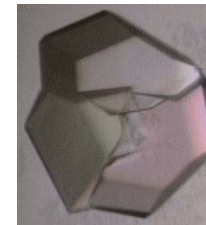




Recipros Wokshop



Paris, June 25th, 2019

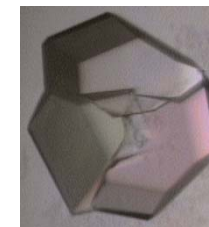
Twinning in Chemical Crystallography (Pseudo-)(Reticular)-Merohedral Examples

rherbst@chemie.uni-goettingen.de

<http://shelx.uni-ac.gwdg.de/~rherbst/twin.html>



Twinning by Pseudo-Merohedry



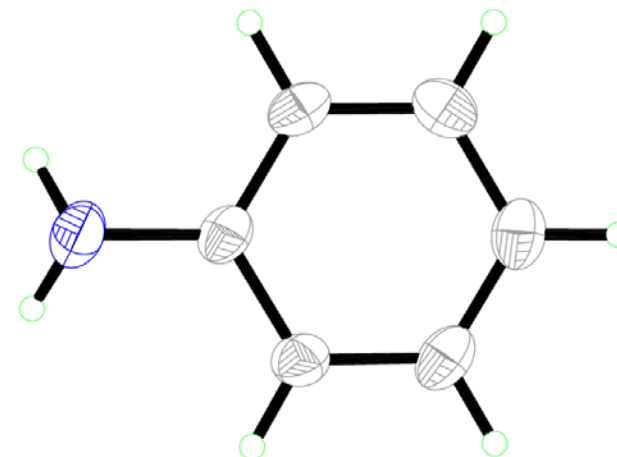
Structure of aniline

cell: 21.645 5.833 8.319 90 101.12 90

space group: $P2_1/c$

$R1 = 0.071$ for 1505 $F_o > 4(F_\sigma)$

$wR2 = 0.198$ for all 1790 data



Analysis of variance for reflections employed in refinement K

$= \text{Mean}[F_o^2] / \text{Mean}[F_c^2]$ for group

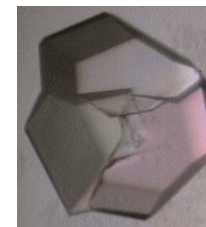
$F_c/F_{c(\max)}$	0.000	0.009	0.017	0.026	0.036	0.047
Number in group	197	164	178	188	173	...
GooF	1.663	1.428	1.579	1.611	1.174	...
K	6.815	1.807	1.486	1.246	1.096	...

s.u. (C - C): 0.004 - 0.005

Residual density maximum: 0.26 e/Å³



Search for Higher Metric Symmetry



Opt. A: FOM = 0.040° orthorhombic C R(int) = 0.300 [5707]
Cell: 8.319 42.477 5.833 90.00 90.00 90.04 V: 2061.20
Matrix: 0.00 0.00 1.00 2.00 0.00 1.00 0.00 1.00 0.00

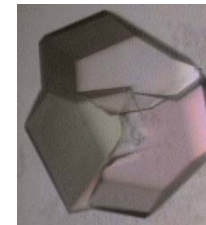
Opt. B: FOM = 0.000° monoclinic P R(int) = 0.110 [4798]
Cell: 8.319 5.833 21.639 90.00 101.04 90.00 V: 1030.60
Matrix: 0.00 0.00 1.00 0.00 1.00 0.00 -1.00 0.00 -1.00

Mean $|E^*E-1| = 0.922$

[expected .968 centrosym and .736 non-centrosym]



TwinRotMat - Output



TwinRotMat

Analysis of Fo/Fc Data for Unaccounted (Non)Merohedral Twinning for: anlln

Cell: 0.71073 21.645 5.833 8.319 90.00 101.12 90.00 Spgr: P21/c
 Criteria: DeltaI/SigmaI .GT. 8.0, DeltaTheta 0.10 Deg., NselMin = 50
 N(refl) = 1790, N(selected) = 50, IndMax = 25, CritI = 0.3, CritT = 0.10

PLATON-Nov 24 10:16:04 2008 - (30506)

2-axls (1 0 0) [2 0 1], Angle () [] = 0.04 Deg, Freq = 39	
(1.000 0.000 1.004) (h1) (h2) Nr Overlap = 1790	
(0.000 -1.000 0.000) * (k1) = (k2) BASF = 0.08	
(0.000 0.000 -1.000) (l1) = (l2) DEL-R = -0.018	
2-axls (1 -2 -2) [0 0 0], Angle () [] = 0.00 Deg, Freq = 10	
(-1.000 -0.670 -0.330) (h1) (h2) Nr Overlap = 479	
(0.000 0.341 0.659) * (k1) = (k2) BASF = 0.06	
(0.000 1.341 -0.341) (l1) = (l2) DEL-R = -0.003	
2-axls (1 -4 -2) [0 0 0], Angle () [] = 0.00 Deg, Freq = 8	
(-1.000 -0.445 -0.109) (h1) (h2) Nr Overlap = 208	
(0.000 0.781 0.438) * (k1) = (k2) BASF = 0.05	
(0.000 0.891 -0.781) (l1) = (l2) DEL-R = -0.001	
2-axls (4 -2 1) [1 -6 2], Angle () [] = 0.56 Deg, Freq = 7	
(-0.549 -2.658 0.880) (h1) (h2) Nr Overlap = 136	
(-0.226 0.329 -0.440) * (k1) = (k2) BASF = 0.00	
(0.113 -0.664 -0.780) (l1) = (l2) DEL-R = 0.000	

101 0-10 00-1
 BASF = 0.08
 DEL-R = -0.018

1
2
3
4

anlln

INPUT INSTRUCTIONS via KEYBOARD or LEFT-MOUSE-CLICKS (HELP with RIGHT CLICKS)

