Structure Determination of Unsolvated Potassium, Rubidium, and Cesium Carbazolate

Robert Dinnebier[a], Hoda Esbak[b], Falk Olbrich[b], and Ulrich Behrens[b]*

[a] Max-Planck Institute for Solid State Research, Heisenbergstrasse 1, D-70569 Stuttgart, Germany

[b] Institut für Anorganische und Angewandte Chemie der Universität Hamburg, Martin-Luther-King-Platz 6, D-20146 Hamburg, Germany

---

* To whom correspondence should be addressed. Fax: +49(0)40/42838-2893. E-mail: ubehrens@chemie.uni-hamburg.de

Supporting Information for the Compounds potassium carbazolate ($KNC_{12}H_8$, 1), rubidium carbazolate ($RbNC_{12}H_8$, 2), and cesium carbazolate ($CsNC_{12}H_8$, 3).

X-ray powder diffraction data of the compounds **1** to **3** were collected at low temperatures with a STOE *Stadi-P* transmission diffractometer (Debye-Scherrer geometry, Ge(111) monochromator, Cu-$K_{\alpha 1}$-radiation, linear PSD) with the samples sealed under Argon in glass capillaries of 0.3 mm diameter. All samples were spun during measurement for better particle statistics.

Data reduction on all three data sets was performed using the GUFI program [1]. Indexing with ITO [2] led to R-centered trigonal unit cells for **1** and **2**, and to a body-centered monoclinic unit cell for **3**. The extinctions found in the powder patterns indicated either *R3c, or R-3c* (**1**), and (**2**), respectively, and *Ia*, or *I*2/*a* for (**3**), as the most probable space groups, of which *R*3*c* and *Ia* could later be confirmed by Rietveld refinements [3] to be the appropriate groups. The peak profiles and precise lattice parameters were determined by LeBail fits [4] using the program GSAS [5]. The background was modelled manually using GUFI. The peak-profile was described by a pseudo-Voigt function [6] in combination with a special function that accounts for the asymmetry due to axial divergence [7]. All powder patterns of the compounds **1** to **3** exhibit some anisotropic peak broadening caused by lattice strain. The phenomenological strain model of Stephens [8] as implemented in GSAS was used to model the anisotropy of the FWHM.

The crystal structures were solved by global optimization in real space using the DASH structure solution package [9] for the laboratory data sets. The measured powder patterns were subjected to Pawley refinements [10] in order to extract correlated integrated intensities from the pattern. Good fits to the data were obtained. An internal coordinate description of the rigid carbazolate moiety was constructed using bond lengths, and angles from the structure determination of $K(NC_{12}H_8)$(18-crown-6) [11]. The position of the alkali cation as well as the position and orientation of the carbazolate anion in the unit cell were postulated and the trial structure was subjected to a global optimization [12]. The structure giving the best fit to the data was validated by Rietveld refinement of the fractional coordinates obtained at the end of the simulated annealing run.

Rietveld refinements employing soft constraints for bond distances (C-C, C-N, C-H), bond angles (C-N-C, C-C-C, C-C-H) and planarity within the carbazolate anion were performed on all powder patterns of (**1**), (**2**), and (**3**) using the program package GSAS. The background and starting values for peak profile were taken from the corresponding LeBail fits. The strength of the soft constraints was lowered during the refinement cycles until no significant improvement in the R-factors could be detected. All refinements converged satisfactorily. Agreement factors (R-values), atomic coordinates, and a selection of intra- and inter-molecular distances and angles are given in the following tables.

References:

[1] Dinnebier, R. E.; Finger, L.: GUFI 5.0., *Z. Krist. Supplement Issue* **15** (1998) 148.

[2] Visser, J. W.: program for finding the unit cell from powder data, *J. Appl. Crystallogr.* **2** (1969) 89-95.

[3] Rietveld, H. M.: profile refinement method for nuclear and magnetic structures, *J. Appl. Crystallogr.* **2** (1969) 65-71.

[4] Le Bail, A.; Duroy, H.; Fourquet, J. L. *Mat. Res. Bull.* **23** (1988) 447-452.

[5] Larson, A. C.; von Dreele, R. B.: GSAS Version 2002, *Los Alamos National Laboratory Report* LAUR 86-748 (1994).
[6] Thompson, P.; Cox, D. E.; Hastings, J. B. *J. Appl. Crystallogr.* **20** (1987) 79-83.
[7] Finger, L. W.; Cox, D. E.; Jephcoat, A. P. *J. Appl. Crystallogr.* **27** (1994) 892-900.
[8] Stephens, P. W. *J. Appl. Crystallogr.* **32** (1999) 281-289.
[9] CCDC, 2004. DASH: A structure solution package for X-ray powder diffraction, developed and distributed between the ISSIS Facility of the Rutherford Appleton Laboratory and the Cambridge Chrystallographic Data Centre. Version 3.0 (2004).
[10] Pawley, G. S. *J. Appl. Crystallogr.* **14** (1981) 357-361.
[11] Esbak, H.; Behrens, U. *Z. Anorg. Allg. Chem.* **631** (2005) 1581 – 1587.
[12] David, W. I. F.; Shankland, K.; Shankland, N. *J. Chem. Soc. Chem. Commun.* (1998) 931 - 932.

Table 1. Crystallographic Data for potassium carbazolate ($KNC_{12}H_8$, **1**)

formula	$C_{12}H_8KN$
FW (g mol^{-1})	205.30
crystal system	trigonal
space group	*R*3*c*; No. 161
temperature (K)	100
cell parameters: a (Å)	25.0827(9)
c (Å)	7.8355(2)
V ($Å^3$)	4269.2(2)
Z	18
density (calc., g cm^{-3})	1.437
2θ range (deg.)	4.0 – 60.1
steps of scan	δ2θ = 0.01 deg. with 60 sec. each step
FWHM 2θ (deg.)	0.122
λ (Å)	1.5406
No. of refl. in refinement	138
No. of refined variables	69
R-values: R_{wp}	0.0562
R_p	0.0442
R_{F2}	0.0992
Diffractometer:	STOE *Stadi-P* transmission diffractometer

Table 2. Atom coordinates and isotropic thermal parameters for $KNC_{12}H_8$ (**1**)

