Supporting Information for "Multiple Single-Crystal-to-Single-Crystal Guest Exchange in a Dynamic 1D Coordination Polymer."

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Content

Experimental

All the chemicals were purchased from Sigma-Aldrich and used without further modification.

X-ray powder diffraction measurements were carried out using a D2 Bruker diffractometer using the reflection mode at room temperature.

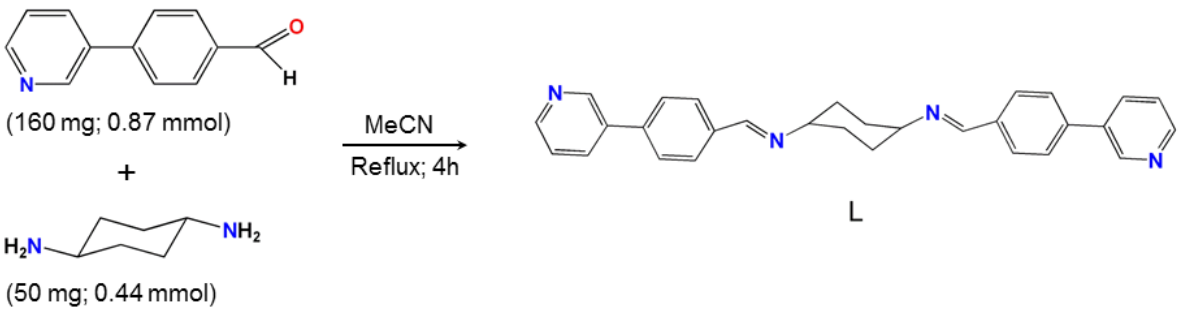
Thermogravimetrical analysis was carried out on a TGA Q500 V20.13 Build 39 instrument. The sample was heated from 26 °C/min (2.00 °C/min) to 700.00 °C.

^{1}H NMR experiments were performed on a Bruker Avance 400 MHz at 300K.

The heating experiments on microcrystalline samples were carried out in a furnace from r.t. to 280 °C at a heating rate of 4 °C/ min.

Synthesis of ligand L.

Ligand **L** was characterized by reacting 1,4-diaminocyclohexane and 4-(3-pyridinyl)benzaldehyde in acetonitrile under refluxing conditions.



Scheme 1. Synthesis of ligand **L**.

^{1}H NMR, elemental analysis and SCXRD. Single crystals were obtained by evaporating a $CHCl_3$ solution (5 mL) of **L** (20 mg) at room temperature. The crystal structure of **L** was determined by single crystal X-ray diffraction. **L** crystallizes in the triclinic *P*-1 space group.

Synthesis of **L**: 160 mg of 3-(4-formyl-phenyl)pyridine (0.87 mmol) and 50 mg of 1,4-diaminocyclohexane (0.44 mmol) were dissolved in 10 mL of MeCN. The solution was maintained under refluxing conditions for 4h and then left under stirring at r.t. overnight. The product, in the form of a

white precipitate, is filtered off and washed with Et_2O. Yield = 75%. ^{1}H-NMR ($CDCl_3$, 400 MHz): δ 8.86 (d, *J* = 1.8 Hz, 2H); δ 8.60 (dd, *J* = 4.8, 1.8 Hz, 2H); δ 8.41 (s, 2H); 7.88 (m, 6H); δ 7.63 (d, *J* = 8.2 Hz, 4H); δ 7.36 (dd, *J* = 7.9, 4.8 Hz, 2H); δ 3.35 (s, 2H); δ 1.88 (m, 8H). ESI-MS: M^+ = 444.23 calcd. for $C_{30}H_{28}N_4$, found 445.42 $[M+H]^+$; Elemental analysis calcd. (%) for $C_{30}H_{28}N_4 \cdot H_2O$ C 77.89; H 6.54; N 12.11; found = C 77.14, H 6.30, N 12.79. Single crystals were grown by slow evaporation of a solution of **L** in trichloromethane. X-ray crystallographic data (100 K): *a* = 5.5990(4) Å *b* = 7.1074(5) Å *c* = 16.1003(11) Å; α = 92.646(4)° β = 98.583(4)° γ = 112.049(3)°; *V* = 583.53(7) Å^3; and triclinic system in the *P*-1 space group.

Formula for the calculation of the parameter R (see manuscript):

$$R = \frac{\text{moles of guest}}{\text{moles of MOF units}} = \frac{\text{MW}_{\text{MOF units}} \cdot \text{Volume} \cdot [\text{guest}]_{\text{NMR}}}{(\text{mg}_{\text{tot}} - \text{MW}_{\text{guest}} \cdot \text{Volume} \cdot [\text{guest}]_{\text{NMR}})}$$

R is the ratio between the moles of guest solvent and the moles of the framework units

$MW_{MOF\ units}$ is the molecular weight of the L-$ZnCl_2$ unit;
MW_{guest} is the molecular weight of the solvent guest;
$[guest]_{NMR}$ corresponds to the concentration of the guest solvent as measured by ^{1}H-NMR by comparison of the integral value of an internal standard;

mg_{tot} is the total amount of material weighted;
Volume is the volume of the DMSO-d6 used (500 μL + 5 μL of internal standard).

