applied sciences

MDPI

# Fluorescence Spectroscopy of Enantiomeric Amide Compounds Enforced by Chiral Light

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## Experimental section

### Chemical synthesis

CDI (162 mg, 1 mmol) and 9-oxo-9*H*-fluorene-2-carboxylic acid (224 mg, 1 mmol) were suspended in 15 mL of ethyl acetate (AcOEt) and the reaction mixture was left under stirring at reflux for 4 hours. Then, (*R*)-1-phenylethanamine or (*S*)-1-phenylethanamine (d = 0.952 g/mL, 127 μL, 1 mmol) was added and the solution was refluxed overnight. After this time, the solvent was evaporated under reduced pressure and the residue was purified by silica gel column chromatography using initially a mixture of petroleum ether and ethyl acetate 7:3 ($R_f$ = 0.25) and subsequently 1:1 ($R_f$ = 0.63) to give a yellowish solid, that was crystallized from ethyl acetate. The compounds *R*-FA1 and *S*-FA1 were obtained with yields of ca. 65% on the basis of isolated products.

*R-FA1*

$^1$H-NMR (DMSO-$d_6$) δ (ppm): 9.04 (d, *J* = 7.6 Hz, 1H); 8.18 (s, 1H); 8.15-8.13 (m, 1H); 7.91-7.87 (m, 2H); 7.68-7.64 (m, 2H); 7.46-7.40 (m, 3H); 7.34 (t, *J* = 7.2 Hz, 2H); 7.25-7.22 (m, 1H); 5.22-5.15 (m, 1H); 1.50 (d, *J* = 6.8 Hz, 3H).

$^{13}$C-NMR (DMF-$d_7$) δ (ppm): 192.8, 164.8, 146.9, 145.3, 143.8, 136.1, 135.7, 134.9, 134.6, 134.0, 130.3, 128.6, 126.9, 126.5, 124.2, 122.7, 122.0, 121.2, 49.4, 22.0.

IR (ν cm$^{-1}$): 3320, 2971, 1720, 1627, 1615, 1531, 1455, 1377, 1363, 1319, 1264, 1212, 1191, 1132, 1039, 1016, 962, 856.

GC-MS, $R_t$ = 56.77 min, m/z (relative intensity): 327 (30%), 207 (100%), 179 (15%), 151 (30%).

$[\alpha]^{20}_D$ = –148.9 (*c* 0.11, DMF).

*S-FA1*

$^1$H-NMR (DMSO-$d_6$) δ (ppm): 9.04 (d, *J* = 7.6 Hz, 1H); 8.18 (s, 1H); 8.15-8.13 (m, 1H); 7.91-7.87 (m, 2H); 7.68-7.64 (m, 2H); 7.46-7.40 (m, 3H); 7.34 (t, *J* = 7.2 Hz, 2H); 7.25-7.22 (m, 1H); 5.22-5.15 (m, 1H); 1.50 (d, *J* = 6.8 Hz, 3H).

