Supporting information available

Thermotropic ionic liquid crystals by molecular-assembly and ion pairing of 4,4'-bipyridinium derivatives and tris(dodecyloxy)benzenesulfonats in a non-polar solvent

by

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The synthesis of compounds: **$MV^{2+}I_2$**, **$V^{3+}Br_3$**, **$MV^{6+}I_6$** and **CsDOBS** are known and described in literature[1-4].

^{1}H- and ^{13}C- NMR Spectra, IR-Spectra, DSC-Analysis

Experimental details corresponding to the synthesis and analytical data of compounds $MV^{2+}I_2$, $V^{3+}Br_3$, $MV^{6+}I_6$ and CsDOBS

Materials and Measurements

4,4´-Bipyridine (99%+, Fluka), Magnesium sulfate, anhydrous (95%+, Alfa Aesar);; acetonitrile (99,9%+, Sigma Aldrich), diethyl ether (99%+, Sigma Aldrich), methyliodide (99%+, Aldrich) 3,4,5-Triydroxybenzene(99 %, Aldrich), 1-bromodecane (99 % Aldrich), sulfuric acid (95,97%, Merck), sodium hydroxide, sodium sulfite, hypochloric acid 0.1 M (ACS reagent), barium acetate (99%, Fluka), Amberlite IR 120 (Fluka), caesium carbonate (99%, Aldrich) were used like received. Ethanol, n-hexane, ethylacetate, toluene, tetrahydrofuran, isopropyl ether were all ACS reagents and used as received. Dichloromethane, diethylether, chloroform and N,N-Dimethylformamide was distillated before to use.

NMR Analysis: ^{1}H-NMR (500 MHz) and ^{13}C-NMR (125 MHz) spectra were recorded on Bruker AMX-500 spectrometer; chemical shift δ in ppm related to tetramethylsilane (TMS) as internal standard in deuterated chloroform or deuterated water at 20°C. *Elemental analyses* were made on VarioMICRO cube and IR Analysis on FT-IR Spektrophotometer Vertex 70. *DSC Analysis*: Differential scanning calorimetry (DSC) measurements were performed on samples of about 4-12 mg. Phase transitions were measured on a Netzsch DSC 204 'Phoenix' differential scanning calorimeter. The heating and cooling rates were 10°C/min. Indium, tin and cyclohexane were used as calibration standards. *Thermo-optical Polarizing Microscopy:a* Zeiss AXIOPLAN 2 polarising microscope was used; equipped with a Mettler FP 80 hot stage d. Pictures were taken using a digital Zeiss AxioCam MRC4 camera with a resolution of 4 megapixels in combination with Zeiss AxioVision software. *Electrochemistry* Analysis: CVs were measured with the potentiostat PGSTAT 302N from AUTOLAB, controlled by PC running under GPES from Windows, version 4.9 (ECO Chemie B.V.). Glass slides (60 mm x 70 mm) coated with ITO (In-doped SnO_2, 20 Ω/cm^2) from BTE Bedampfungstechnik (Elsoff, Germany) was used for CV. The electroactive film with an area of 0.42 cm^2 and a thickness of 5 μm was obtained between two pieces of SnO_2 conducting glass and measured in two electrode system without electrolyte.

*Synthesis of 1,1´-dimethyl(4-pyridin-4-ylpyridinium) diiodide (**MV^{2+}**I_2)*[1]

To a solution of 5 g (32 mmol) 4,4´-bipyridine dissolved in 30 ml MeCN, 4 ml (64 mmol) of methyl iodide was added. The mixture was stirred at 70°C for 30 h, the resulting precipitate was filtered off, and thoroughly washed with dry diethyl ether and toluene. The crystallization of the product proceeded slowly (48 h at 4°C) from an ethanol-ether mixture (1:1,5 vol %). The resulting orange-red powder was dried for 12 h in an oil pump vacuum to yield 4.93 g (11.2 mmol) product as iodide, yield 35%. ^{1}H-NMR (500 MHz, D_2O, 20°C, TMS, δ/ppm): 9.07 (d, 4H, *J*=5.0 Hz), 8.55 (d, 4H, *J*=7.0 Hz) 4.53 (s, 6H). ^{13}C-NMR (125 MHz, D_2O, 20 °C, TMS, δ/ppm): 149.93 (s), 146.41 (d), 126.84 (d), 48.57 (q). IR (ATR, cm^{-1}) 3085(m), 2990(s), 1633(s), 1555(m), 1500(m), 1434(m), 1348(m), 1267(m), 1227(w), 1175(s), 1108(w), 965(w), 811(s), 700(w). Elemental analysis: calculated for $C_{12}H_{14}I_2N_2$ (wt %), C 32.75, H 3.21, N 6.37; found, C 32.77, H 3.55, N 6.50.

*Synthesis of 1,1´,1´´- [benzene-1,3,5-triyltris(methylene)tris](4-pyridin-4-ylpyridinium) tribromide (**V^{3+}**Br_3)*[2]

14 g (89 mmol) 4,4´-Bipyridine were dissolved in 80 mL of warm MeCN, and a solution of 5 g (5,8 mmol) 1,3,5-tris(bromomethyl)benzene[3] in 50 mL MeCN was

added within 7h at a rate of 0.118 mL/minute. The mixture was stirred at 70°C for 30 h under reflux. Subsequently the reaction mixture was cooled to 20°C, filtered and washed for three times with 45 mL of CH_2Cl_2. The filter cake was dissolved in water and extracted four times with 200 mL of CH_2Cl_2 each. The water was evaporated and the residue was dissolved in 30 ml methanol and then filtered. Diethyl ether was added drop wise to initiate the precipitation. A yellow powder was obtained and dried in an oil pump vacuum to yield 3.63 g (4.4 mmol) product as bromide, 76 %. TGA: decomposition beyond 215°C. ^{1}H-NMR (500 MHz, 20°C, TMS, δ/ppm): 6.09 (s, 6H), 7.54 (s, 3H), 7.91 (d, 6H, J=3.0 Hz), 8.50 (d, 6H, J=7.0 Hz), 8.79 (d, 6H, J=3.0 Hz), 9.11 (d, 6H, J=6.5 Hz). ^{13}C-NMR (125 MHz, D_2O, 20 °C, TMS,δ/ppm): 63.2 (t), 122.3 (d), 126.2 (d), 129.2 (d) 136.0 (s), 142.0 (s), 145.4 (d) 150.1 (d), 154.4 (s). IR (ATR, cm^{-1}) 3346(br), 3022(m), 2966(m), 1633(vs), 1597(m), 1542(m), 1521(m), 1490(m), 1459(m), 1405(s), 1350(m), 1216(m), 1157(m), 1069(w), 993(w), 870(w), 813(s), 748(m), 714(w). Elemental analysis: calculated for $C_{39}H_{33}Br_3N_6$ (wt %), C 56.75, H 4.03, N 10.18; found, C 55.92, H 4.22, N 8.62.

*Synthesis of 1,1´,1´´- [benzene-1,3,5-triyltris(methylene)tris](1’methyl 4-pyridin-4-ylpiridinium) hexaiodide (**MV^{6+}**I_6)*

To a solution of 0.5 g (0.5 mmol) 1,1´,1´´- [benzene-1,3,5-triyltris(methylene)tris](4-pyridin-4-ylpyridinium) trihexafluorophosphate ($V^{3+}(PF_6)_3$) dissolved in 8 mL MeCN was added 1 ml (16 mmol) of methyl iodide. After a few minutes from start the solution become red-dark and a precipitate was obtained. The mixture was stirred at 70 °C for 48 h. The resulting precipitate was filtered and thoroughly washed with MeCN. The resulting red powder was dried in an oil pump vacuum at 50^{o}C for 12 h to result 0.300 g (0.4 mmol) as iodide, yield 44 %. ^{1}H NMR (500 MHz, D_2O, 20 °C, TMS, δ/ppm): 9.51(*d*, 6 H, *J* (H,H) = 6.5 Hz), 9.29 (*d*, 6 H, *J* (H,H) = 6.5 Hz), 8.85 (*d*, 6 H, *J* (H,H) = 6.5 Hz), 8.78 (*d*, 6 H, *J* (H,H) = 6.5 Hz), 7.89 (s, 3H), 6.05 (s, 6H), 4.46 (s, 9H). ^{13}C NMR (125 MHz, D_2O, 20 °C, TMS, δ/ppm): δ = 48.10 (q), 62.23 (t), 126.18(d), 127.01(d), 130.36(d), 135.37(s), 145.91 (d), 146.51(d), 147.89(s), 148.98(s). Elemental analysis: calculated for $C_{42}H_{42}I_6N_6$ (wt %), C 36.23, H 3.04, N 6.04; found, C 36,10, H 3.46, N 6,26;0

Caesium 3,4,5-*tris(dodecyloxy)benzenesulfonate* (***CsDOBS***)[4]

5.0 g (7.03 mmol) 3,4,5-Trihydroxybenzenesulfonic acid and 23.8 g (73.05 mmol) Cs_2CO_3 were mixed with 60 ml of dried DMF under a nitrogen atmosphere. At 100 °C, 10 ml (41.6 mmol) of 1-bromododecane were added dropwise. The reaction mixture was stirred for 48 h at 100 °C and subsequently slowly poured into 50 ml of ice water. The precipitate was isolated by filtration and recrystalised twice from 100 ml of methanol to give a white powder. Yield: 1.8 g (2.1 mmol) 29,8%. M.p. 116.9 °C (by DSC). TLC (KG-60, $CHCl_3$: methanol = 6 vol : 1 vol): R_f = 0.46. ^{1}H NMR (500 MHz,$CDCl_3$, 20 °C, TMS; δ/ppm): 7.02 (s, 2H, aromatic), 3.95-3.91 (m, 6H, $O\underline{CH_2}$), 1.71-1.70 (m, 6H, $OCH_2\underline{CH_2}$), 1.45-1.41 (m, 6H, $O(CH_2)_2\underline{CH_2}$), 1.26 (overlapped peaks, 48H, $O(CH_2)_3(\underline{CH_2})_8$), 0,88 (t, 9H, CH_3, *J* = 5.0 Hz). ^{13}C NMR (125 MHz, $CDCl_3$, 20 °C, TMS; δ/ppm): 153.25(s), 140.19(s), 140.06(s), 104.59(d), 73.68(t), 69.65(t), 32.17(t), 30.64(t), 29.92(t), 29.73(t), 26.50(t), 22.91(t), 14.30(q). Elemental analysis: calculated for $C_{42}H_{77}CsO_6S$ (wt %), C 59.84, H 9.21; found, C 59.51, H 9.11.

References

(1) Ross, J.H.; Krieger, R.I.; *J. Agric. Food Chem*. **1980**, *28*, 1026-1031.

