Supporting information

Lithium aluminate flotation by pH- and light-switchable collectors based on the natural product punicine

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Contents

1. NMR spectra of punicine **1** 2
2. NMR spectra of puncine **2** 3
3. NMR spectra of punicine **3** 5
4. NMR spectra of punicine **4** 6
5. NMR spectra of punicine **5** 8
6. UV spectrum of punicine **2** 10
7. UV spectrum of punicine **3** 10
8. UV spectrum of punicine **4** 11
9. Zeta potential measurements 11
10. Crystallographic data 14
11. Flotation of $LiAlO_2$ using punicine **5** at pH 2.....22
12. XRD of Lithium aluminate and Gehlenite mix.....22
13. Wettability measurements 23
14. Contact angle measurements of lithium aluminate and gehlenite with punicine **2**-**5**....24
15. Specification of the LEDs..... 25

1. NMR spectra of punicine **1**

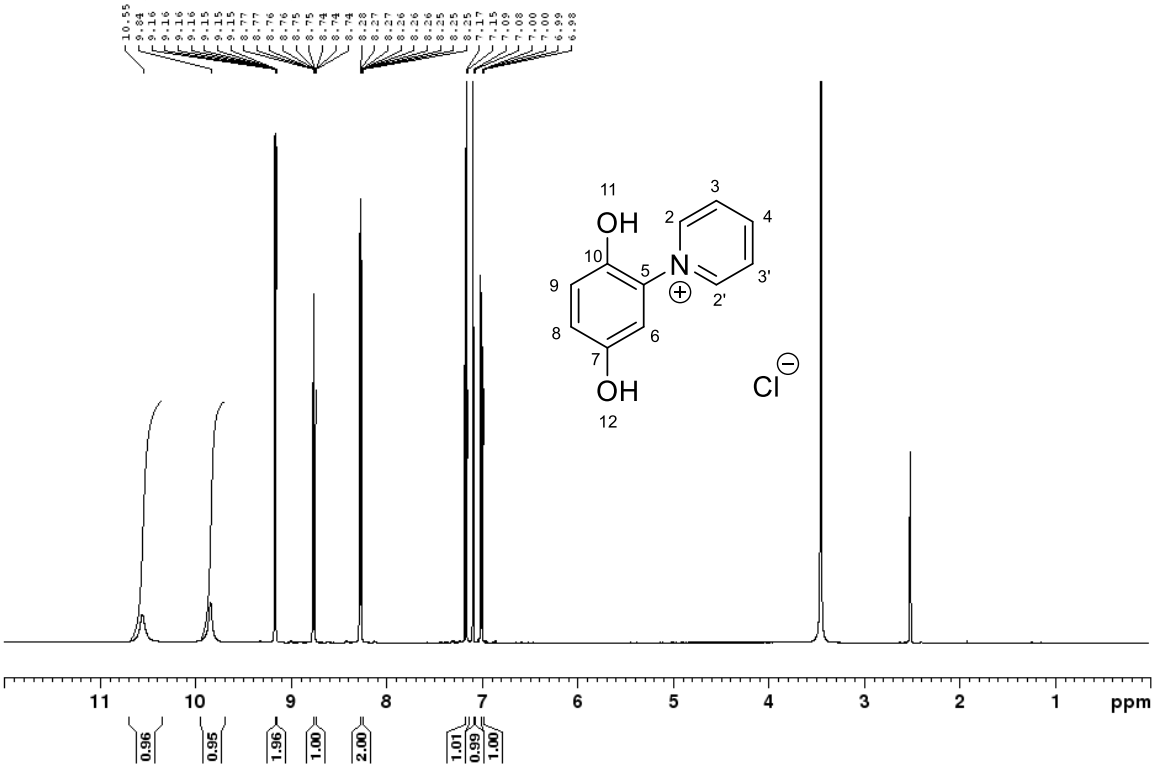


Figure 1: ^{1}H-NMR of 1.

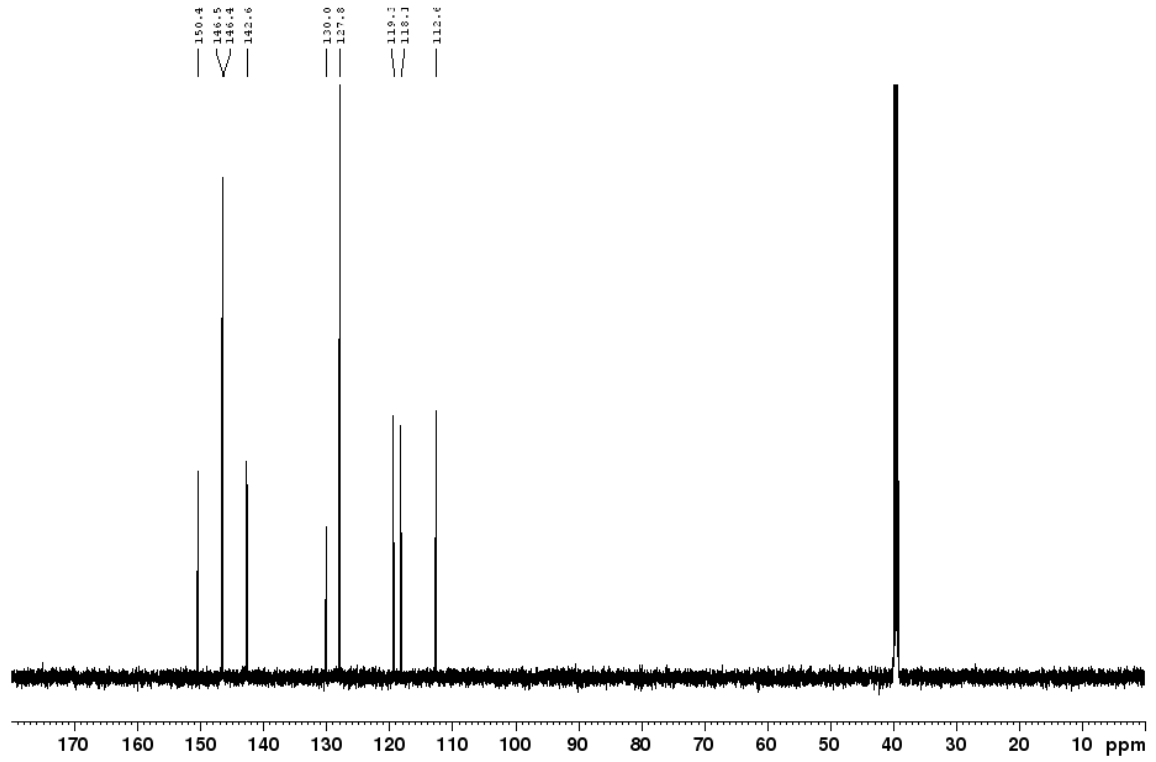


Figure 2: ^{13}C- NMR of 1.

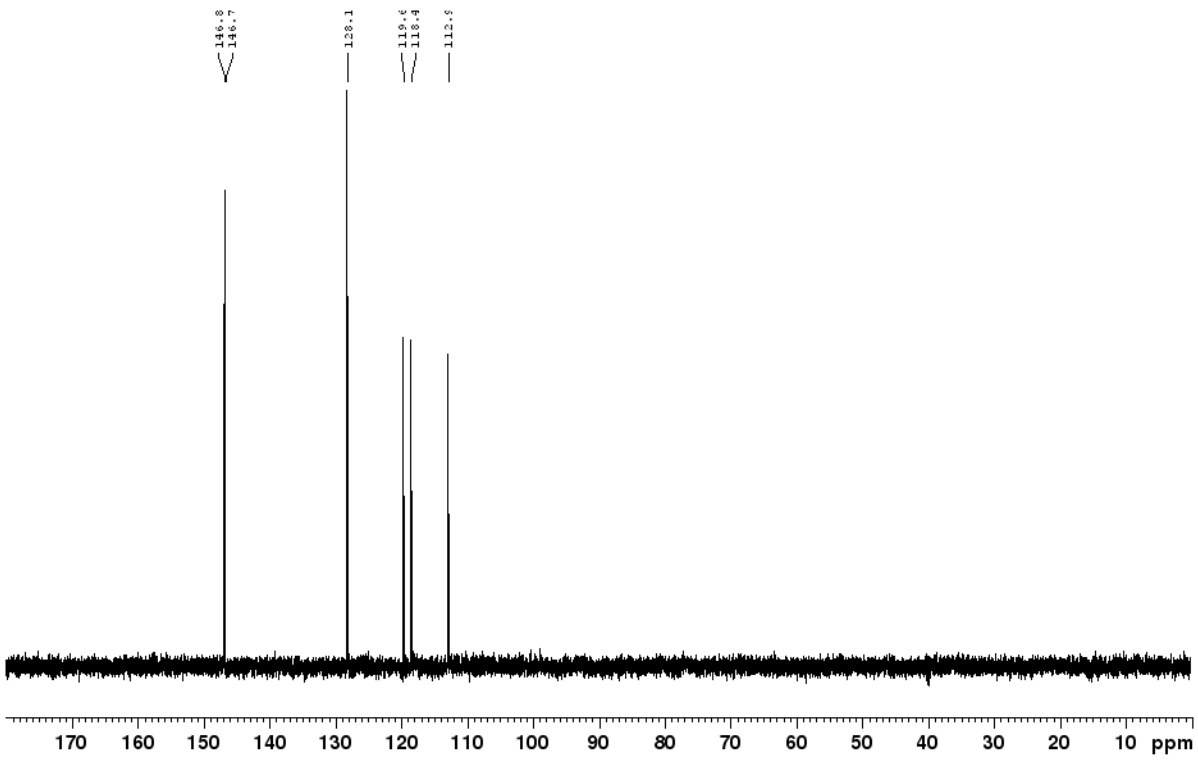


Figure 3: DEPT-135-spectrum of 1.

2. NMR spectra of punicine 2

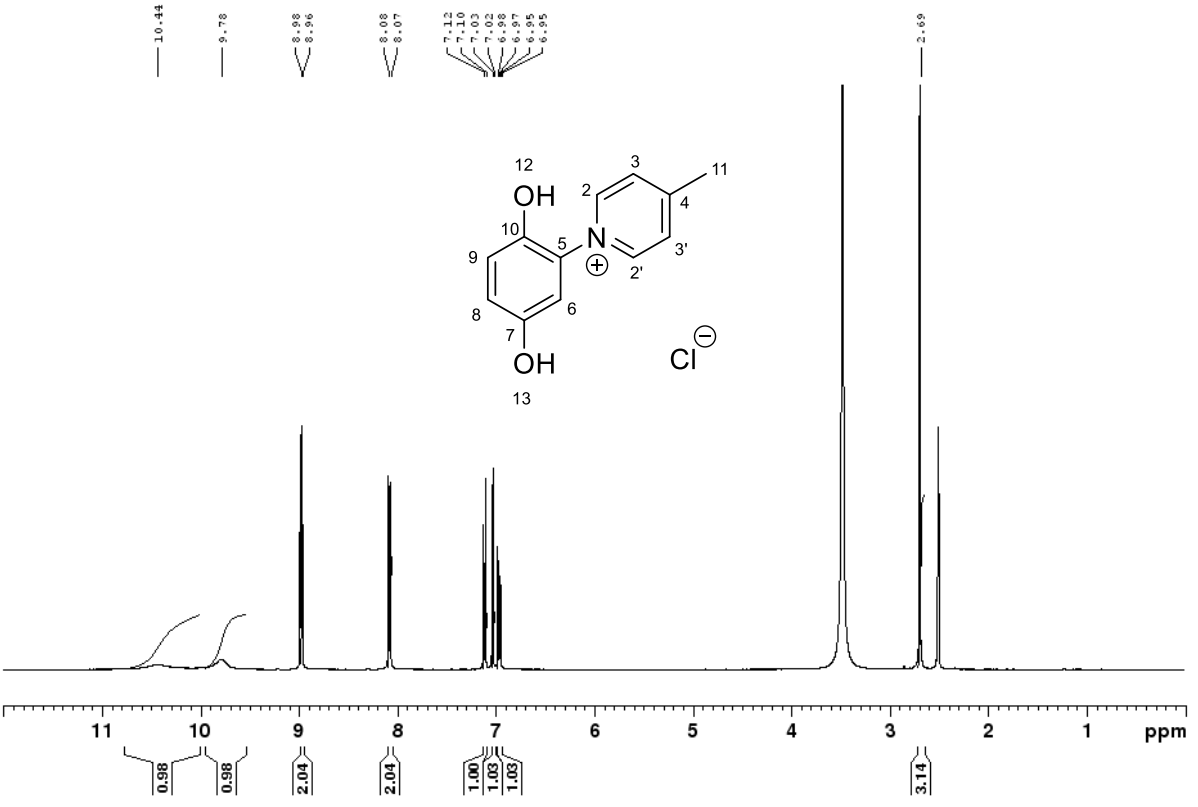


Figure 4: ^{1}H-NMR of 4.

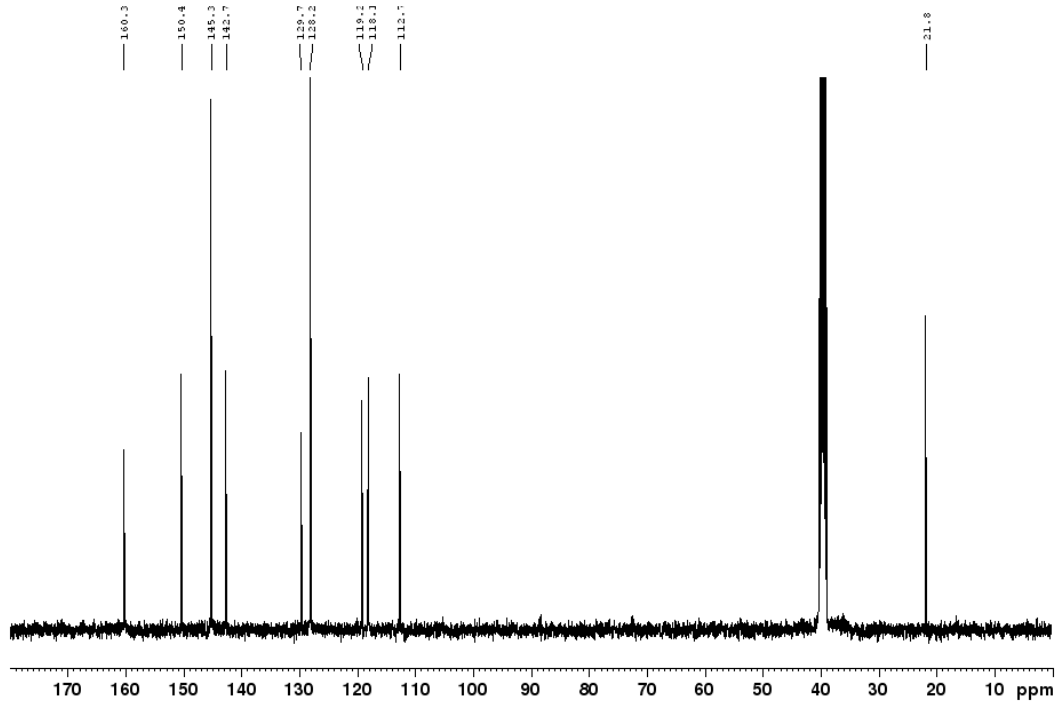


Figure 5: ^{13}C- NMR of 4.

