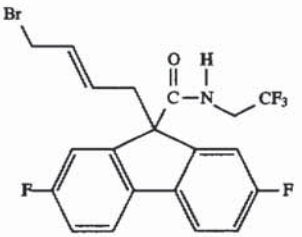
added dropwise n-BuLi (0.7 ml, 2.5M in hexanes), with a red colored solid falling from solution after all the base was added. After 10 min, Part A compound (325 mg, 0.864 mmol) was added and the reaction stirred an additional 2 h. The brown reaction mixture was quenched with sat. NH_4Cl and the aqueous layer was extracted twice with EtOAc, the combined organics dried over Na_2SO_4, and concentrated to a brown colored oil (400 mg). The residue was purified by flash column chromatography (SiO_2, 5 by 9.5 cm), eluting with 27 and 35% CH_3CN:CH_2Cl_2, then 4 and 10% iPrOH:CH_2Cl_2, to give title compound (184.5 mg, 46% yield) as a colorless solid. mp 93.5°–96° C.

MS: (CI, M+H$^+$) : m/z 501. Anal. Calc. for $C_{26}H_{41}N_2O_4P$: C, 67.18; H, 8.25; N, 5.60; P 6.19. Found: C, 67.24; H, 8.28; N, 5.61; P 5.83.

EXAMPLE 203

(E)-9-[4-(Dibutoxyphosphinyl)-2-butenyl]-2,7-difluoro-N-(2,2,2-trifluoroethyl)-9H-fluorene-9-carboxamide

A.



The Example 195 Part B carboxylic acid (465 mg, 1.23 mmol) was dissolved in 10 ml of dichloromethane and DMF (50 µl) was added. The mixture was cooled to 0° C. under an argon atmosphere and oxalyl chloride (165 mg, 1.3 mmol) was added and the mixture allowed to warm to ambient temperature and stir for 2.5 hrs. The mixture was evaporated several times from dichlormethane yielding the crude acid chloride as a pale yellow solid.

The acid chloride was dissolved in 5 ml of THF and cooled to 0° C. under an argon atmosphere. Triethylamine (142 mg, 1.4 mmol) was added followed by the addition of 2,2,2-trifluoroethylamine (139 mg, 1.4 mmol). The reaction was allowed to warm to ambient temperature and stir overnight. The reaction was quenched by adding sat. sodium bicarbonate and extracted with ethyl acetate (3×20 ml). The crude product was purified on a Merck EM silica column eluting with 10% ethyl acetate/hexane yielding 230 mg (38%) of title compound as a pale yellow solid, (Mass Spec, M+H=461).

B. (E)-9-[4-(Dibutoxyphosphinyl)-2-butenyl]-2,7-difluoro-N-(2,2,2-trifluoroethyl)-9H-fluorene-9-carboxamide

A solution of Part A compound (230 mg, 0.5 mmol) in tributyl phosphite (3 ml) was heated at 110° C. overnight. Excess tributyl phosphite was removed under vacuum at 100° C. and the oily residue was purified on a Merck EM silica column eluting with 3% isopropanol/dichloromethane yielding 186 mg (68%) of title compound as a colorless solid, m.p. 142°–144° C.

MS (CI, +ions) 574 (M+H). Anal Calc'd for $C_{28}H_{33}NF_5PO_4$+0.3H_2O: C, 58.63; H, 5.80; N, 2.44; F, 16.56; P, 5.40. Found: C, 58.91; H, 5.88; N, 2.47; F, 16.24; P, 5.50.

EXAMPLES 204–205

9-[4-[4-[[(2-Phenoxyphenyl)carbonyl]amino]phenyl]butyl]-N-propyl-9H-fluorene-9-carboxamide

To a solution of 2-phenoxybenzoic acid (Aldrich Chemical Co.) (111 mg, 0.518 mmol) and DMF (2 drops) in CH_2Cl_2 (1.5 mL) was added oxalyl chloride (389 µL, 2.0M in CH_2Cl_2, 0.777 mmol). The reaction bubbled for 10 min, then was stirred at RT under argon for 1.5 h. The reaction was concentrated in vacuo, and the resulting residue was dissolved in CH_2Cl_2 (1.5 mL) and added dropwise to a solution of Example 204 Part A compound (172 mg, 0.432 mmol) and triethylamine (90 µL, 0.648 mmol) in CH_2Cl_2 (1.5 mL) at 0° C. under argon. The reaction was stirred at 0° C. for 10 min, diluted with CH_2Cl_2 (20 mL), washed with saturated $NaHCO_3$ (5 mL) and brine (5 mL), then dried over Na_2SO_4. Evaporation gave a yellow oil, which was dissolved in a minimum amount of CH_2Cl_2 and purified by flash chromatography on silica gel (50 g) eluting with 30% EtOAc/hexane to provide title compound (211 mg, 82%) as a yellow gum.