Atom	x	y	z	U_i/U_e
K1	0.35634(19)	0.26900(23)	0.5277(6)	0.0234(17)
N1	0.23826(5)	0.26129(28)	0.53328(13)	0.0178(26)
C1	0.19091(10)	0.20617(23)	0.58973(34)	0.0178(26)
C2	0.15641(5)	0.21256(21)	0.72543(20)	0.0178(26)
C3	0.10655(9)	0.16003(25)	0.7962(4)	0.0178(26)
C4	0.09035(19)	0.10162(22)	0.7330(7)	0.0178(26)
C5	0.12392(24)	0.09541(22)	0.5993(9)	0.0178(26)
C6	0.17401(19)	0.14750(27)	0.5276(7)	0.0178(26)
C7	0.23410(7)	0.30412(22)	0.63203(25)	0.0178(26)
C8	0.18451(7)	0.27751(21)	0.75189(22)	0.0178(26)
C9	0.17447(15)	0.31573(30)	0.8613(5)	0.0178(26)
C10	0.21312(23)	0.37966(28)	0.8540(8)	0.0178(26)
C11	0.26186(23)	0.40574(20)	0.7371(8)	0.0178(26)
C12	0.27240(14)	0.36812(22)	0.6264(5)	0.0178(26)

H3	0.08340(7)	0.1640(4)	0.88782(29)	0.0178(26)
H4	0.05605(22)	0.06568(26)	0.7815(9)	0.0178(26)
H5	0.11253(31)	0.05521(24)	0.5567(11)	0.0178(26)
H6	0.19687(23)	0.1430(4)	0.4360(8)	0.0178(26)
H9	0.14113(15)	0.2981(4)	0.9417(5)	0.0178(26)
H10	0.20619(30)	0.4058(4)	0.9294(10)	0.0178(26)
H11	0.28817(29)	0.44962(20)	0.7328(10)	0.0178(26)
H12	0.30587(14)	0.38624(30)	0.5465(6)	0.0178(26)

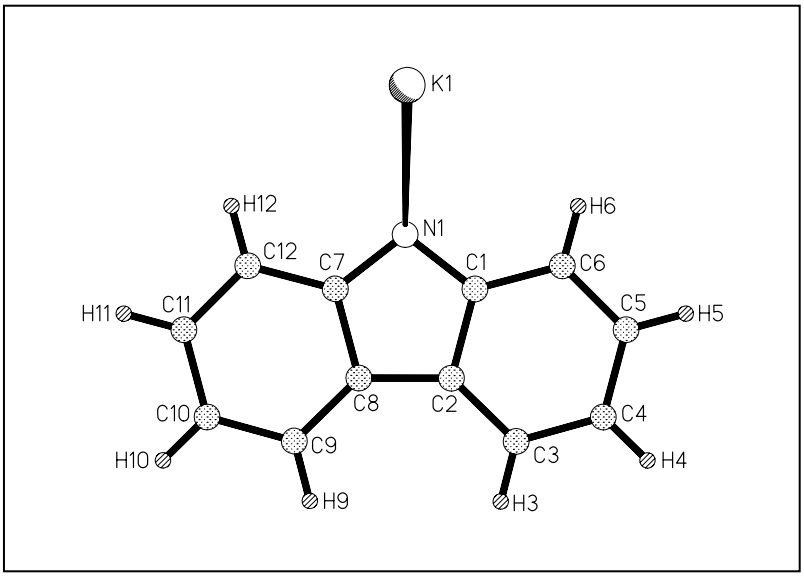


Fig. 1: Asymmetric unit of $KNC_{12}H_8$ (**1**)

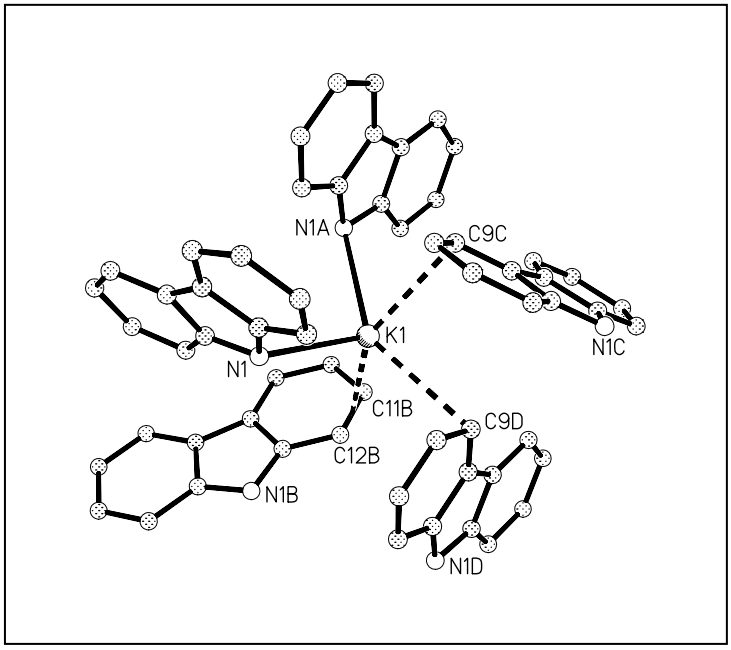


Fig. 2: Coordination of five $NC_{12}H_8^-$ anions to a K^+-ion in $KNC_{12}H_8$ (**1**). Symmetry operations, A: -y+2/3, x-y+1/3, z+1/3; B: -x+y+1/3, -x+2/3, z-1/3; C: -y+2/3, -x+1/3, z-1/6; D: -y+2/3, x-y+1/3, z-2/3.

Fig. 3 – 5: K-C interactions, up to 3.40 Å, in **1**.

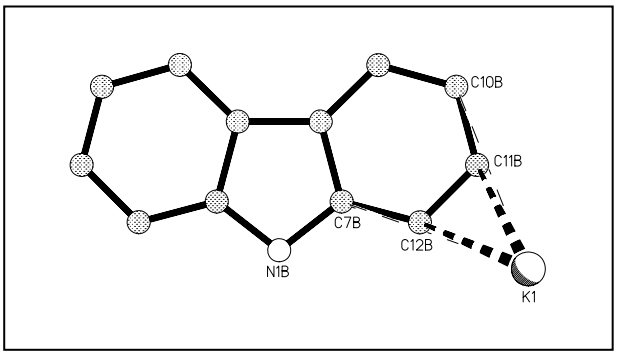


Fig. 3

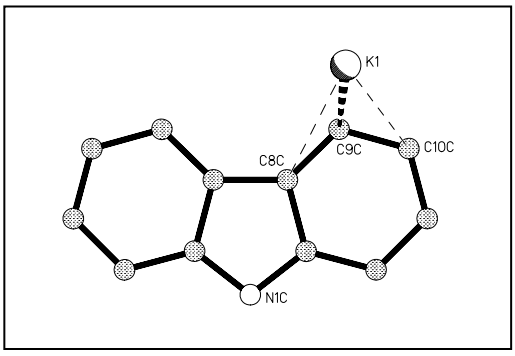


Fig. 4

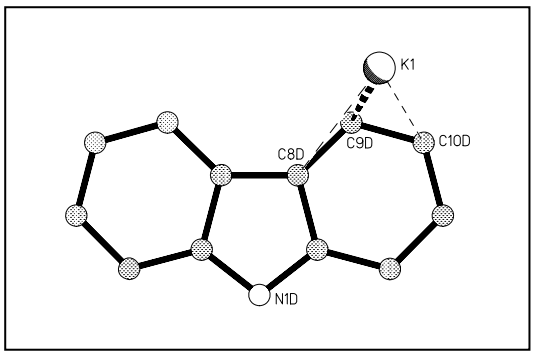


Fig. 5

Table 3. Bond lengths [Å] and angles [deg.] for $KNC_{12}H_8$ (**1**)