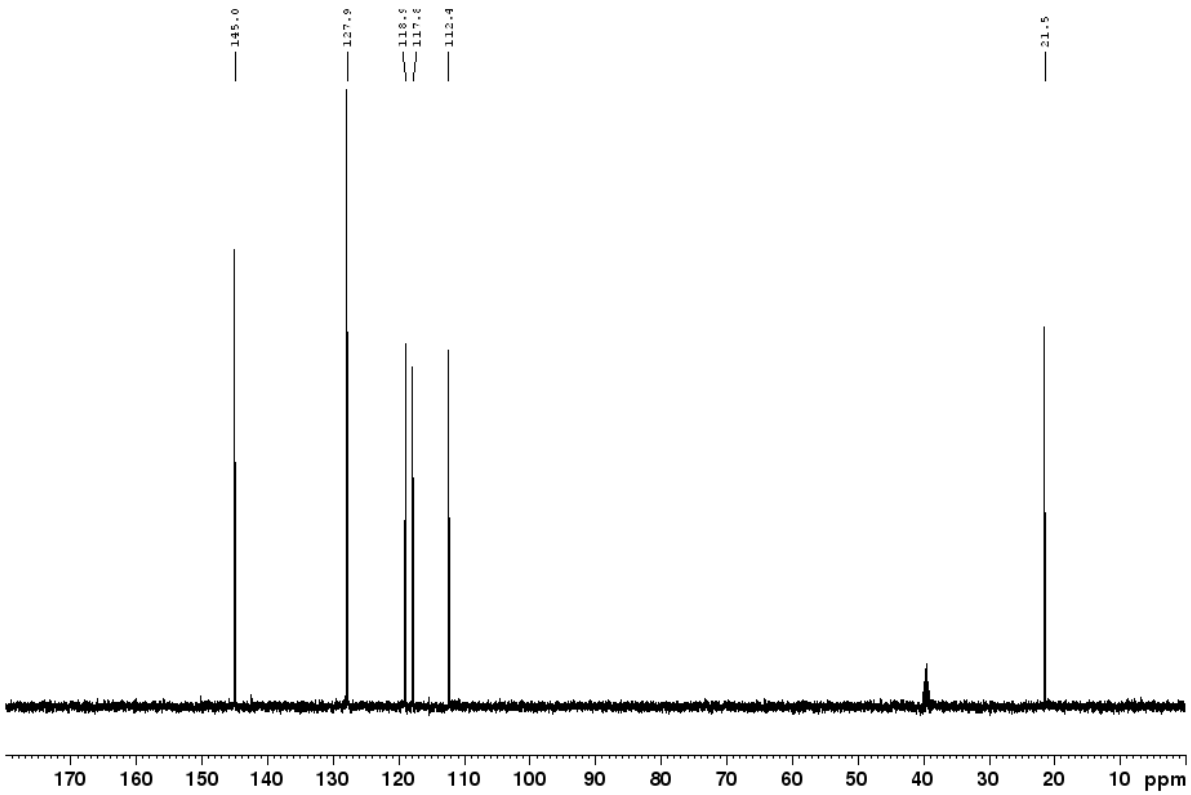


Figure 6: DEPT-135-spectrum of 4.

3. NMR spectra of punicine 3

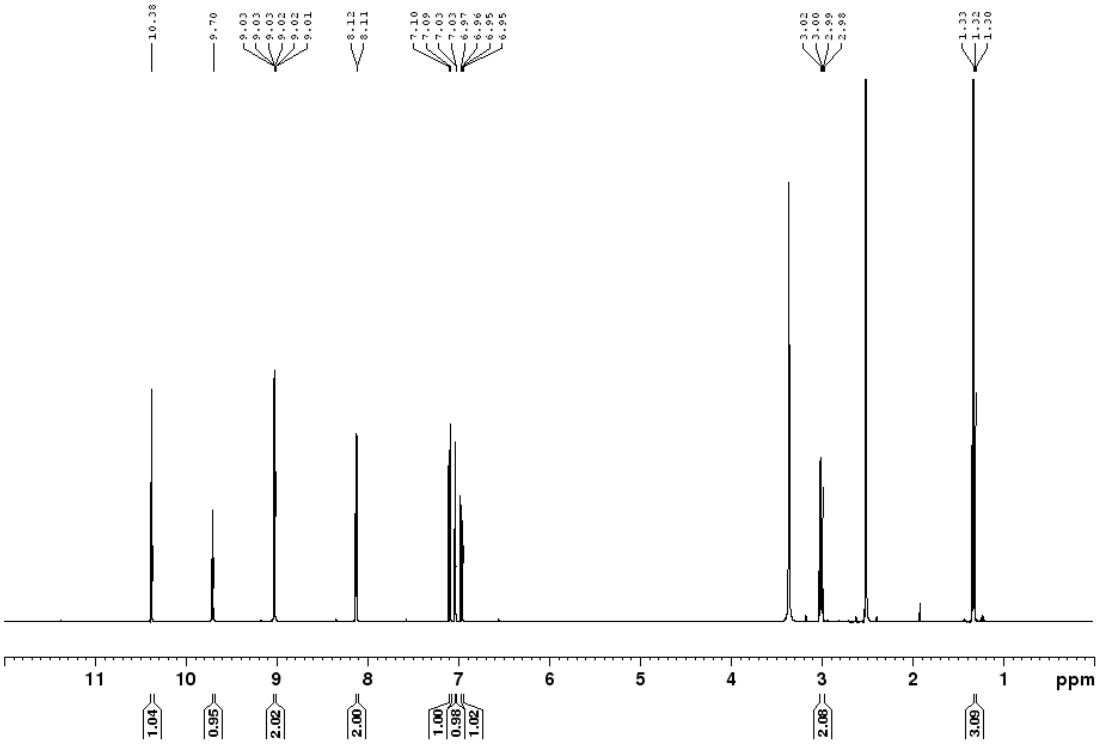


Figure 7: ^{1}H-NMR of 3.

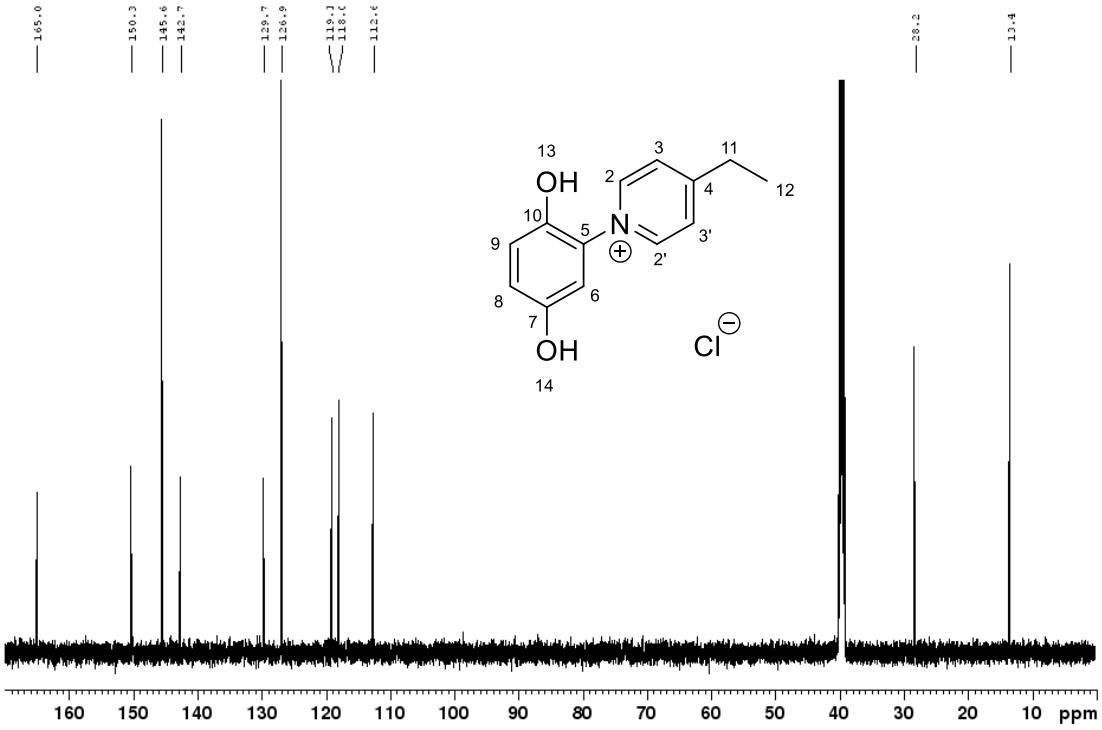


Figure 8: ^{13}C- NMR of 3.

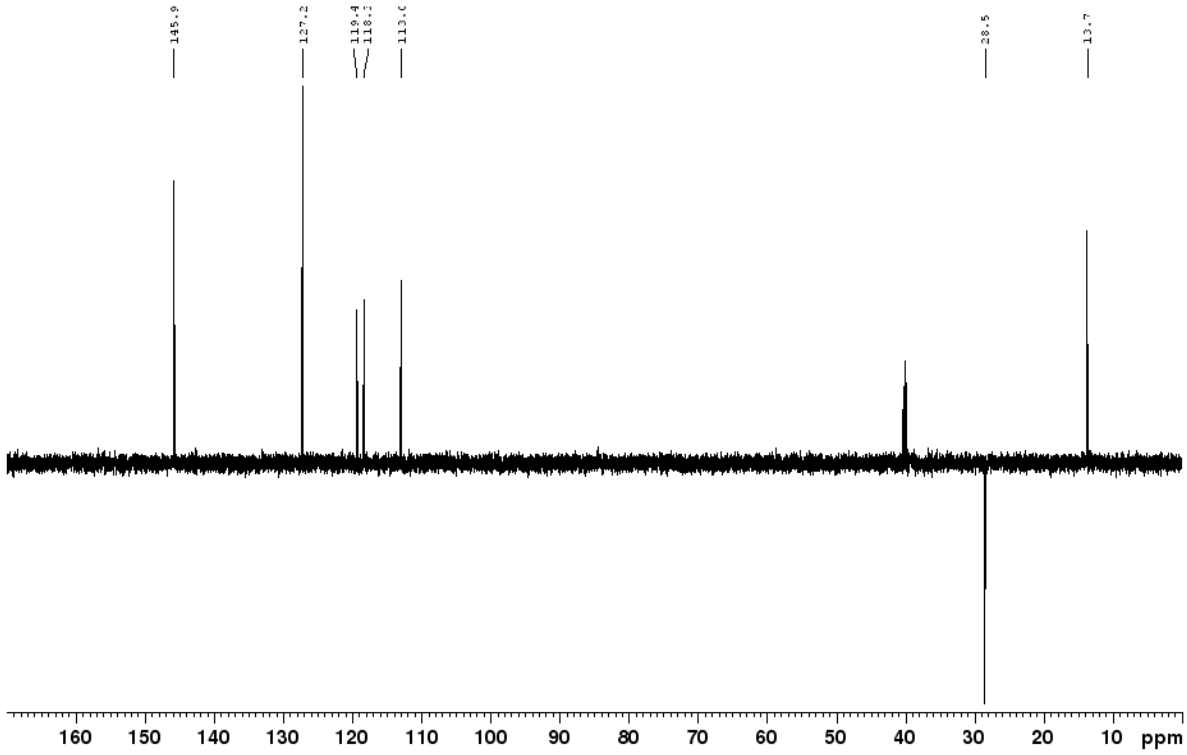


Figure 9: DEPT-135-spectrum of 3.

4. NMR spectra of punicine 4

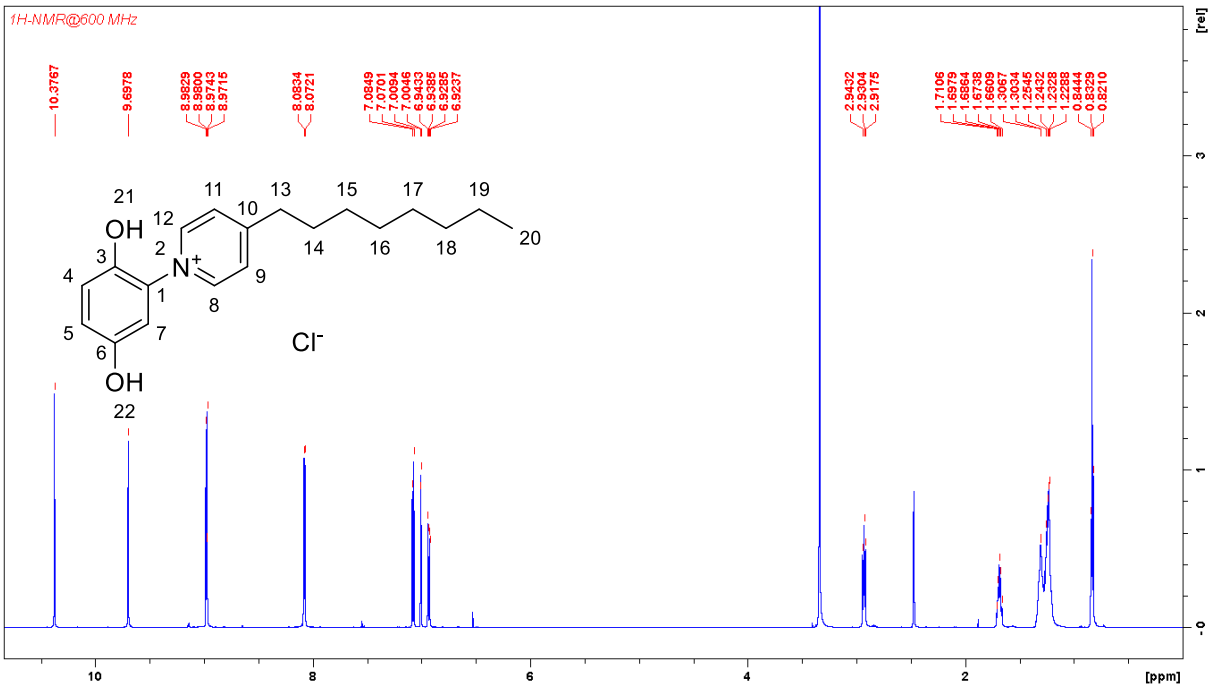


Figure 10: ^{1}H-NMR of 4.