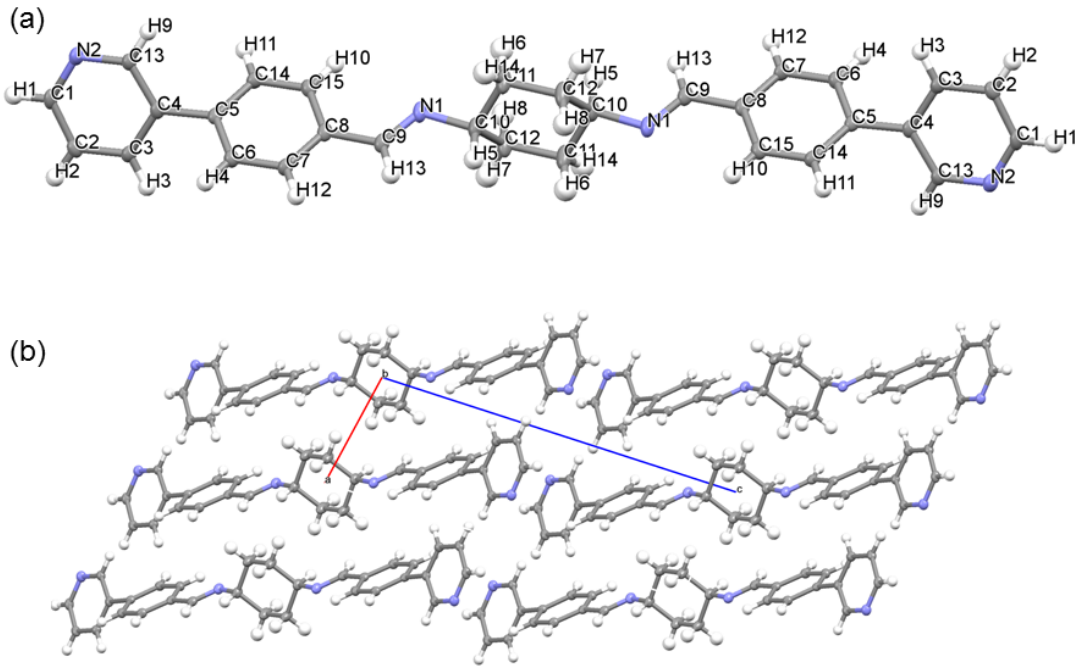


Figure S1. Crystal structure of **L**. (a) View of one molecule of **L**. (b) Crystal packing of **L** viewed along the *b*-axis.

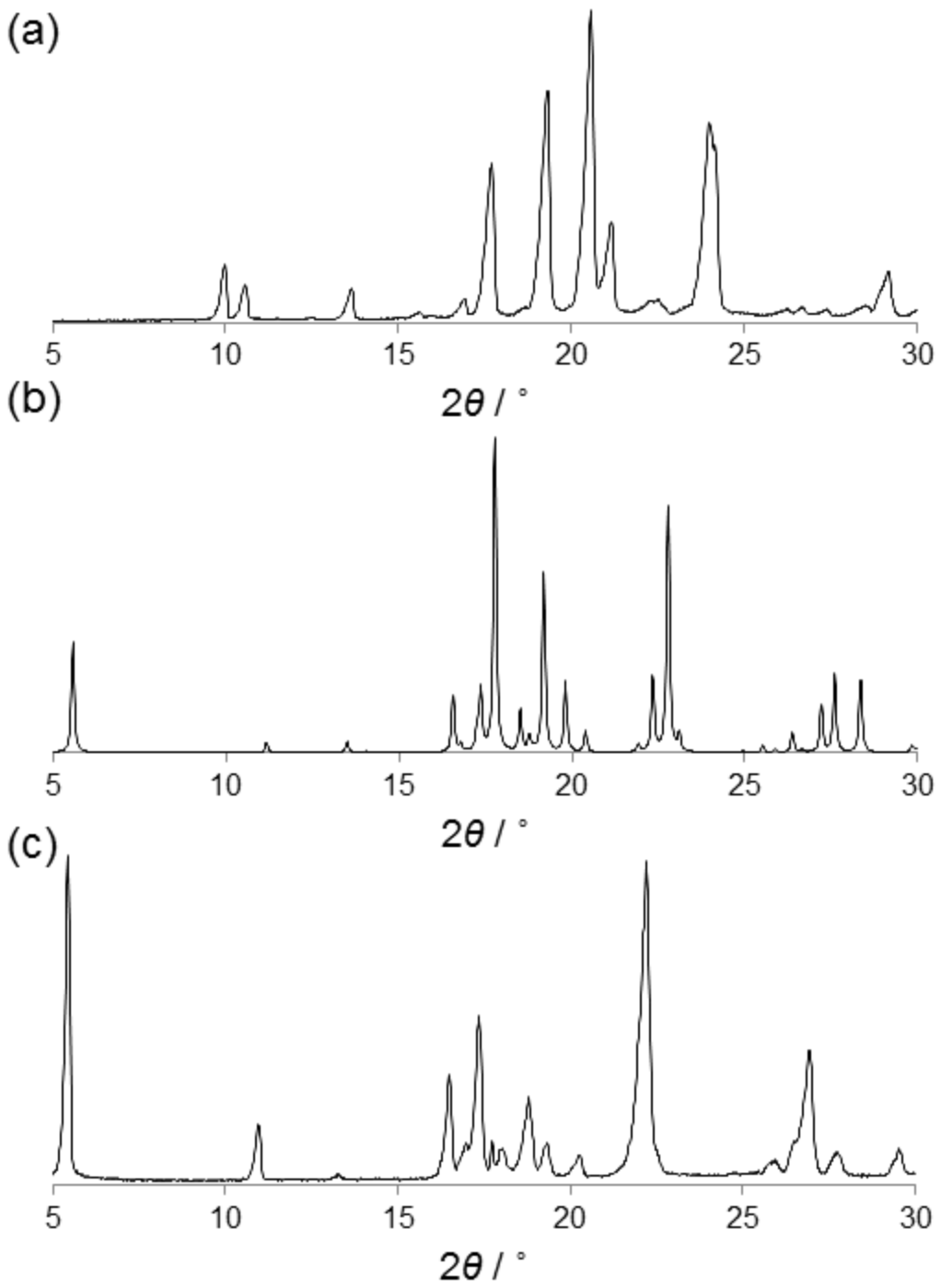


Figure S2. (a) Experimental XRPD pattern of the as-synthesized **L** (300 K). (b) Simulated XRPD pattern of **L** (100 K). (c) Experimental XRPD pattern of **L** (300 K) recrystallized using chloroform showing a good match with the simulated diffraction pattern from single crystal XRD data, indicating that the single crystal structure is representative of the bulk material.