vv

TwRoMt MENU

NRefSelMin

DeltaI/SigI

MaxIndexUVW

DeltaTheta

FullListing

EPS-TwinLaw

DspTwinMat1

DspTwinMat2

DspTwinMat3

DspTwinMat4

EPS-TwinLat

Resolution>

Zone-H,K,L

Up Down

RacemicTwin

SelectTMat1

SelectTMat2

SelectTMat3

SelectTMat4

HKLF5-CritI

HKLF5-CritT

HKLF5-Gener

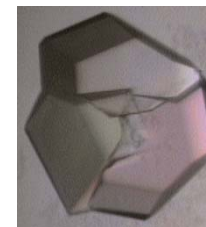
End

Exit

MenuActive



Twin Refinement



```
TWIN 1 0 1 0 -1 0 0 0 -1  
BASF .2
```

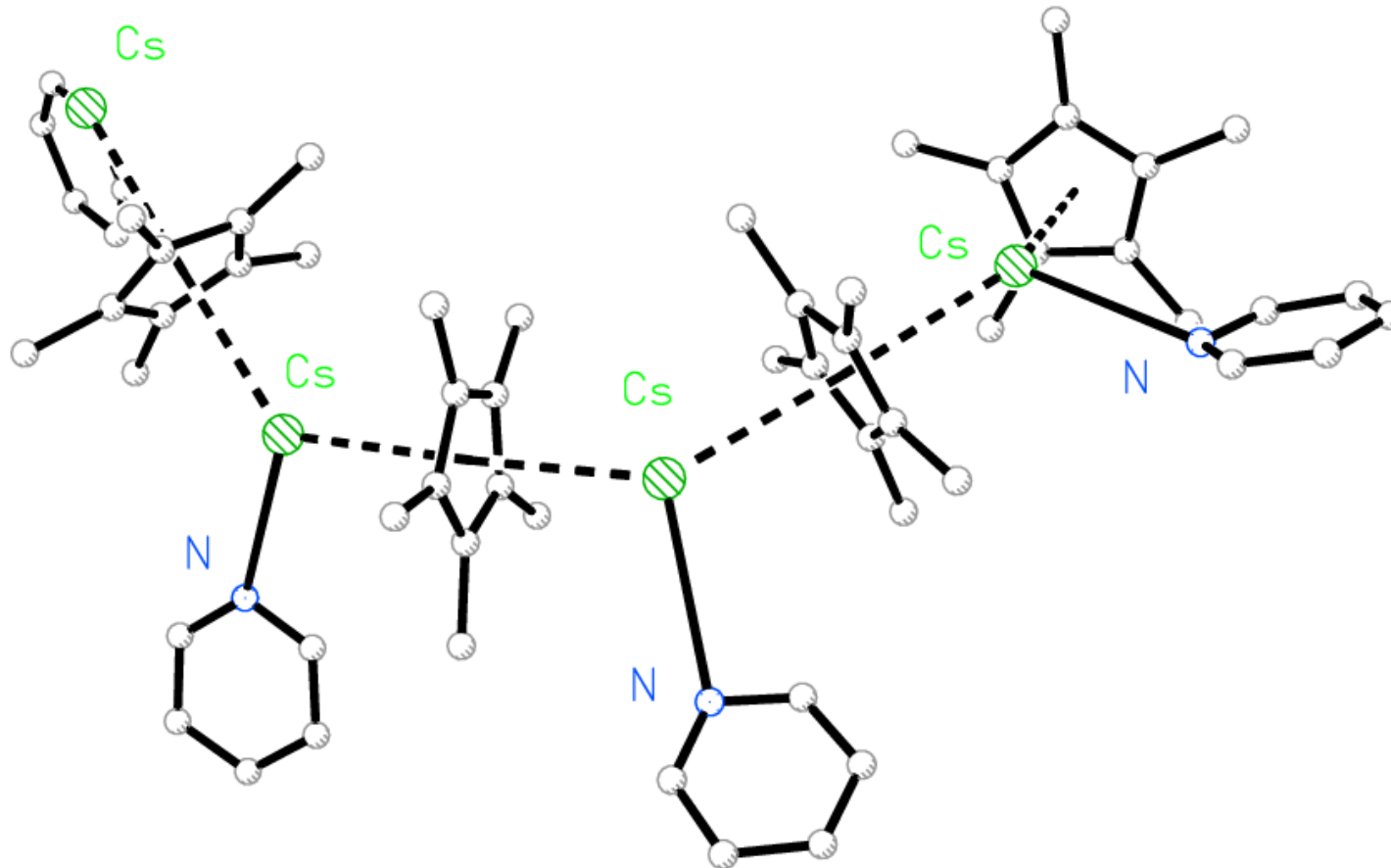
Comparison of the results:

	without TWIN	with TWIN
R1 ($F > 4\sigma(F)$)	0.071	0.047
wR2 (all data)	0.198	0.123
k_2	-	0.0734(1)
Res. electron density	0.26	0.20
s.u.(C - C)	0.004 - 0.005	0.003
K (weakest reflections)	6.815	0.956

P. Müller, R. Herbst-Irmer, A. L. Spek, T. R. Schneider, M. R. Sawaya, Crystal Structure Refinement – A Crystallographer's Guide to SHELXL, Oxford University Press 2006



Merohedral Twin

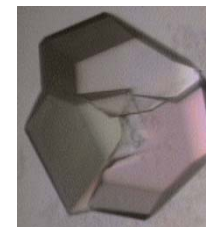


cell: 11.370 11.370 20.496 90.00 90.00 120.00

R. Herbst-Irmer, *Z. Kristallogr.* **2016**, 231, 573-581.



Space Group Determination



Crystal system H and Lattice type P selected

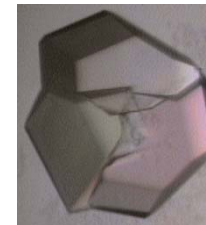
Mean $|E^*E-1| = 0.529$ [expected .968 centrosym and .736 non-centrosym]

Systematic absence exceptions:

	$6_1/6_5$	$6_2=3_1$	6_3	-C-	--C	
N	15	12	9	553	301	
N I>3 σ	1	1	1	535	279	
<I>	1.3	1.3	1.4	265.5	253.1	
<I/ σ >	1.1	1.1	1.3	42.6	40.3	
Opt.	Space Gr.	CSD	R(int)	N(eq)	Syst. Abs.	CFOM
[A]	P6 ₁	62	0.059	4946	1.3 / 34.8	6.18
[B]	P6 ₅	62	0.059	4946	1.3 / 34.8	6.18
[C]	P6 ₁ 22	20	0.148	5326	1.3 / 34.8	78.62
[D]	P6 ₅ 22	20	0.148	5326	1.3 / 34.8	78.62



XPREP – Test for Merohedral Twinning



[1] -3 / -31m:

R(int) 0.060(4612)/0.142(898), $\langle |E^2-1| \rangle$ 0.505/0.487

TWIN 0 -1 0 -1 0 0 0 -1 BASF 0.382 [C] or 0.350 [NC]

[2] -3 / -3m1:

R(int) 0.060(4612)/0.156(824), $\langle |E^2-1| \rangle$ 0.505/0.486

TWIN 0 1 0 1 0 0 0 -1 BASF 0.365 [C] or 0.328 [NC]

[3] -3 / 6/m:

R(int) 0.060(4612)/0.012(962), $\langle |E^2-1| \rangle$ 0.505/0.514

TWIN -1 0 0 0 -1 0 0 1 BASF 0.481 [C] or 0.475 [NC]

[4] -31m / 6/mmm:

R(int) 0.142(898)/0.021(444), $\langle |E^2-1| \rangle$ 0.487/0.505

TWIN -1 0 0 0 -1 0 0 1 BASF 0.479 [C] or 0.474 [NC]

[5] -3m1 / 6/mmm:

R(int) 0.156(824)/0.022(518), $\langle |E^2-1| \rangle$ 0.486/0.506

TWIN -1 0 0 0 -1 0 0 1 BASF 0.481 [C] or 0.476 [NC]

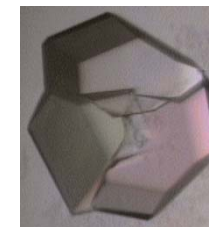
[6] 6/m / 6/mmm:

R(int) **0.012(962)/0.183(380)**, $\langle |E^2-1| \rangle$ 0.514/0.505

TWIN **0 1 0 1 0 0 0 -1** BASF **0.365 [C] or 0.328 [NC]**



Solution



Space Group $P6_1$

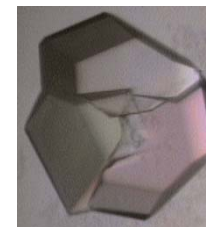
- **SHELXS:**
CFOM 0.0528, RE = 0.198 for C_{20} Cs
- **SHELXD:**
 C_{12} Cs best final CC 79.34
- **SHELXD with TWIN 0 1 0 1 0 0 0 0 -1 and BASF 0.33:**
 C_{12} Cs best final CC 87.04

- **SHELXT:**

R1	Alpha	Space group	[Flack(x)]	Formula
0.202	0.037	P6/m		C54 Cs
0.182	0.032	P-6	0.49	C58 N95
0.209	0.036	P6	0.44	C90 N72



Solution and Refinement



SHELXS in $P6_1$: 1 Cs

```
TWIN 0 1 0 1 0 0 0 0 -1  
BASF 0.4
```

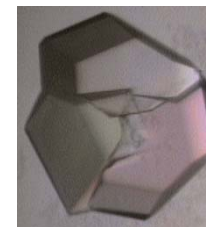
Difference Fourier with these coordinates:

	without	with TWIN
R1(F > 4 σ (F))	0.25	0.13
wR2 (all data)	0.62	0.38
k ₂		0.355(9)
E-density	only few peaks	nearly the whole structure interpretable

R1 = 0.022

wR2 = 0.057

K2 = 0.341(2)



BUT:

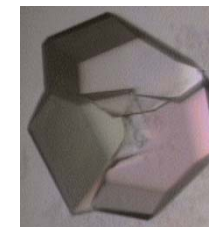
**** Absolute structure probably wrong –
invert and repeat refinement****

Flack x: 0.83(5)

Parsons 0.67(6)



Additional Twinning by Inversion?