K1–K1a	4.293(1)	K1–N1	2.870(5)	K1–N1a	2.892(6)
K1–C9c	2.998(5)	K1–C9d	3.208(5)	K1–C12b	3.293(5)
K1–C11b	3.378(6)	N1–C1	1.370	N1–C7	1.370
C1–C2	1.430	C1–C6	1.400	C2–C3	1.400
C2–C8	1.430	C3–C4	1.400	C4–C5	1.400
C5–C6	1.400	C7–C8	1.430	C7–C12	1.400
C8–C9	1.400	C9–C10	1.400	C10–C11	1.400
C11–C12	1.400	C–H	0.960		

N1–K1–N1a	104.5(2)	N1–K1–C9d	103.9(2)
N1–K1–C9c	111.2(2)	N1–K1–X1a	107.4(2)
N1–K1–C11b	116.6(2)	N1–K1–C12b	97.7(2)
N1a–K1–C9c	75.2(2)	N1a–K1–C11b	84.1(1)
N1a–K1–C12b	103.7(2)	C9c–K1–C9d	80.3(1)
C9d–K1–X1a	92.3(2)	C9d–K1–C11b	96.9(2)
C9d–K1–C12b	87.5(1)	X1a–K1–N1a	93.8(2)
K1–N1–K1a	96.3(2)	K1–N1–C1	114.7(3)

K1–N1–C7	115.9(2)	K1a–N1–C1	124.5(2)
K1a–N1–C7	101.6(2)	C1–N1–C7	104.0

X1a = center between C11b and C12b. Symmetry operations, A: -y+2/3, x-y+1/3, z+1/3; B: -x+y+1/3, -x+2/3, z-1/3; C: -y+2/3, -x+1/3, z-1/6; D: -y+2/3, x-y+1/3, z-2/3.

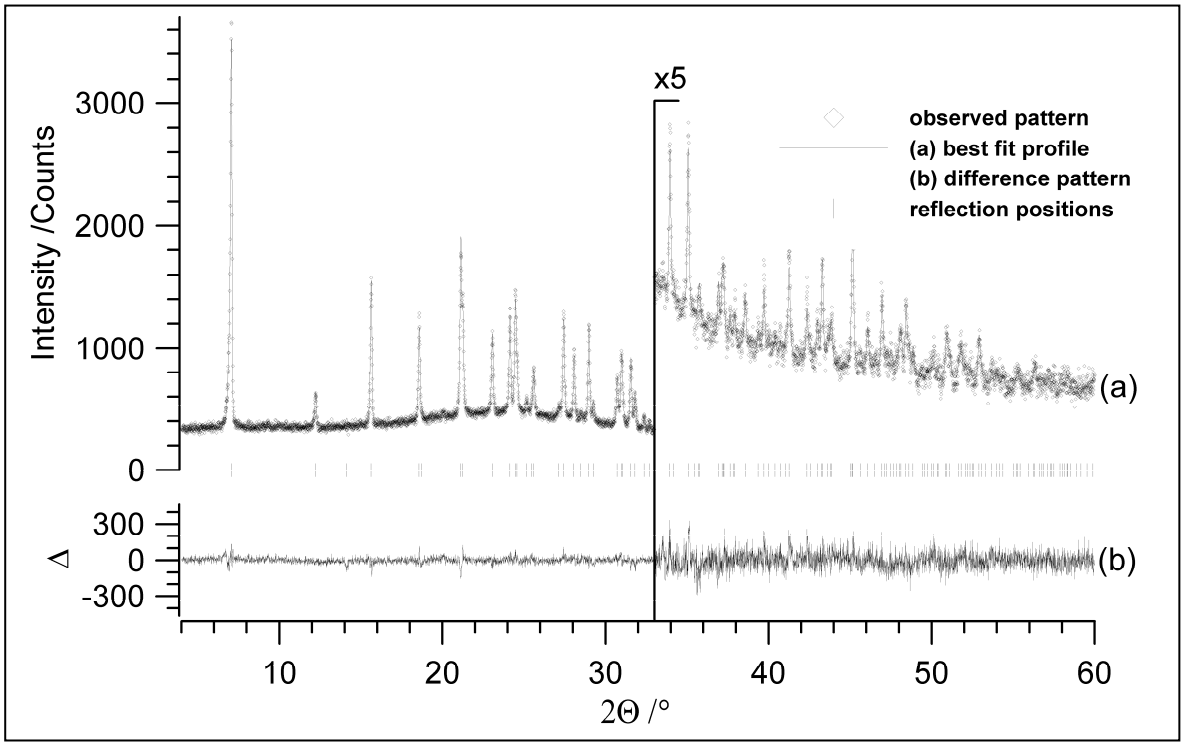


Fig. 6: Rietveld plot of $KNC_{12}H_8$ (**1**). The high angle part is enlarged by a factor of five.

Table 4. Crystallographic Data for rubidium carbazolate ($RbNC_{12}H_8$, **2**)

formula	$C_{12}H_8NRb$
FW (g mol^{-1})	251.67
crystal system	trigonal
space group	*R*3*c*; No. 161
temperature (K)	100
cell parameters: a (Å)	24.9084(11)
c (Å)	8.1932(4)
V ($Å^3$)	4402.3(3)
Z	18
density (calc., g cm^{-3})	1.709
2θ range (deg.)	3.0 – 50.1
steps of scan	δ2θ = 0.01 deg. with 70 sec. each step
FWHM 2θ (deg.)	0.151
λ (Å)	1.5406
No. of refl. in refinement	90
No. of refined variables	77
R-values: R_{wp}	0.0502
R_p	0.0393
R_{F2}	0.1011
Diffractometer:	STOE *Stadi-P* transmission diffractometer

Table 5. Atom coordinates and isotropic thermal parameters for $RbNC_{12}H_8$ (**2**)