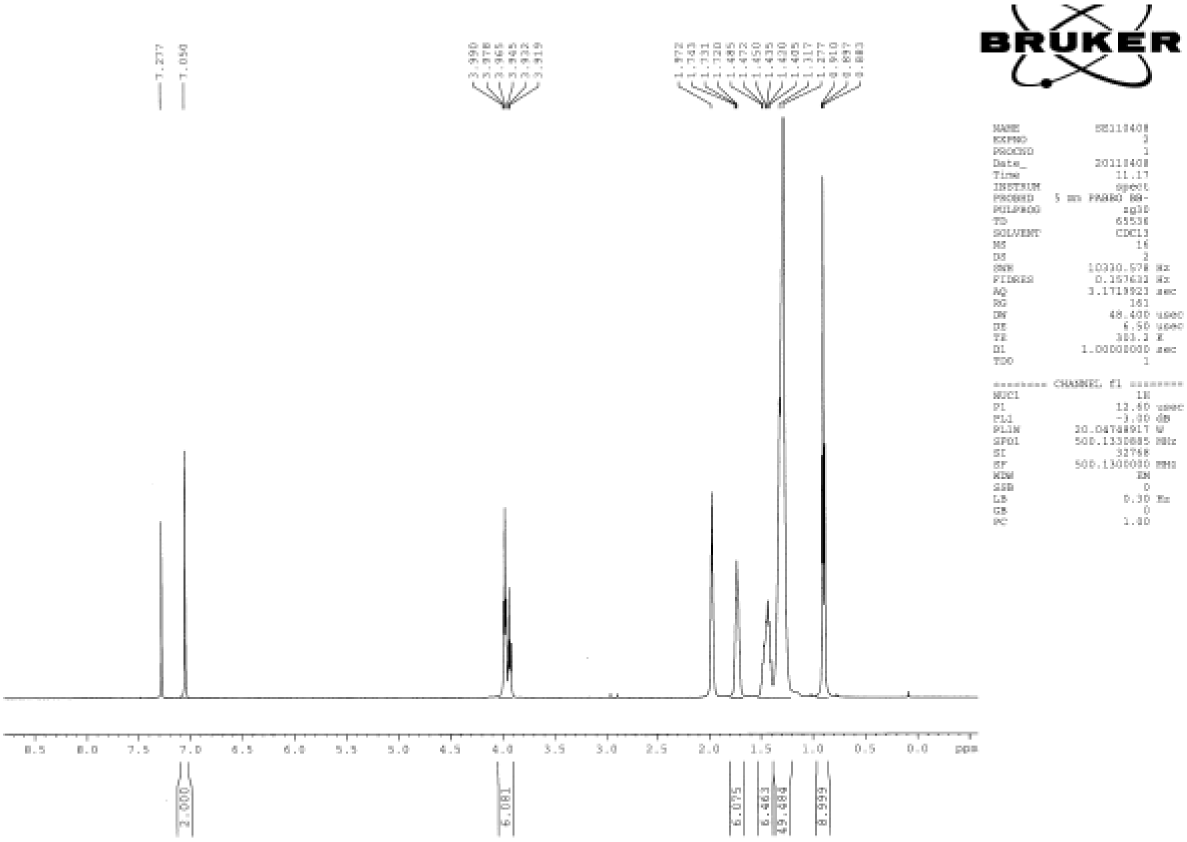
(2) Amabilino, D.B.; Ashton, P.R.; Belöhradsky, M.; Raymo, M.; Stoddart, J.F.; *J. Chem. Soc. Chem. Comm*., **1995**, *7*, 751-753.

(3) (a) Cochrane,W.; Pauson P.L.; Stevens T.S.; *J. Chem. Soc., C-Organic*, **1968**, 630-632. (b) Vögtle, F**.;** Zuber, M.; Lichtenthaler**,** R. G.; *Chem. Ber*. **1973**, *106*, 717−718.

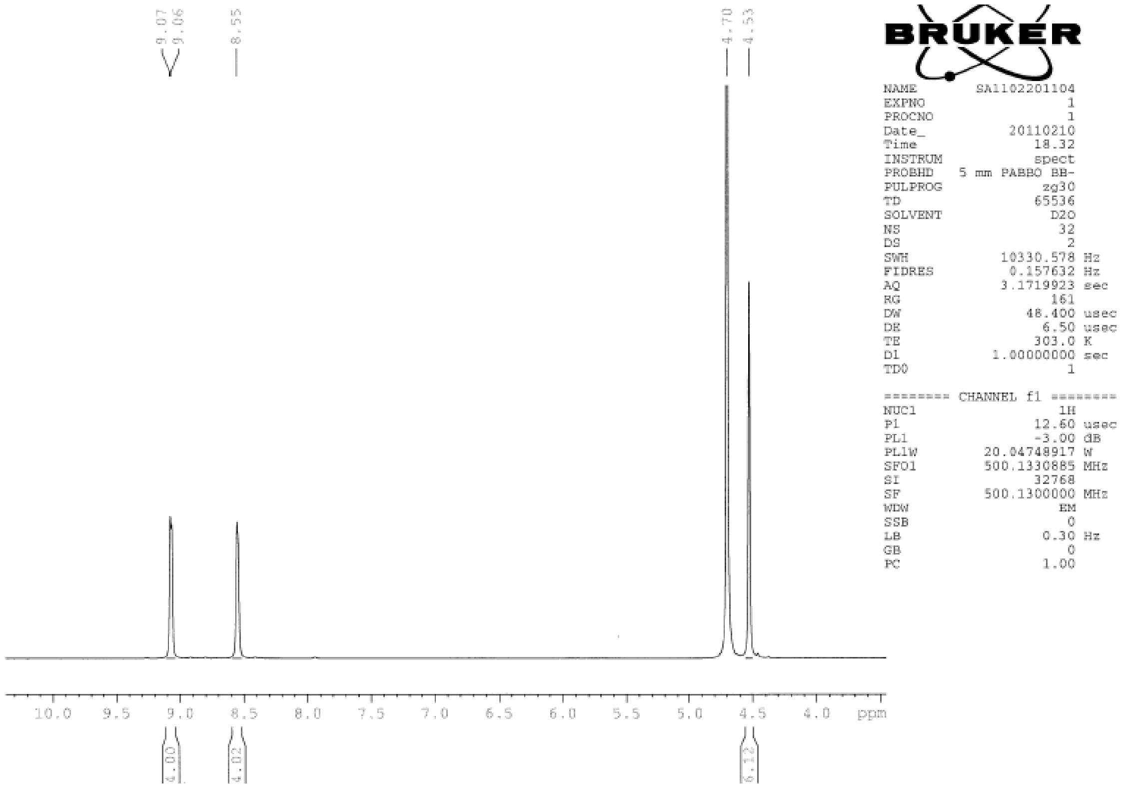
(4) Beginn, U.; Yan, L.; Chvalun, S. N.; Shcherbina, M.A.; Bakirov, A.; Moeller, M.; *Liq. Cryst*., **2008**, *35*, 1073-1093.

^{1}H-NMR

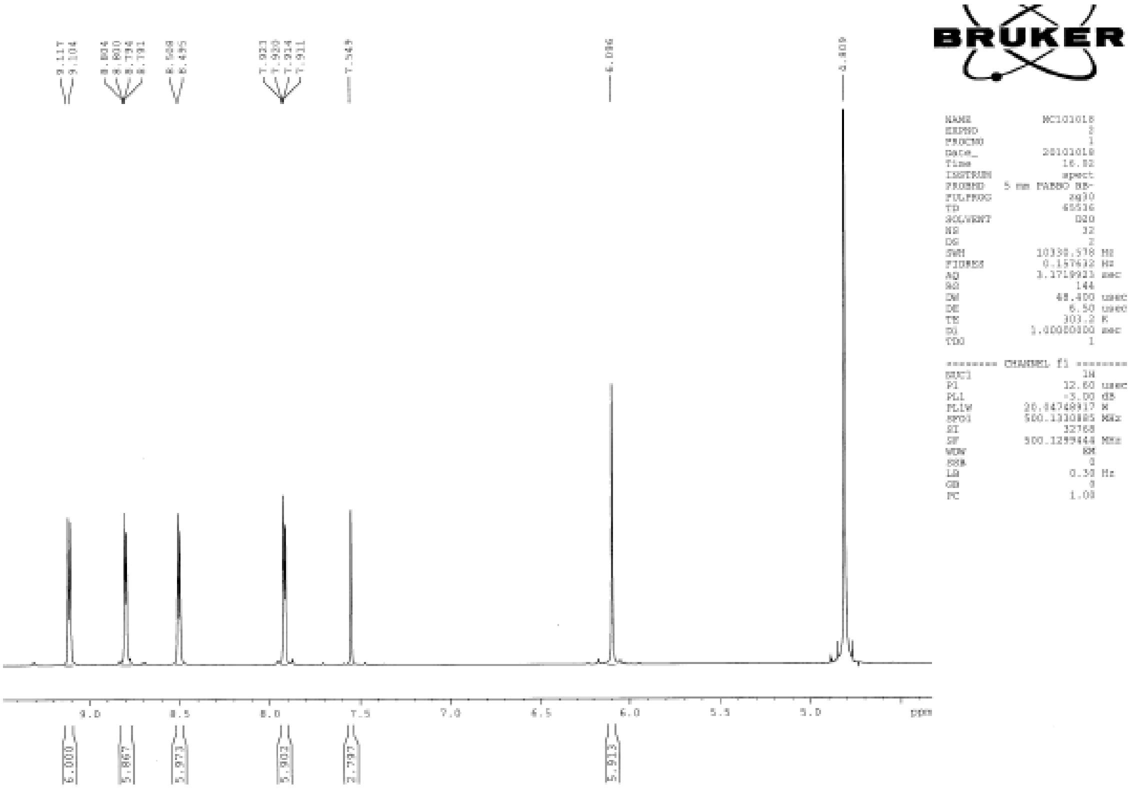
Caesium 3,4,5-tris(dodecyloxy)benzene sulfonate (**DOBS**)



1,1'-Dimethyl (4-pyridin-4-ylpyridinium) diiodide (**MV^{2+}I$_2$**)



1,1',1"-[Benzene-1,3,5-triyltris(methylene)]tris (4-pyridin-4-ylpyridinium) tribromide (**V^{3+}Br$_3$**)



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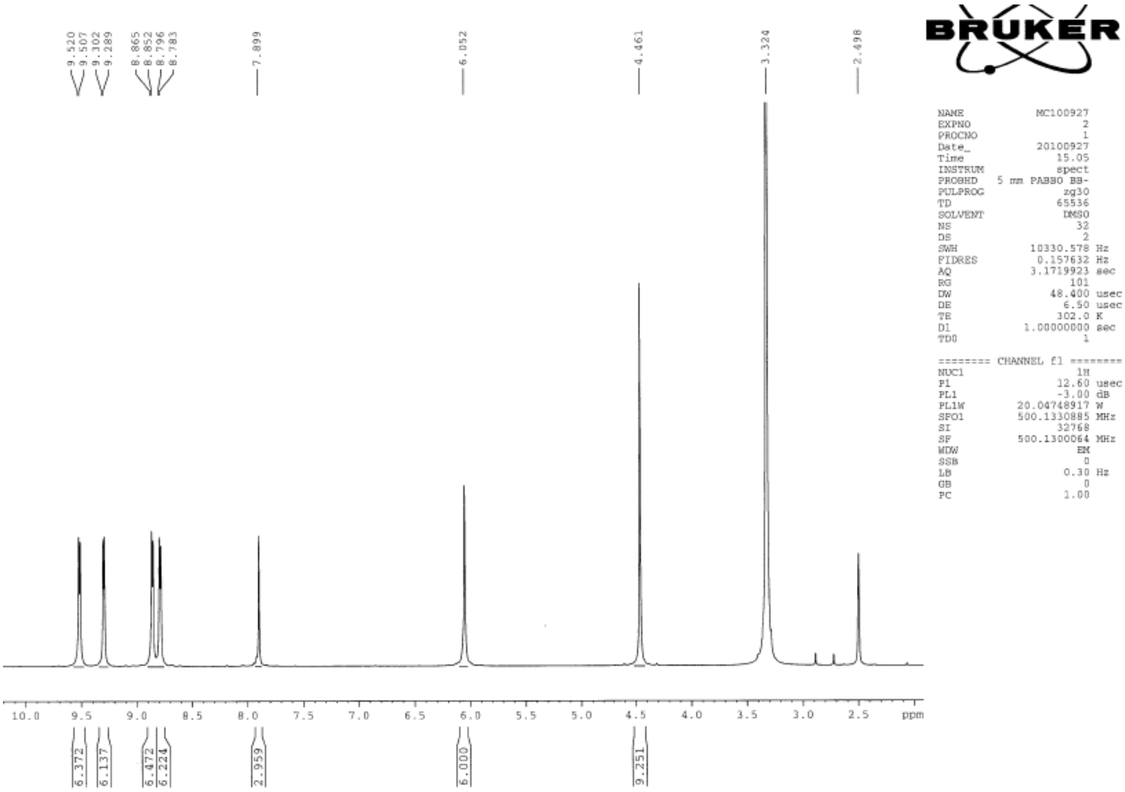