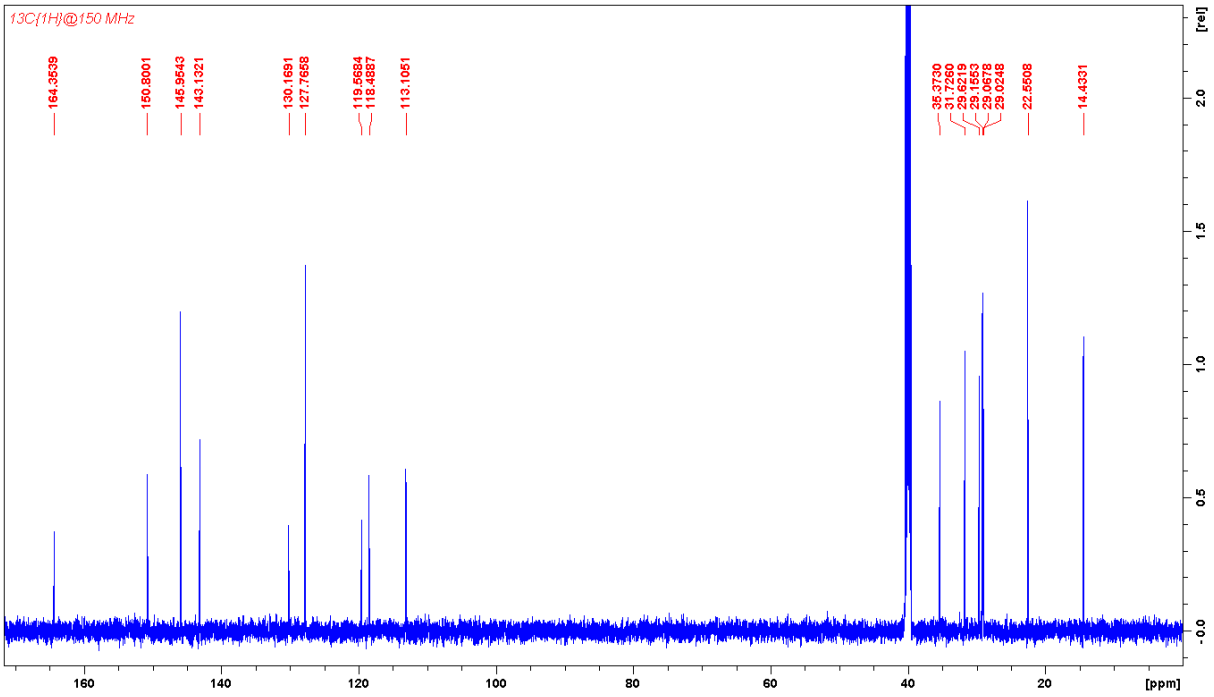


Figure 11:^{13}C-NMR of 4.

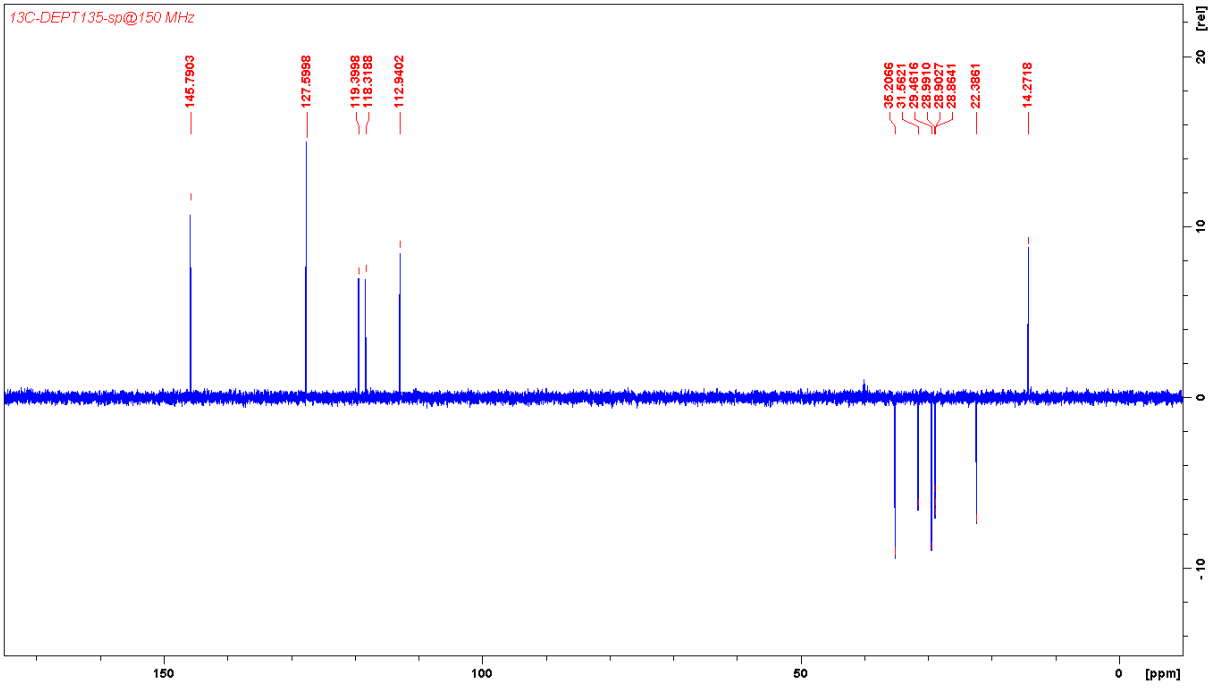


Figure 12: DEPT-135-spectrum of 4.

5. NMR spectra of punicine 5

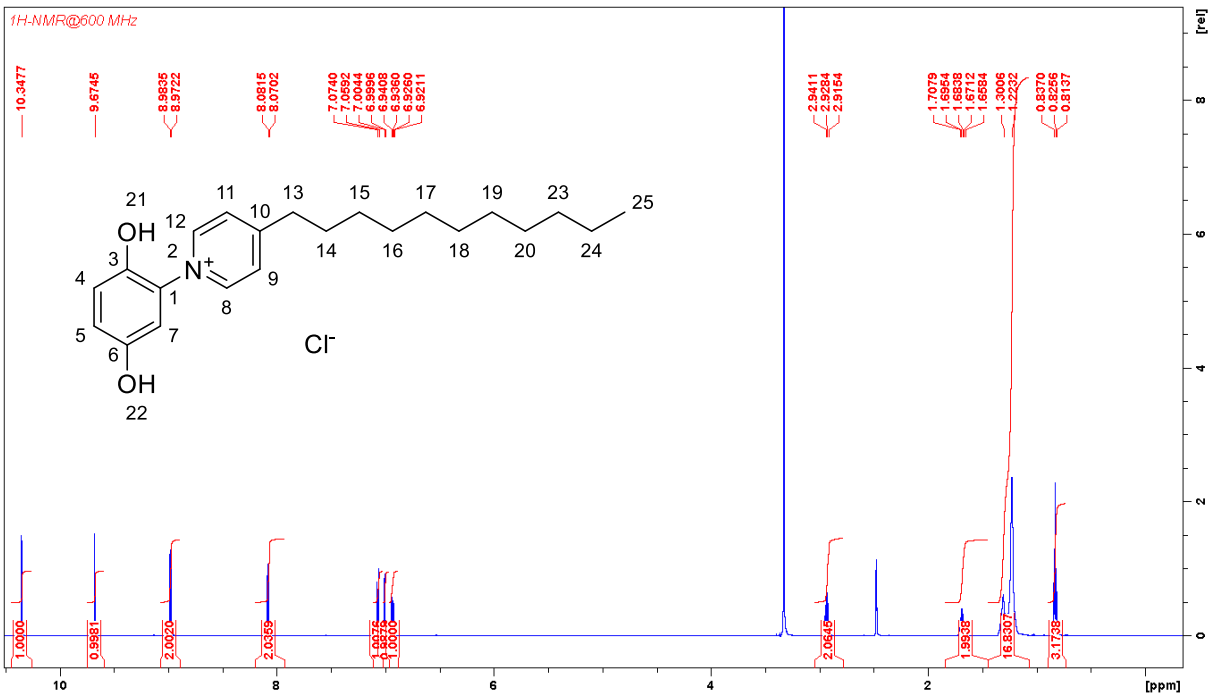


Figure 13: ^{1}H-NMR of 5.

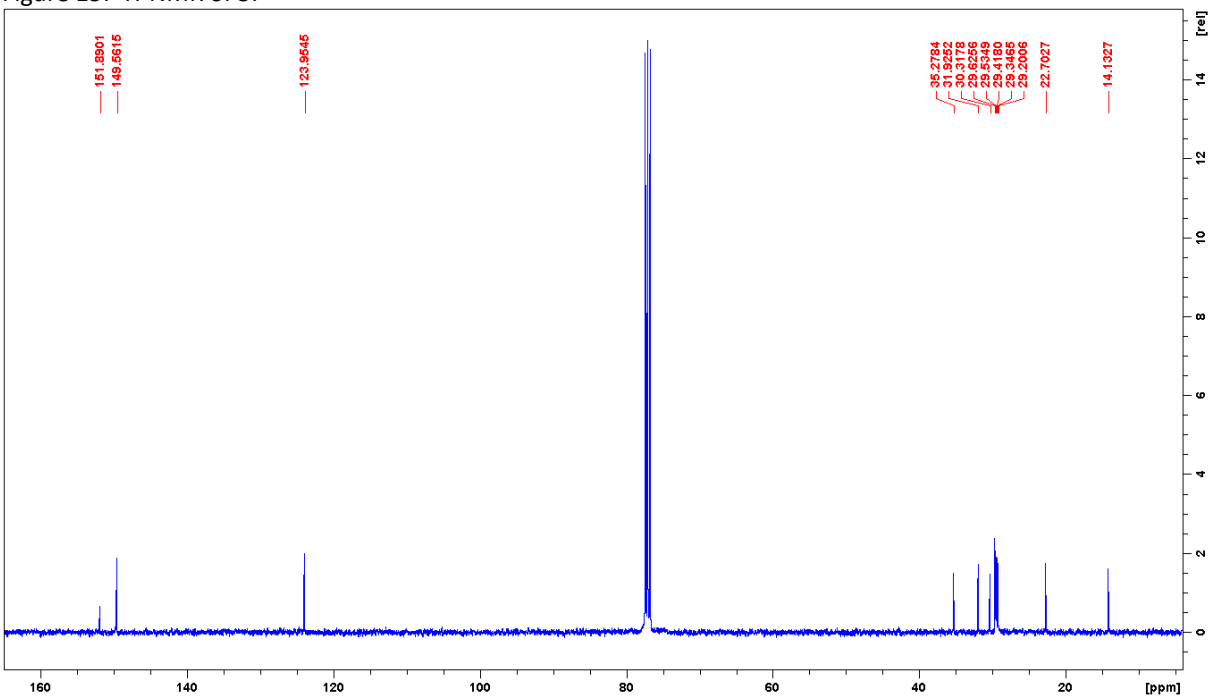


Figure 14: ^{13}C-NMR of 5.

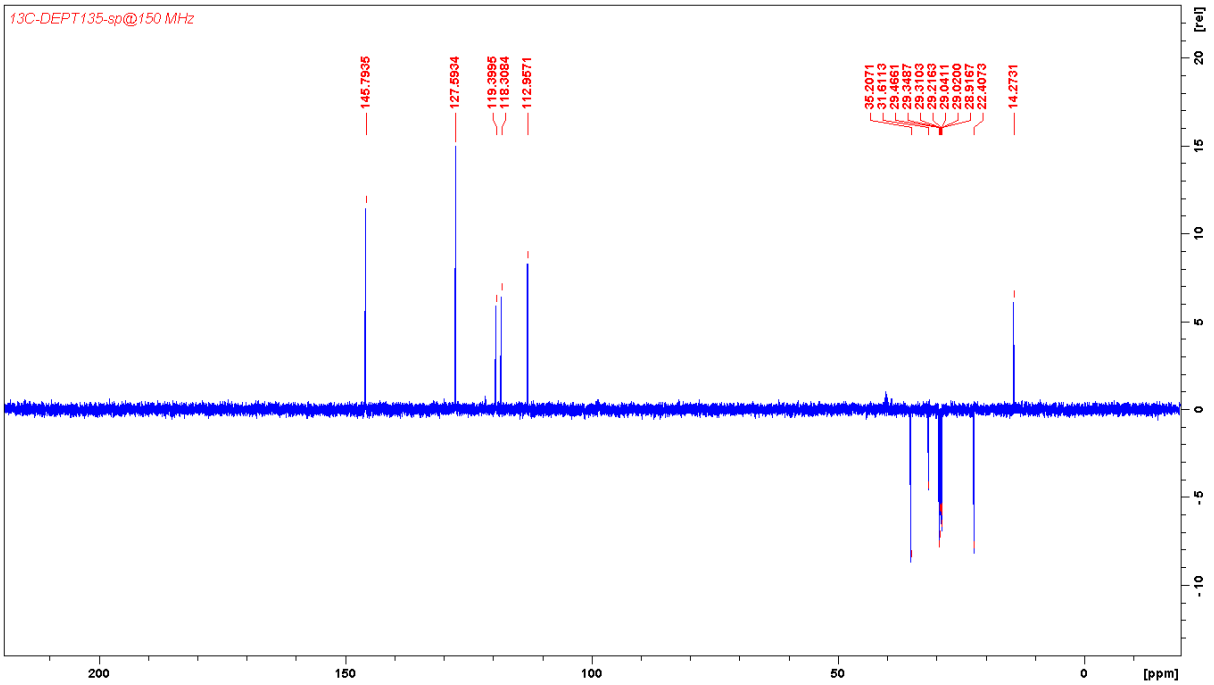


Figure 15: DEPT-135-spectrum of 5.

