Supplementary Material (ESI) for Organic & Biomolecular Chemistry

General information

HPLS analysis were performed with Spherisorb-NH_2, 3 μm, 4.6 × 100 mm analytical column.
The purity of the compounds was determined by integration of peak areas on an HPLC chromatogram.

3,8-dibromoacenaphthylene (3)
HPLC (2% (i-Pr)$_2$NEt in hexane)
99.9 %, k′ = 2.87, $\lambda_{(max)}$= 347

7,10-diphenyl-8,9-diazafluoranthene (4a)
HPLC (A:B= 17:3)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2,5% MeOH + 2% (i-Pr)$_2$NEt in CH_2Cl_2
99.6 %, k′ = 5.43, $\lambda_{(max)}$= 368

1,6-dimethyl-7,10-diphenyl-8,9-diazafluoranthene (4b)
HPLC (A:B= 17:3)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2,5% MeOH + 2% (i-Pr)$_2$NEt in CH_2Cl_2
98.9 % , k′ = 7.82, $\lambda_{(max)}$= 380

7,10-bis(2-hydroxyphenyl)-8,9-diazafluoranthene (5a)
HPLC (A:B:C= 1:5:4)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2% (i-Pr)$_2$NEt in ethyl acetate
C = 2% (i-Pr)$_2$NEt in MeOH
99.0 % , k′ = 3.08, $\lambda_{(max)}$= 322

7,10-bis(2-hydroxyphenyl)-1,6-dimethyl-8,9-diazafluoranthene (5b)
HPLC (A:B:C= 1:5:4)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2% (i-Pr)$_2$NEt in ethyl acetate
C = 2% (i-Pr)$_2$NEt in MeOH
98.0 %, k′ = 2.71, $\lambda_{(max)}$= 336

7,10-bis(4-bromophenyl)-8,9-diazafluoranthene (6a)
HPLC (A:B:C= 5:10:0.5)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2% (i-Pr)$_2$NEt in acetone
C = 2% (i-Pr)$_2$NEt in MeOH
Purity – 97.2 % , k′ = 4.39, $\lambda_{(max)}$= 369

7,10-bis(4-bromophenyl)-1,6-dimethyl-8,9-diazafluoranthene (6b)
HPLC (A:B:C= 84:15:1)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2% (i-Pr)$_2$NEt in ethyl acetate
C = 2% (i-Pr)$_2$NEt in MeOH
98.3 %, k′ = 5.42, $\lambda_{(max)}$= 302

7,10-bis(3,5-dimethyl-1H-pyrazol-1-yl)-8,9-diazafluoranthene (7a)
HPLC (A:B = 4:1)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2% (i-Pr)$_2$NEt in ethyl acetate
99.4 %, k′ = 6.27, $\lambda_{(max)}$= 326

7,10-bis(3,5-dimethyl-1H-pyrazol-1-yl)-1,6-dimethyl-8,9-diazafluoranthene (7b)
HPLC (A:B:C= 89:10:1)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2% (i-Pr)$_2$NEt in acetone

C = 2% (i-Pr)$_2$NEt in MeOH
96.7 % , k′ = 6.98, $\lambda_{(max)}$= 380

7,10-di(thiophen-2-yl)-8,9-diazafluoranthene (8a)
HPLC (A:B= 17:3)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2,5% MeOH + 2% (i-Pr)$_2$NEt in CH_2Cl_2
99.0 %, k′ = 7.98, $\lambda_{(max)}$= 319

7,10-di(thiophen-2-yl)-1,6-dimethyl-8,9-diazafluoranthene (8b)
HPLC (A:B= 17:3)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2,5% MeOH + 2% (i-Pr)$_2$NEt in CH_2Cl_2
95.7 %, k′ = 7.69, $\lambda_{(max)}$= 325

7,10-di(furan-2-yl)-8,9-diazafluoranthene (9a)
HPLC (A:B:C= 79:20:1)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2% (i-Pr)$_2$NEt in ethyl acetate
C = 2% (i-Pr)$_2$NEt in MeOH
99.9 %, k′ = 8.01, $\lambda_{(max)}$= 334