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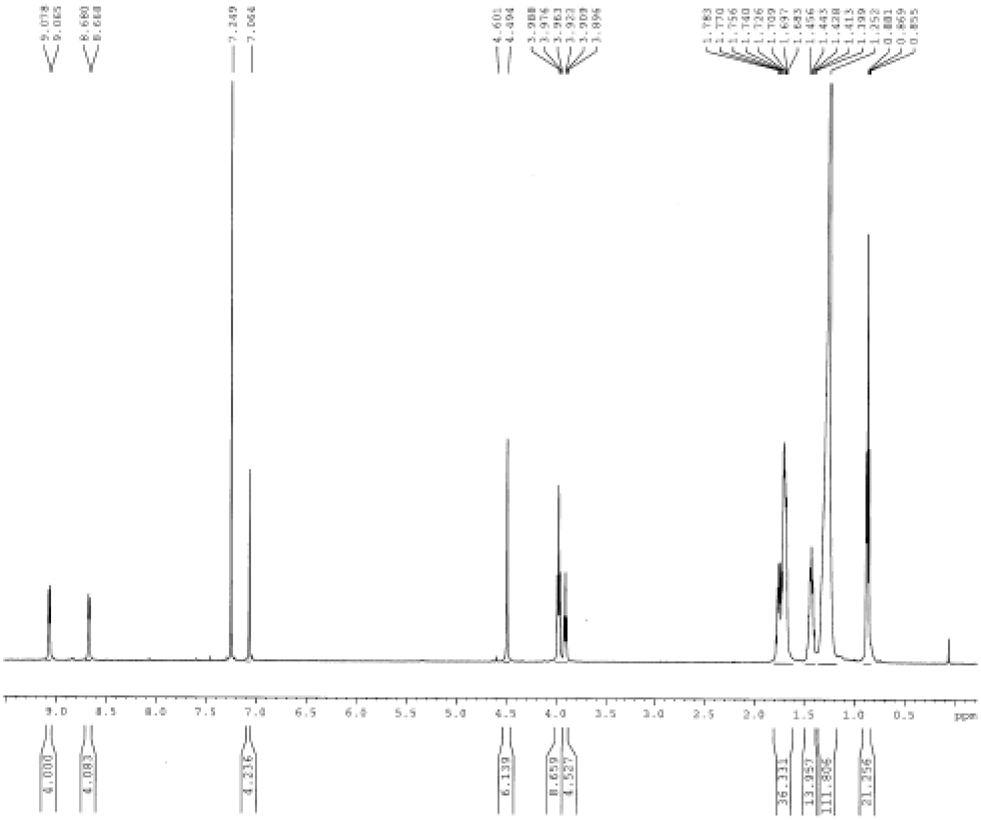
$MV^{2+}(DOBS)_2$



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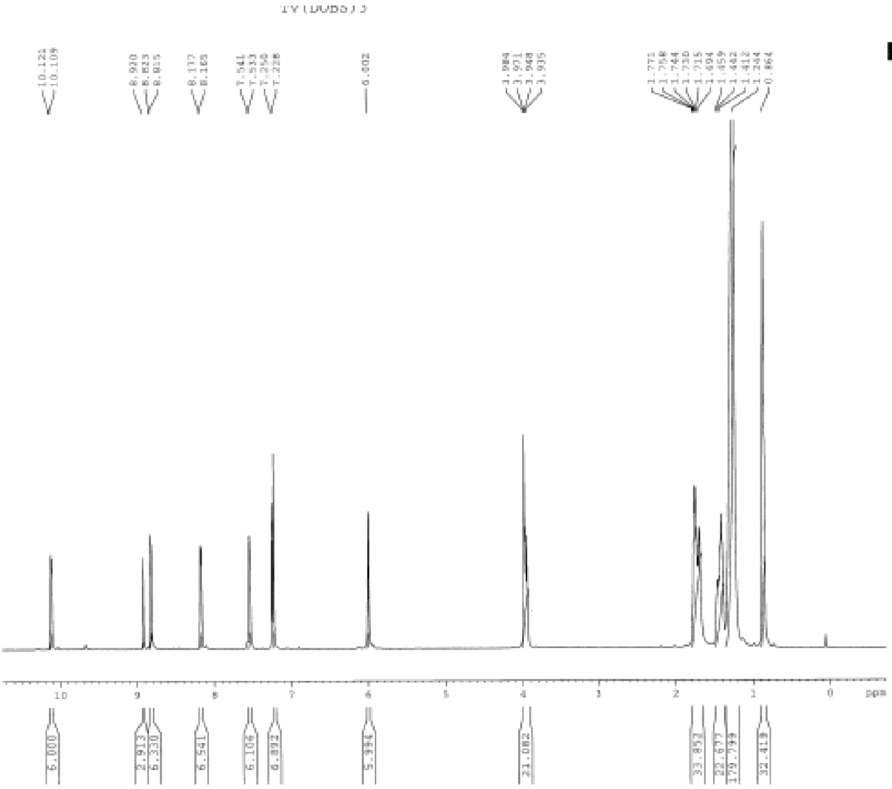
$V^{3+}(DOBS)_3$



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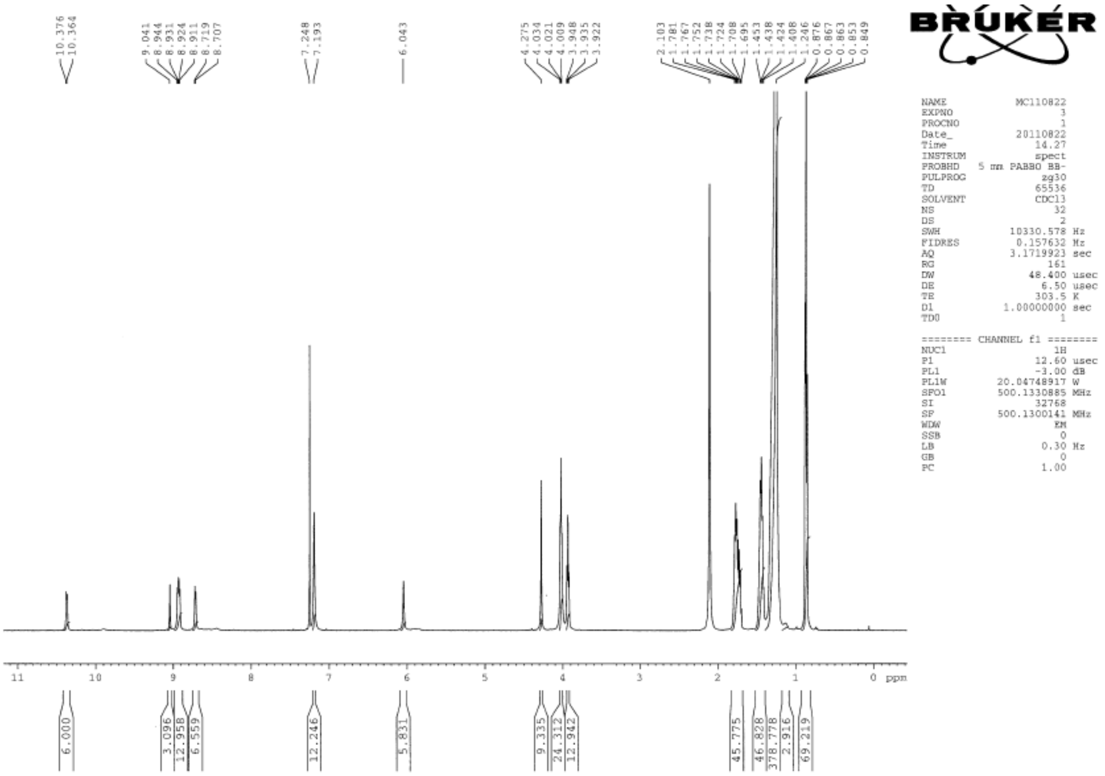
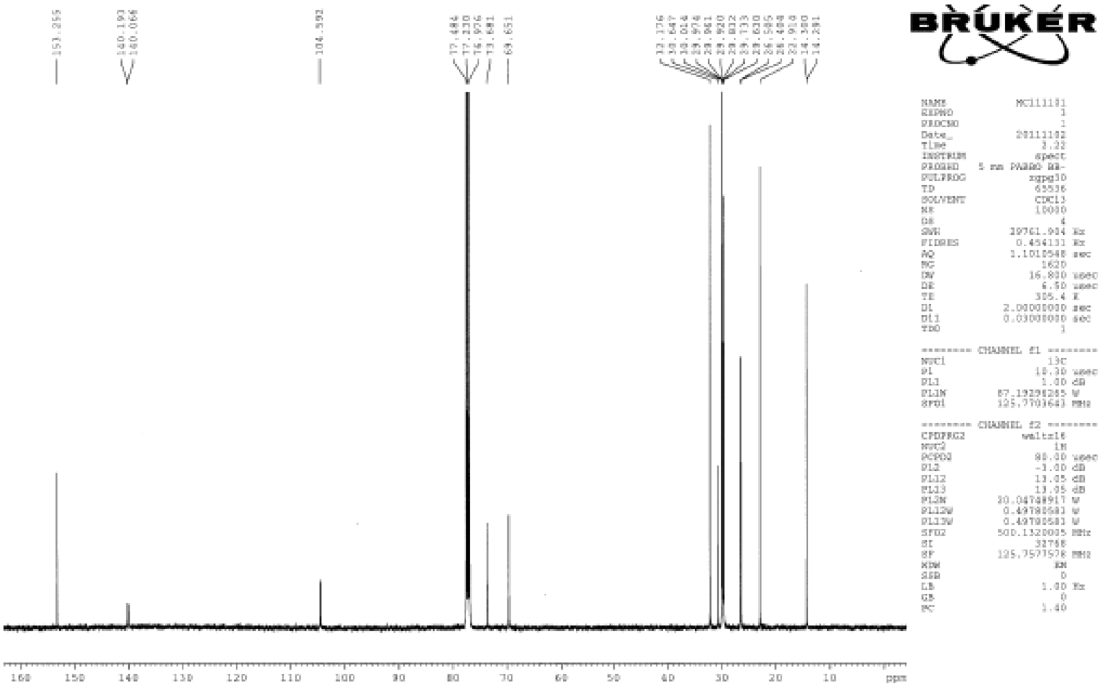
MV^{6+}(DOBS)$_6$