MS (CI, +ions) m/z 595 (M+H). Anal. Calcd. for $C_{40}H_{38}N_2O_3$.0.4 CH_2Cl_2: C, 77.18; H, 6.22; N, 4.46. Found: C, 77.18; H, 6.20; N, 4.87.

EXAMPLES 206–208

9-[3-[4-(Benzoylamino)]phenyl]-N-propyl-9H-fluorene-9-carboxamide

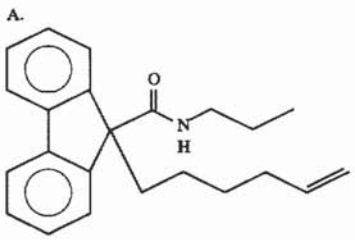
To a solution of Example 207 Part A compound (100 mg, 0.26 mmol) and triethylamine (0.04 mL, 0.39 mmol) in dichloromethane at 0° C. was added dropwise a solution of benzoyl chloride (0.04 mL, 0.31 mmol) in dichloromethane (1 mL). The reaction was stirred at 0° C. for 20 min. Ethyl acetate (50 mL) was added and the solution was washed with saturated sodium bicarbonate solution (2×30 mL), water (2×30 mL), brine (2×30 mL) and dried over $MgSO_4$. Purification was performed by flash chromatography on silica gel (50 g), loaded and eluted with 30% ethyl acetate in hexane. Pure fractions were combined and evaporated to give a solid. The resulting solid was recrystallized from ethyl acetate/hexane to give title compound (52 mg, 41%) as a white solid.

m.p. 187°–190° C. MS (CI, +ions) m/z 489 (M+H). Anal. Calc. for $C_{33}H_{32}N_2O_2$.1.0H_2O: C, 78.23; H, 6.76; N, 5.53. Found: C, 78.44; H, 6.54; N, 5.43.

EXAMPLES 209–210

9-[5-[(6-Ethoxy-2-benzothiazolyl)thio]pentyl]-N-propyl-9H-fluorene-9-carboxamide

A.

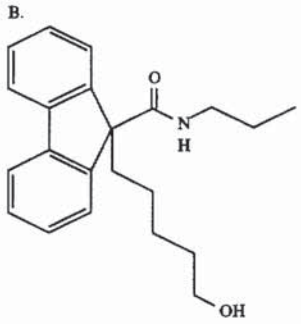


To a mixture of 3.0 g (11.95 mmol) of Example 11 Part C compound in 30 mL of THF, under argon at 0° C., was added 9.4 mL (23.90 mmol) of n-BuLi (2.5 M in hexanes)

dropwise. The dianion was stirred for 0.5 h at which time 1.9 mL (14.34 mmol) of 6-bromo-1-hexene (Aldrich) was added dropwise. The reaction gradually warmed to RT and was stirred for 6 days. The reaction was diluted with a 1:1 mixture of ethyl acetate/water and separated. The organics were washed with brine, dried (Na_2SO_4) and evaporated. Flash chromatography was performed on 200 g of silica gel eluting with 4:1 hexanes/ethyl acetate to provide 3.0 g (77%) of title compound as a pale yellow solid.

mp 54°–56° C. TLC Silica gel (4:1 hexanes/ethyl acetate) R_f=0.27. MS (CI-NH_3, +ions) m/e 334 (M+H). Anal. Calc. for $C_{23}H_{27}NO$: C, 82.84; H, 8.16; N, 4.20. Found: C, 82.90; H, 8.18; N, 4.59.

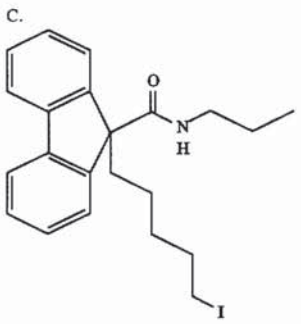
B.



To a solution of 2.0 g (6.00 mmol) of Part A compound in 20 mL of methanol, under nitrogen at −78° C., was bubbled O_3 for 0.5 h. The solution was purged with nitrogen and treated with 718 mg (18.89 mmol) of sodium borohydride (~5 pellets). The mixture was gradually warmed to room temperature and was stirred for 18 h, at which time the reaction was diluted with ether and quenched with NH_4Cl. The organics were washed with water, brine, dried (Na_2SO_4) and evaporated. Flash chromatography was performed on 200 g of silica gel eluting with 1:1 hexanes/ethyl acetate to provide 1.6 g (80%) of title compound as a colorless oil.