6. UV spectrum of punicine 2

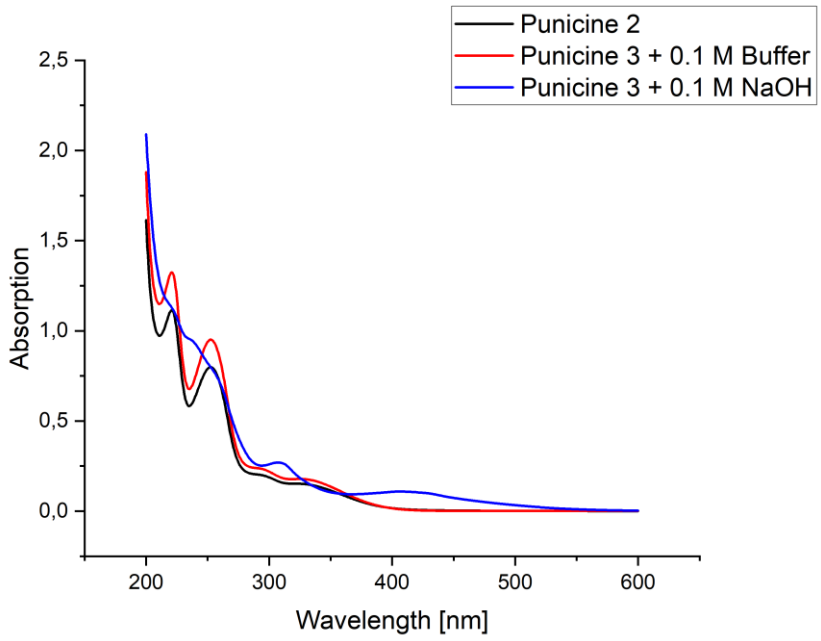


Figure 16: UV spectrum of punicine 3.

7. UV spectrum of punicine 3

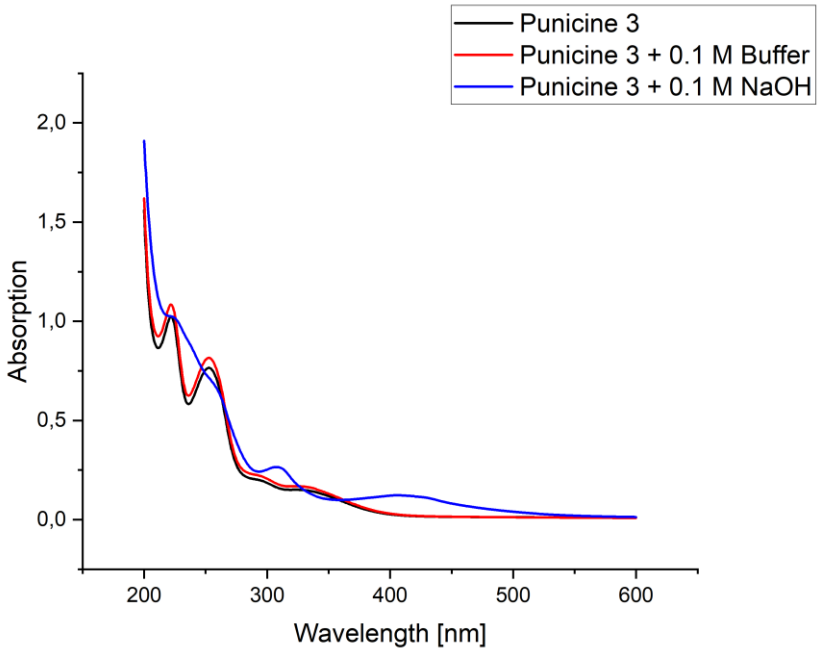


Figure 17: UV spectrum of punicine 3.

8. UV spectrum of punicine 4

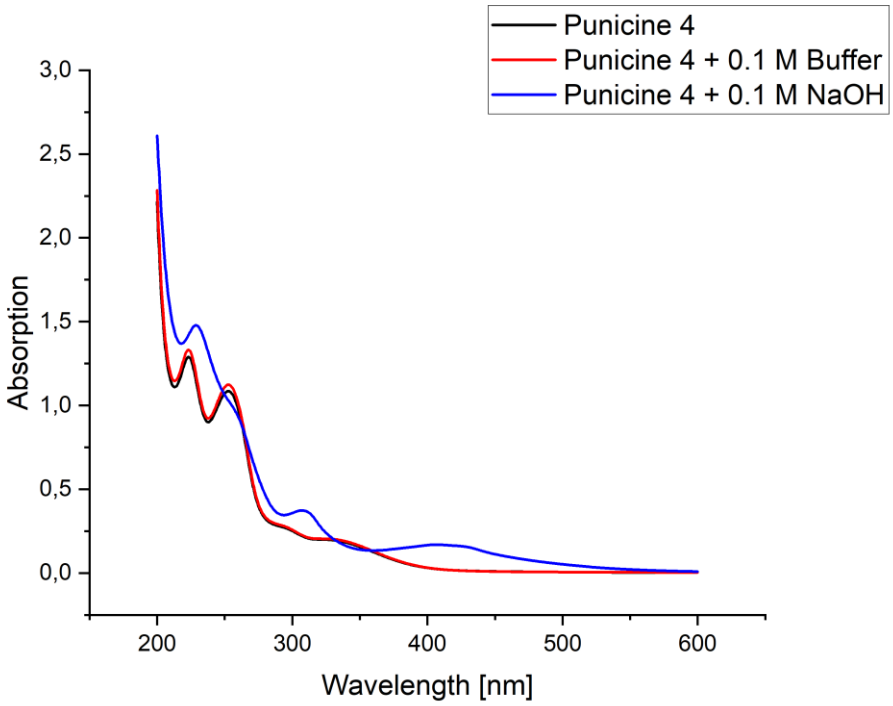


Figure 18: UV spectrum of punicine 4.

9. Zeta potential measurements

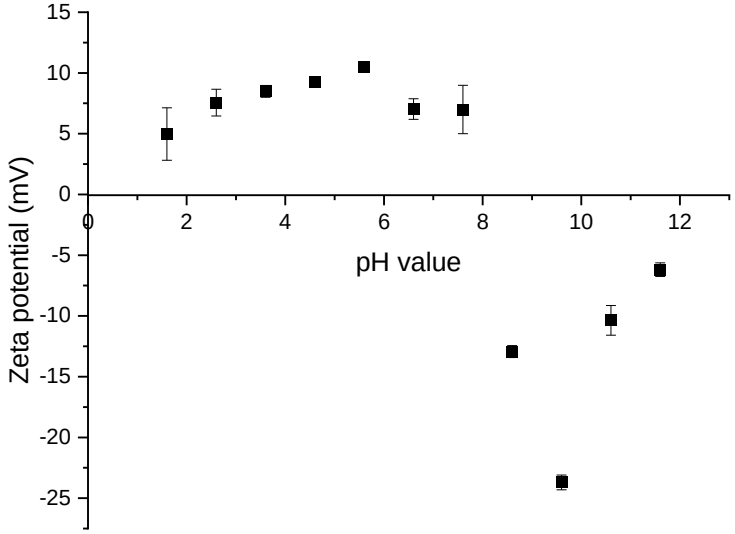


Figure 19: Zeta potential of the pure lithium aluminate.

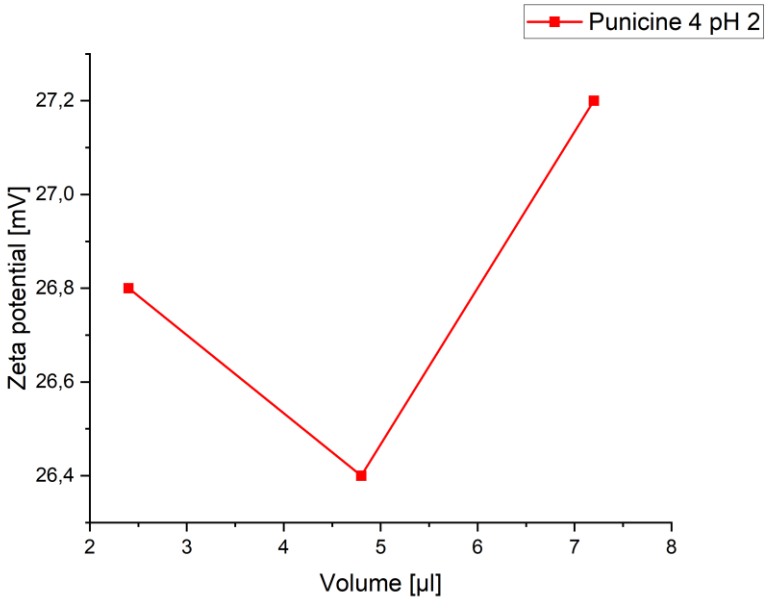


Figure 20: Zeta potential of punicine 4 + LiAlO2 pH 2 at different concentrations.

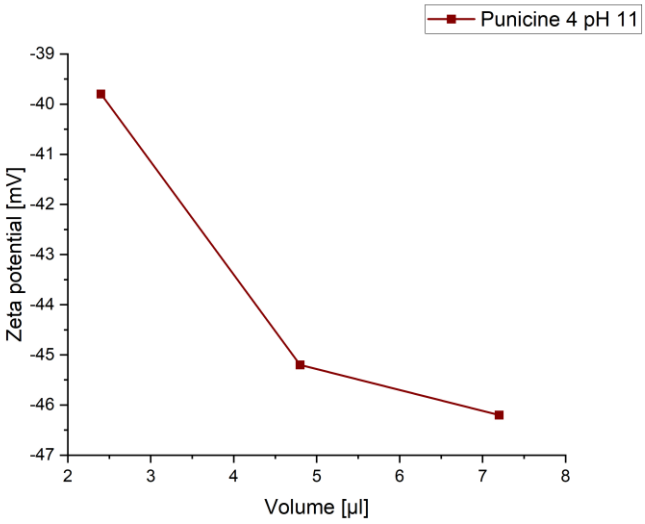


Figure 21: Zeta potential of punicine 4 + LiAlO2 pH 11 at different concentrations.

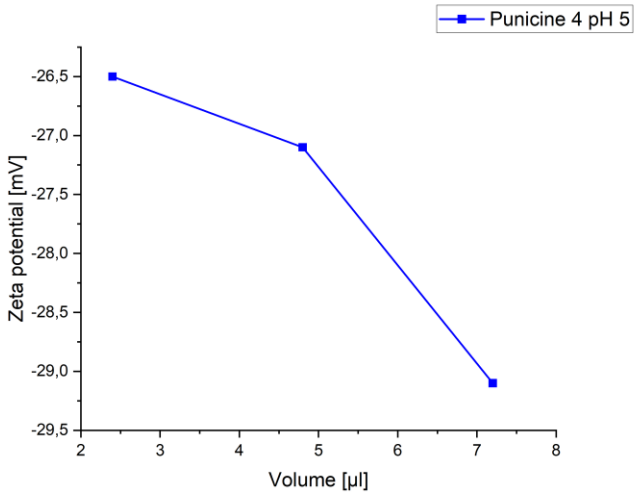


Figure 22: Zeta potential of punicine 4 + LiAlO2 pH 5 at different concentrations.

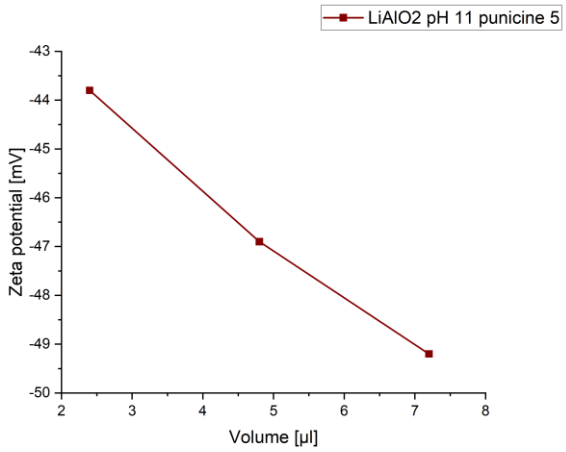


Figure 23: Zeta potential of punicine 5 + LiAlO2 pH 11 at different concentrations.

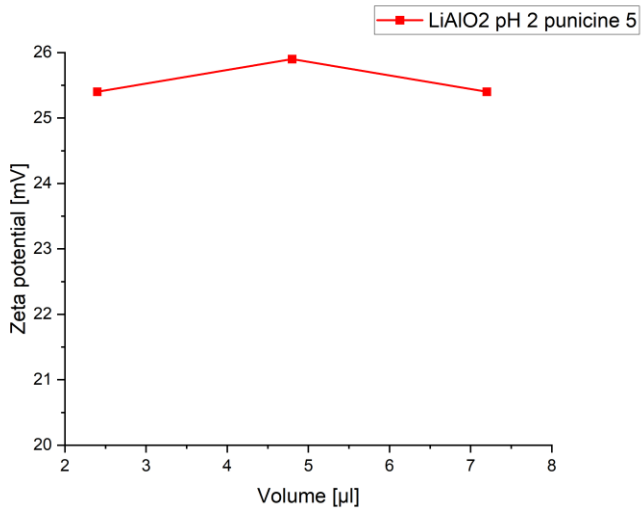


Figure 24: Zeta potential of punicine 5 + LiAlO2 pH 2 at different concentrations.