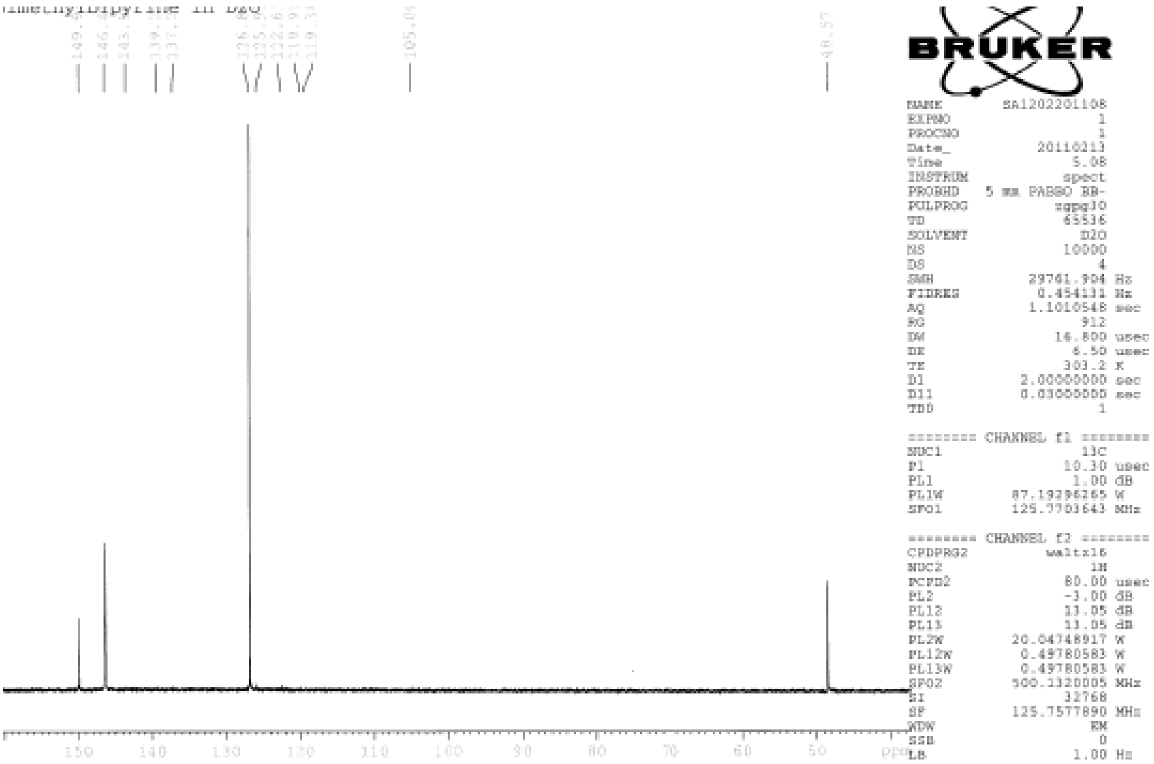


^{13}C-NMR

Caesium 3,4,5-tris(dodecyloxy)benzene sulfonate (**DOBS**)



1,1’-Dimethyl (4-pyridin-4-ylpyridinium) diiodide (**MV^{2+}I$_2$**)



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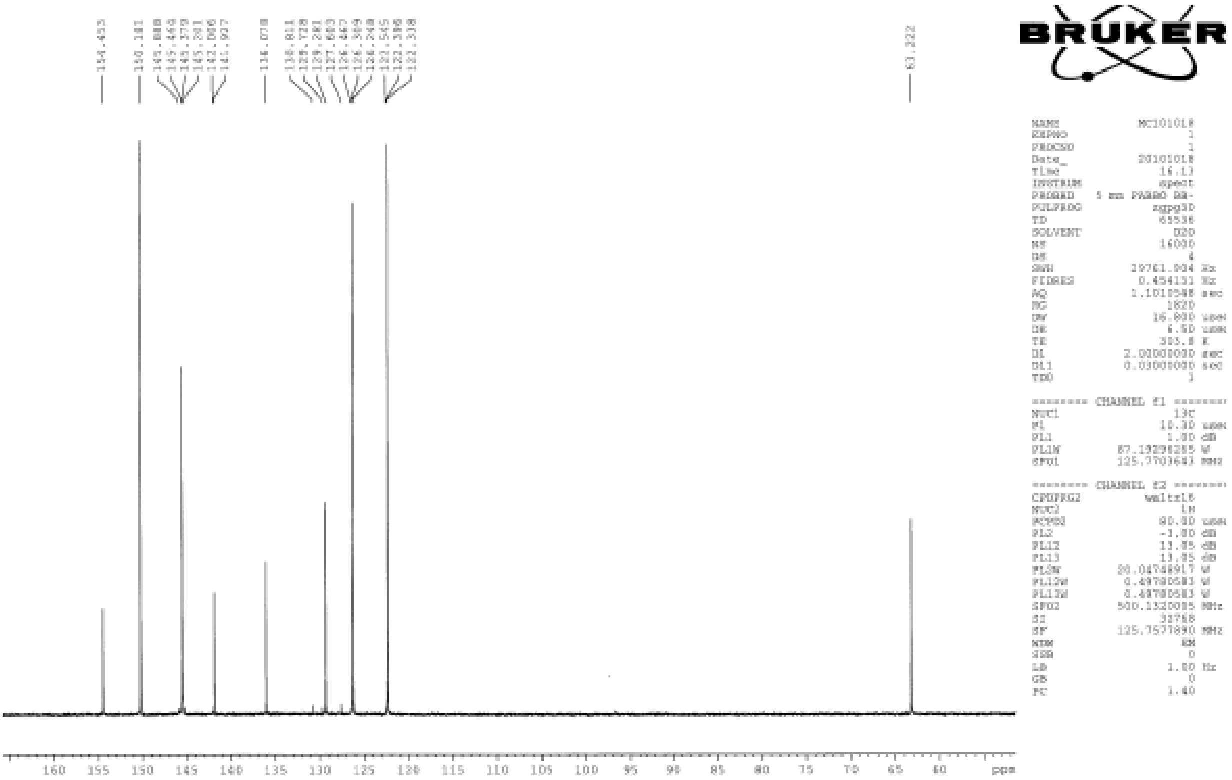
1,1’,1”-[Benzene-1,3,5-triyltris(methylene)]tris (4-pyridin-4-ylpyridinium) tribromide (**V^{3+}Br$_3$**)



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======== CHANNEL f1 ========
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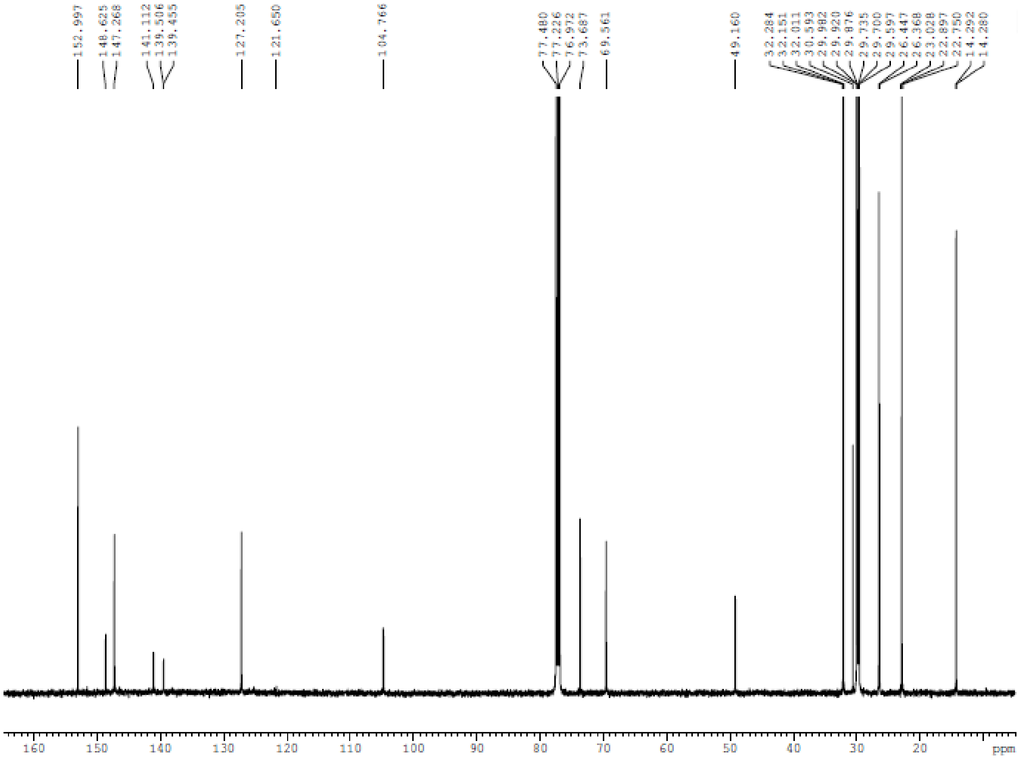
1,1’,1”-[benzene-1,3,5-triyltris(methylene)]tris[(methyl) 4-pyridin-4-ylpyridinium)] hexaiodide (**MV^{6+}I$_6$**)



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RG                103
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D11        0.0300000
TD0

======== CHANNEL f1 ===
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PL1W       87.192962
SFO1     125.770364

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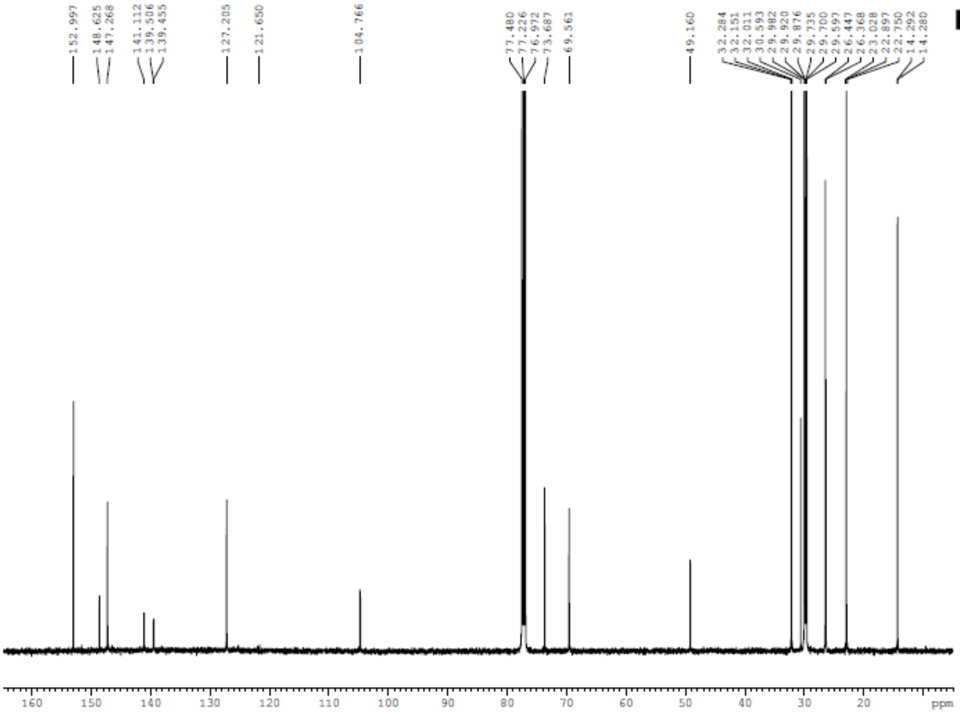
MV^{2+}(DOBS)$_2$



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AQ          1.101054
RG                103
DW              16.80
DE               6.5
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D11        0.0300000
TD0

======== CHANNEL f1 ===
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P1               10.3
PL1              1.0
PL1W       87.192962
SFO1     125.770364