Perhaps four twin domains with following indices:

h, k, l

$k, h, -l$ TWIN matrix

$-h, -k, -l$ inversion

$-k, -h, l$ TWIN matrix and inversion

```
TWIN 0 1 0 1 0 0 0 0 -1 -4
BASF .2 .2 .2
```

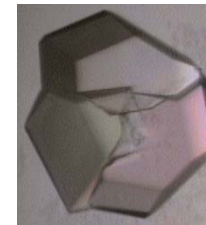
Parameter	Value	s.u.	Indices
k_1	$1-(k_2+k_3+k_4)$		h, k, l
k_2	0.00	0.03	$k, h, -l$
k_3	0.70	0.04	$-h, -k, -l$
k_4	0.34	0.03	$-k, -h, l$

correct space group $P6_5$:

```
MOVE 1 1 1 -1
TWIN 0 1 0 1 0 0 0 0 -1
```



Different Refinements

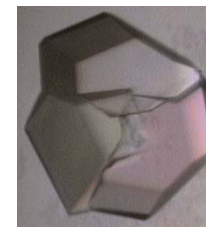


- A: Space group $P6_1$
TWIN $0\ 1\ 0\ 1\ 0\ 0\ 0\ -1$
- B: Space group $P6_1$
TWIN $0\ -1\ 0\ -1\ 0\ 0\ 0\ 1$
- C: Space group $P6_5$
TWIN $0\ -1\ 0\ -1\ 0\ 0\ 0\ 1$
- D: Space group $P6_5$
TWIN $0\ 1\ 0\ 1\ 0\ 0\ 0\ -1$

	R1	wR2	K_2	Flack x	bond precision (C - C)
A	0.022	0.057	0.341(2)	0.83(5)	0.0163
B	0.021	0.054	0.341(2)		0.0160
C	0.020	0.049	0.340(2)		0.0145
D	0.018	0.046	0.340(1)	-0.02(4)	0.0138



Pseudo-Merohedral Twin: Example 2



Very instable compound, difficult to mount,

But good crystal quality

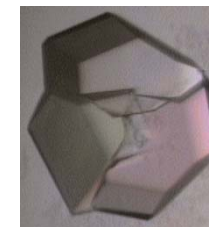
unknown Al-compound, possible composition $C_{27}H_{26}AlN_2$,
perhaps some I from the starting material

cell: 16.934 16.934 12.603 90.00 90.00 120.00

hexagonal metric



Crystal System



Search for higher metric symmetry

Option A: FOM = 0.00 deg. HEXAG. P-lattice R(sym) = 0.653 [10906]
Cell: 16.934 16.934 12.603 90.00 90.00 120.00 Volume: 3129.81
Matrix: 1.000 0.000 0.000 0.000 1.000 0.000 0.000 0.000 1.000

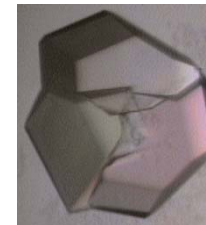
Option B: FOM = 0.00 deg. ORTHO. C-lattice R(sym) = 0.493 [11989]
Cell: 16.934 29.330 12.603 90.00 90.00 90.00 Volume: 6259.63
Matrix: 0.000 1.000 0.000 2.000 1.000 0.000 0.000 0.000 -1.000

Option C: FOM = 0.00 deg. ORTHO. C-lattice R(sym) = 0.493 [12004]
Cell: 16.934 29.330 12.603 90.00 90.00 90.00 Volume: 6259.63
Matrix: 1.000 1.000 0.000 -1.000 1.000 0.000 0.000 0.000 1.000

Option D: FOM = 0.00 deg. ORTHO. C-lattice R(sym) = 0.033 [12024]
Cell: 16.934 29.330 12.603 90.00 90.00 90.00 Volume: 6259.63
Matrix: 1.000 0.000 0.000 1.000 2.000 0.000 0.000 0.000 1.000



Orthorhombic?



Crystal system O and Lattice type C selected

Mean $|E^*E-1| = 0.702$ [expected .968 centrosym and .736 non-centrosym]

Systematic absence exceptions:

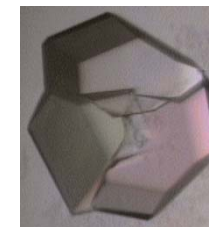
	c--	n--	-c-	-n-	--a	--b	--21
N	1259	1259	785	785	1602	1602	41
N I>3s	891	891	548	548	0	0	0
<I>	29.2	29.2	65.3	65.3	0.2	0.2	0.1
<I/s>	28.0	28.0	35.0	35.0	0.3	0.3	0.3

Opt. Space Gr.	No.	CSD	R(sym)	N(eq)	Syst. Abs.	CFOM
----------------	-----	-----	--------	-------	------------	------

No acceptable space group - change tolerances or unset chiral flag
or possibly change input lattice type, then recheck cell using H-option



Monoclinic Space Group?



Cell: 16.934 12.603 16.934 90.00 120.00 90.00

Monoclinic P

$R_{\text{int}} = 0.026$

Crystal system M and Lattice type P selected

Mean $|E^*E-1| = 0.714$ [expected .968 centrosym and .736 non-centrosym]

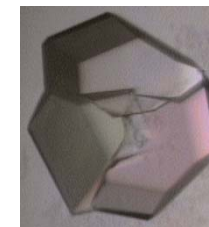
Systematic absence exceptions:

	-21-	-a-	-c-	-n-
N	41	1732	1726	1602
N ($I > 3\sigma$)	0	839	839	0
$\langle I \rangle$	0.1	35.3	35.4	0.2
$\langle I/\sigma \rangle$	0.3	17.9	18.0	0.3

Opt. [A]	Space Gr.	No. #	CSD	R(sym)	N(eq)	Syst. Abs.	CFOM
	P2(1)/n	14	19410	0.026	7746	0.3 / 17.9	7.05



Solution

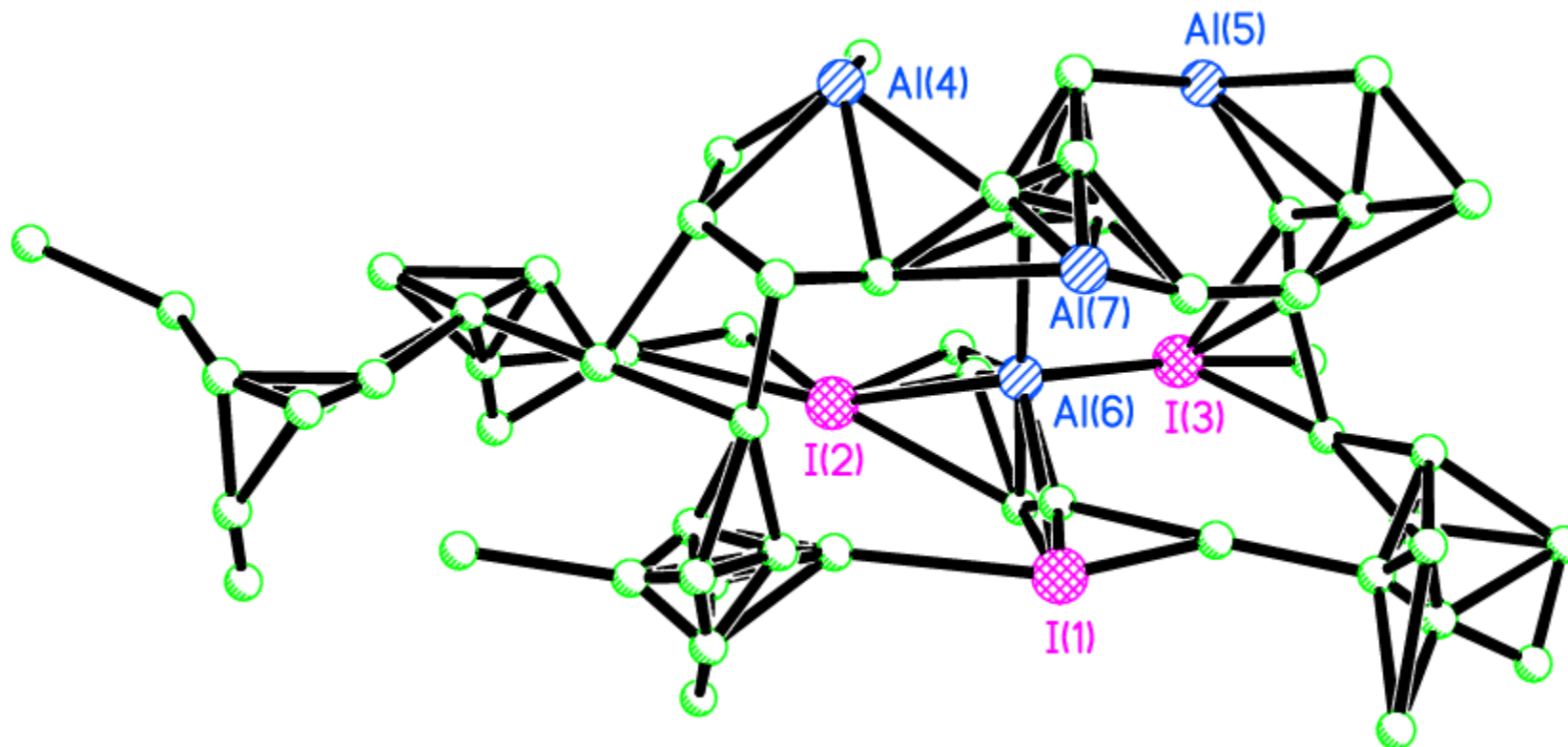
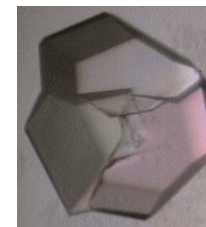