Atom	x	y	z	U_i/U_e
Rb1	0.36109(15)	0.27087(18)	0.5259(6)	0.0444(12)
N1	0.23686(6)	0.2601(4)	0.53752(17)	0.025(4)
C1	0.18896(13)	0.20563(29)	0.5959(4)	0.025(4)
C2	0.15583(6)	0.21414(29)	0.72637(23)	0.025(4)
C3	0.10560(11)	0.1624(4)	0.7983(5)	0.025(4)
C4	0.08788(23)	0.10276(32)	0.7420(9)	0.025(4)
C5	0.12025(30)	0.09444(27)	0.6139(11)	0.025(4)
C6	0.17063(24)	0.14570(33)	0.5409(8)	0.025(4)
C7	0.23469(8)	0.30473(28)	0.63047(30)	0.025(4)
C8	0.18594(9)	0.27983(30)	0.74870(28)	0.025(4)
C9	0.17798(19)	0.3199(4)	0.8525(6)	0.025(4)
C10	0.21772(30)	0.3841(4)	0.8406(10)	0.025(4)
C11	0.26553(29)	0.40847(28)	0.7249(10)	0.025(4)
C12	0.27407(18)	0.36896(29)	0.6201(7)	0.025(4)

H3	0.08326(8)	0.1679(5)	0.88610(33)	0.025(4)
H4	0.05338(27)	0.0674(4)	0.7914(10)	0.025(4)
H5	0.1078(4)	0.05339(29)	0.5760(14)	0.025(4)
H6	0.19266(30)	0.1397(5)	0.4531(10)	0.025(4)
H9	0.14528(20)	0.3035(5)	0.9320(6)	0.025(4)
H10	0.2122(4)	0.4115(5)	0.9120(13)	0.025(4)
H11	0.2926(4)	0.45250(28)	0.7175(13)	0.025(4)
H12	0.30691(18)	0.3859(4)	0.5410(7)	0.025(4)

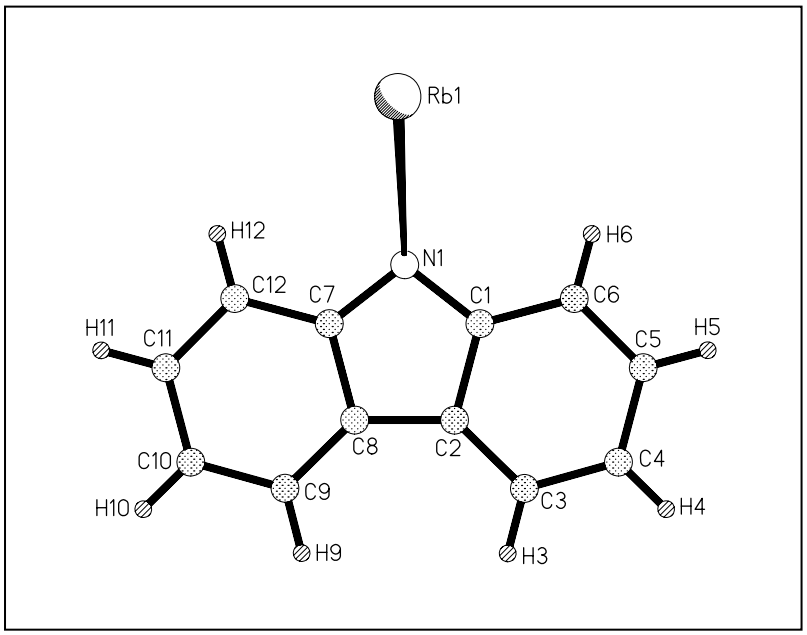


Fig. 7: Asymmetric unit of $RbNC_{12}H_8$ (**2**)

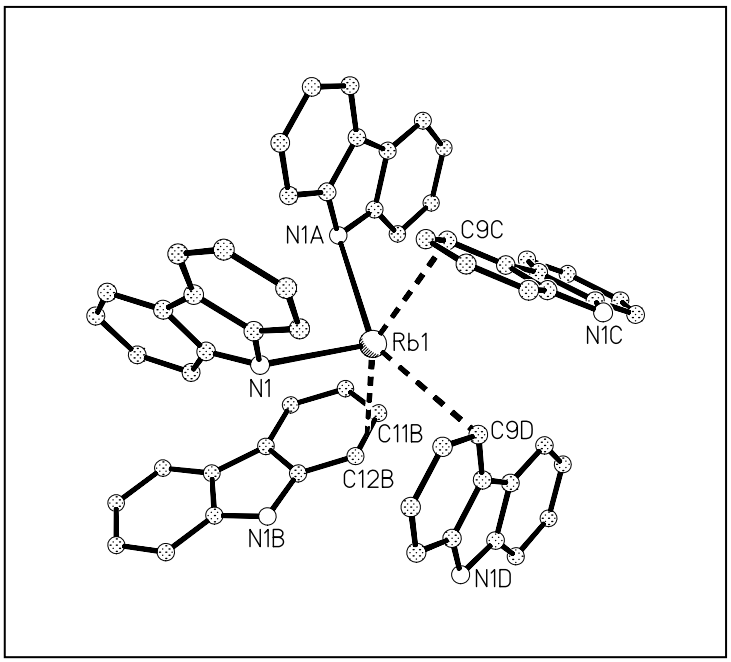


Fig. 8: Coordination of five $NC_{12}H_8^-$ anions to a Rb^+ ion in $RbNC_{12}H_8$ (**2**). Symmetry operations, A: -y+2/3, x-y+1/3, z+1/3; B: -x+y+1/3, -x+2/3, z-1/3; C: -y+2/3, -x+1/3, z-1/6; D: -y+2/3, x-y+1/3, z-2/3.

Fig. 9 – 11: Rb-C interactions, up to 3.40 Å, in **2**.