Synthesis of 1·TCM. The 1D coordination network was prepared by treating **L** with $ZnCl_2$ in a chloroform-nitrobenzene-methanol gradient solution and separated as single crystals with formula {[($ZnCl_2$)(L)]·(TCM)}*n* (1·**TCM**, *n* = denotes a polymeric structure). The triple-layered solution was prepared by placing a chloroform solution (5mL) of **L** (0.045 mmol) at the bottom of the crystallization tube, a nitrobenzene layer in the middle (1 mL), and a methanol solution (1 mL) of $ZnCl_2$ (0.045 mmol) as the top layer. Within one week large colourless single crystals were grown and isolated by filtration.

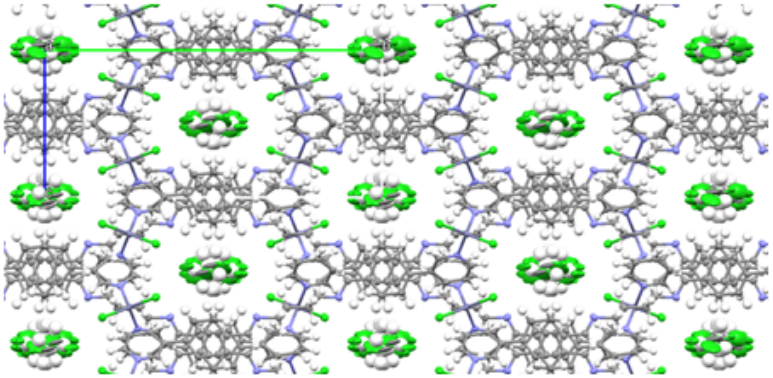


Figure S3. SCXRD structure of **1·DCM** viewed along the *a* -axis respectively.

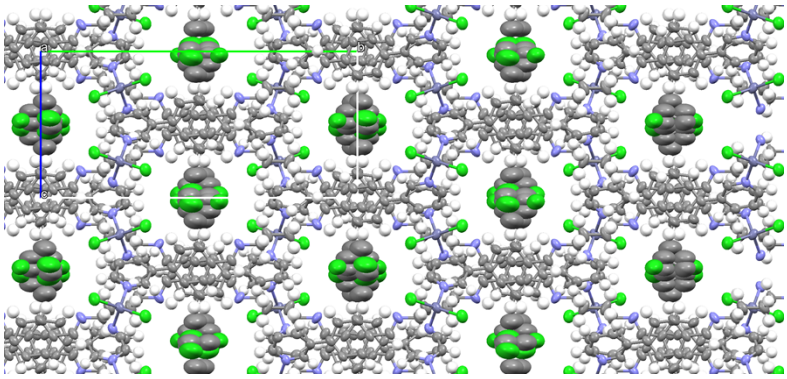


Figure S4. SCXRD structure of **1·DCE** viewed along the *a*-axis respectively.

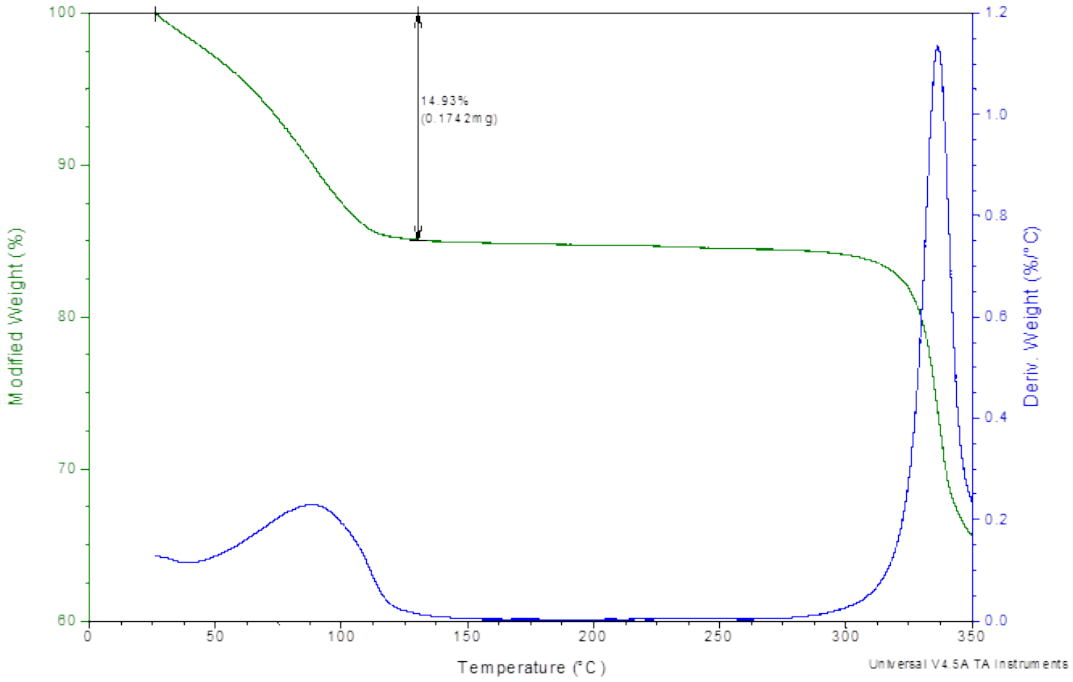


Figure S5. TG plot of **1·TCM**. The calculated weight loss obsd (calcd) and temperature are as follows: $ChCl_3$ 15% (17 %), 30 ˚C-150 ˚C.