Space group $P2_1/n$

- **SHELXS:**
CFOM 0.1293, RE = 0.359 for C_{40} Al₅ I₃
- **SHELXD:**
 C_{24} Al I₃ best final CC 70.67
- **SHELXD with TWIN 0 0 1 0 -1 0 1 0 0 and BASF 0.5:**
 C_{23} Al I₃ best final CC 83.80
- **SHELXT :**
space group **Pn**
R1 = 0.257, Alpha = 0.062, Flack x= 0.49, C_{107} Al₁₀ I₇



SHELXS-Solution in $P2_1/n$

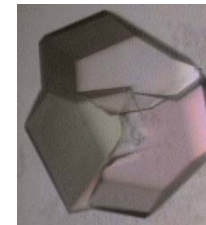


RE = 0.359 for 48 atoms and 2982 E-values

AlI_3 unit can be identified



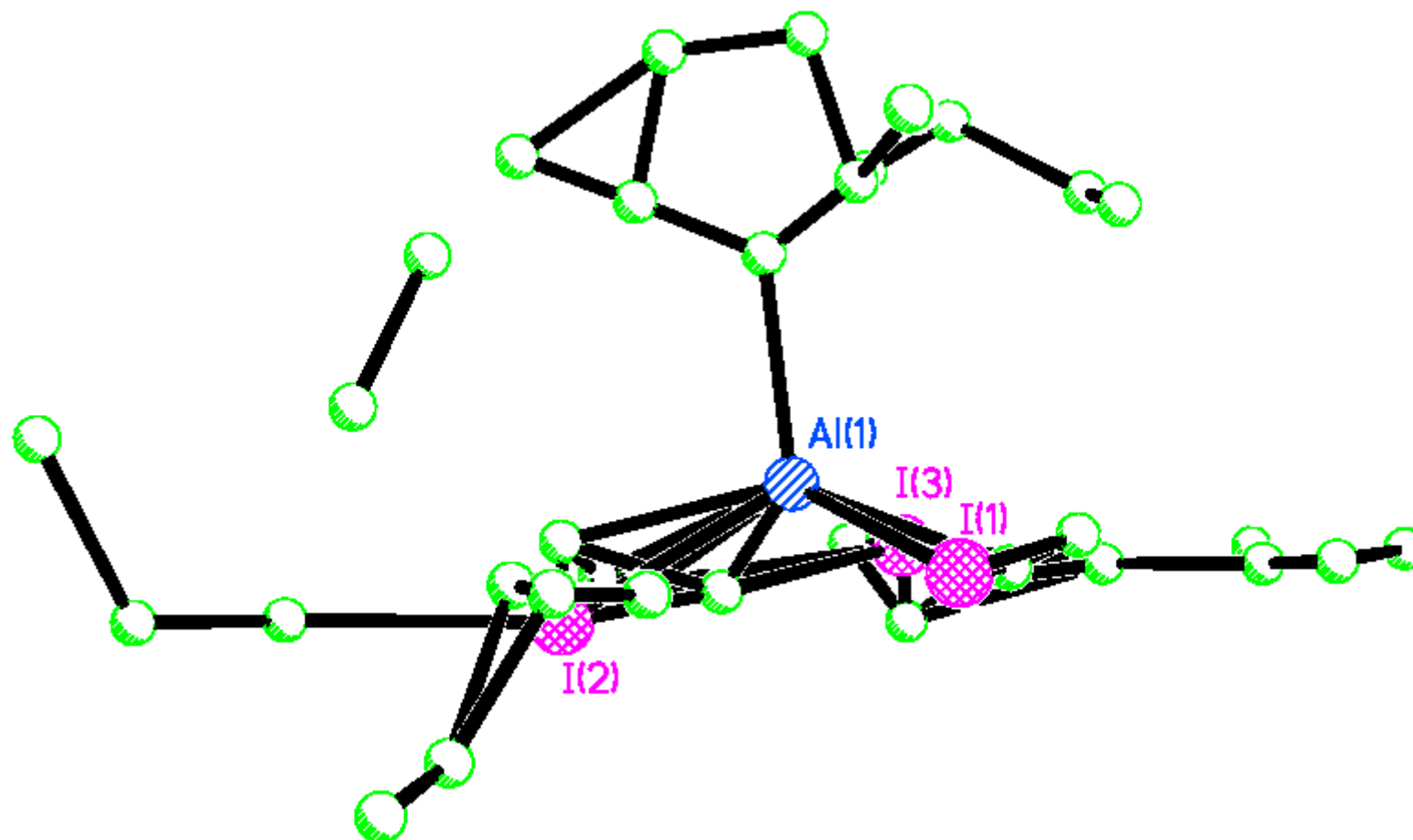
Refinement in $P2_1/n$



Refinement of the AlI_3 unit

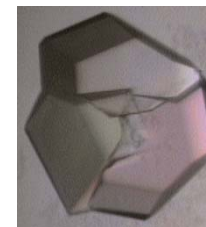
$R1 = 0.341$

$wR2 = 0.760$





Twin Law



TwinRotMat

Analysis of Fo/Fc Data for Unaccounted (Non)Merohedral Twinning for: mono1

Cell: 0.71073 16.934 12.603 16.934 90.00 120.00 90.00 Spgr: P21/n
 Criteria: $\Delta \theta = 0.10$ Deg., NselMin = 50
 N(refl) = 5, CrItI = 0.1, CrItT = 0.10

2-axls (h k l) [h' k' l']	Angle (deg)	Freq	Nr Overlap	BASF	DEL-R	
(0.000 0.000 0.000) [0.000 0.000 0.000]	0.00	52	8654	0.50	-0.207	1
(-1.000 0.000 0.000) [1.000 0.000 0.000]	0.00	52	8420	0.17	-0.033	2
(1.000 0.000 0.000) [2.000 0.000 1.000]	0.00	53	8464	0.17	-0.032	3
(-0.575 1.150 -0.425) [0.425 0.150 -0.425]	2.28	13	1444	0.15	-0.006	4