7,10-di(furan-2-yl)-1,6-dimethyl-8,9-diazafluoranthene (9b)
HPLC (A:B:C= 79:20:1)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2% (i-Pr)$_2$NEt in ethyl acetate
C = 2% (i-Pr)$_2$NEt in MeOH
99.9 %, k′ = 6.91, $\lambda_{(max)}$= 323

7,10-di(pyridin-2-yl)-8,9-diazafluoranthene (10a)
HPLC (A:B:C= 29:10:1)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2,5% MeOH + 2% (i-Pr)$_2$NEt in CH_2Cl_2
C = 2% (i-Pr)$_2$NEt in MeOH
99.9 % , k′ = 2.75,.$\lambda_{(max)}$= 281

Dimethyl 8,9-diazafluoranthene-7,10-dicarboxylate (11a)
HPLC (A:B= 4:1)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2,5% MeOH + 2% (i-Pr)$_2$NEt in CH_2Cl_2
99.9 %, k′ = 4.69, $\lambda_{(max)}$= 299

Dimethyl-1,6-dimethyl-8,9-diazafluoranthene-7,10-dicarboxy-late (11b)
HPLC (A:B= 4:1)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2,5% MeOH + 2% (i-Pr)$_2$NEt in CH_2Cl_2
99.0 %, k′ = 5.67, $\lambda_{(max)}$= 366

Dimethyl-8,9-diazafluoranthene-7,10-dicarboxamide (12a)
Compound **12a** is soluble only in DMSO at 100°C

2,5-di-tert-butyl-8,9-diazafluoranthene-7,10-dicarboxamide (12b)
HPLC (A:B:C= 27:12:11)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2,5% MeOH + 2% (i-Pr)$_2$NEt in CH_2Cl_2
C = 2% (i-Pr)$_2$NEt in MeOH
99.2 %, k′ = 4.81, $\lambda_{(max)}$= 310

7,10-dimethyl-8,9-diazafluoranthene (13a)
HPLC (A:B = 4:1)
A = 2% (i-Pr)$_2$NEt in hexane
B = 2,5% MeOH + 2% (i-Pr)$_2$NEt in CH_2Cl_2

99.2 %, k′ = 7.9, $\lambda_{(max)}$= 349

1,6,7,10-tetramethyl-8,9-diazafluoranthene (13b)

HPLC (A:B = 4:1)
A = 2% (*i*-Pr)$_2$NEt in hexane
B = 2,5% MeOH + 2% (*i*-Pr)$_2$NEt in CH_2Cl_2
98.0 % , k′ = 7.07, $\lambda_{(max)}$= 366

7,10-bis(methylthio)-8,9-diazafluoranthene (14a)

HPLC (A:B = 99:1)
A = 2% (*i*-Pr)$_2$NEt in hexane
B = 2% (*i*-Pr)$_2$NEt in ethyl acetate
99.9 %, k′ = 5.63, $\lambda_{(max)}$= 327

P.S. Compound 14a was obtained in a higher yield (75%) by purification on column chromatography, using aluminium oxide and hexane /ethyl acetate (20:1→10:1) as a eluent. Reaction was carried out in *p*-xylene, reflux, 1 day.

1,6-dimethyl-7,10-bis(methylthio)-8,9-diazafluoranthene (14b)

HPLC (A:B = 99:1)
A = 2% (*i*-Pr)$_2$NEt in hexane
B = 2% (*i*-Pr)$_2$NEt in ethyl acetate
99.9 %, k′ = 6.97, $\lambda_{(max)}$= 347

7,10-bis(4-chlorobenzyl)-8,9-diazafluoranthene (15a)

HPLC (A:B:C= 79:20:1)
A = 2% (*i*-Pr)$_2$NEt in hexane
B = 2% (*i*-Pr)$_2$NEt in acetone
C = 2% (*i*-Pr)$_2$NEt in MeOH
99.6 %, k′ = 4.09, $\lambda_{(max)}$= 352

Dimethyl-3,4-dichloro-8,9-diazafluoranthene-7,10-dicarboxylate (18)

HPLC (A:B:C= 79:20:1)
A = 2% (*i*-Pr)$_2$NEt in hexane
B = 2% (*i*-Pr)$_2$NEt in ethyl acetate
C = 2% (*i*-Pr)$_2$NEt in MeOH
99.4 %, k′ = 7.92, $\lambda_{(max)}$= 373