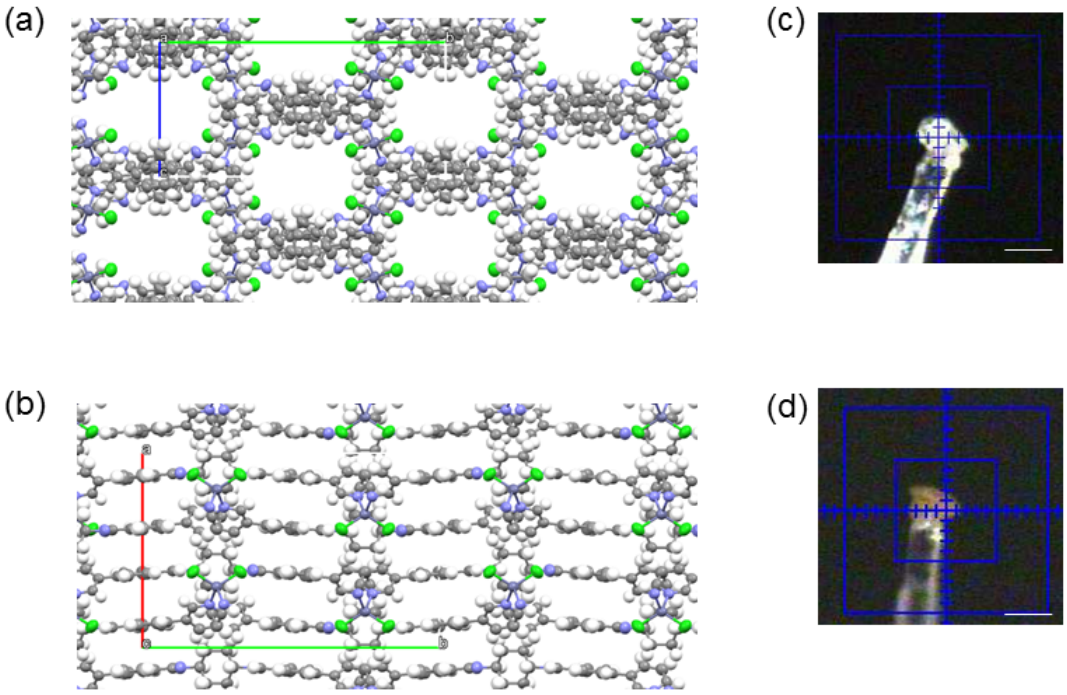


Figure S6. SCXRD structure of **1.** (a) and (c) packing viewed along the *a*- and *c*-axis respectively. (c) and (d) actual single crystal used for the heating experiment. Scale bar 100 μm.

Absorption Correction Details:

The absorption correction was carried out using SADABS program implemented in the Bruker APEX2 Software Suite.

L: Number of reflections was 6554. The R(int) was 0.1433 before and 0.0562 after correction. The Ratio of minimum to maximum transmission is 0.6887.

1·TCE: Number of reflections was 11178. The R(int) was 0.1152 before and 0.0541 after correction. The Ratio of minimum to maximum transmission is 0.7965.

1·DCM: Number of reflections was 34992. The R(int) was 0.1038 before and 0.0502 after correction. The Ratio of minimum to maximum transmission is 0.6667.

1·DCE: Number of reflections was 11886. The R(int) was 0.1052 before and 0.0541 after correction. The Ratio of minimum to maximum transmission is 0.7602.

1: Number of reflections was 12502. The R(int) was 0.0553 before and 0.0314 after correction. The Ratio of minimum to maximum transmission is 0.8447.

checkCIF (standard) running

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checkCIF/PLATON (standard)

You have not supplied any structure factors. As a result the full set of tests cannot be run.

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

Datablock: jm13_100k_0m(L)

Bond precision: C-C = 0.0020 A Wavelength=1.54178

Cell: a=5.5990(4) b=7.1074(5) c=16.1003(11)

alpha=92.646(4) beta=98.583(4) gamma=112.049(3)

Temperature: 100 K

	Calculated	Reported
Volume	583.53(7)	583.53(7)
Space group	P -1	P-1
Hall group	-P 1	?
Moiety formula	C30 H28 N4	?
Sum formula	C30 H28 N4	C30 H28 N4
Mr	444.56	444.56
Dx,g cm-3	1.265	1.265
Z	1	1
Mu (mm-1)	0.584	0.584
F000	236.0	236.0
F000'	236.62	
h,k,lmax	6,8,19	6,8,19
Nref	2060	1997
Tmin,Tmax	0.900,0.916	0.518,0.753
Tmin'	0.890	

Correction method= # Reported T Limits: Tmin=0.518
Tmax=0.753 AbsCorr = MULTI-SCAN

Data completeness= 0.969 Theta(max)= 66.170

R(reflections)= 0.0394(1805) wR2(reflections)= 0.1299(1997)

S = 1.173 Npar= 154

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

Alert level C

PLAT029_ALERT_3_C _diffrn_measured_fraction_theta_full Low ..... 0.969 Note

PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor .... 2.1 Note
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd. # 1 Note
C30 H28 N4

Alert level G

PLAT005_ALERT_5_G No _iucr_refine_instructions_details in the CIF Please Do !
PLAT093_ALERT_1_G No su's on H-positions, refinement reported as . mixed Check
PLAT899_ALERT_4_G SHELXL97 is Deprecated and Succeeded by SHELXL 2014 Note