======== CHANNEL f2 ===
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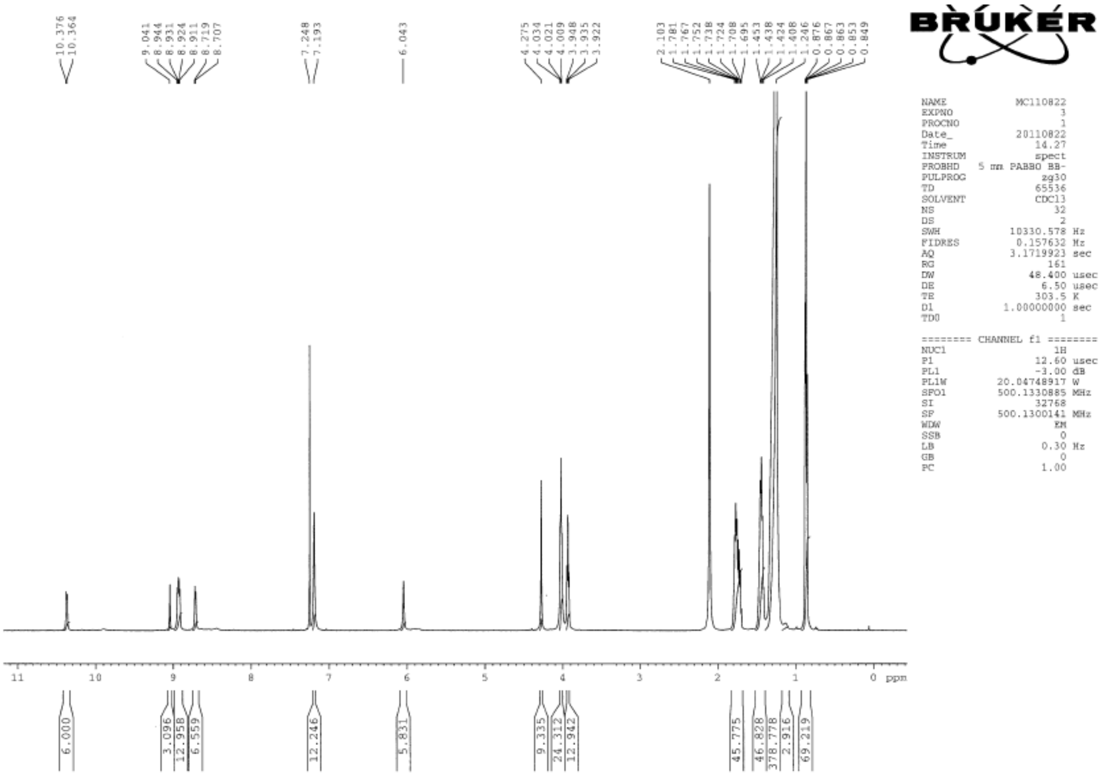
$V^{3+}(DOBS)_3$



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NS                   32
DS                    2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                  161
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DE                 6.50 usec
TE                303.5 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                12.60 usec
PL1               -3.00 dB
PL1W        20.04748917 W
SFO1        500.1330885 MHz
SI                32768
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WDW                  EM
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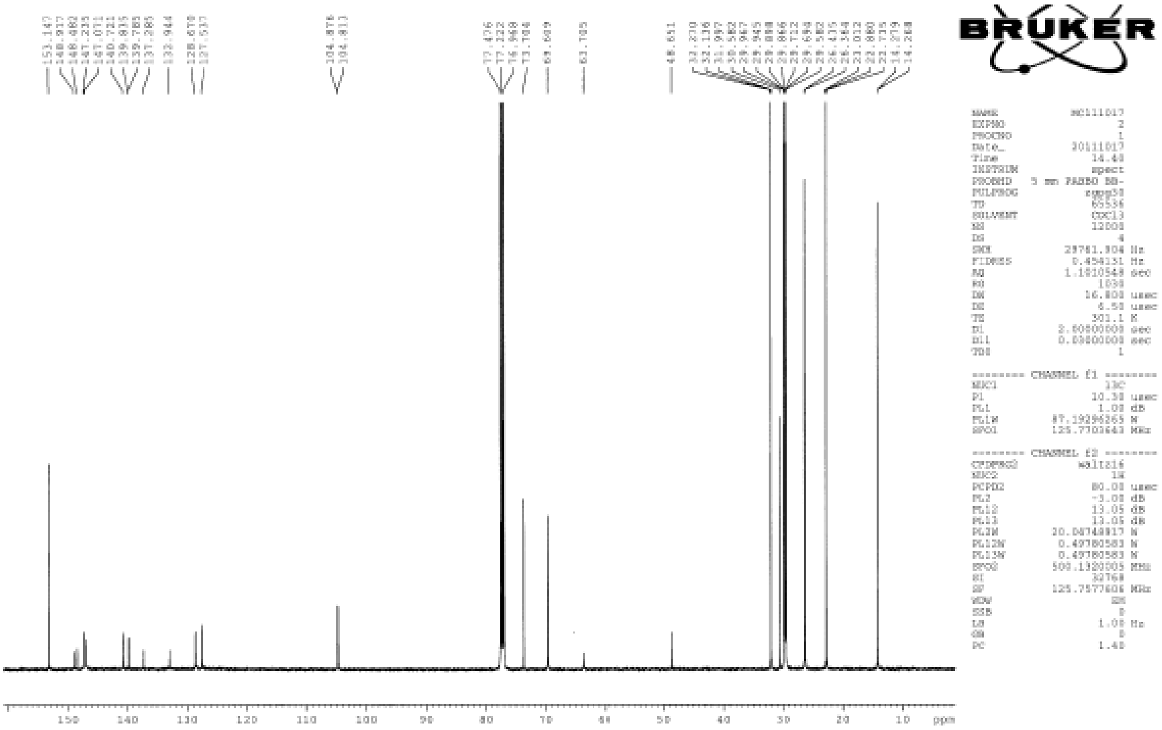
$MV^{6+}(DOBS)_6$



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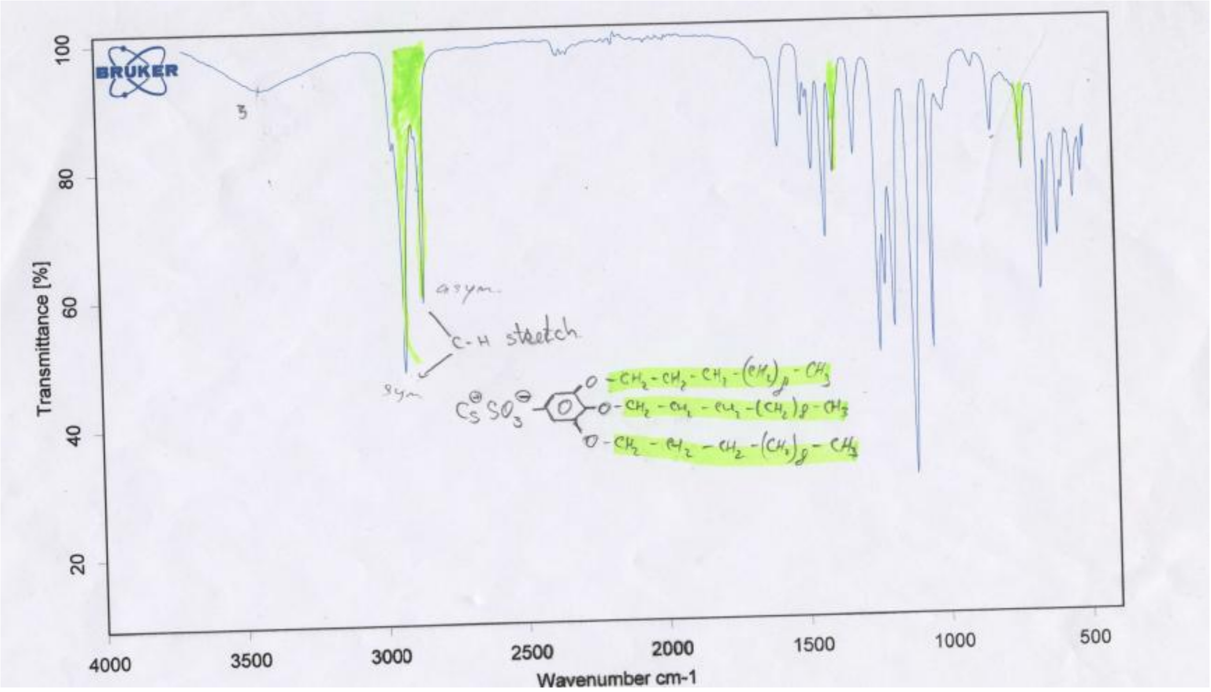
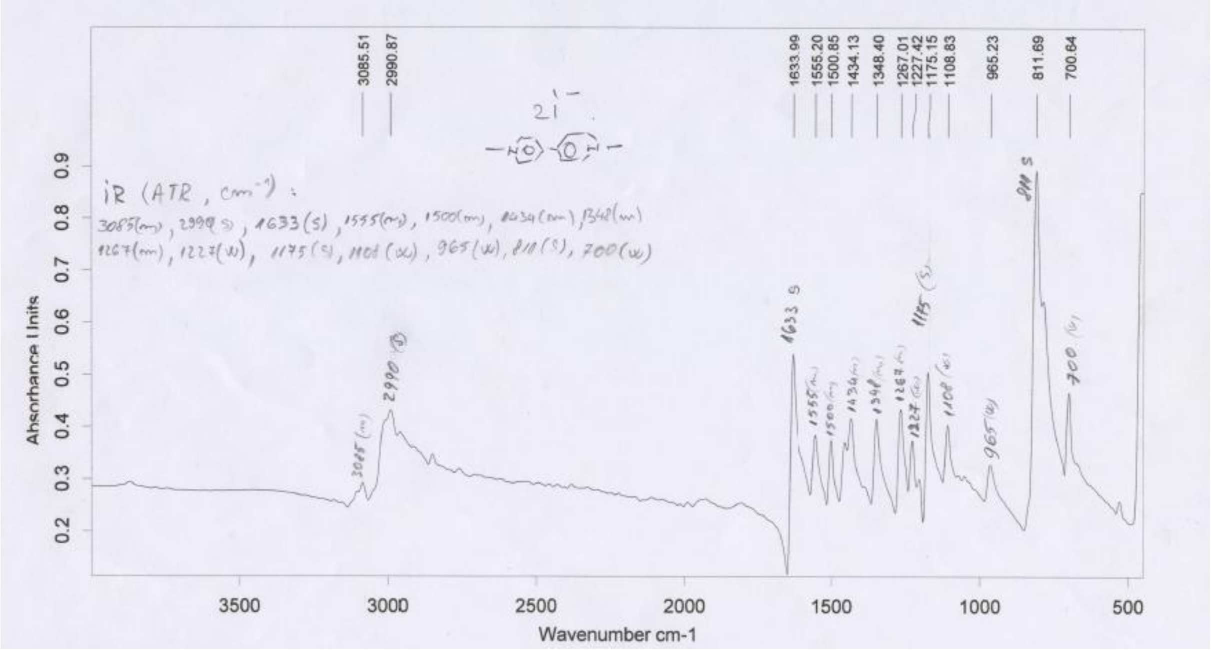
Date_          20111017
Time              14.48
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                12000
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010548 sec
RG                 1030
DW               16.800 usec
DE                 6.50 usec
TE                301.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