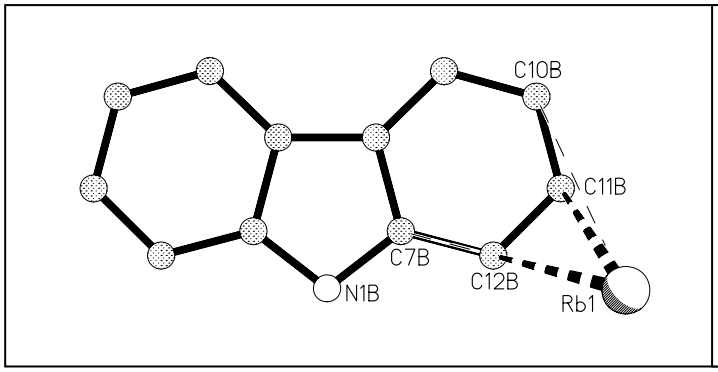


Fig. 9

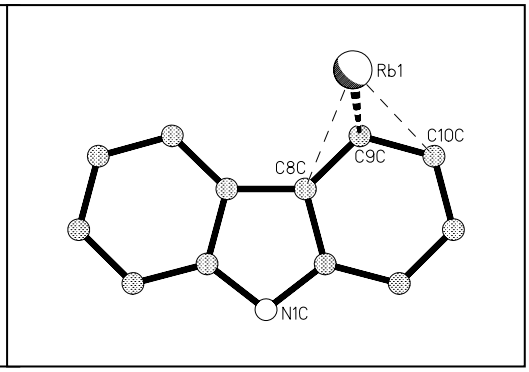


Fig. 10

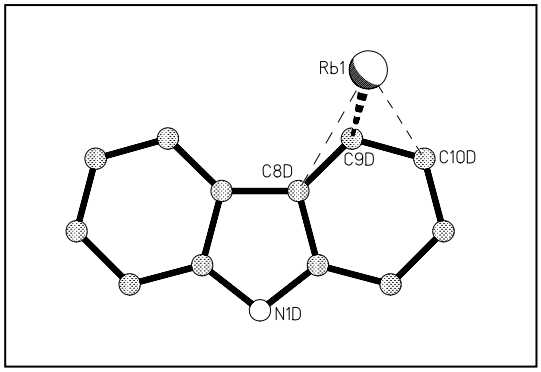


Fig. 11

Table 6. Bond lengths [Å] and angles [deg.] for $RbNC_{12}H_8$ (**2**)

Rb1–Rb1a	4.403(1)	Rb1–N1	2.971(5)	Rb1–N1a	3.020(5)
Rb1–C9c	3.016(5)	Rb1–C11b	3.265(5)	Rb1–C12c	3.280(4)
Rb1–C9d	3.332(6)	N1–C1	1.370	N1–C7	1.370
C1–C2	1.430	C1–C6	1.400	C2–C3	1.400
C2–C8	1.430	C3–C4	1.400	C4–C5	1.400
C5–C6	1.400	C7–C8	1.430	C7–C12	1.400
C8–C9	1.400	C9–C10	1.400	C10–C11	1.400
C11–C12	1.400	C–H	0.960		

N1–Rb1–N1a	101.8(2)	N1–Rb1–C9d	100.1(2)
N1–Rb1–C9c	106.2(2)	N1–Rb1–C11b	117.3(2)
N1–Rb1–C12	97.4(1)	N1–Rb1–X1a	107.5(2)
N1a–Rb1–C9c	75.4(2)	N1a–Rb1–C11b	89.6(1)
N1a–Rb1–C12b	109.0(1)	N1a–Rb1–X1a	99.4(1)
C9d–Rb1–C9c	82.5(1)	C9d–Rb1–C11b	95.1(2)
C9d–Rb1–C12b	84.5(2)	C9d–Rb1–X1a	89.8(2)
Rb1–N1–Rb1a	94.6(2)	Rb1–N1–C1	116.9(3)

Rb1–N1–C7	114.0(2)	Rb1a–N1–C1	123.7(2)
Rb1a–N1–C7	103.4(3)	C1–N1–C7	104.0

X1a = center between C11b and C12b. Symmetry operations, A: -y+2/3, x-y+1/3, z+1/3; B: -x+y+1/3, -x+2/3, z-1/3; C: -y+2/3, -x+1/3, z-1/6; D: -y+2/3, x-y+1/3, z-2/3.

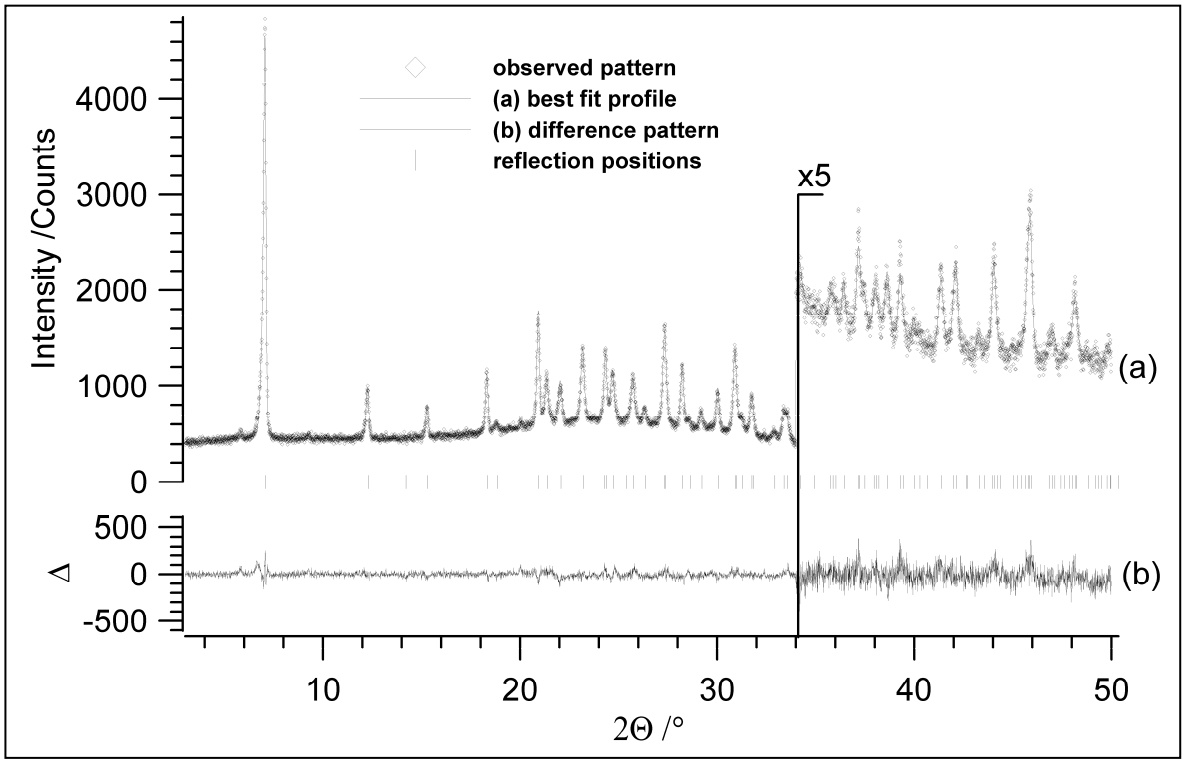


Fig. 12: Rietveld plot of $RbNC_{12}H_8$ (**2**). The high angle part is enlarged by a factor of five.