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

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

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

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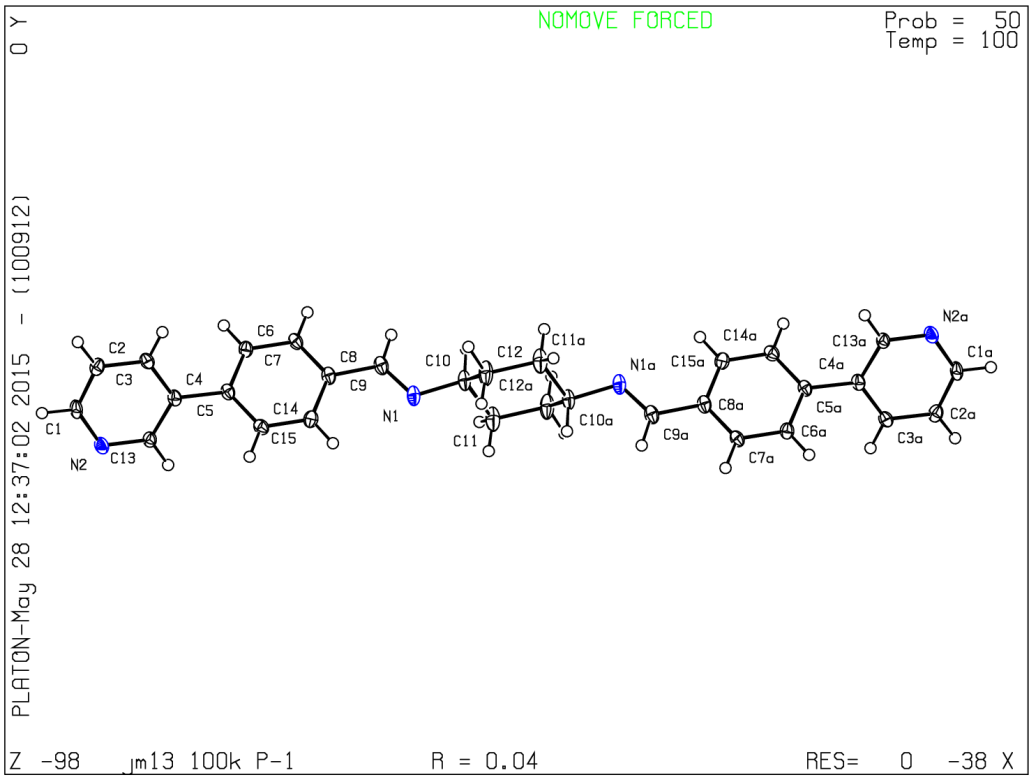
PLATON version of 21/04/2015; check.def file version of 09/03/2015

Datablock jm13_100k_0m(L) - ellipsoid plot



checkCIF (standard) running

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

checkCIF/PLATON (standard)

Structure factors have been supplied for datablock(s) 1_CHCl3

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

Datablock: 1_CHCl3

Bond precision: C-C = 0.0062 A Wavelength=1.54178

Cell: a=14.6608(9) b=22.7977(16) c=9.9218(7)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	3316.2(4)	3316.2(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, 2(C0.50 Cl0.50), 2(Cl)	?
Sum formula	C31 H28 Cl5 N4 Zn	C31 H28 Cl5 N4 Zn
Mr	699.21	699.19
Dx,g cm-3	1.401	1.400
Z	4	4
Mu (mm-1)	4.944	4.944
F000	1428.0	1428.0
F000'	1431.70	
h,k,lmax	17,27,11	17,26,11
Nref	2909	2870
Tmin,Tmax	0.616,0.743	0.600,0.753
Tmin'	0.251	

Correction method= # Reported T Limits: Tmin=0.600
Tmax=0.753 AbsCorr = EMPIRICAL

Data completeness= 0.987 Theta(max)= 66.183

R(reflections)= 0.0760(2638) wR2(reflections)= 0.2268(2870)

S = 1.106 Npar= 221

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

PLAT018_ALERT_1_B _diffrn_measured_fraction_theta_max .NE. _full ! Check

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Alert level C

PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.0062 Ang.
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd. # 1 Note
C30 H28 Cl2 N4 Zn
PLAT906_ALERT_3_C Large K value in the Analysis of Variance ..... 2.929 Check
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.593 38 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much smaller I(calc) . 2 Check
PLAT939_ALERT_3_C Large Value of Not (SHELXL) Weight Optimized S . 10.53

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 6 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 6 Report
PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 1 Info
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large. 0.14 Report
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large. 7.44 Why ?
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 1 Report
PLAT300_ALERT_4_G Atom Site Occupancy of *Cl11 is Constrained at 0.500 Check
PLAT300_ALERT_4_G Atom Site Occupancy of *C51S is Constrained at 0.500 Check
PLAT302_ALERT_4_G Anion/Solvent Disorder ..... Percentage = 100 Note
PLAT432_ALERT_2_G Short Inter X...Y Contact Cl2X .. C51S .. 1.73 Ang.
And 8 other PLAT432 Alerts
More ...
PLAT434_ALERT_2_G Short Inter HL..HL Contact Cl1 .. Cl2X .. 3.04 Ang.
PLAT764_ALERT_4_G Overcomplete CIF Bond List Detected (Rep/Expd) . 1.36 Ratio
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... # 76 Check
CL2X -C51S -CL3X 1.555 1.555 4.555 31.10 Deg.
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 68 Note
PLAT909_ALERT_3_G Percentage of Observed Data at Theta(Max) still 87 %
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Th(Min) ... 1 Report