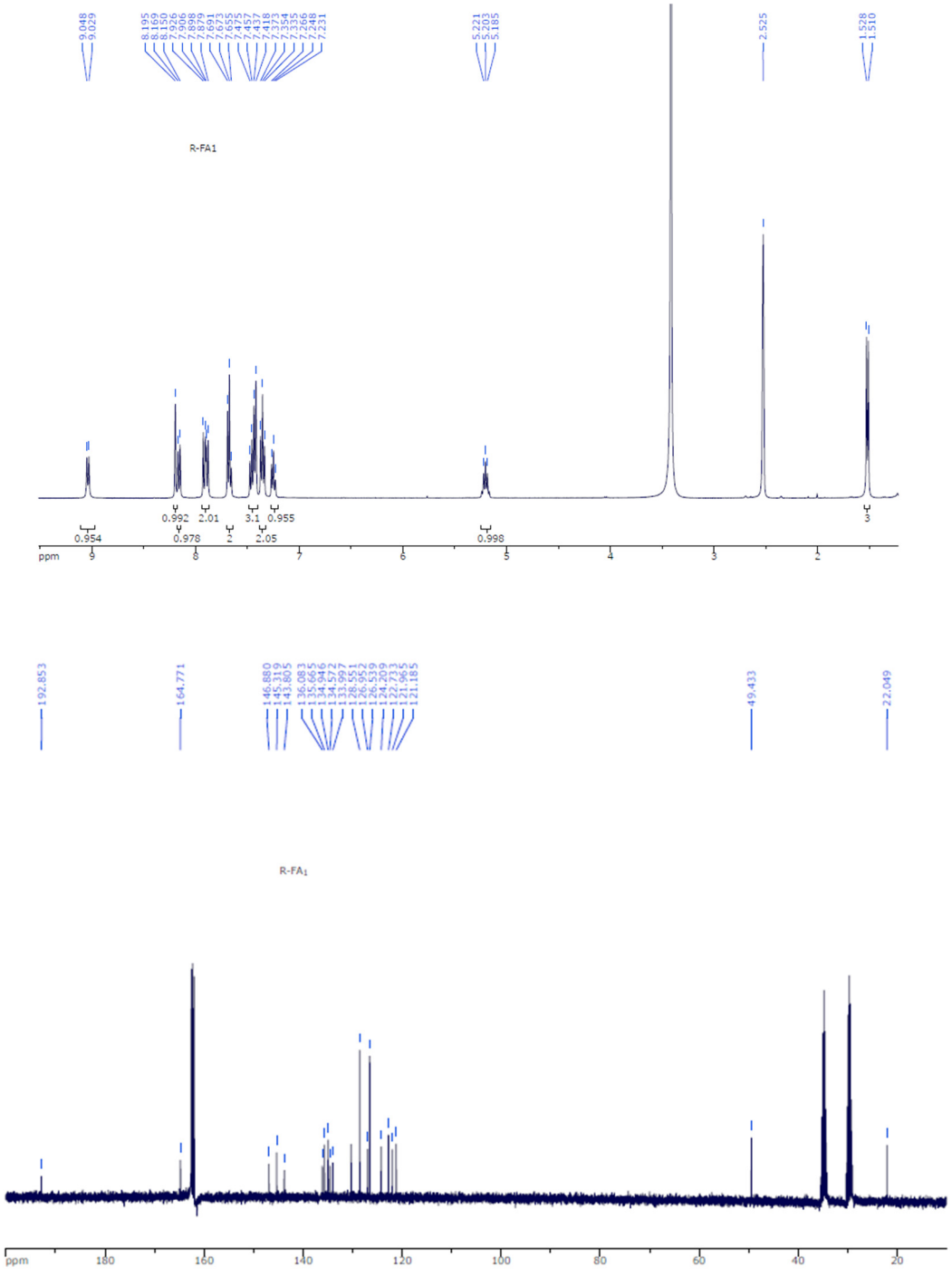
$^{13}$C-NMR (DMF-$d_7$) δ (ppm): 192.8, 164.8, 146.9, 145.3, 143.8, 136.1, 135.7, 134.9, 134.6, 134.0, 130.3, 128.6, 126.9, 126.5, 124.2, 122.7, 122.0, 121.2, 49.4, 22.0.

IR (ν cm$^{-1}$): 3320, 2971, 1720, 1627, 1615, 1532, 1455, 1377, 1363, 1319, 1264, 1212, 1191, 1132, 1039, 1015, 962, 856.