PLATON-day, 18 June 2019 - (230917)
 Fc from Coordinates
 mono1 P 21/n R = 0.34

$0\ 0\ -1\ 0\ -1\ 0\ -1\ 0\ 0$
 BASF = 0.50
 DEL-R = -0.207

$-1\ 0\ 0\ 0\ -1\ 0\ 1\ 0\ 1$
 BASF = 0.17
 DEL-R = -0.033

$1\ 0\ 1\ 0\ -1\ 0\ 0\ 0\ -1$
 BASF = 0.17
 DEL-R = -0.032

TwinRotM 25

NRefSelMin

Delta/Sigl

MaxIndexUVW

DeltaTheta

FullListing

EPS-TwinLaw

DspTwinMat1

DspTwinMat2

DspTwinMat3

DspTwinMat4

EPS-TwinLat

Resolution>

localFromFCF

Zone-H,K,L

Up Down

RacemicTwin

SelectTMat1

SelectTMat2

SelectTMat3

SelectTMat4

HKLF5-CritI

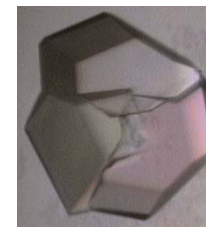
HKLF5-CritT

HKLF5-Gener

End

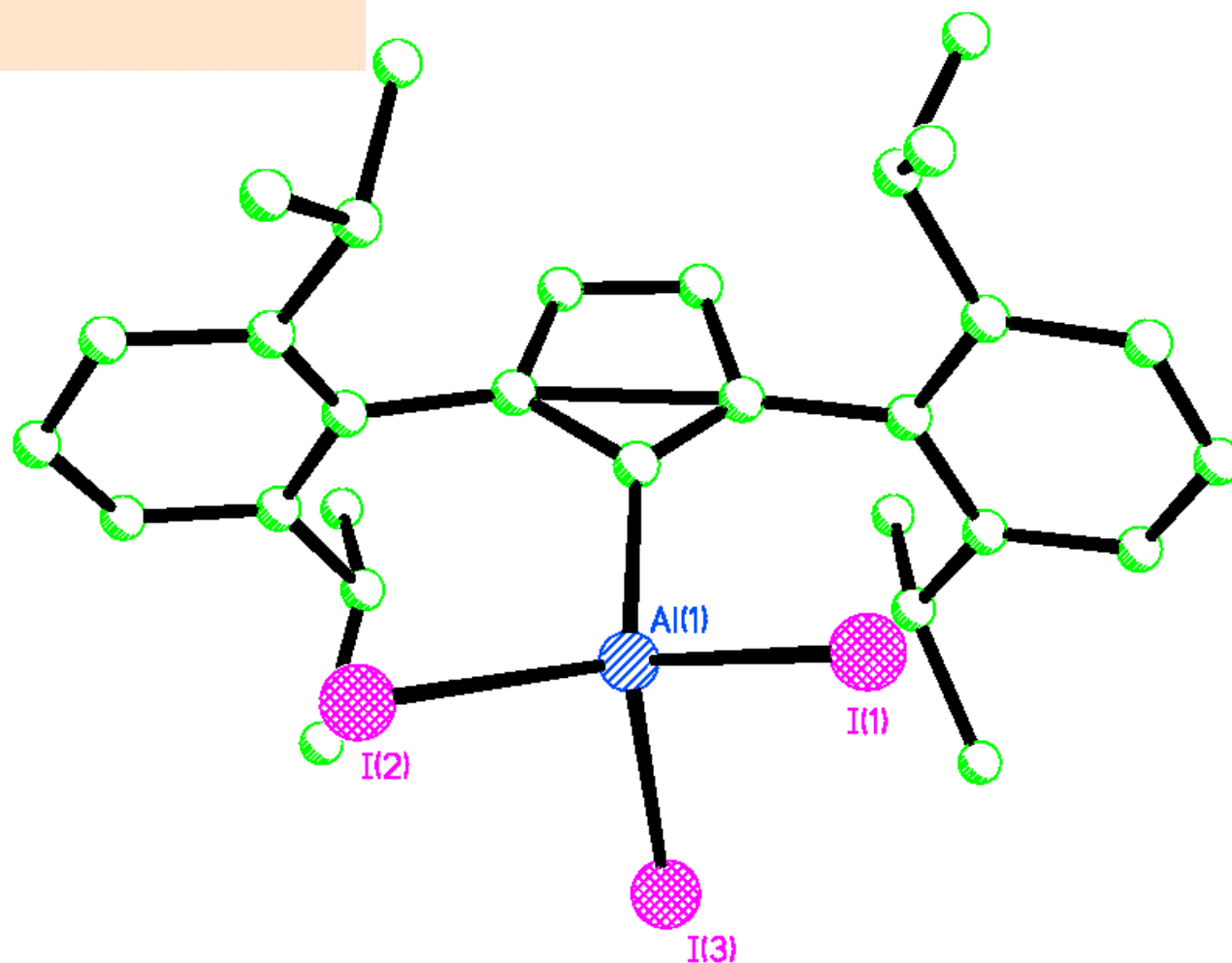


Refinement as Twin



TWIN 1 0 1 0 -1 0 0 0 -1

BASF 0.5



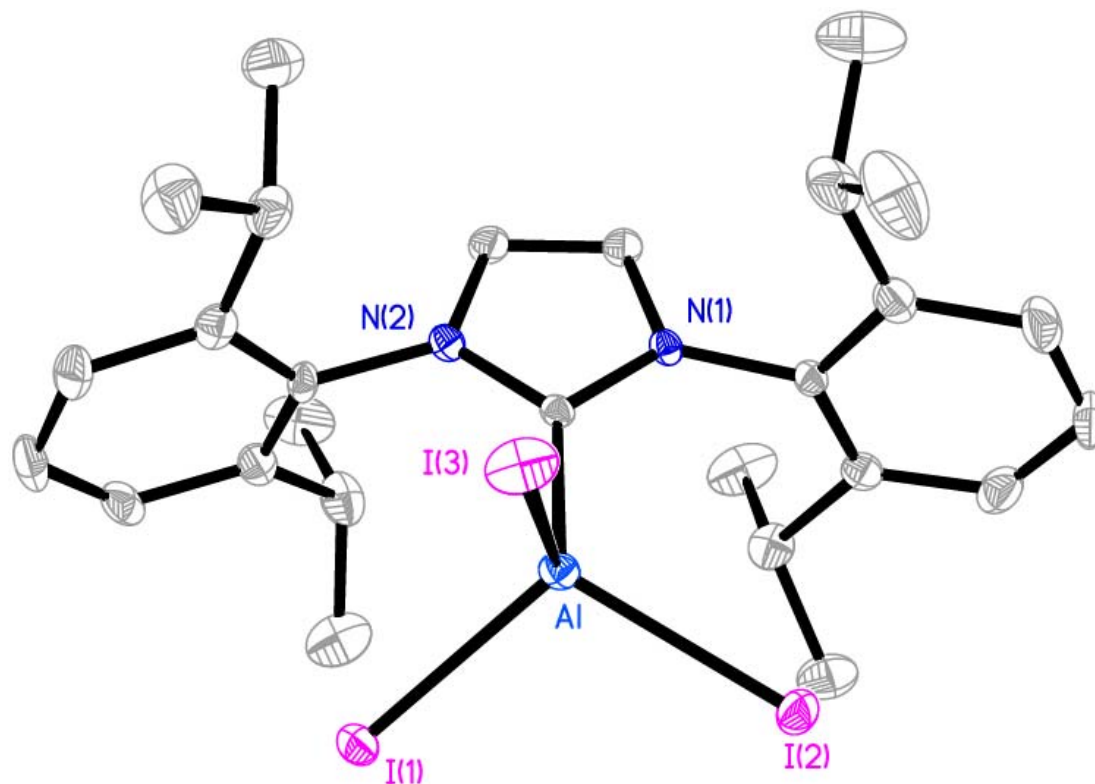
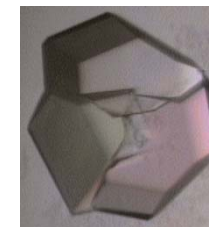
R1 = 0.139

wR2 = 0.398

Definition



Final Results



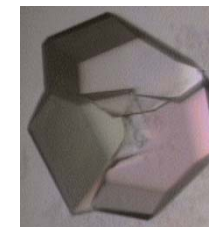
R1 ($F > 4\sigma(F)$)	0.022	wR2 (all data)	0.046
k_2	0.4982(4)	Parameter	307
Data	8918	Residual Density [$e\text{\AA}^{-3}$]	1.07