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0 **ALERT level A** = Most likely a serious problem - resolve or explain
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6 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
24 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
14 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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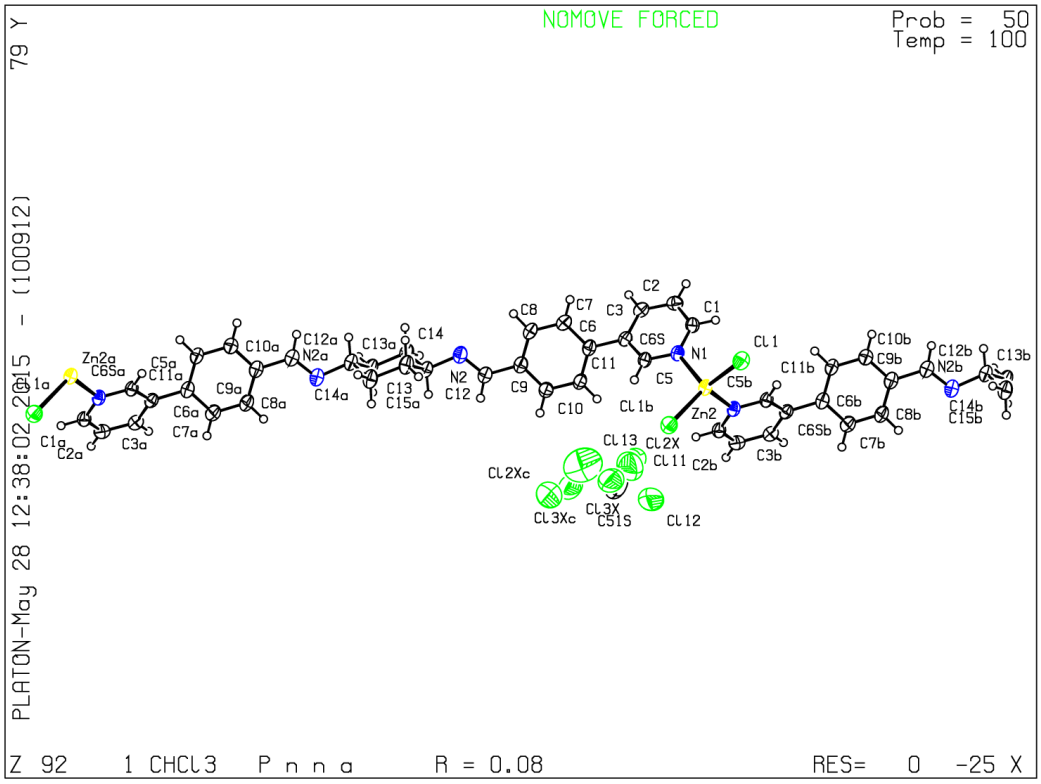
PLATON version of 21/04/2015; check.def file version of 09/03/2015

Datablock 1_CHCl3 - ellipsoid plot



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checkCIF (standard) running

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

checkCIF/PLATON (standard)

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

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No syntax errors found.
Please wait while processing ....
Structure factor report

CIF dictionary
Interpreting this report

Datablock: 1_DCM

Bond precision: C-C = 0.0053 A Wavelength=1.54178

Cell: a=14.6639(9) b=22.7525(14) c=9.8535(6)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	3287.5(3)	3287.5(3)
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, 0.387(C2 H4 Cl4), 0.226(C H2 Cl2)	?
Sum formula	C31 H30 Cl4 N4 Zn	C62 H60 Cl8 N8 Zn2
Mr	665.78	1331.52
Dx,g cm-3	1.345	1.345
Z	4	2
Mu (mm-1)	4.228	4.228
F000	1368.0	1368.0
F000'	1370.21	
h,k,lmax	17,26,11	17,26,11
Nref	2870	2848
Tmin,Tmax	0.632,0.776	0.502,0.753
Tmin'	0.268	

Correction method= # Reported T Limits: Tmin=0.502
Tmax=0.753 AbsCorr = EMPIRICAL

Data completeness= 0.992 Theta(max)= 65.999

R(reflections)= 0.0696(2672) wR2(reflections)= 0.1885(2848)

S = 0.970 Npar= 240

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

SHFSU01_ALERT_2_A The absolute value of parameter shift to su ratio > 0.20
Absolute value of the parameter shift to su ratio given 1.446
Additional refinement cycles may be required.
Author Response: This is due to the disordered guest.

PLAT080_ALERT_2_A Maximum Shift/Error ..... 1.45 Why ?
Author Response: This is due to the disordered guest.

Alert level B

PLAT018_ALERT_1_B _diffrn_measured_fraction_theta_max .NE. _full ! Check

---

Alert level C

RINTA01_ALERT_3_C The value of Rint is greater than 0.12
Rint given 0.171
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd. # 1 Note
C30 H28 Cl2 N4 Zn
PLAT906_ALERT_3_C Large K value in the Analysis of Variance ..... 2.087 Check
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.593 22 Report
PLAT913_ALERT_3_C Missing # of Very Strong Reflections in FCF .... 1 Note

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Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 9 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 9 Report
PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 1 Info
PLAT045_ALERT_1_G Calculated and Reported Z Differ by ..... 2.00 Ratio
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large. 0.12 Report
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large. 10.27 Why ?
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 1 Report
PLAT302_ALERT_4_G Anion/Solvent Disorder ..... Percentage = 100 Note
PLAT434_ALERT_2_G Short Inter HL..HL Contact Cl2 .. Cl1Q .. 3.12 Ang.
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..... 6 Note
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 144 Note
PLAT909_ALERT_3_G Percentage of Observed Data at Theta(Max) still 83 %
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Th(Min) ... 1 Report

---

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Publication of your CIF in other journals