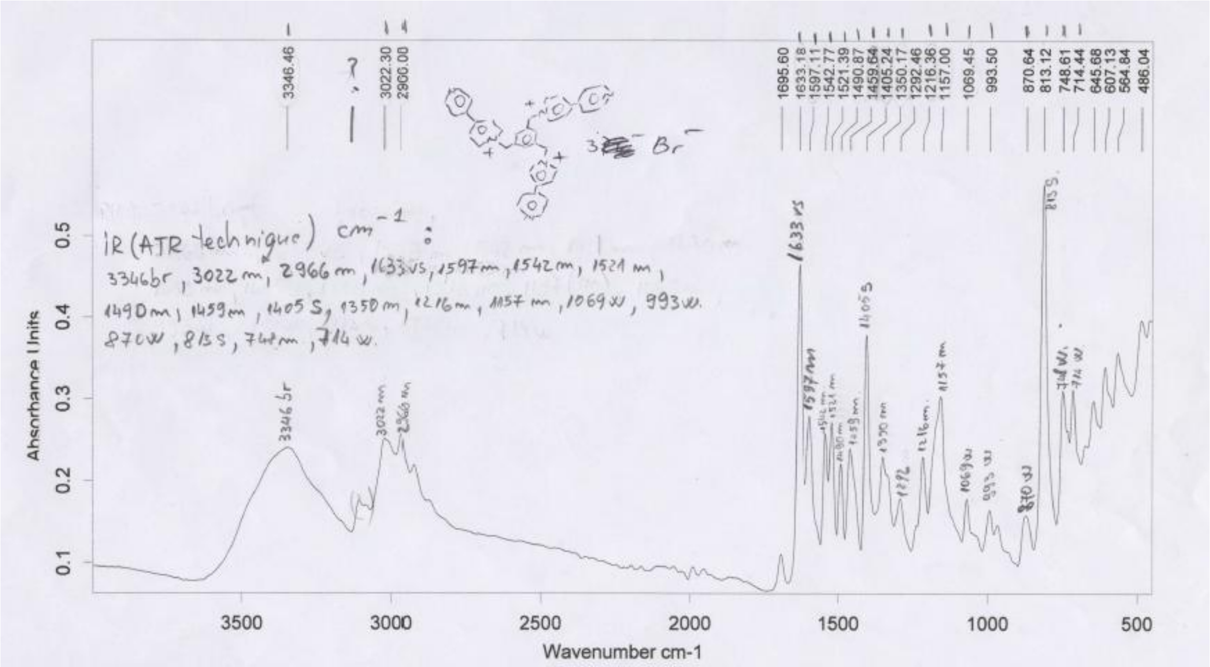
======== CHANNEL f1 ========
NUC1                13C
P1                10.30 usec
PL1                1.00 dB
PL1W        87.19296265 W
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              13.05 dB
PL13              13.05 dB
PL2W        20.04748917 W
PL12W        0.49780583 W
PL13W        0.49780583 W
SFO2        500.1320005 MHz
SI                32768
SF          125.7577606 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

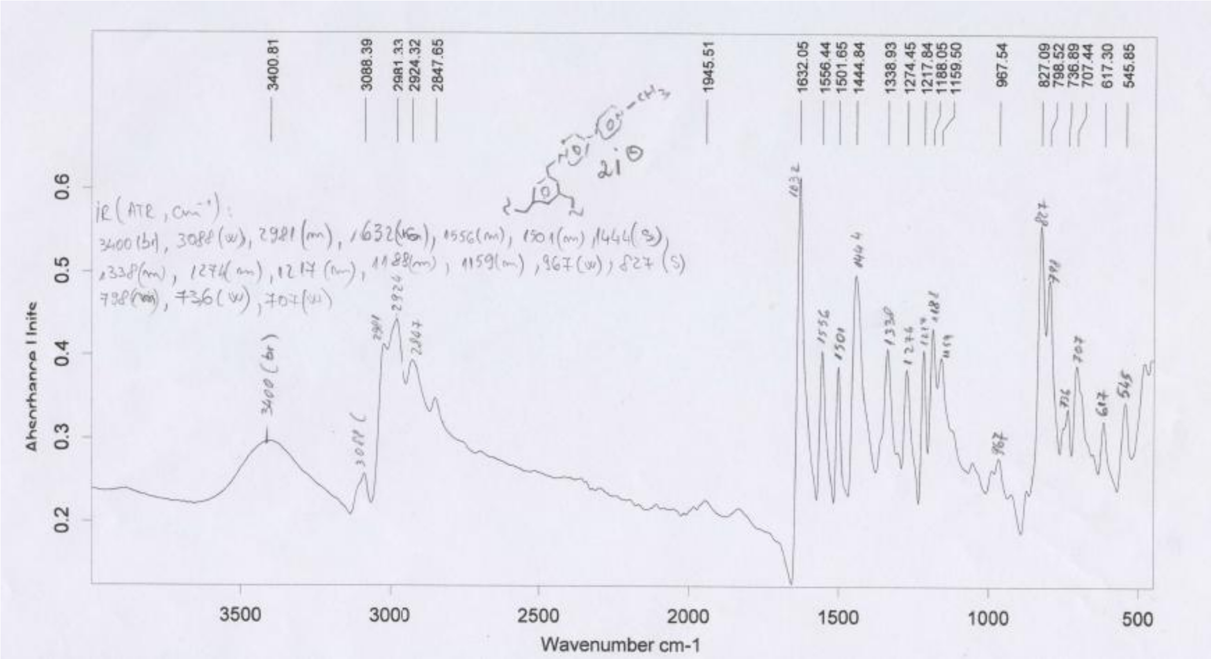




iR (ATR, cm⁻¹):
3085(m), 2990(s), 1633(s), 1555(m), 1500(m), 1434(m), 1348(m)
1267(m), 1227(w), 1175(s), 1108(w), 965(w), 811(s), 700(w)

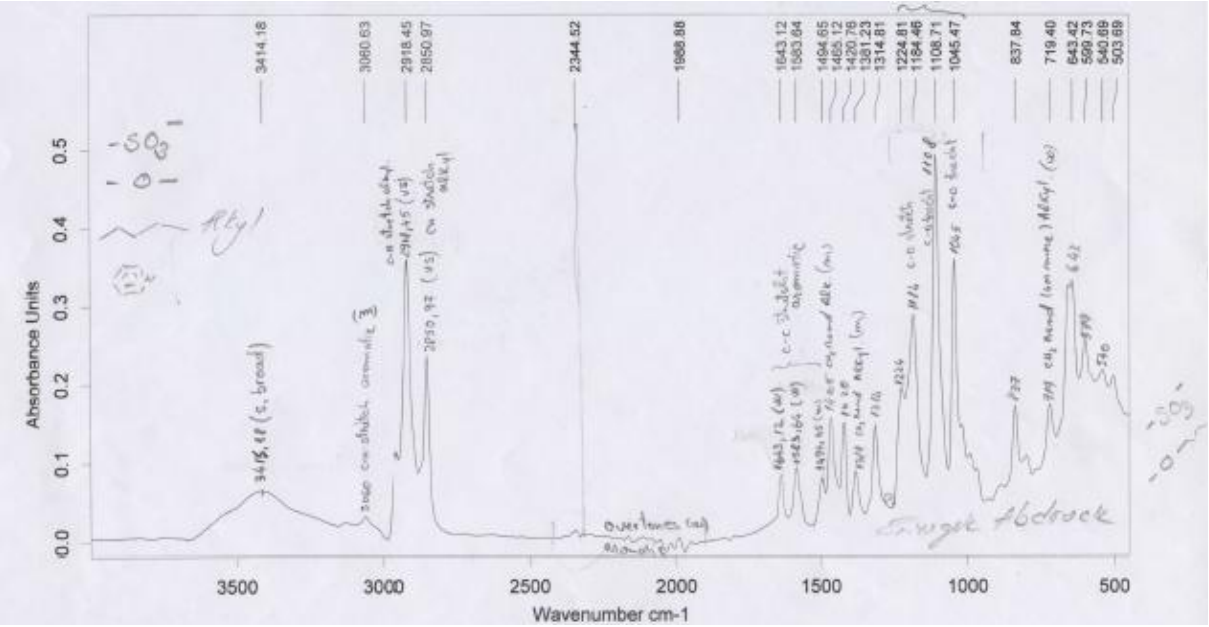


IR (ATR technique) cm^{-1} :
3346br, 3022 m, 2966 m, 1633 vs, 1597 m, 1542 m, 1521 m, 1490 m, 1459 m, 1405 S, 1350 m, 1216 m, 1157 m, 1069 w, 993 w. 870 w, 815 S, 748 m, 714 w.

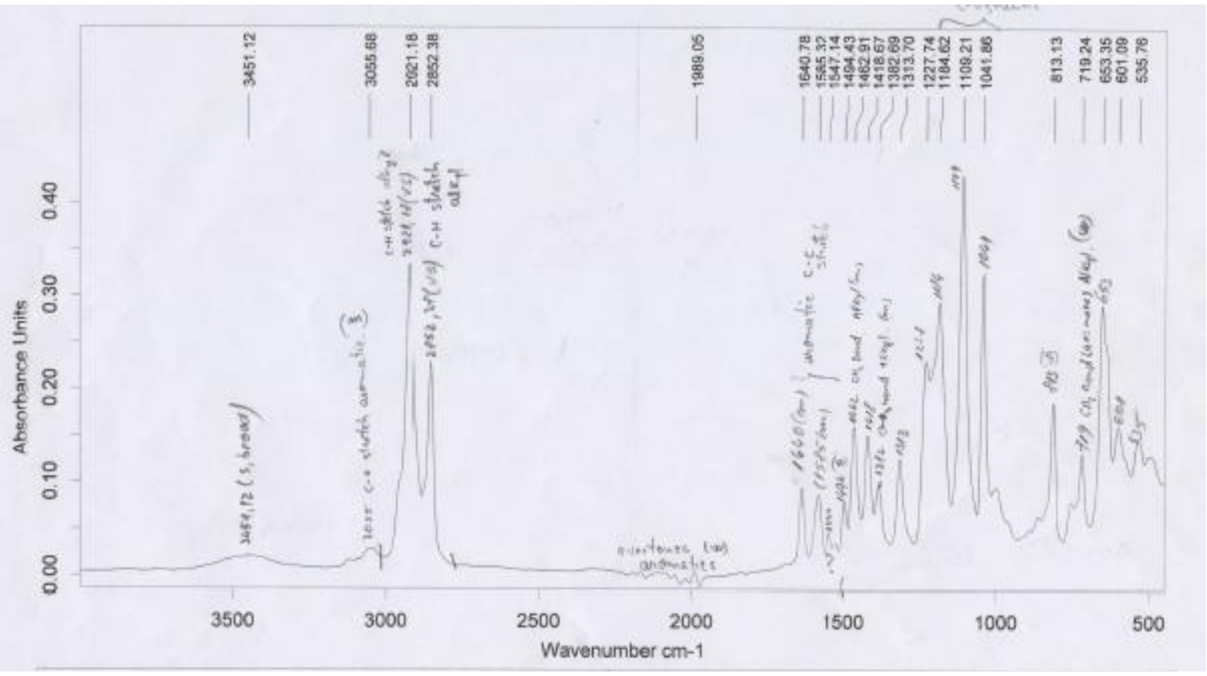


iR (ATR, cm^{-1}):
3400 (br), 3088 (w), 2981 (m), 1632 (vs), 1556 (m), 1501 (m), 1444 (s), 1338 (m), 1274 (m), 1217 (m), 1188 (m), 1159 (m), 967 (w), 827 (S) 798 (m), 736 (w), 707 (w)