R. S. Ghadwal, H. W. Roesky, R. Herbst-Irmer, P. G. Jones, *Z. Anorg. Allgem. Chem.*, **2009**, 635, 431-433.

R. Herbst-Irmer, *Z. Kristallogr.*, **2016**, 231, 573-581.

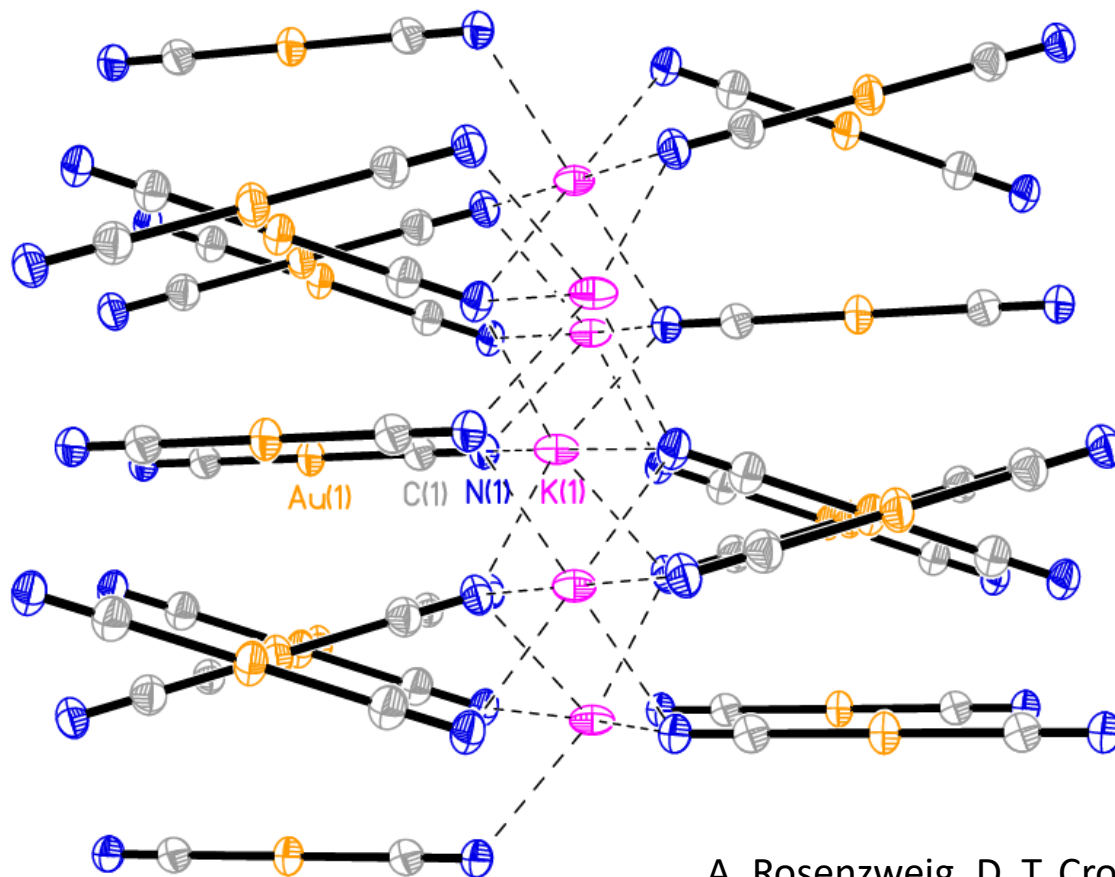


Twinning by Reticular Merohedry



Structure of $\text{K}[\text{Au}(\text{CN})_2]$

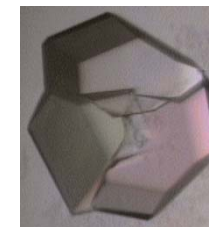
cell: 7.240 7.240 26.445 90 90 120, space group $R\bar{3}$



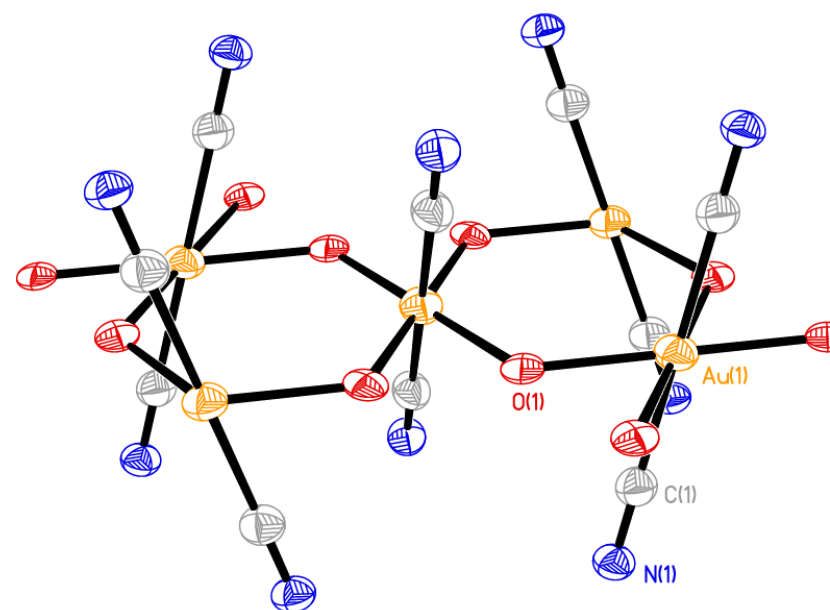
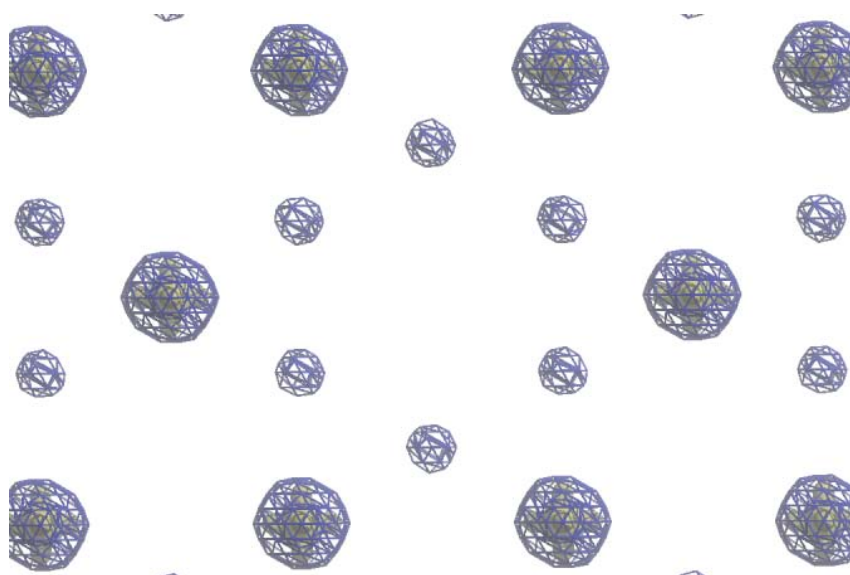
A. Rosenzweig, D. T. Cromer, *Acta Crystallogr.* **12**, 709, 1959.



