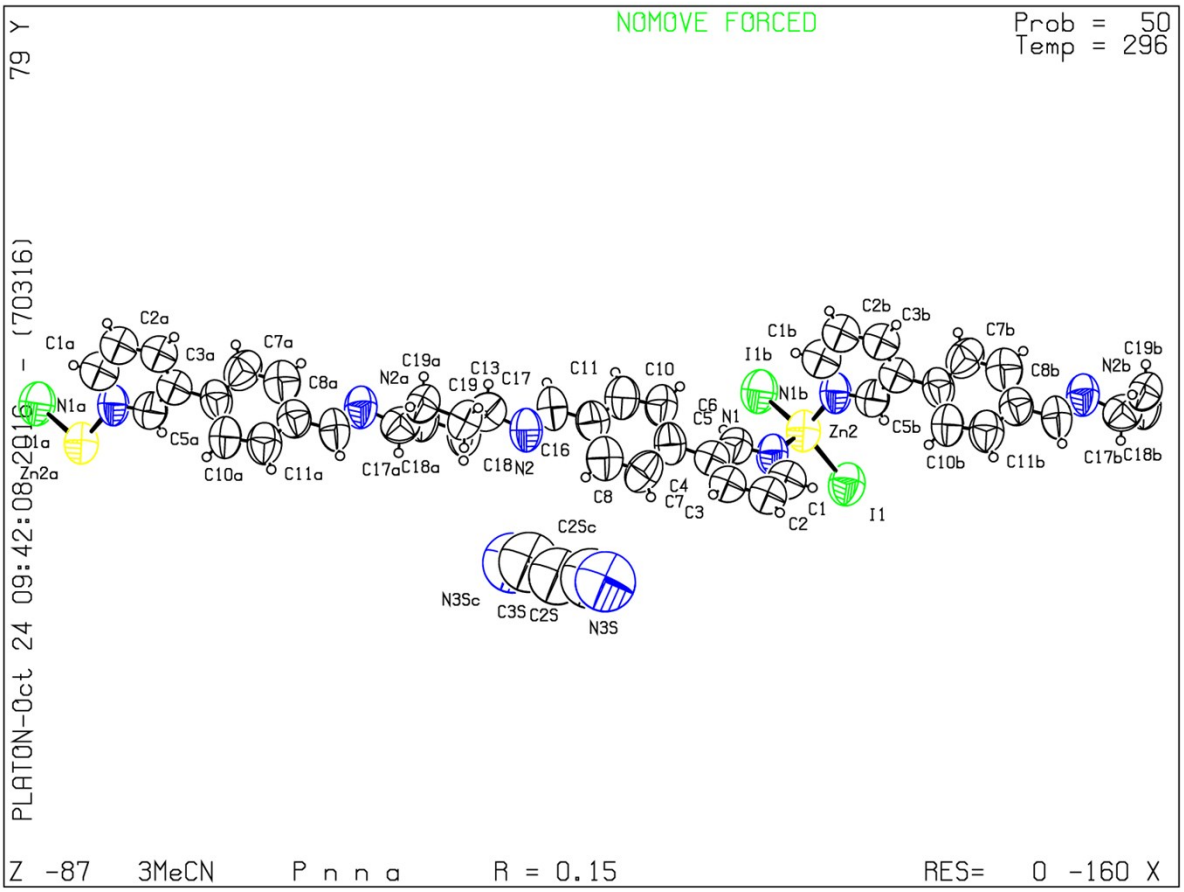
PLATON version of 11/08/2016; check.def file version of 04/08/2016

Datablock 3MeCN - ellipsoid plot



checkCIF (basic structural check) running

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

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) 3MeCN_100K

Datablock: 3MeCN_100K

Bond precision: C-C = 0.0103 A Wavelength=1.54178

Cell: a=14.9803(14) b=22.7330(19) c=10.0998(8)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	3439.5(5)	3439.5(5)
Space group	P n n a	P n n a
Hall group	-P 2a 2bc	-P 2a 2bc
Moiety formula	C30 H28 I2 N4 Zn, C2 N	?
Sum formula	C32 H28 I2 N5 Zn	C16.50 H17 I N2.50 Zn0.50
Mr	801.78	409.91
Dx,g cm-3	1.548	1.583
Z	4	8
Mu (mm-1)	15.287	15.298
F000	1564.0	1612.0
F000'	1558.98	
h,k,lmax	17,26,11	0,0,0
Nref	2997	2940
Tmin,Tmax	0.199,0.217	0.009,0.017
Tmin'	0.081	