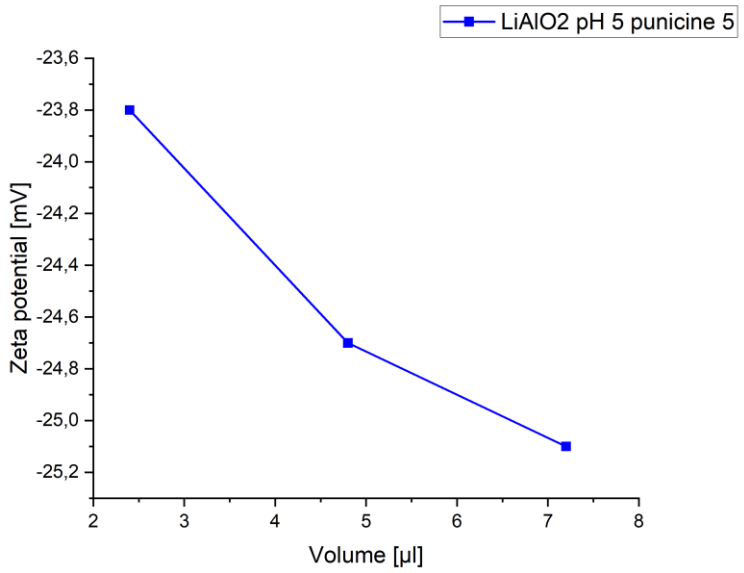


Figure 25: Zeta potential of punicine 5 + LiAlO2 pH 5 at different concentrations.

10. Crystallographic data

Crystal structure determination of punicine 3 [1-(2,5-dihydroxyphenyl)-4-ethylpyridin-1-ium chloride water solvate]

The single-crystal X-ray diffraction study was carried out on a Bruker D8 Venture diffractometer with PhotonII detector at 173(2) K using Mo-K radiation (= 0.71073 Å). Dual space methods (SHELXT) [G. M. Sheldrick, *Acta Crystallogr.* 2015, **A71**, 3-8] were used for structure solution and refinement was carried out using SHELXL-2014 (full-matrix least-squares on F^2) [G. M. Sheldrick, *Acta Crystallogr.* 2015, **C71**, 3-8]. Hydrogen atoms were localized by difference electron density determination and refined using a riding model (H(O) free). A semi-empirical absorption correction was applied.

3: yellow crystals, $C_{13}H_{14}NO_2^+ \cdot Cl^- \cdot H_2O$, M_r = 269.72, crystal size 0.35 × 0.20 × 0.15 mm, monoclinic, space group $P2_1/n$ (No. 14), a = 11.9361(3) Å, b = 7.3938(2) Å, c = 14.4601(4) Å, β = 93.592(1)°, V = 1273.64(6) Å^3, Z = 4, ρ = 1.407 Mg/m^{-3}, μ(Mo-K$_\alpha$) = 0.30 mm^{-1}, F(000) = 568, T = 173(2) K, $2\theta_{max}$ = 55.2°, 31472 reflections, of which 2944 were independent (R_{int} = 0.054), 175 parameters, 6 restraints, R_1 = 0.030 (for 2801 I > 2σ(I)), wR_2 = 0.083 (all data), S = 1.05, largest diff. peak / hole = 0.28 / -0.19 e Å^{-3}.

CCDC 2291748 (**3**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

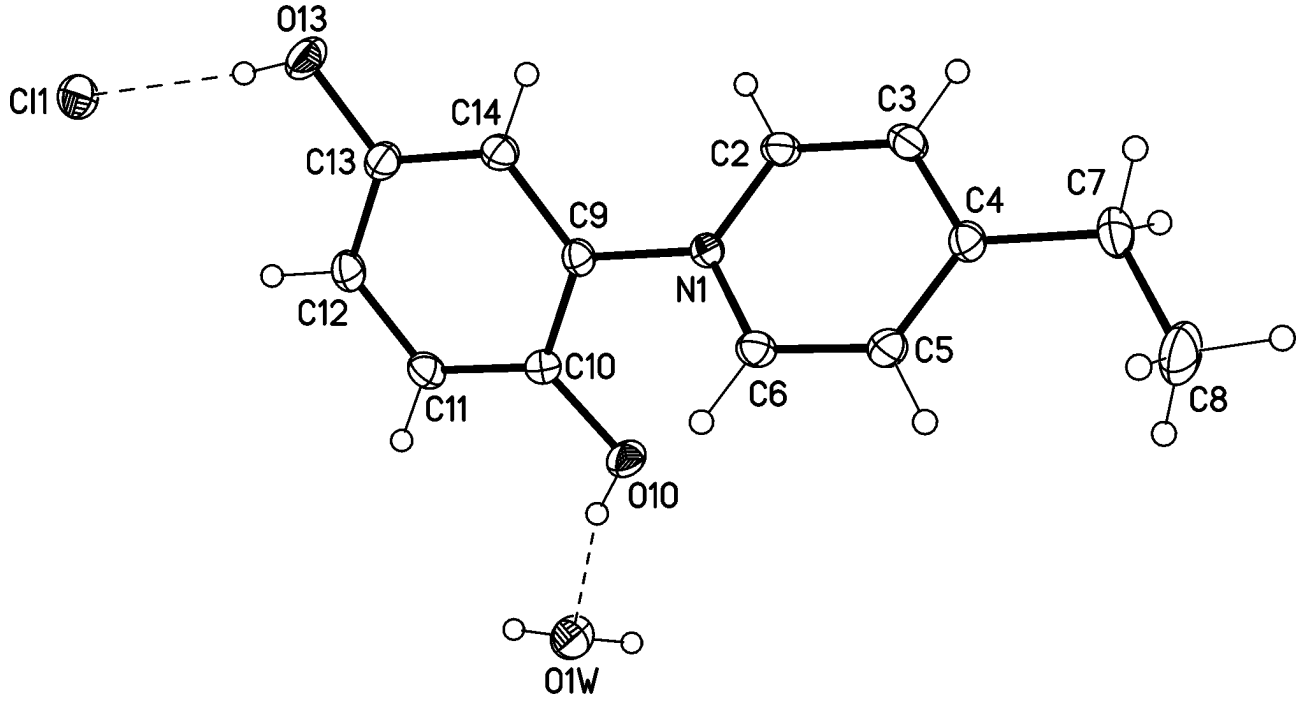
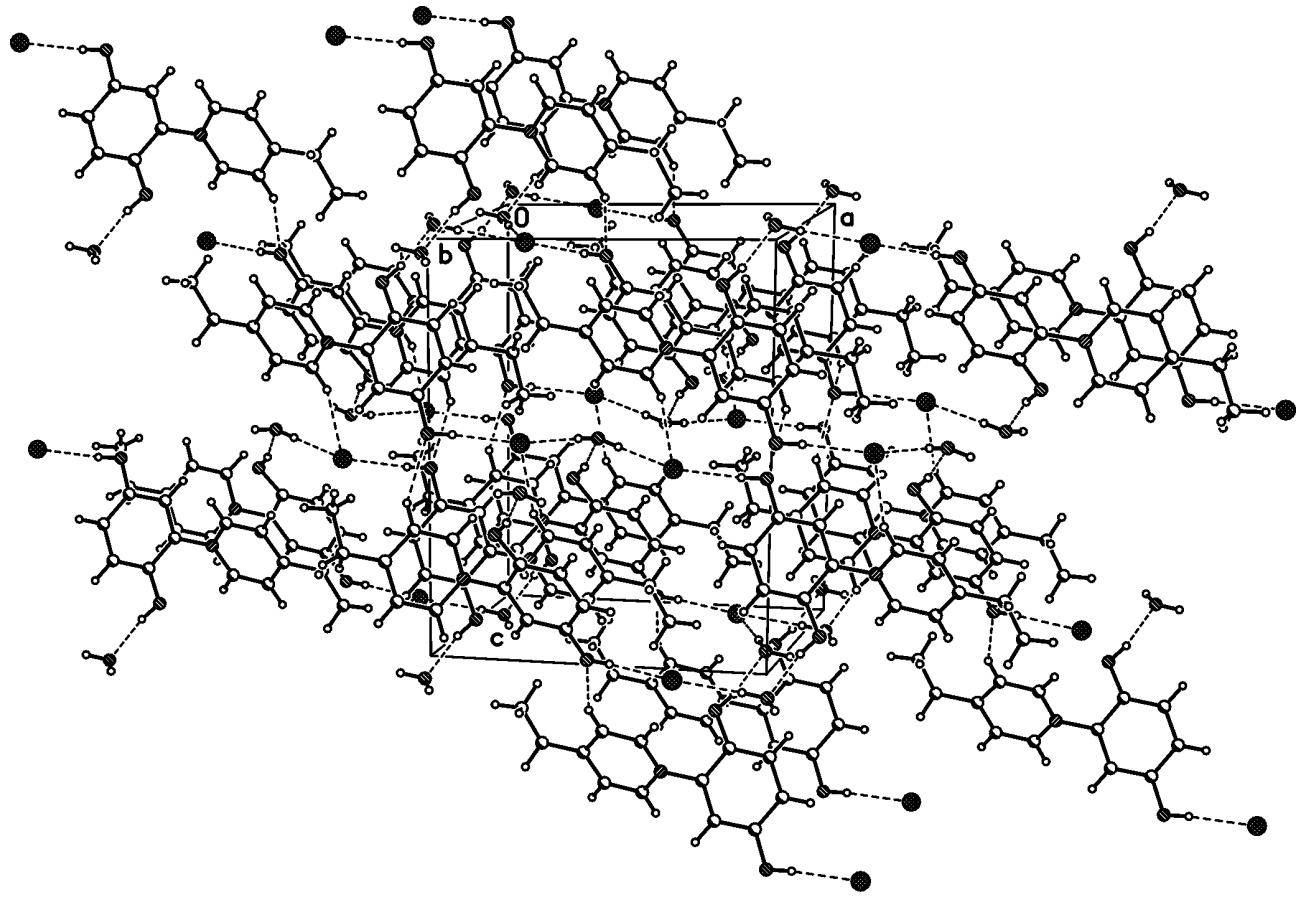
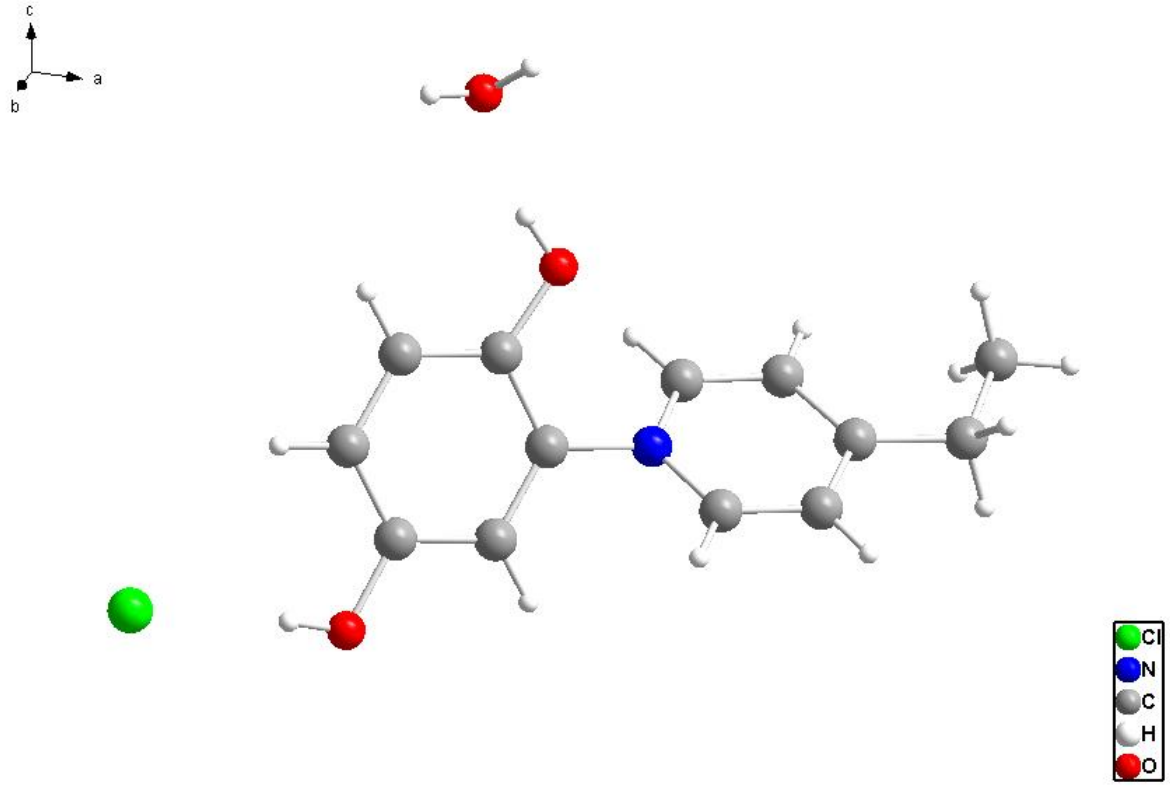
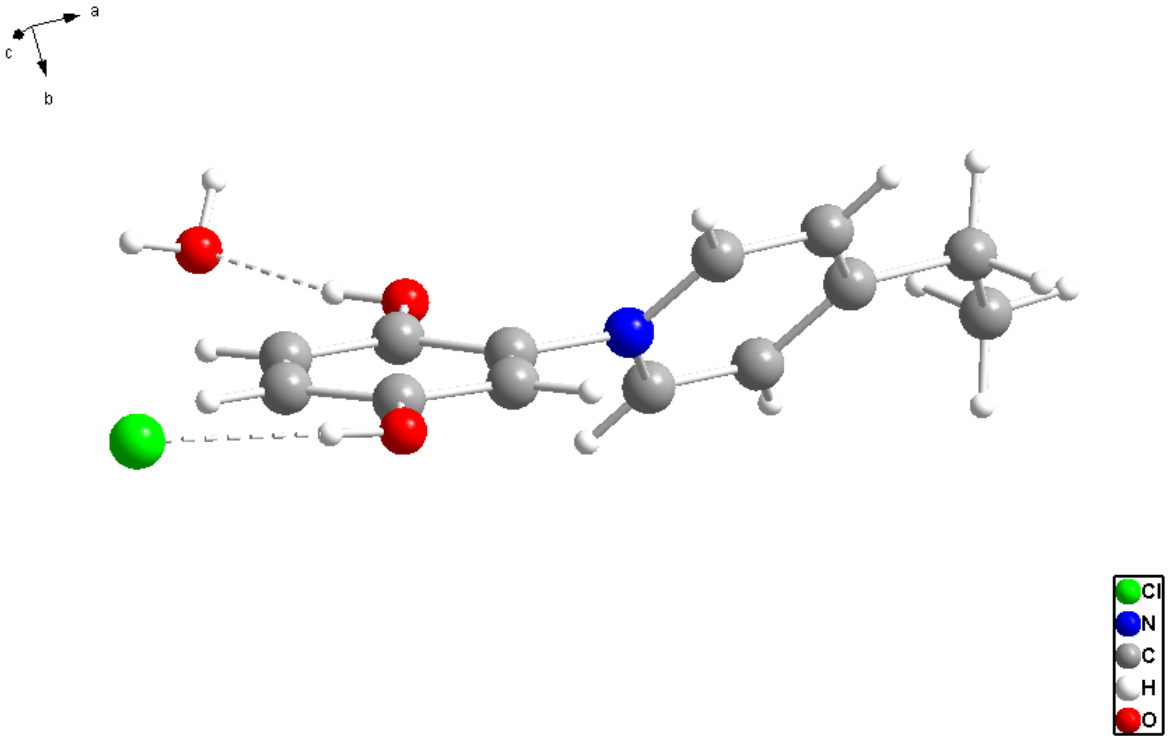
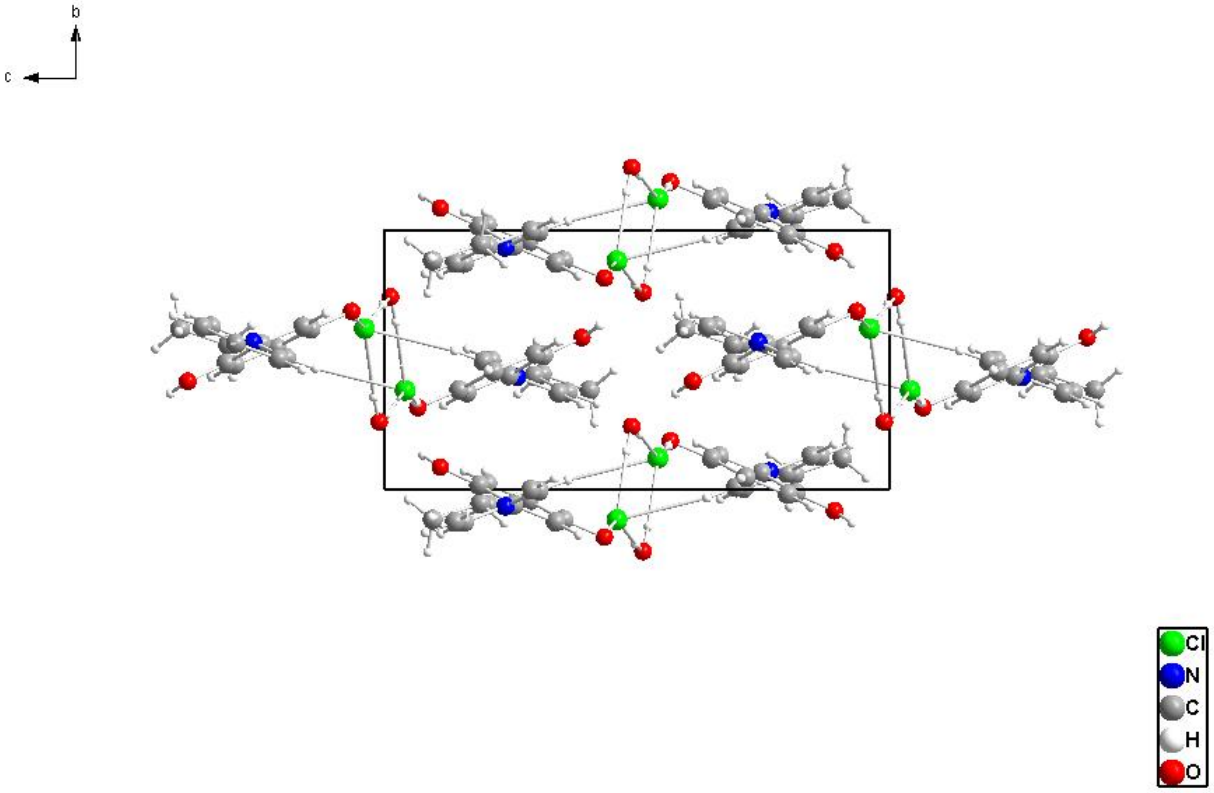