$K[Au(CN)_2]$



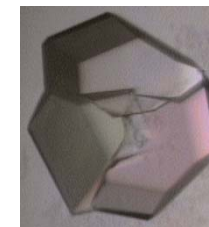
R1 = **0.074** for $640 F_o > 4\sigma(F_o)$, wR2 = **0.172** for all 648 data



R1 = **0.027** for $640 F_o > 4\sigma(F_o)$, wR2 = **0.076** for all 648 data
Residual density: 1.18/-1.48 e/A³



Warning Signs



Systematic Absences Violations:

2	0	0	8.08	2.00	observed but should be systematically absent
-1	0	1	507.42	32.65	observed but should be systematically absent
-1	0	1	610.89	37.97	observed but should be systematically absent
1	0	-1	517.12	34.48	observed but should be systematically absent
0	-1	-1	540.26	33.43	observed but should be systematically absent
1	-1	1	512.14	35.24	observed but should be systematically absent
10	-1	-1	557.75	34.37	observed but should be systematically absent

...

Most Disagreeable Reflections

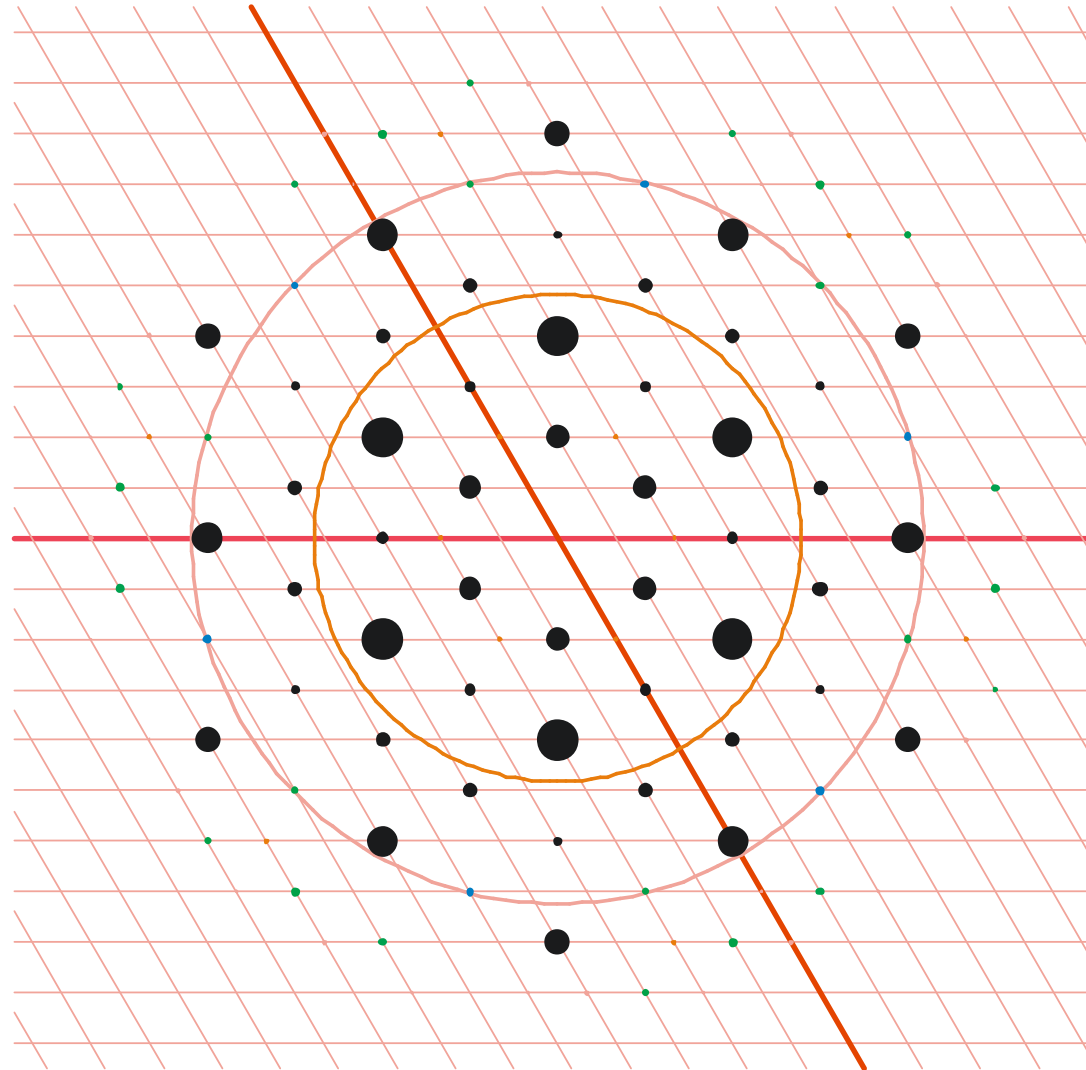
h	k	l	F_o^2	F_c^2	$\Delta(F^2)/\sigma$	F_c/F_{cmax}	Res.(Å)
0	3	0	1907.04	407.73	11.79	0.026	2.09
-1	2	6	7075.12	11145.69	6.78	0.137	2.80
1	1	6	1275.08	818.27	3.69	0.037	2.80
-1	2	0	27026.22	32870.20	3.53	0.235	3.60
-1	2	3	47884.52	56252.36	2.98	0.307	3.35
-1	2	12	7698.09	9417.93	2.98	0.126	1.88
-5	4	6	642.68	966.24	2.77	0.040	1.31

...