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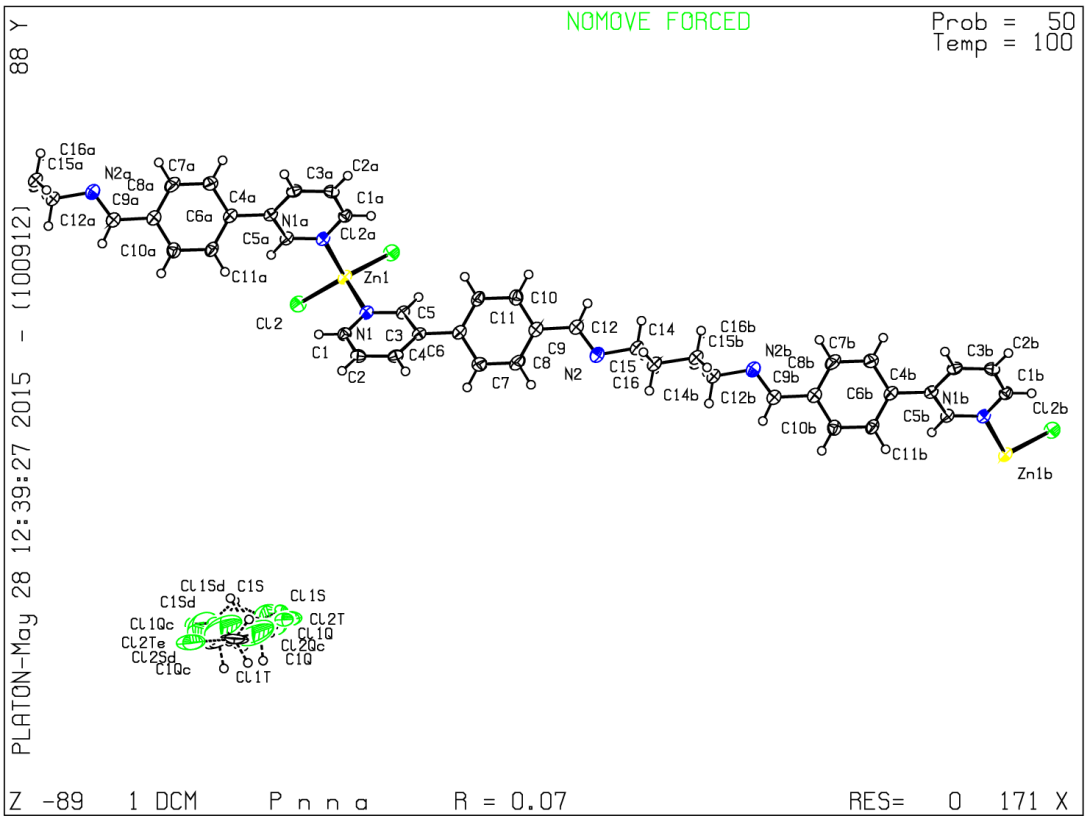
PLATON version of 21/04/2015; check.def file version of 09/03/2015

Datablock 1_DCM - ellipsoid plot



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checkCIF (standard) running

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

checkCIF/PLATON (standard)

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

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

CIF dictionary
Interpreting this report

Datablock: 1_dce

Bond precision: C-C = 0.0058 A Wavelength=1.54178

Cell: a=14.7255(9) b=22.4827(15) c=10.2852(7)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	3405.1(4)	3405.1(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, 0.398(C4), 0.204(C2), 2(Cl)	?
Sum formula	C32 H28 Cl4 N4 Zn	C32 H28 Cl4 N4 Zn
Mr	675.77	675.75
Dx,g cm-3	1.318	1.318
Z	4	4
Mu (mm-1)	4.092	4.092
F000	1384.0	1384.0
F000'	1386.28	
h,k,lmax	17,26,12	17,26,11
Nref	2944	2844
Tmin,Tmax	0.659,0.782	0.577,0.753
Tmin'	0.322	

Correction method= # Reported T Limits: Tmin=0.577
Tmax=0.753 AbsCorr = EMPIRICAL

Data completeness= 0.966 Theta(max)= 65.608

R(reflections)= 0.0765(2288) wR2(reflections)= 0.2187(2844)

S = 1.053 Npar= 225

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 0.184
PLAT018_ALERT_1_B _diffrn_measured_fraction_theta_max .NE. _full ! Check
PLAT020_ALERT_3_B The value of Rint is greater than 0.12 ..... 0.184 Report

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Alert level C

PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density .... 2.07 Report
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= 0.591 91 Report

---

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 6 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 6 Report
PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 1 Info
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large. 0.15 Report
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 2 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records 1 Report
PLAT302_ALERT_4_G Anion/Solvent Disorder ..... Percentage = 100 Note
PLAT432_ALERT_2_G Short Inter X...Y Contact C7 .. C4T .. 3.19 Ang.
PLAT432_ALERT_2_G Short Inter X...Y Contact C8 .. C4T .. 3.13 Ang.
PLAT764_ALERT_4_G Overcomplete CIF Bond List Detected (Rep/Expd) . 1.20 Ratio
PLAT773_ALERT_2_G Check long C-C Bond in CIF: C4S -- C4T . 1.80 Ang.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... # 36 Check
C3S -C3S -C3T 2.665 1.555 2.665 28.00 Deg.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... # 40 Check
C3T -CL3 -C3S 1.555 1.555 1.555 24.00 Deg.
PLAT779_ALERT_4_G Suspect or Irrelevant (Bond) Angle in CIF .... # 43 Check
C3T -C3T -C3S 2.665 1.555 2.665 28.00 Deg.
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 70 Note
PLAT909_ALERT_3_G Percentage of Observed Data at Theta(Max) still 52 %
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Th(Min) ... 1 Report