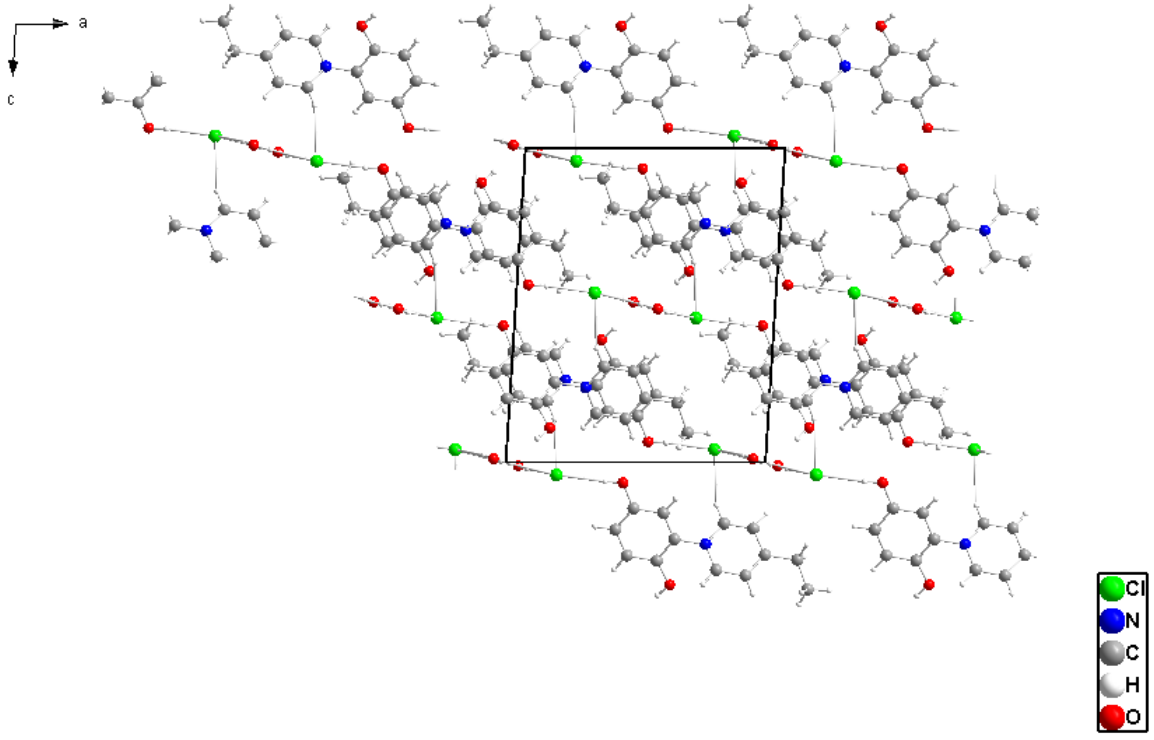
Fig. 1x. Molecular structure of **3** (displacement parameters are drawn at 50 % probability level).

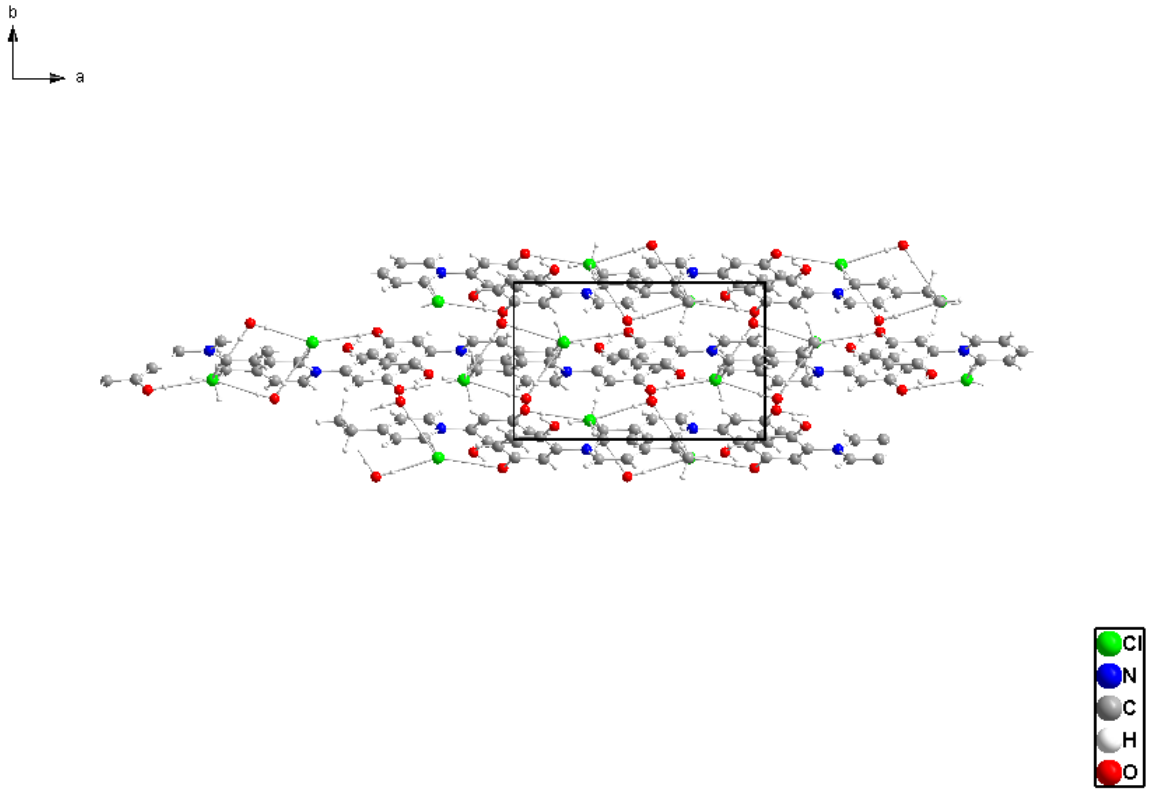



c
a
b
Cl
N
C
H
O


a
c
b
Cl
N
C
H
O


b
c
Cl
N
C
H
O


a
c
Cl
N
C
H
O



Computing details

Data collection: *APEX3* (Bruker AXS Inc., 2019); cell refinement: *APEX3* (Bruker AXS Inc., 2019); data reduction: *SAINT* (Bruker AXS Inc., 2019); program(s) used to solve structure: SHELXT (Sheldrick, 2015); program(s) used to refine structure: *SHELXL2014*/7 (Sheldrick, 2014); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Special details

Experimental. dx = 40 mm, 1 deg., 4+1 runs, 764 frames, 12 sec./frame
Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for X

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.19866 (2)	0.61923 (4)	0.04035 (2)	0.02514 (10)
N1	0.78934 (7)	0.56721 (11)	0.26148 (5)	0.01361 (17)
C2	0.85552 (8)	0.50638 (13)	0.19512 (6)	0.01613 (19)
H2	0.8224	0.4659	0.1374	0.019*
C3	0.96977 (8)	0.50307 (14)	0.21095 (7)	0.0182 (2)
H3	1.0152	0.4600	0.1640	0.022*
C4	1.02056 (8)	0.56230 (14)	0.29536 (7)	0.0166 (2)
C5	0.95049 (8)	0.62683 (13)	0.36169 (7)	0.0170 (2)
H5	0.9820	0.6697	0.4195	0.020*
C6	0.83581 (8)	0.62854 (13)	0.34358 (7)	0.01588 (19)

H6	0.7888	0.6731	0.3891	0.019*
C7	1.14621 (9)	0.55629 (16)	0.31085 (8)	0.0237 (2)
H7A	1.1790	0.6323	0.2631	0.028*
H7B	1.1712	0.4304	0.3011	0.028*
C8	1.19361 (10)	0.61850 (18)	0.40521 (9)	0.0316 (3)
H8A	1.2757	0.6093	0.4081	0.047*
H8B	1.1718	0.7444	0.4152	0.047*
H8C	1.1640	0.5420	0.4533	0.047*
C9	0.66816 (8)	0.56199 (13)	0.24537 (7)	0.01433 (19)
C10	0.60495 (8)	0.48489 (13)	0.31364 (6)	0.01552 (19)
O10	0.66046 (6)	0.41581 (11)	0.39031 (5)	0.02168 (17)
H10	0.6159 (13)	0.369 (2)	0.4248 (10)	0.033*
C11	0.48865 (8)	0.47994 (14)	0.29713 (7)	0.0181 (2)
H11	0.4434	0.4311	0.3428	0.022*
C12	0.43826 (8)	0.54555 (14)	0.21485 (7)	0.0183 (2)
H12	0.3589	0.5412	0.2047	0.022*
C13	0.50296 (8)	0.61802 (13)	0.14668 (7)	0.0173 (2)
O13	0.45648 (6)	0.68575 (12)	0.06532 (5)	0.02552 (18)
H13	0.3875 (11)	0.666 (2)	0.0618 (11)	0.038*
C14	0.61900 (8)	0.62732 (13)	0.16210 (7)	0.0162 (2)
H14	0.6640	0.6774	0.1166	0.019*
O1W	0.54826 (7)	0.24137 (12)	0.51117 (6)	0.02601 (18)
H1W1	0.4818 (11)	0.212 (2)	0.4960 (11)	0.039*
H1W2	0.5859 (12)	0.145 (2)	0.5218 (12)	0.039*