R. Herbst-Irmer, G. M. Sheldrick, Refinement of obverse/reverse twins, *Acta Crystallogr.* **2002**, B58, 477-481.

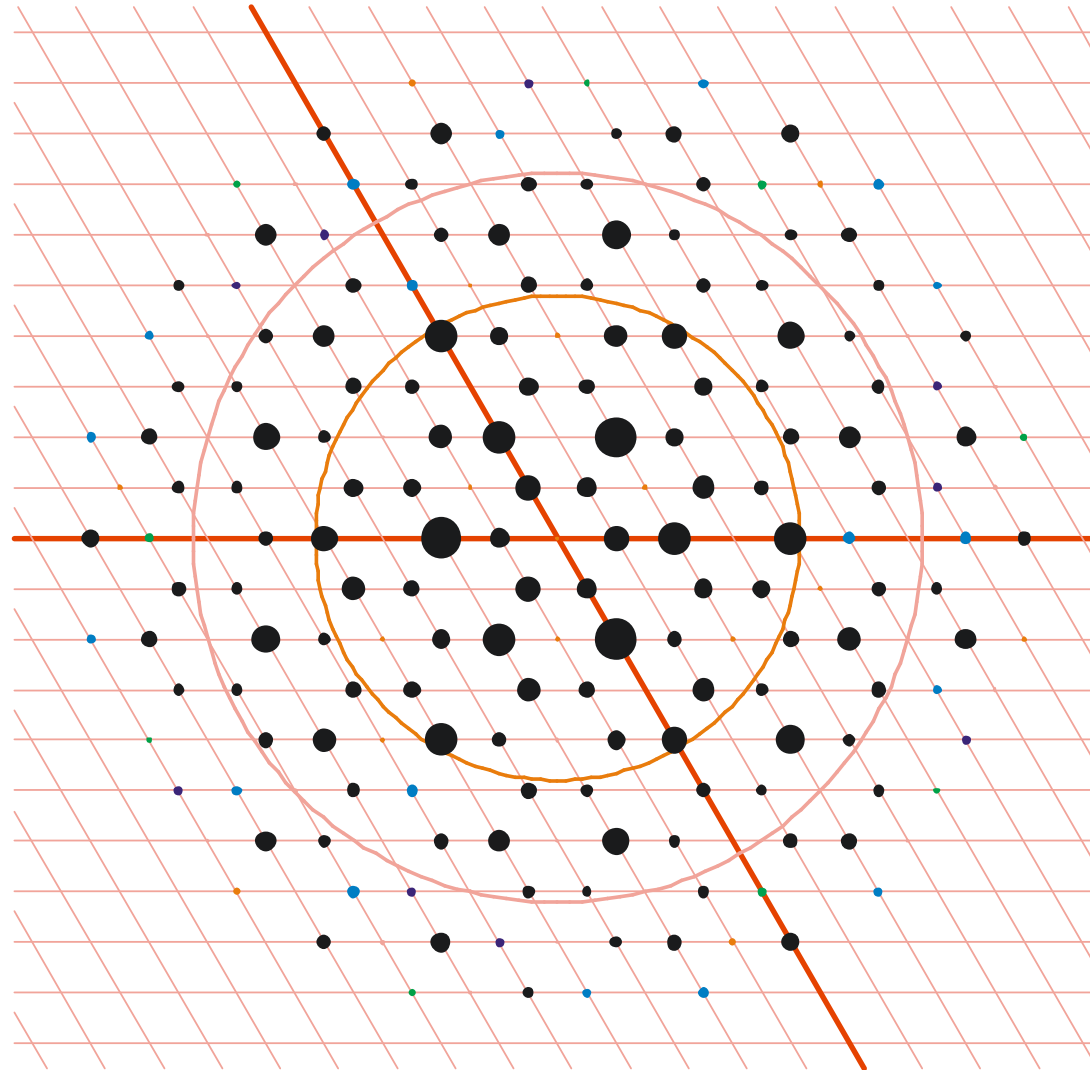


Reciprocal Space Plot $l = 0$



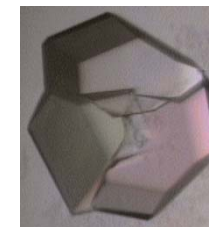


Reciprocal Space Plot $l = 2$





Obverse/Reverse Twinning



	P	A	B	C	I	F	Obv	Rev	All
N	0	4252	4264	4282	4258	6399	5663	5715	8516
N I > 3 σ	0	2177	2189	2180	2198	3273	1698	1887	4447
<I>	0.0	303.7	302.2	132.1	303.5	245.8	93.4	246.6	341.6
<I/ σ >	0.0	6.0	6.0	5.4	6.0	5.8	3.3	4.2	6.1

Obverse/reverse test for trigonal/hexagonal lattice

Mean I: obv only **619.5**, rev only **252.1**, neither obv nor rev **0.5**,

Preparing dataset for refinement with BASF **0.289** and TWIN **-1 0 0 0 -1 0**

0 0 1

Reflections absent for both components will be removed



Possible Twin Laws



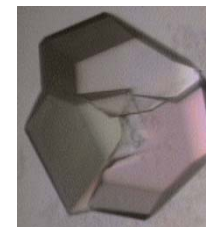
True Laue Group	Apparent	Twin Law								
$\bar{3}$	6/mmm	0	-1	0	-1	0	0	0	0	-1
		0	1	0	1	0	0	0	0	-1
		-1	0	0	0	-1	0	0	0	1

$R\bar{3}$ \Rightarrow systematic absences: $-h + k + l = 3n$

0	1	0	1	0	0	0	0	-1	\Rightarrow	merohedral twin
0	-1	0	-1	0	0	0	0	-1	\Rightarrow	obverse reverse twin
-1	0	0	0	-1	0	0	0	1	\Rightarrow	obverse reverse twin



Obverse/Reverse Twinning



P.L.A.T.O.N.

TwinRotMat

Analysis of Fo/Fc Data for Unaccounted (Non)Merohedral Twinning for: hp33

Cell: 0.71073 7.240 7.240 26.445 90.00 90.00 120.00 Spgr: R-3
 Criteria: DeltaI/SigmaI .GT. 4.0, DeltaTheta 0.10 Deg., NselMin = 50
 N(refl) = 648, N(selected) = 50, IndMax = 5, CritI = 0.1, CritT = 0.10

2-axls (0 1 0) [1 2 0], Angle () [] = 0.00 Deg, Freq = 50	0 -1 0 -1 0 0 0 0 -1
(-1.000 0.000 0.000) (h1) (h2) (1.000 1.000 0.000) * (k1) = (k2) (0.000 0.000 -1.000) (l1) (l2) DEL-R = -0.044	
2-axls (0 0 1) [0 0 1], Angle () [] = 0.00 Deg, Freq = 46	-1 0 0 0 -1 0 0 0 1
(-1.000 0.000 0.000) (h1) (h2) (0.000 -1.000 0.000) * (k1) = (k2) (0.000 0.000 1.000) (l1) (l2) DEL-R = -0.039	
2-axls (1 -2 0) [0 -1 0], Angle () [] = 0.00 Deg, Freq = 50	0 1 0 1 0 0 0 0 -1
(-1.000 -1.000 0.000) (h1) (h2) (0.000 1.000 0.000) * (k1) = (k2) (0.000 0.000 -1.000) (l1) (l2) DEL-R = 0.000	

PLATON-Sep 02 09:57:00 2011 - (300311)
 Fc from Fo/Fc File

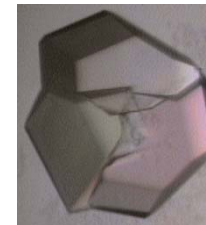
hp33

TwinRoMt 25

NRefSelMin
 Delta/Sigl
 MaxIndexUVW
 Delta Theta
 FullListing
 EPS-TwinLaw
 DspTwinMat1
 DspTwinMat2
 inMat3
 inMat4
 EPS-TwinLat
 Resolution>
 mCIF
 Zone-H,K,L
 Up Down
 cTwin
 Mat1
 SelectTMat2
 SelectTMat3
 SelectTMat4
 HKLF5-CritI
 HKLF5-CritT
 HKLF5-Gener
 End



Refinement as Twin



SHELXL-97:

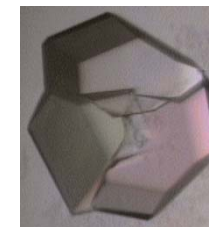
```
BASF 0.3  
HKLF 5
```

SHELXL-13

```
TWIN 0 -1 0 -1 0 0 0 0 -1  
BASF 0.3  
HKLF 4
```




Refinement as Twin

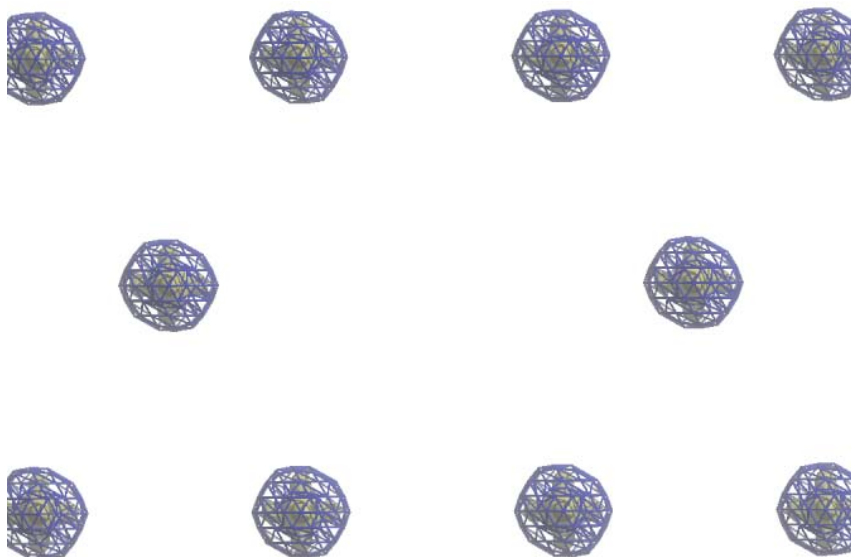


TWIN 0 -1 0 -1 0 0 0 0 -1
BASF 0.3

TWIN -1 0 0 0 -1 0 0 0 1
BASF 0.3

R1 = 0.018
wR2 = 0.043
 k_2 = 0.287(3)
Res. Dens. = 1.0 e/Å³

0.030
0.089
0.219(5)
3.2 e/Å³



S. R. Hettiarachchi, B. K. Schaefer, R. L. Yson,
R. J. Staples, R. Herbst-Irmer, H. H.
Patterson, *Inorg. Chem.*, **2007**, **46**, 6997-
7004.
R. Herbst-Irmer, *Z. Kristallogr.*, **2016**, **231**,
573-581.



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