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

1 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
8 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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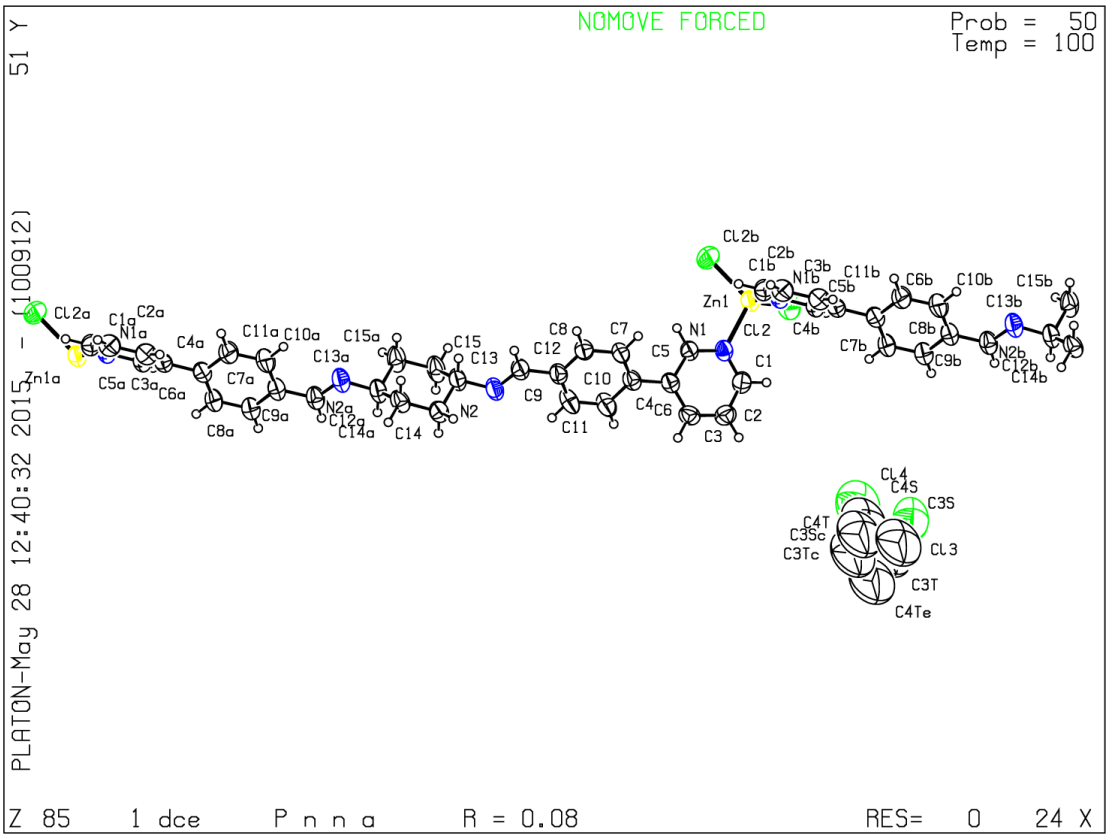
Datablock 1_dce - ellipsoid plot



checkCIF (standard) running

checkCIF/PLATON (standard)

You have not supplied any structure factors. As a result the full set of tests cannot be run.

No syntax errors found. CIF dictionary

Please wait while processing .... Interpreting this report

Datablock: 400k(1)

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

Cell: a=14.7556(10) b=22.5021(14) c=10.5208(7)

alpha=90 beta=90 gamma=90

Temperature: 399 K

	Calculated	Reported
Volume	3493.2(4)	3493.2(4)
Space group	P n n a	Pnna
Hall group	-P 2a 2bc	?
Moiety formula	C30 H28 Cl2 N4 Zn	?
Sum formula	C30 H28 Cl2 N4 Zn	C30 H28 Cl2 N4 Zn
Mr	580.85	580.83
Dx,g cm-3	1.105	1.104
Z	4	4
Mu (mm-1)	2.539	2.539
F000	1200.0	1200.0
F000'	1199.18	
h,k,lmax	17,26,12	17,26,12
Nref	3058	3008
Tmin,Tmax	0.808,0.881	0.636,0.753
Tmin'	0.737	

Correction method= # Reported T Limits: Tmin=0.636
Tmax=0.753 AbsCorr = EMPIRICAL

Data completeness= 0.984 Theta(max)= 66.110

R(reflections)= 0.1020(1849) wR2(reflections)= 0.3315(3008)

S = 1.131 Npar= 168

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test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level A

PLAT601_ALERT_2_A Structure Contains Solvent Accessible VOIDS of . 471 Ang3

Author Response: This is expected as an avacuated channel upon heating to 400K"

Alert level C

RFACG01_ALERT_3_C The value of the R factor is > 0.10
R factor given 0.102
RFACR01_ALERT_3_C The value of the weighted R factor is > 0.25
Weighted R factor given 0.331
PLAT084_ALERT_3_C High wR2 Value (i.e. > 0.25) ..... 0.33 Report
PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density .... 2.74 Report
PLAT234_ALERT_4_C Large Hirshfeld Difference C8 -- C9 .. 0.17 Ang.
PLAT241_ALERT_2_C High Ueq as Compared to Neighbors for ..... C8 Check
PLAT242_ALERT_2_C Low Ueq as Compared to Neighbors for ..... Zn1 Check
PLAT341_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.0103 Ang.

Alert level G

PLAT004_ALERT_5_G Polymeric Structure Found with Maximum Dimension 1 Info
PLAT005_ALERT_5_G No _iucr_refine_instructions_details in the CIF Please Do !
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large. 0.20 Report
PLAT093_ALERT_1_G No su's on H-positions, refinement reported as . mixed Check
PLAT899_ALERT_4_G SHELXL97 is Deprecated and Succeeded by SHELXL 2014 Note

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Datablock 400k(1) - ellipsoid plot