TLC Silica gel (1:1 hexanes/ethyl acetate) R_f=0.13. Anal. Calcd. for $C_{22}H_{27}NO_2$+0.40 mol H_2O+0.15 mol CH_2Cl_2. C, 74.44; H, 7.92; N, 3.92. Found: C, 74.50; H, 7.62; N, 3.73.

C.



To a solution of 1.4 g (4.15 mmol) of Part B compound in 20 mL of THF, under argon at 0° C., was added 620 mg (9.13 mmol) of imidazole and 1.4 g (5.40 mmol) of triphenylphosphine. This mixture was stirred at 0° C. for 0.5 h, at which time 1.4 g (5.40 mmol) of iodine in 10 mL of THF was added dropwise. The reaction was stirred for 1.5 h, at 0° C., at which time it was diluted with hexanes and washed with sodium bisulfite, $NaHCO_3$, brine, dried (Na_2SO_4) and evaporated. Flash chromatography was performed on 50 g of silica gel eluting with 1:1 hexanes/ethyl acetate to provide 1.57 g (84%) of title compound as a white solid.

TLC: Silica gel (1:1 hexanes/ethyl acetate). R_f=0.63. MS (ES, +ions) m/e 448 (M+H).

D. 9-[5-[(6-Ethoxy-2-benzothiazolyl)thio]pentyl]-N-propyl-9H-fluorene-9-carboxamide

To a solution of 200 mg (0.45 mmol) of Part C compound in 5 mL of DMF, under argon at RT, was added 125 mg (0.90 mmol) of K_2CO_3 followed by 114 mg (0.54 mmol) of 6-ethoxy-2-mercaptobenzothiazole. The reaction was stirred for 18 h at which time it was diluted with ether and the organics were washed with water, brine, dried (Na_2SO_4) and evaporated. Flash chromatography was performed on 50 g of silica gel eluting with 95:5 dichloromethane/isopropanol to provide 120 mg (50%) of title compound as a biege solid.

mp 67°–70° C. TLC Silica gel (95:5 dichloromethane/isopropanol). R_f=0.35. MS (CI-NH_3, +ions) m/e 531 (M+H). Anal. Calcd. for $C_{31}H_{34}N_2O_2S_2$: C, 70.15; H, 6.46; N, 5.28; S, 12.08. Found: C, 69.95; H, 6.20; N, 5.22; S, 12.11.

EXAMPLE 211

9-[4-[4-(Benzoylamino)phenyl]butyl]-N-propyl-9H-fluorene-9-carboxamide

Benzoyl chloride (156 μL, 1.35 mmol) was added dropwise to a solution of Example 207 Part A compound (490 mg, 1.23 mmol) and triethylamine (257 μL, 1.85 mmol) in CH_2Cl_2 (4 mL) at 0° C. under argon. The reaction was stirred at 0° C. for 30 min, diluted with CH_2Cl_2 (20 mL) and $CHCl_3$ (20 mL), washed with 1N KOH (2×10 mL) and water (10 mL), then dried over $MgSO_4$. Evaporation gave a yellow solid, which was adsorbed onto silica gel (10 g), then purified by flash chromatography on silica gel (150 g) eluting with 5% EtOAc/CH_2Cl_2 to give a solid. The product was dried under high vacuum at 50° C. overnight to provide title compound (412 mg, 67%) as a white solid.

mp 171°–173° C. Anal. Calcd. for $C_{34}H_{34}N_2O_2$.0.4H_2O: C, 81.24; H, 6.82; N, 5.57. Found: C, 80.88; H, 6.83; N, 5.33.

EXAMPLE 212

9-[5-(Dibutoxyphosphinyl)pentyl]-N-propyl-9H-fluorene-9-carboxamide

To 400 mg (0.89 mmol) of Example 209 Part A compound, under argon, was added 1.2 mL (4.45 mmol) of tributylphosphite (neat). The mixture was heated to 120° C. for 18 h and bulb to bulb distilled (5 mm, 100° C.) to remove lower boiling impurities and provide a pale yellow oil. Flash chromatography was performed on 75 g of silica gel eluting with 95:5 dichloromethane/isopropanol to provide 440 mg (96%) of title compound as a pale yellow oil.

TLC Silica gel (95:5 dichloromethane/isopropanol) R_f=0.29. IR 3434, 2959, 2934, 2872, 1665, 1508, 1449, 1244, 1024, 978, 743 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) is consistent with the indicated compound. MS (CI-NH_3, +ions) m/e 514 (M+H). Anal. Calcd. for $C_{30}H_{44}NO_4P$: C, 70.15; H, 8.63; P, 6.03. Found: C, 70.60; H, 8.80; P, 5.86. ^{13}C NMR (75 MHz, $CDCl_3$) is consistent with the indicated compound.