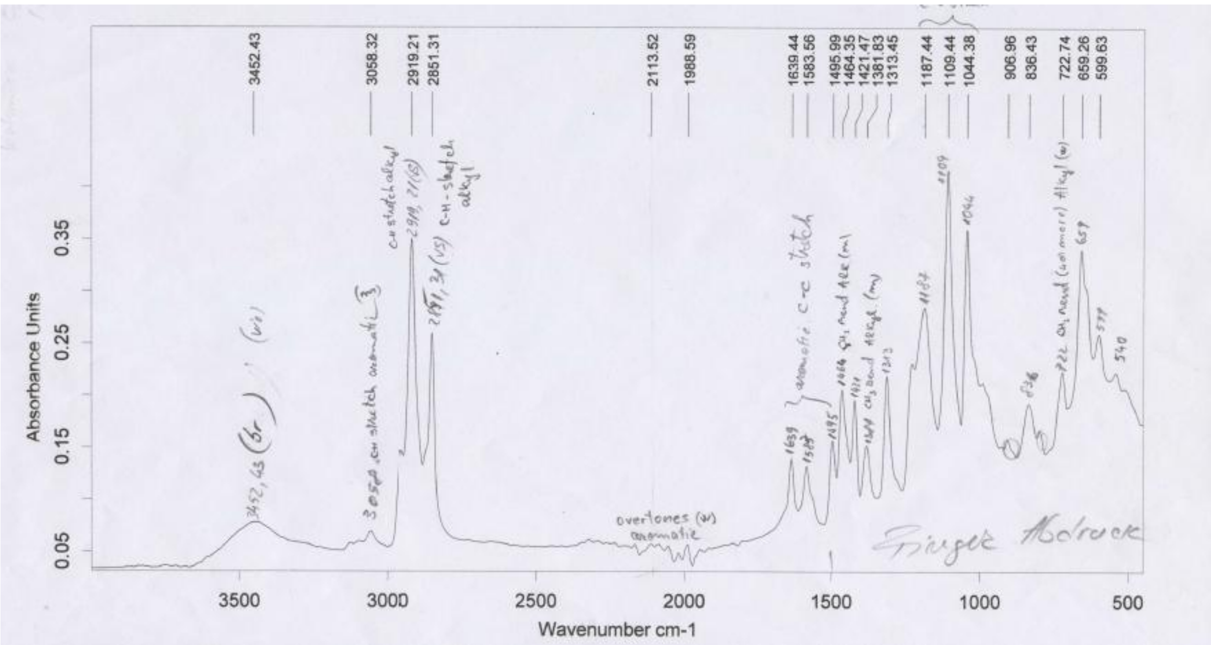
$MV^{2+}(DOBS)_2$



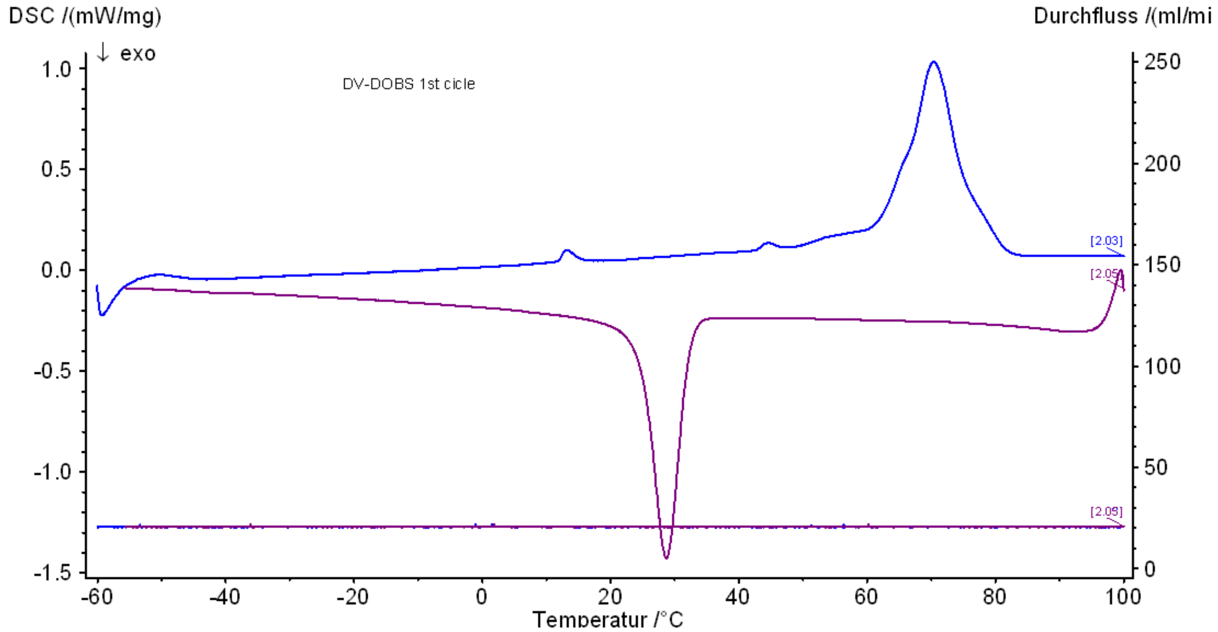
$V^{3+}(DOBS)_3$

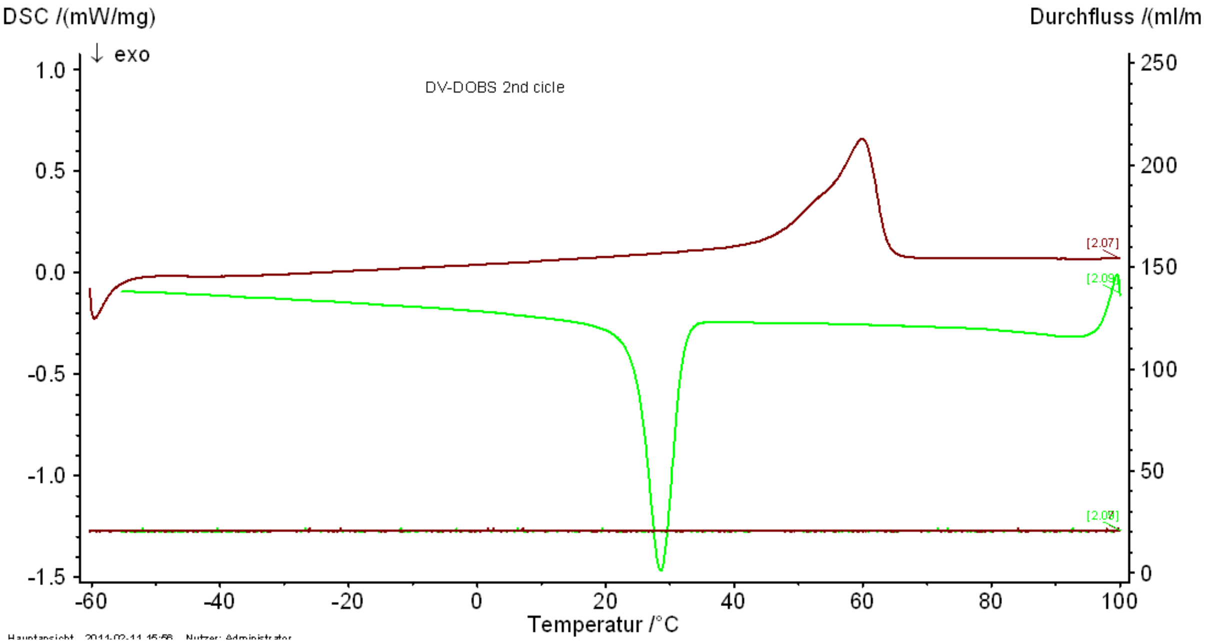


$MV^{6+}(DOBS)_6$

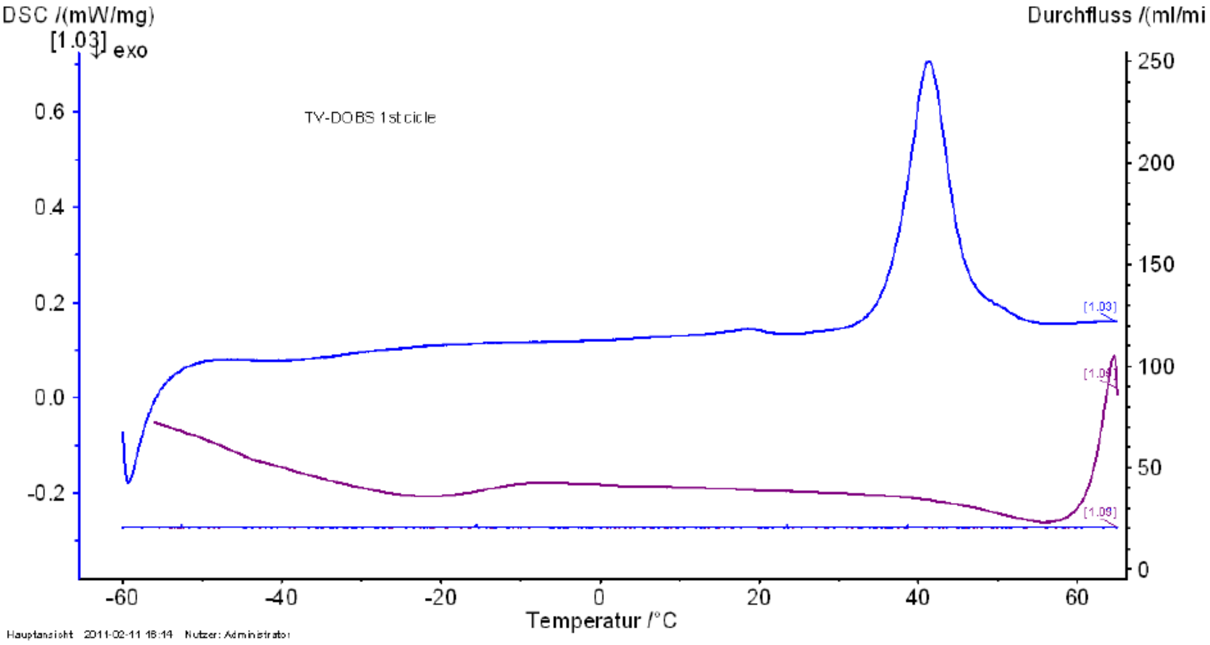


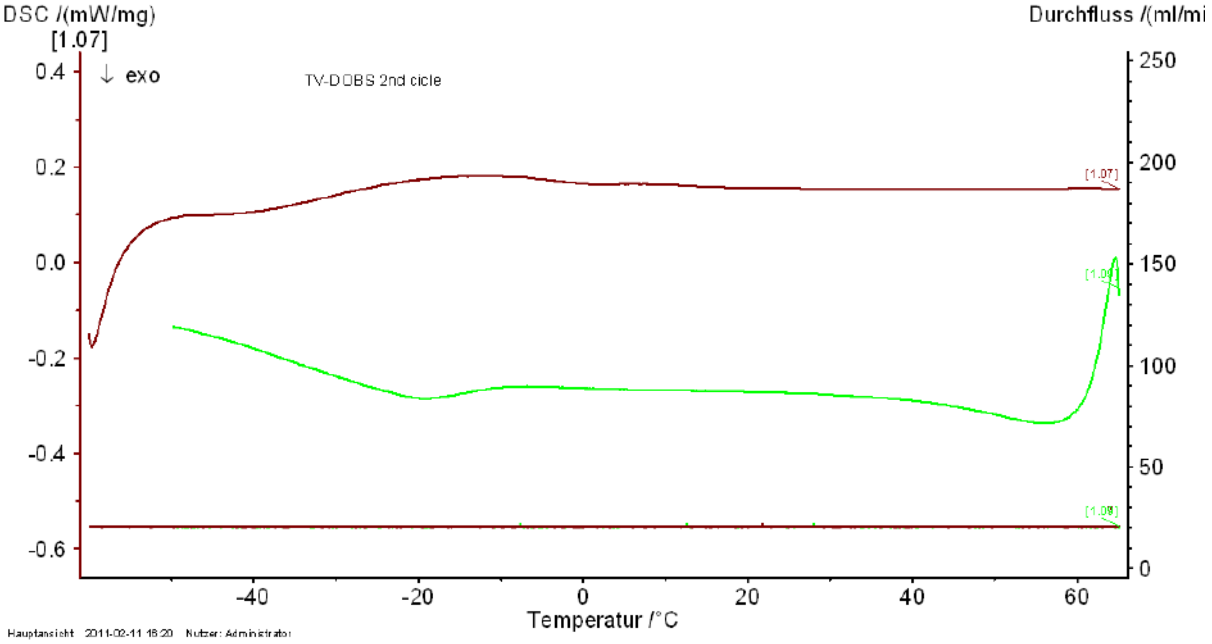
$MV^{2+}(DOBS)_2$



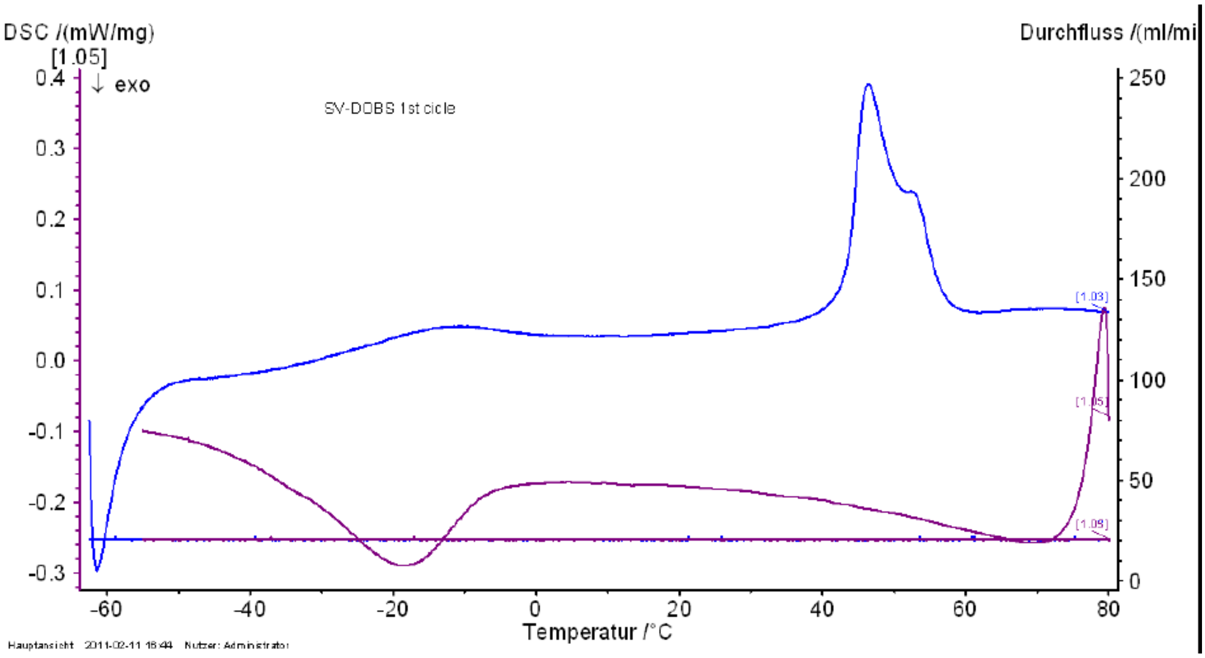

DSC /(mW/mg)
↓ exo
Durchfluss /(ml/m
DV-DOBS 2nd cicle
1.0
0.5
0.0
-0.5
-1.0
-1.5
250
200
150
100
50
0
[2.07]
[2.09]
[2.07]
-60
-40
-20
0
20
40
60
80
100
Temperatur /°C
Hauptansicht 2011-02-11 15:56 Nutzer: Administrator

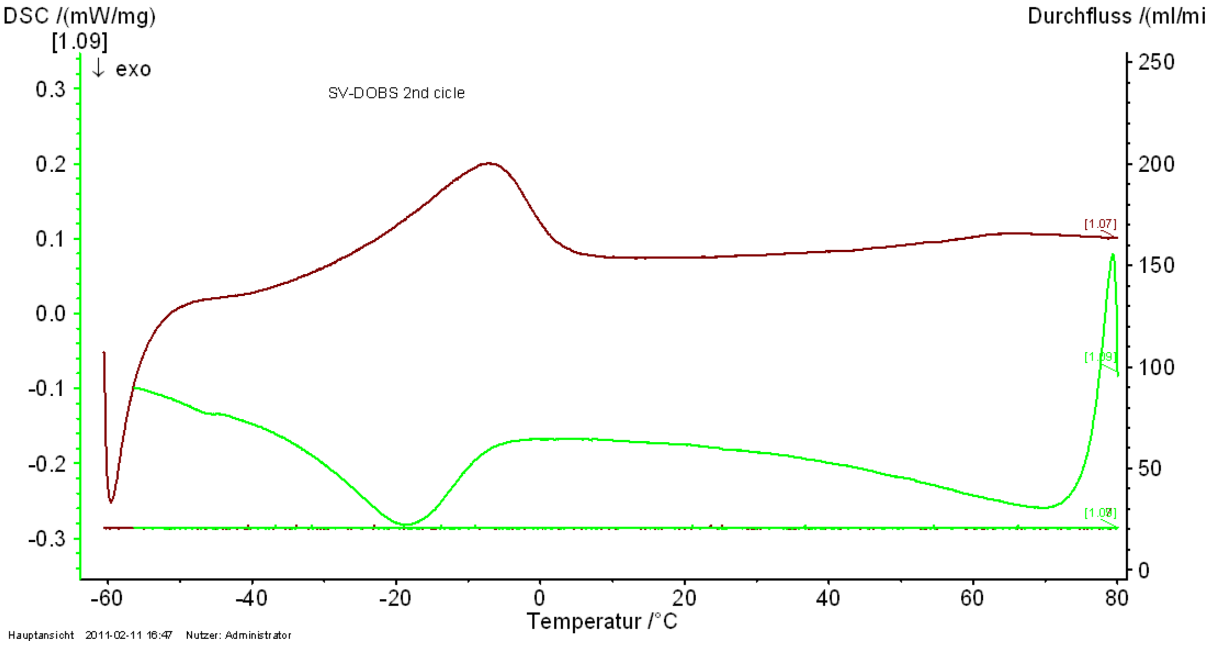
$V^{3+}(DOBS)_3$

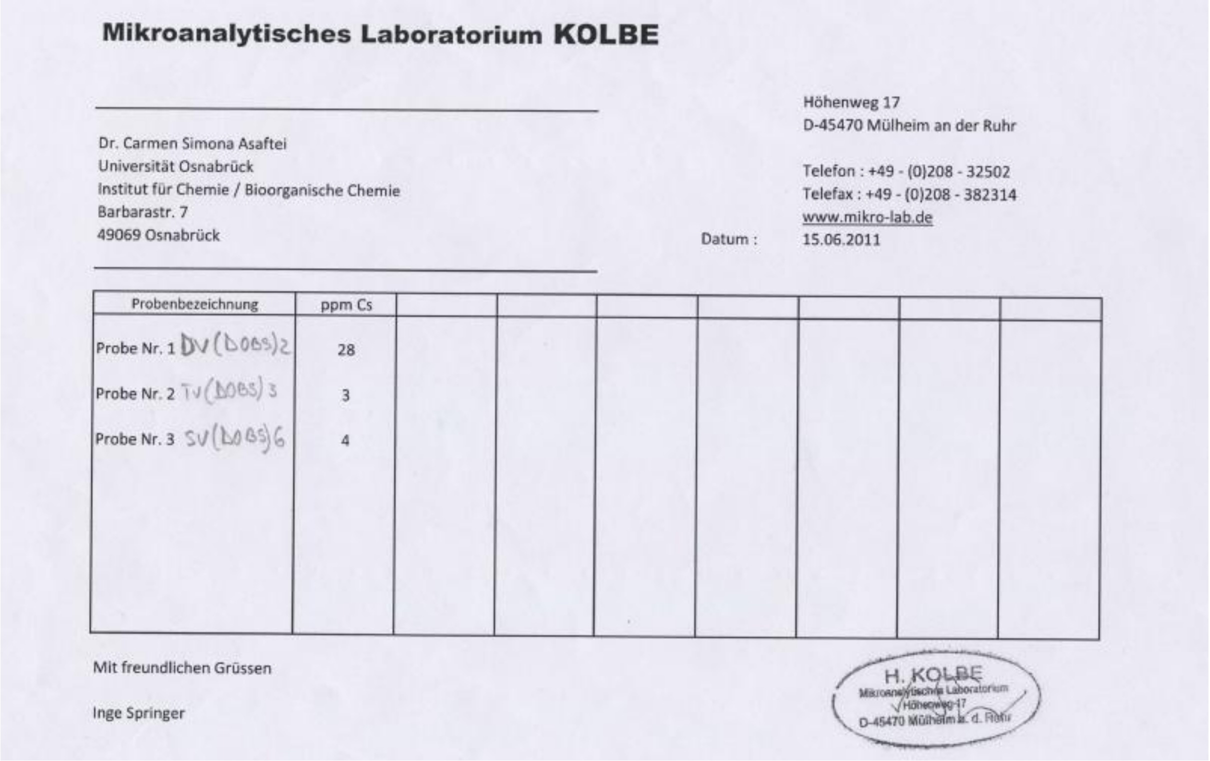

DSC /(mW/mg)
[1.03]
↓ exo
Durchfluss /(ml/mi
TV-DOBS 1st cicle
0.6
0.4
0.2
0.0
-0.2
250
200
150
100
50
0
[1.03]
[1.09]
[1.09]
-60
-40
-20
0
20
40
60
Temperatur /°C