Atomic displacement parameters (Å^2) for X

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01603 (14)	0.03575 (17)	0.02321 (15)	-0.00048 (9)	-0.00215 (10)	-0.00159 (10)
N1	0.0119 (4)	0.0156 (4)	0.0133 (4)	-0.0007 (3)	0.0010 (3)	0.0009 (3)
C2	0.0165 (4)	0.0196 (5)	0.0124 (4)	-0.0019 (3)	0.0018 (3)	-0.0011 (3)
C3	0.0162 (5)	0.0229 (5)	0.0159 (4)	-0.0004 (4)	0.0048 (3)	-0.0016 (4)
C4	0.0134 (4)	0.0184 (4)	0.0181 (5)	-0.0015 (3)	0.0010 (4)	0.0014 (4)
C5	0.0159 (5)	0.0203 (5)	0.0145 (4)	-0.0017 (3)	-0.0005 (4)	-0.0015 (3)
C6	0.0168 (5)	0.0171 (4)	0.0138 (4)	-0.0004 (3)	0.0019 (3)	-0.0016 (3)
C7	0.0125 (4)	0.0325 (6)	0.0262 (5)	-0.0002 (4)	0.0010 (4)	-0.0017 (4)
C8	0.0184 (5)	0.0397 (7)	0.0354 (7)	-0.0018 (5)	-0.0080 (5)	-0.0056 (5)
C9	0.0113 (4)	0.0157 (4)	0.0160 (4)	-0.0007 (3)	0.0008 (3)	-0.0006 (3)
C10	0.0157 (4)	0.0170 (4)	0.0139 (4)	0.0004 (3)	0.0012 (3)	0.0010 (3)
O10	0.0164 (3)	0.0314 (4)	0.0171 (3)	-0.0021 (3)	0.0004 (3)	0.0096 (3)
C11	0.0158 (5)	0.0204 (5)	0.0185 (5)	-0.0006 (4)	0.0049 (4)	0.0016 (4)
C12	0.0124 (4)	0.0213 (5)	0.0211 (5)	0.0002 (3)	0.0010 (4)	0.0003 (4)
C13	0.0159 (5)	0.0189 (5)	0.0166 (5)	0.0007 (3)	-0.0012 (4)	0.0016 (3)
O13	0.0158 (3)	0.0383 (5)	0.0217 (4)	-0.0019 (3)	-0.0044 (3)	0.0113 (3)
C14	0.0151 (4)	0.0180 (5)	0.0156 (4)	-0.0015 (3)	0.0019 (3)	0.0020 (3)
O1W	0.0192 (4)	0.0337 (4)	0.0250 (4)	-0.0065 (3)	0.0007 (3)	0.0073 (3)

Geometric parameters (Å, º) for X

N1—C6	1.3565 (12)	C8—H8C	0.9800
N1—C2	1.3573 (12)	C9—C14	1.3924 (13)
N1—C9	1.4511 (12)	C9—C10	1.4007 (13)
C2—C3	1.3688 (13)	C10—O10	1.3553 (12)
C2—H2	0.9500	C10—C11	1.3942 (13)
C3—C4	1.3986 (14)	O10—H10	0.828 (13)
C3—H3	0.9500	C11—C12	1.3867 (14)
C4—C5	1.3956 (14)	C11—H11	0.9500

C4—C7	1.5031 (13)	C12—C13	1.3967 (14)
C5—C6	1.3775 (14)	C12—H12	0.9500
C5—H5	0.9500	C13—O13	1.3641 (12)
C6—H6	0.9500	C13—C14	1.3908 (13)
C7—C8	1.5156 (16)	O13—H13	0.835 (13)
C7—H7A	0.9900	C14—H14	0.9500
C7—H7B	0.9900	O1W—H1W1	0.838 (13)
C8—H8A	0.9800	O1W—H1W2	0.853 (13)
C8—H8B	0.9800		
C6—N1—C2	120.36 (8)	H8A—C8—H8B	109.5
C6—N1—C9	119.88 (8)	C7—C8—H8C	109.5
C2—N1—C9	119.74 (8)	H8A—C8—H8C	109.5
N1—C2—C3	120.37 (9)	H8B—C8—H8C	109.5
N1—C2—H2	119.8	C14—C9—C10	122.29 (9)
C3—C2—H2	119.8	C14—C9—N1	119.43 (8)
C2—C3—C4	120.88 (9)	C10—C9—N1	118.24 (8)
C2—C3—H3	119.6	O10—C10—C11	124.10 (9)
C4—C3—H3	119.6	O10—C10—C9	118.20 (9)
C5—C4—C3	117.46 (9)	C11—C10—C9	117.68 (9)
C5—C4—C7	122.89 (9)	C10—O10—H10	110.5 (10)
C3—C4—C7	119.64 (9)	C12—C11—C10	120.76 (9)
C6—C5—C4	120.24 (9)	C12—C11—H11	119.6
C6—C5—H5	119.9	C10—C11—H11	119.6
C4—C5—H5	119.9	C11—C12—C13	120.70 (9)
N1—C6—C5	120.68 (9)	C11—C12—H12	119.7
N1—C6—H6	119.7	C13—C12—H12	119.7
C5—C6—H6	119.7	O13—C13—C14	117.86 (9)
C4—C7—C8	115.97 (9)	O13—C13—C12	122.45 (9)
C4—C7—H7A	108.3	C14—C13—C12	119.66 (9)
C8—C7—H7A	108.3	C13—O13—H13	109.5 (11)
C4—C7—H7B	108.3	C13—C14—C9	118.88 (9)
C8—C7—H7B	108.3	C13—C14—H14	120.6
H7A—C7—H7B	107.4	C9—C14—H14	120.6
C7—C8—H8A	109.5	H1W1—O1W—H1W2	108.2 (14)
C7—C8—H8B	109.5		
C6—N1—C2—C3	1.23 (14)	C2—N1—C9—C10	130.00 (10)
C9—N1—C2—C3	-177.11 (9)	C14—C9—C10—O10	176.40 (9)
N1—C2—C3—C4	-0.12 (15)	N1—C9—C10—O10	-1.35 (13)
C2—C3—C4—C5	-0.90 (15)	C14—C9—C10—C11	-1.95 (14)
C2—C3—C4—C7	179.74 (10)	N1—C9—C10—C11	-179.71 (8)
C3—C4—C5—C6	0.84 (15)	O10—C10—C11—C12	-176.79 (10)
C7—C4—C5—C6	-179.82 (9)	C9—C10—C11—C12	1.46 (15)
C2—N1—C6—C5	-1.29 (14)	C10—C11—C12—C13	0.02 (16)
C9—N1—C6—C5	177.05 (9)	C11—C12—C13—O13	-179.24 (10)
C4—C5—C6—N1	0.23 (15)	C11—C12—C13—C14	-1.10 (15)
C5—C4—C7—C8	2.23 (16)	O13—C13—C14—C9	178.86 (9)
C3—C4—C7—C8	-178.45 (10)	C12—C13—C14—C9	0.64 (15)
C6—N1—C9—C14	133.84 (10)	C10—C9—C14—C13	0.91 (15)
C2—N1—C9—C14	-47.82 (12)	N1—C9—C14—C13	178.64 (8)
C6—N1—C9—C10	-48.34 (12)		

Hydrogen-bond geometry (Å, º) for X

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···Cl1^{i}	0.95	2.64	3.5494 (10)	160
C5—H5···O13^{ii}	0.95	2.40	3.2510 (12)	149
C6—H6···Cl1^{ii}	0.95	2.93	3.8519 (10)	164
O10—H10···O1*W*	0.83 (1)	1.80 (1)	2.6086 (11)	167 (2)
O13—H13···Cl1	0.84 (1)	2.28 (1)	3.1150 (8)	175 (2)
O1*W*—H1*W*1···Cl1^{iii}	0.84 (1)	2.29 (1)	3.1275 (8)	177 (2)
O1*W*—H1*W*2···Cl1^{iv}	0.85 (1)	2.38 (1)	3.2271 (9)	175 (2)

Symmetry codes: (i) -*x*+1, -*y*+1, -*z*; (ii) *x*+1/2, -*y*+3/2, *z*+1/2; (iii) -*x*+1/2, *y*-1/2, -*z*+1/2; (iv) *x*+1/2, -*y*+1/2, *z*+1/2.

11. Flotation of lithium aluminate using punicine **5** at pH 2.

Carbon Chain length	**Daylight**	**Darkness**	**UV**	**Daylight**	**Darkness**	**UV**
	Yield in %	Yield in %	Yield in %	Confidence interval %	Confidence interval	Confidence interval
0	28.56	27.22	27.00	1.89	6.69	6.85
1	32.71	42.96	44.06	4.36	6.47	7.46
2	38.33	43.51	40.42	7.19	6.17	6.32
8	45.37	53.22	50.42	5.39	5.21	5.97
11	44.09	47.73	54.85	2.63	1.56	2.00

Table 4: Flotation of lithium aluminate using different punicine derivatives as collector at pH 2, and under different light conditions. The conditioning here was as following: 60 µl collector, 1 min mixing, 30 µl foamer, 1 min mixing, stirring speed 500 rpm, air flow rate 32 ml/min using 250 mL distilled water and flotation time 3 min.

12. XRD of Lithium aluminate and Gehlenite mix

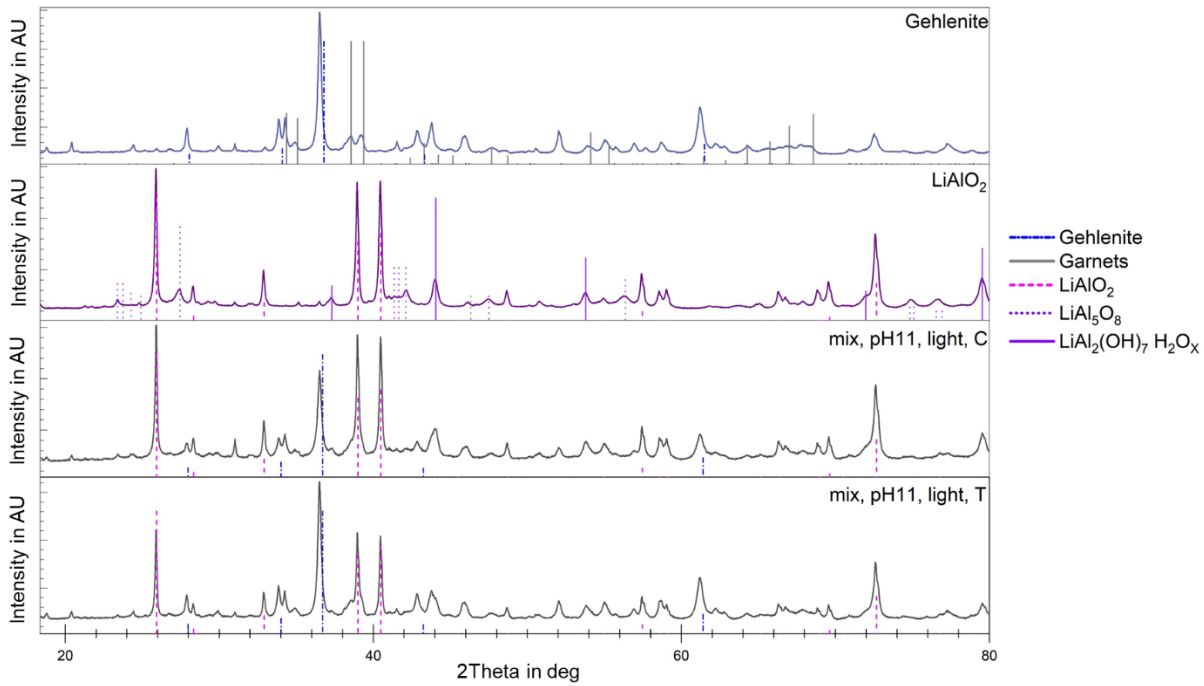


Figure 28: The figure shows the XRD results from the top of gehlenite, lithium aluminate and the floated mixture 1:1 by weight gehlenite and lithium aluminate where C (Concentrate) and T (Tailing) with the conditions punicine 5, pH11 and light. The difference in Intensity of the reflexes typical for lithium aluminate (40.5 deg) and for gehlenite (36.7) picture the different amount of each compound.

13. Wettability measurements

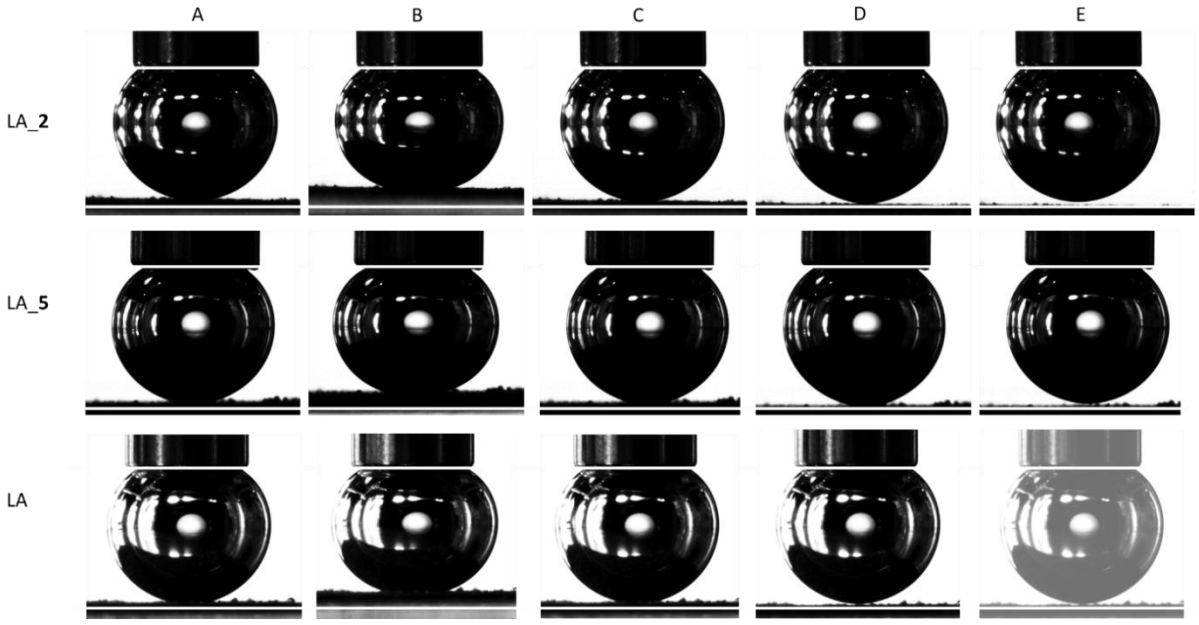


Figure 29: The figure pictures the wettability measurements for conducting the contact angle of lithium aluminate (LA) conditions with punicine **2** (LA_2) and **5** (LA_5) as well as the pinning behavior of bubbles on the particle bed. (A) contact angle beginning, (B) pressing particle bed against bubble with a certain pressure, (C) apparent contact angle, (D) pinning of bubble, (E) detaching of bubble from particle bed. In LA no pinning occurred in (D) so the bubble instantly detached from the particle bed (E).