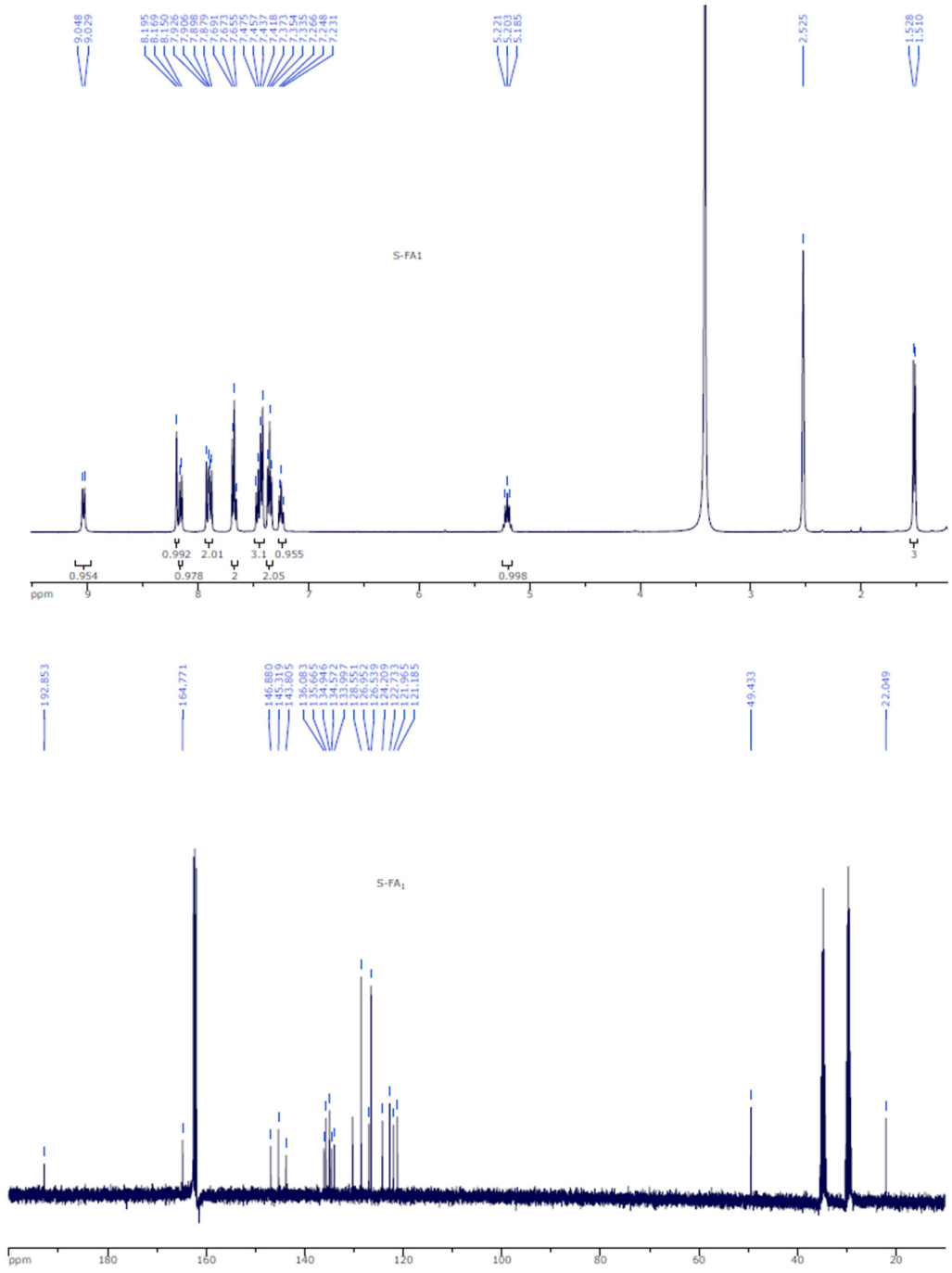
GC-MS, $R_t$ = 56.77 min, m/z (relative intensity): 327 (30%), 207 (100%), 179 (15%), 151 (30%).

$[\alpha]^{20}_D$ = +148.9 (*c* 0.09, DMF).

**1. H-NMR and $^{13}$C-NMR Spectra of R-FA1**




**$^{1}$H-NMR and $^{13}$C-NMR Spectra of S-FA1**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8.169
8.160
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7.691
7.673
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7.437
7.418
7.373
7.354
7.335
7.266
7.248
7.231
5.221
5.203
5.185
2.525
1.528
1.510
S-FA1
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0.978
0.992
2.01
2
3.1
2.05
0.955
0.998
3
ppm
9
8
7
6
5
4
3
2
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145.319
143.805
136.083
135.665
134.946
134.672
133.997
128.551
126.952
126.359
124.209
122.733
121.965
121.185
49.433
22.049
S-FA1
ppm
180
160
140
120
100
80
60
40
20