Hauptansicht 2011-02-11 16:14 Nutzer: Administrator


DSC /(mW/mg)
[1.07]
↓ exo
TV-DOBS 2nd cicle
Durchfluss /(ml/mi
0.4
0.2
0.0
-0.2
-0.4
-0.6
250
200
150
100
50
0
[1.07]
[1.09]
[1.09]
-40
-20
0
20
40
60
Temperatur /°C
Hauptansicht 2011-02-11 16:20 Nutzer: Administrator

$MV^{6+}(DOBS)_6$


DSC /(mW/mg)
[1.05]
↓ exo
SV-DOBS 1st cicle
Durchfluss /(ml/mi
0.4
0.3
0.2
0.1
0.0
-0.1
-0.2
-0.3
250
200
150
100
50
0
[1.03]
[1.05]
[1.08]
-60
-40
-20
0
20
40
60
80
Temperatur /°C
Hauptansicht 2011-02-11 16:44 Nutzer: Administrator





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49069 Osnabrück

Datum : 15.06.2011

Probenbezeichnung	ppm Cs							
Probe Nr. 1 DV(DOBS)2	28							
Probe Nr. 2 TV(DOBS)3	3							
Probe Nr. 3 SV(DOBS)6	4							

Mit freundlichen Grüssen

Inge Springer

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