Table 7. Crystallographic Data for cesium carbazolate ($CsNC_{12}H_8$, **3**)

formula	$C_{12}H_8CsN$
FW (g mol^{-1})	299.11
crystal system	monoclinic
space group	*Ia*; No. 9
temperature (K)	100
cell parameters: a (Å)	15.3978(3)
b (Å)	12.7373(3)
c (Å)	11.3843(2)
angle β (deg.)	103.5032(13)
V ($Å^3$)	2171.04(8)
Z	8
density (calc., g cm^{-3})	1.830
2θ range (deg.)	5.0 – 59.9
steps of scan	δ2θ = 0.01 deg. with 60 sec. each step
FWHM 2θ (deg.)	0.105
λ (Å)	1.5406
No. of refl. in refinement	305
No. of refined variables	149
R-values: R_{wp}	0.0581
R_p	0.0464
R_{F2}	0.0955
Diffractometer:	STOE *Stadi-P* transmission diffractometer

Table 8. Atom coordinates and isotropic thermal parameters for $CsNC_{12}H_8$ (**3**)

Atom	x	y	z	U_i/U_e
Cs1	0.7654(4)	0.93836(29)	0.65348	0.0722(22)
N1	0.6895(7)	0.6979(8)	0.9202(6)	0.055(5)
C1	0.7684(8)	0.7364(7)	0.9022(5)	0.055(5)
C2	0.8027(6)	0.6771(8)	0.8163(6)	0.055(5)
C3	0.8830(6)	0.7073(11)	0.7887(9)	0.055(5)
C4	0.9292(7)	0.7955(12)	0.8446(12)	0.055(5)
C5	0.8955(11)	0.8537(9)	0.9284(11)	0.055(5)
C6	0.8153(11)	0.8244(7)	0.9571(7)	0.055(5)
C7	0.6717(6)	0.6132(8)	0.8442(7)	0.055(5)
C8	0.7382(6)	0.5960(7)	0.7772(5)	0.055(5)
C9	0.7289(10)	0.5122(7)	0.6956(5)	0.055(5)
C10	0.6547(12)	0.4456(7)	0.6801(8)	0.055(5)

C11	0.5896(9)	0.4623(10)	0.7458(11)	0.055(5)
C12	0.5980(6)	0.5459(11)	0.8276(10)	0.055(5)
H3	0.9064(6)	0.6676(13)	0.7313(10)	0.055(5)
H4	0.9843(7)	0.8161(16)	0.8254(15)	0.055(5)
H5	0.9276(14)	0.9141(10)	0.9664(13)	0.055(5)
H6	0.7925(14)	0.8646(7)	1.0146(7)	0.055(5)
H9	0.7733(12)	0.5004(9)	0.6503(5)	0.055(5)
H10	0.6485(15)	0.3882(7)	0.6241(9)	0.055(5)
H11	0.5390(10)	0.4163(12)	0.7346(14)	0.055(5)
H12	0.5531(6)	0.5570(14)	0.8724(13)	0.055(5)
Cs2	0.5517(4)	0.69881(26)	0.5512(4)	0.0511(5)
N2	0.4988(7)	0.8948(10)	0.7750(7)	0.076(6)
C21	0.4356(6)	0.8728(9)	0.6716(10)	0.076(6)
C22	0.4371(6)	0.9439(9)	0.5750(6)	0.076(6)
C23	0.3762(6)	0.9311(11)	0.4636(8)	0.076(6)
C24	0.3139(6)	0.8491(12)	0.4473(14)	0.076(6)
C25	0.3123(6)	0.7795(10)	0.5421(18)	0.076(6)
C26	0.3729(7)	0.7913(8)	0.6540(16)	0.076(6)
C27	0.5416(6)	0.9817(10)	0.7455(6)	0.076(6)
C28	0.5073(6)	1.0162(9)	0.6240(6)	0.076(6)
C29	0.5445(7)	1.1044(9)	0.5812(11)	0.076(6)
C30	0.6150(6)	1.1584(9)	0.6571(16)	0.076(6)
C31	0.6486(5)	1.1246(11)	0.7761(15)	0.076(6)
C32	0.6121(6)	1.0365(12)	0.8203(9)	0.076(6)
H23	0.3770(8)	0.9786(13)	0.3984(6)	0.076(6)
H24	0.2722(6)	0.8405(14)	0.3709(16)	0.076(6)
H25	0.2694(6)	0.7235(10)	0.5302(23)	0.076(6)
H26	0.3715(9)	0.7433(9)	0.7186(19)	0.076(6)
H29	0.5217(8)	1.1279(10)	0.4997(12)	0.076(6)
H30	0.6404(7)	1.2188(9)	0.6275(20)	0.076(6)
H31	0.6970(6)	1.1620(13)	0.8277(19)	0.076(6)
H32	0.6354(8)	1.0137(14)	0.9020(9)	0.076(6)

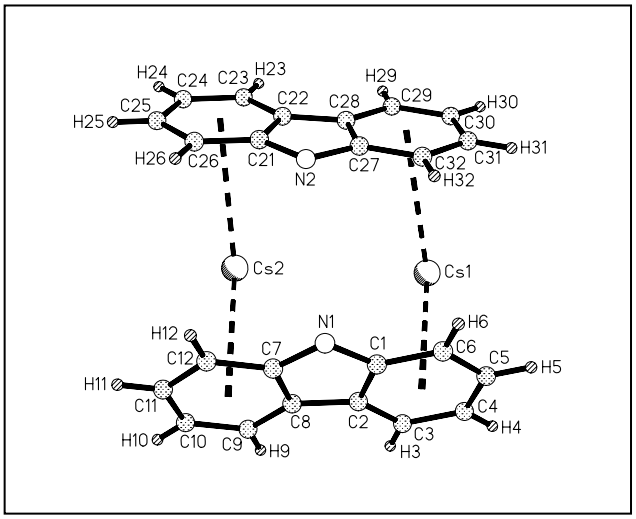


Fig. 13: Asymmetric unit of $CsNC_{12}H_8$ (**3**)

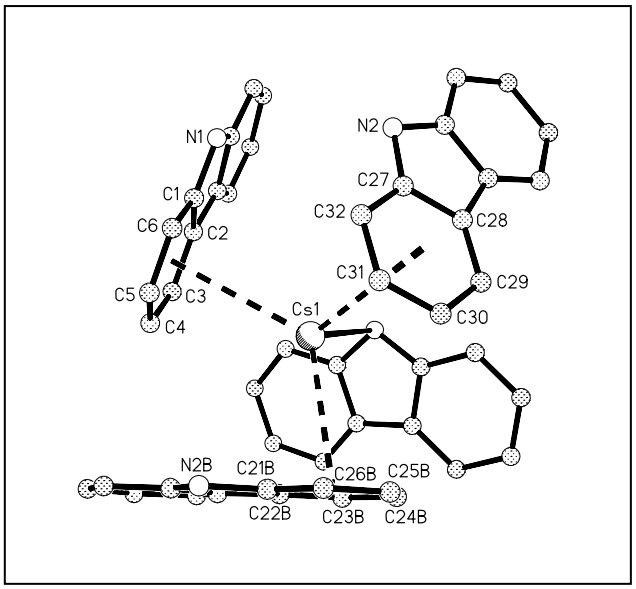


Fig. 14: : Coordination of four $NC_{12}H_8^-$ anions to the $Cs1^+$-ion in $CsNC_{12}H_8$ (**3**). Symmetry operations, A: x, -y+3/2, z-1/2; B: x+1/2, 2-y, z.