Correction method= # Reported T Limits: Tmin=0.009
Tmax=0.017 AbsCorr = REFDELF

```
Data completeness= 0.981        Theta(max)= 65.867

R(reflections)= 0.0513( 2285)      wR2(reflections)= 0.1370( 2940)

S = 1.154              Npar= 175
```

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

PLAT043_ALERT_1_B Calculated and Reported Mol. Weight Differ by .. 18.04 Check

---

Alert level C

CHEMW03_ALERT_2_C The ratio of given/expected molecular weight as
calculated from the _atom_site* data lies outside
the range 0.99 <> 1.01
From the CIF: _cell_formula_units_Z 8
From the CIF: _chemical_formula_weight 409.91
TEST: Calculate formula weight from _atom_site_*

atom	mass	num	sum
C	12.01	16.00	192.18
H	1.01	14.00	14.11
N	14.01	2.50	35.02
Zn	65.39	0.50	32.69
I	126.90	1.00	126.90

Calculated formula weight 400.91

PLAT041_ALERT_1_C Calc. and Reported SumFormula Strings Differ Please Check
PLAT068_ALERT_1_C Reported F000 Differs from Calcd (or Missing)... Please Check
PLAT342_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.01031 Ang.
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd. # 1 Note
C30 H28 I2 N4 Zn
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.592 57 Report
PLAT975_ALERT_2_C Check Calcd Residual Density 0.99A From N1S 0.61 eA-3
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density 0 Note

---

Alert level G

FORMU01_ALERT_2_G There is a discrepancy between the atom counts in the
_chemical_formula_sum and the formula from the _atom_site* data.
Atom count from _chemical_formula_sum:C16.5 H17 I1 N2.5 Zn0.5
Atom count from the _atom_site data: C16 H14 I1 N2.5 Zn0.5
CELLZ01_ALERT_1_G Difference between formula and atom_site contents detected.
CELLZ01_ALERT_1_G ALERT: Large difference may be due to a
symmetry error - see SYMMG tests
From the CIF: _cell_formula_units_Z 8
From the CIF: _chemical_formula_sum C16.50 H17 I N2.50 Zn0.50
TEST: Compare cell contents of formula and atom_site data

atom	Z*formula	cif sites	diff
C	132.00	128.00	4.00
H	136.00	112.00	24.00

I	8.00	8.00	0.00
N	20.00	20.00	0.00
Zn	4.00	4.00	0.00

PLAT002_ALERT_2_G	Number of Distance or Angle Restraints on AtSite	3	Note
PLAT003_ALERT_2_G	Number of Uiso or Uij Restrained non-H Atoms ...	3	Report
PLAT004_ALERT_5_G	Polymeric Structure Found with Maximum Dimension	1	Info
PLAT045_ALERT_1_G	Calculated and Reported Z Differ by a Factor ...	0.50	Check
PLAT083_ALERT_2_G	SHELXL Second Parameter in WGHT Unusually Large	17.70	Why ?
PLAT172_ALERT_4_G	The CIF-Embedded .res File Contains DFIX Records	2	Report
PLAT300_ALERT_4_G	Atom Site Occupancy of *N1S is Constrained at	0.5	Check
PLAT300_ALERT_4_G	Atom Site Occupancy of *C2S is Constrained at	0.5	Check
PLAT302_ALERT_4_G	Anion/Solvent Disorder ..... Percentage =	67	Note
PLAT860_ALERT_3_G	Number of Least-Squares Restraints	2	Note
PLAT909_ALERT_3_G	Percentage of Observed Data at Theta(Max) Still	54	%

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **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
4 ALERT type 3 Indicator that the structure quality may be low
5 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 11/08/2016; check.def file version of 04/08/2016

Datablock 3MeCN_100K - ellipsoid plot