14. Contact angle of lithium aluminate and gehlenite with punicine **2-5**.

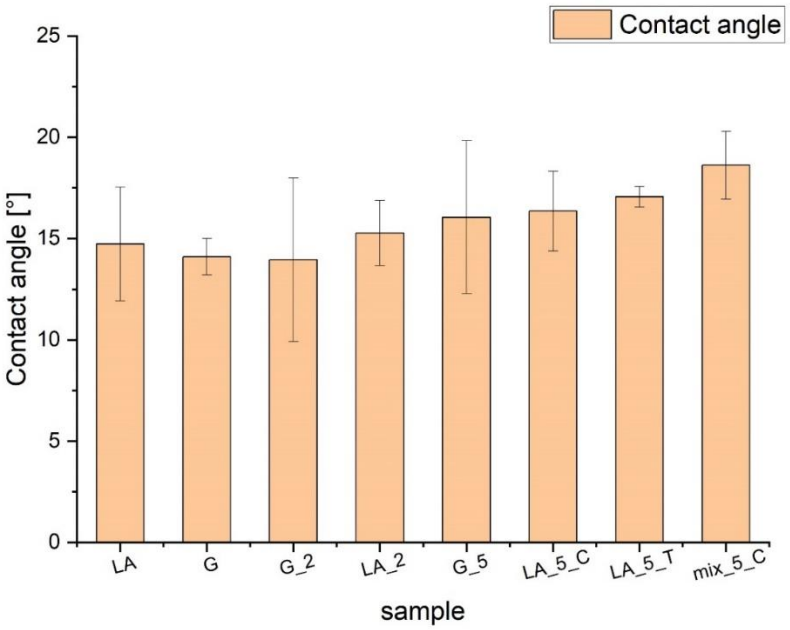


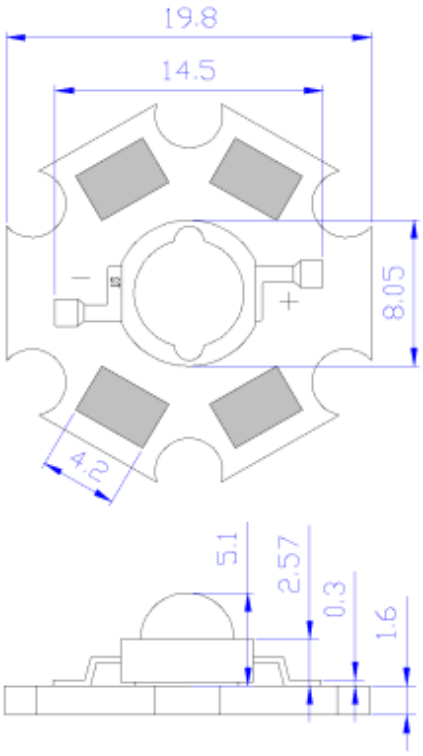
Figure 30: Apparent contact angle of lithium aluminate (LA) and gehlenite (G) and mix (1:1 gehlenite and lithium aluminate), conditioned with punicines 2 and 5 as well as samples resulting from flotation at pH 11. C (Concentrate), T (Tailing). Confidence interval is 95%.

15. Specification of the LEDs

Source: Avonec - 3W High Power LED auf Starplatine 390nm - 400nm UV-A, retrieved on 02.02.2023.

Outline Dimensions

1、Dome Type



2、Circuit diag ram

INRERNAL CIRCUIT DIAGRAM

Anode Cathode

Notes

1. All dimensions are in millimeters.(tolerance:±0.2)
2. Dimension Scale:1:1

*The appearance and specifications of the product may be changed for improvement without notice.

Parameters

Electrical-Optical Characteristics at IF=750mA，Ta=25℃

Parameter	Symbol	Min	Typ	Max	Unit
Radiation flux	Φe	900	~	1000	mw
Wavelength	λD	390	~	400	nm
Forward Voltage	V_F	3.5	~	4.5	V
Power Dissipation	P_D	2.63	~	3.38	W
View Angle	2θ1/2	~	120	~	deg.
Thermal Resistance	$R\theta_{J-B}$	~	12	~	℃/W

Absolute Maximum Ratings

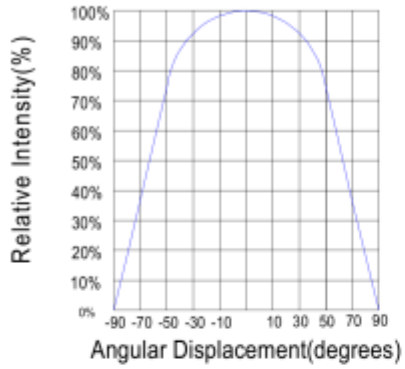
Parameter	Symbol	Value	Unit
Forward Current	I_F	750	mA
Junction Temperature	T_j	115	℃
Operating Temperature	T_{opr}	-40~+60	℃
Storage Temperature	T_{stg}	0~+60	℃
ESD Sensitivity	~	±2,000V HBM	~
Temperature Coefficient of voltage	~	-5	mV/℃
DC Pulse Current(@ 1 KHz,10% duty cycle)	I_{FP}	1000	mA
Reverse Voltage	V_R	Not designed for reverse operation	

*Notes

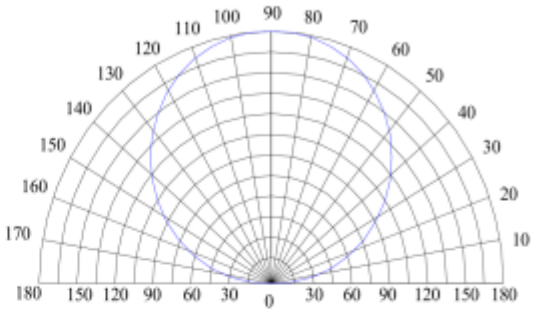
1. Tolerance of Luminous Flux is ±3%.
2. Tolerance of Forward Voltage is ±0.1V.

Typical Characteristic Curves(1)

1. Typical Light Distribution Curve

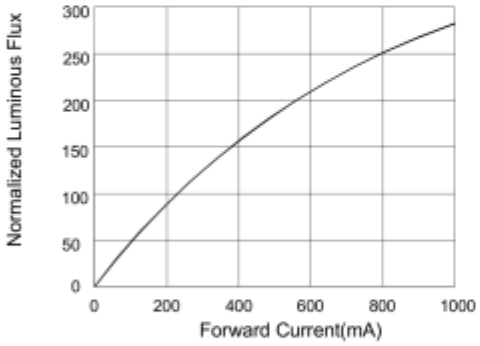


2. Typical Light-Emitting Angle Radiation Pattern

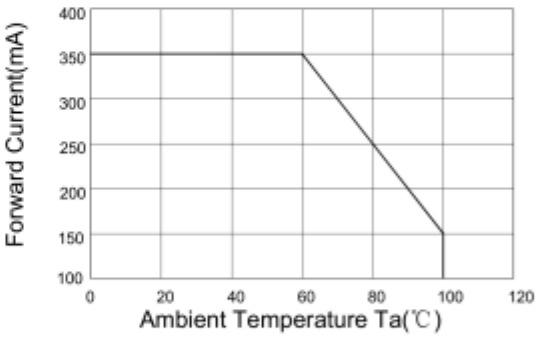


Typical Polar Radiation Pattern for White Lambertian

3. Forward Current vs.Relative Luminous Flux Curve

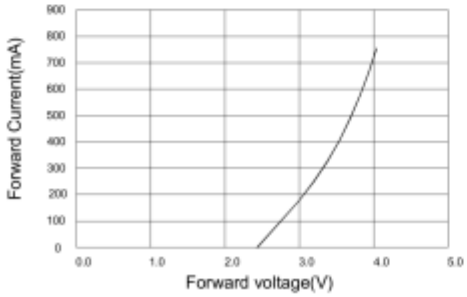


4. Forward Current Derating Curve,Derating based on Timax=125℃

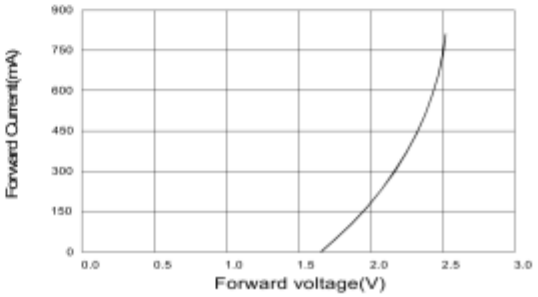


5. Electrical Characteristics Curve

5-1. White,Royal Blue , Blue, Green

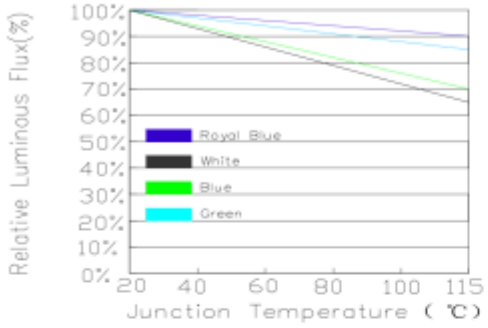


5-2. Amber, Red

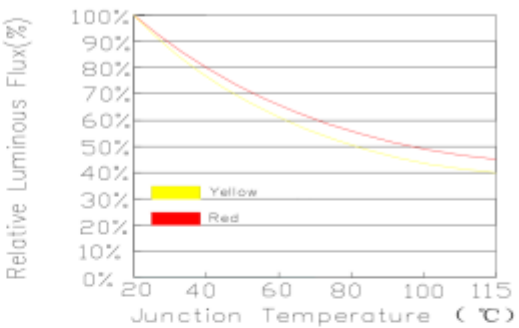


Typical Characteristic Curves(2)

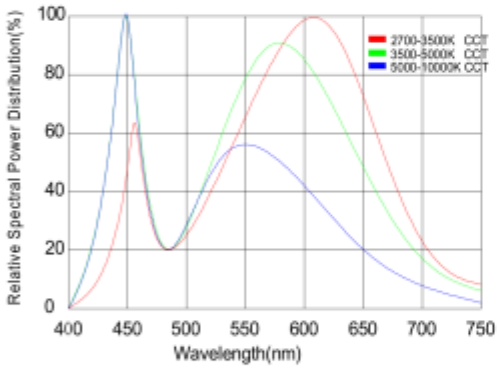
6-1. Relative Flux vs. Junction Temperature (If = 350 mA) White, Royal Blue, Blue, Green



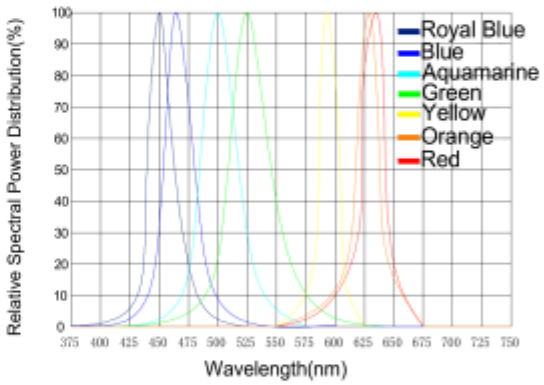
6-2. Relative Flux vs.Junction Temperature(If =400mA) Amber, Red



7. Typical white spectral distribution

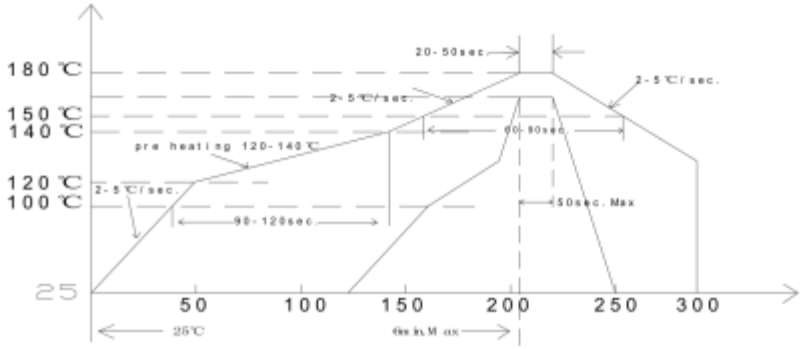


8. Relative Spectral Power Distribution



9. Reflow temperature time curve

Reflow Soldering Profile — Lead Free Solder



Reliability Test Items And Conditions

Test Items	Test Condition	Test Hours Cyles	Sample Size	Ac/Re
DC Aging	Ta=25℃ IF=750mA	1000H	22	0/1
Hot and cold shock	-40℃/30min +100℃/30min	100Cycles	22	0/1
High Temperature Storage	Ta=100℃	1000H	22	0/1
High Temperature High Humidity	85℃/85%RH	1000H	22	0/1
Low TemPerature Storage	Ta=-40℃	1000H	22	0/1
ESD(HBM)	2000V HBM	1Time	10	0/1

Criteria For Judging the Damage

Items	Symbol	Test Condition	Criteria For Judging Damage
Forward Voltage	V_F	I_F=750mA	Initial Data±10%
Reverse Current	I_R	V_R=5V	$I_R \leqslant 10\mu A$
Luminous Flux	ϕ_V	I_F=750mA	Average ϕ_V degradation≤20% Single LED ϕ_V degradation≤30%

Soldering Condition

Reflow Soldering			Manual Welding	
	High temperature PC lens	Molding products	Temperature	Soldering time
Preheat	100-140℃	180-200℃	Highest 350℃	3ses once
Heatup time	120sec Max	120sec Max		
Peak temperature	180℃ Max	260℃ Max		
Condition of Soldering time	50sec Max	10sec Max		

*Notes

Conventional PC lens products don't use reflow soldering.