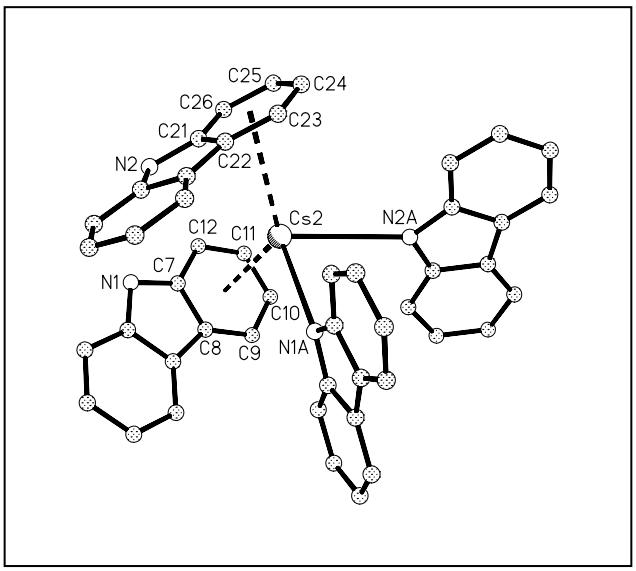
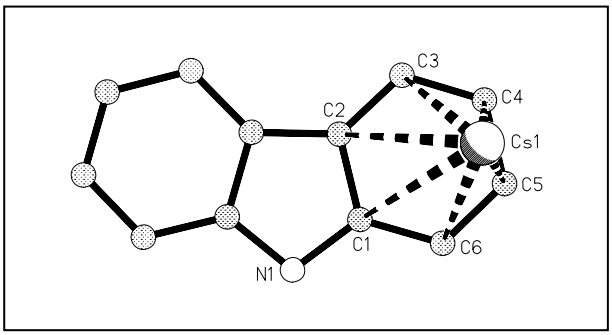


Fig. 15: Coordination of four $NC_{12}H_8^-$ anions to the $Cs2^+$ ion in $CsNC_{12}H_8$ (**3**). Symmetry operation, A: x, -y+3/2, z-1/2; B: x+1/2, 2-y, z.

Fig. 17 – 22: Cs-C interactions, up to 4.10 Å, in **3**.



Fi. 17:

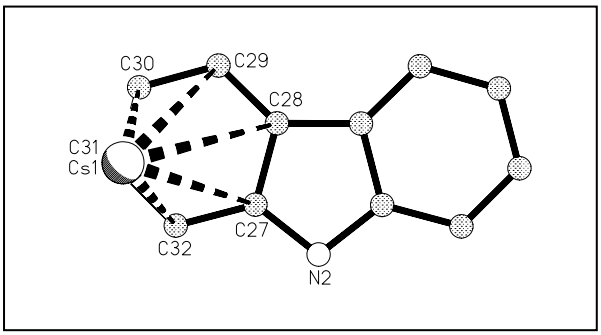


Fig. 18:

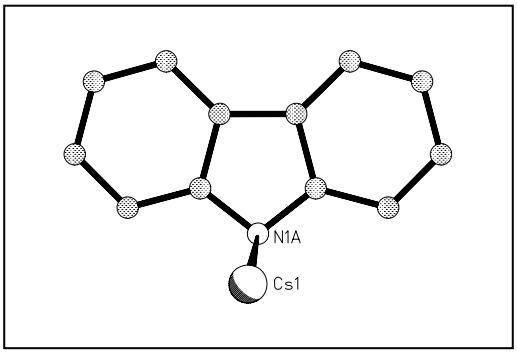


Fig. 19:

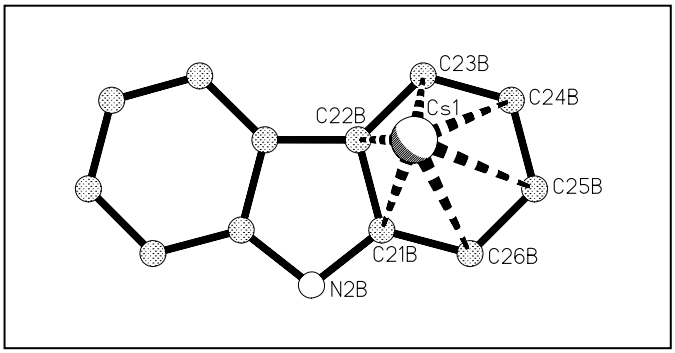


Fig. 20:

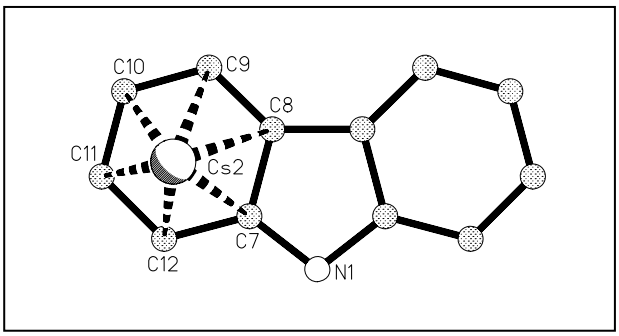


Fig. 21:

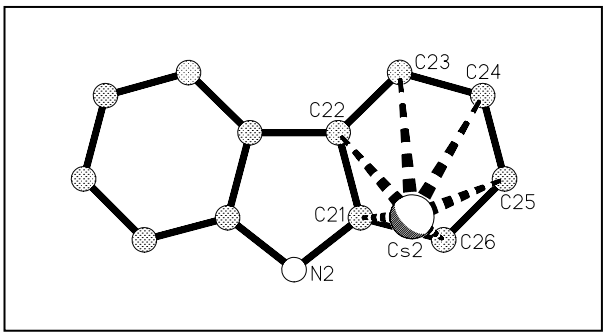


Fig. 22:

Table 9. Bond lengths [Å] and angles [deg.] for $CsNC_{12}H_8$ (**3**)

Cs1–Cs2	4.439(2)	Cs1–N1a	3.160(2)		
Cs1–C1	3.818(4)	Cs1–C2	3.787(5)	Cs1–C3	3.607(7)
Cs1–C4	3.442(8)	Cs1–C5	3.470(5)	Cs1–C6	3.661(5)
Cs1–C27	3.868(5)	Cs1–C28	4.033(5)	Cs1–C29	3.926(7)
Cs1–C30	3.642(6)	Cs1–C31	3.462(7)	Cs1–C32	3.583(6)
Cs1–C21b	3.528(6)	Cs1–C22b	3.338(5)	Cs1–C23b	3.473(5)
Cs1–C24b	3.770(8)	Cs1–C25b	3.933(7)	Cs1–C26b	3.820(6)
Cs2–N1a	3.152(9)	Cs2–N2a	3.283(9)		

Cs2–C7	3.585(5)	Cs2–C8	3.624(5)	Cs2–C9	3.702(7)
Cs2–C10	3.740(6)	Cs2–C11	3.704(7)	Cs2–C12	3.627(7)
Cs2–C21	3.336(7)	Cs2–C22	3.627(6)	Cs2–C23	3.974(7)
Cs2–C24	4.056(7)	Cs2– C25	3.805(6)	Cs2– C26	3.441(8)

N1–C1	1.370	N1–C7	1.370	N2–C27	1.370
N2–C21	1.370				
C1–C6	1.400	C1–C2	1.430	C2–C3	1.400
C2–C8	1.430	C2–C1	1.430	C3–C4	1.400
C4–C5	1.400	C5–C6	1.400	C7–C12	1.400
C7–C8	1.430	C8–C9	1.400	C9–C10	1.400
C10–C11	1.400	C11–C12	1.400	C21–C26	1.400
C21–C22	1.430	C22–C23	1.400	C22–C28	1.430
C23–C24	1.400	C24–C25	1.400	C25–C26	1.400
C27–C32	1.400	C27–C28	1.430	C28–C29	1.400
C29–C30	1.400	C30–C31	1.400	C31–C32	1.400
C–H	0.960				

X1a–Cs1–X1b	112.9(2)	X1a–Cs1–X1d	127.0(2)
X1a–Cs1–N1a	105.6(2)	X1b–Cs1–X1d	100.2(2)
X1b–Cs1–N1a	103.7(2)	X1d–Cs1–N1a	105.2(2)
N1a–Cs2–N2a	75.1(1)	X1c–Cs2–X1e	129.9(1)
X1c–Cs2–N1a	108.3(1)	X1c–Cs2–N2a	114.7(1)
X1e–Cs2–N1a	112.7(1)	X1e–Cs2–N2a	102.8(1)
Cs1–N1a–Cs2	89.4(2)	Cs1–N1a–C1a	98.9(3)
Cs1–N1a–C7a	94.5(3)	C1a–N1a–C7a	104.0
C1a–N1a–Cs2	129.8(6)	C7a–N1a–Cs2	124.8(6)
Cs2–N2a–C21a	135.7(6)	Cs2–N2a–C27a	120.1(6)
C21a–N2a–C27a	104.0		

X1a = center of C1 – C6; X1b = center of C27 – C32; X1c = center of C7 – C12; X1d = center of C21b – C26b; X1e = center of C21 – C26. Symmetry operations, A: x, -y+3/2, z-1/2; B: x+1/2, 2-y, z.

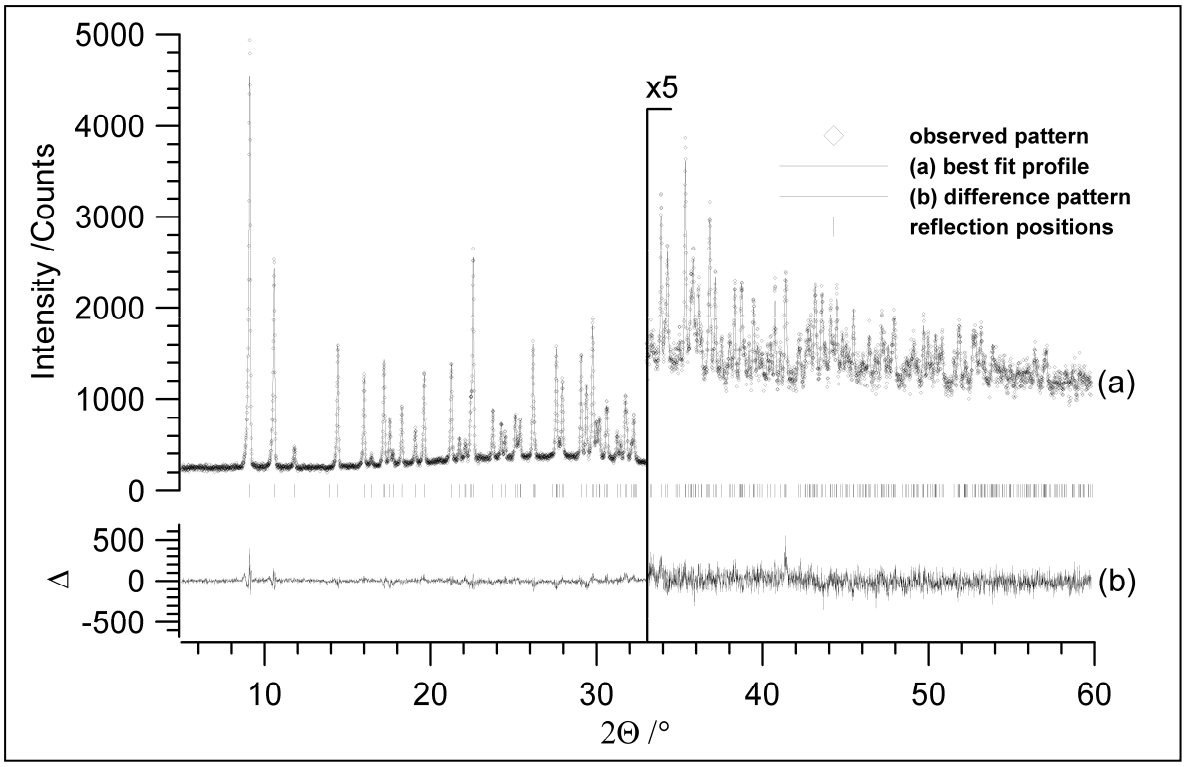


Fig. 23: Rietveld plot of $CsNC_{12}H_8$ (**3**). The high angle part is enlarged by a factor of five.