The following compounds were prepared employing procedures as described hereinbefore.

EXAMPLE 213

N,N-Diethyl-9-(2-propenyl)-9H-fluorene-9-carboxamide

MS (CI, M+H)$^+$ m/z 306 Anal. Calcd for $C_{21}H_{23}NO$.0.14H_2O: C, 81.90; H, 7.62; N, 4.55. Found: C, 82.11; H, 7.52; N, 4.34. mp 84°–86° C.

EXAMPLE 214

N-Ethyl-9-propyl-9H-fluorene-9-carboxamide

MS (CI, M+H)$^+$ m/z 280. Anal. Calcd for $C_{19}H_{21}NO$: C, 81.68; H, 7.58; N, 5.01. Found: C, 81.45; H, 7.77; N, 5.06. mp 96°–97.5° C.

EXAMPLE 215

N-Ethyl-9-(2-propenyl)-9H-xanthene-9-carboxamide

MS (CI-NH_3, +ions) m/e 311 (M+NH_4), 294 (M+H). Anal. Calcd for $C_{19}H_{19}O_2N$: C, 77.79; H, 6.53; N, 4.77. Found: C, 77.87; H, 6.57; N, 4.77. mp 111°–112° C.

EXAMPLE 216

N-Ethyl-9-(3-phenylpropyl)-9H-xanthene-9-carboxamide

MS (CI-NH_3, +ions) m/e 372 (M+H). Anal. Calcd for $C_{25}H_{25}NO_2$: C, 80.83; H, 6.78; N, 3.77. Found: C, 80.77; H, 6.88; N, 3.83. mp 130° C.

EXAMPLES 217–218

9-Hexyl-N-propyl-9H-xanthene-9-carboxamide

MS (CI-NH_3, +ions) m/e 352 (M+H). Anal. Calcd for $C_{23}H_{29}NO_2$: C, 78.60; H, 8.32; N, 3.98. Found: C, 78.64; H, 8.46; N, 3.96. mp 76°–77.5° C.

EXAMPLE 219

N-Methoxy-N-methyl-9-propyl-9H-fluorene-9-carboxamide

CI-Mass Spec. (M+H)=296. Anal. Calcd for $C_{19}H_{21}NO_2$: C, 77.26; H, 7.17; N, 4.74. Found: C, 77.12; H, 7.04; N, 4.68. mp 73.75° C.

EXAMPLE 220

10,11-Dihydro-5-(3-phenyl-2-propenyl)-N-propyl-5H-dibenzo[a,d]cycloheptene-5-carboxamide

MS (CI-NH_3, +ions) m/e 396 (M+H). Anal. Calcd for $C_{28}H_{29}NO$: C, 85.02; H, 7.39; N, 3.54. Found: C, 84.66; H, 7.46; N, 3.46. mp 159° C.

EXAMPLE 221

N-Methyl-9-propyl-9H-fluorene-9-carboxamide

CI-Mass Spec. (M+H)=266. Anal. Calcd for $C_{18}H_{19}NO$+0.12H_2O: C, 80.82; H, 7.25; N, 5.24. Found: C, 80.90; H, 7.26; N, 5.16. mp 145°–146° C.

EXAMPLE 222

1-(9-Propyl-9H-fluoren-9-yl)-1-pentanone

CI-Mass Spec. (M+H)=293. Anal. Calcd for $C_{21}H_{24}O$: C, 86.20; H, 8.24. Found: C, 85.86; H, 8.14. mp 56°–58° C.

EXAMPLE 223

α-Butyl-9-propyl-9H-fluorene-9-methanol

CI-Mass Spec. (M+NH_4)=312$^+$. Anal. Calcd for $C_{21}H_{26}O$+0.12H_2O: C, 85.05; H, 8.92. Found: C, 85.05; H, 8.87. mp 88°–90° C.

EXAMPLE 224

1-(9-Propyl-9H-fluoren-9-yl)-1-butanone

CI-Mass Spec. (M+H)=279. Anal. Calcd for $C_{20}H_{22}O$+0.1H_2O: C, 85.79; H, 7.98. Found: C, 85.79; H, 8.15. mp 65°–67° C.

EXAMPLE 225

α,9-Dipropyl-9H-fluorene-9-methanol

CI-Mass Spec. (M+NH_3)=298. Anal. Calcd for $C_{20}H_{24}O$+0.1H_2O: C, 85.15; H, 8.64. Found: C, 85.15; H, 8.72. mp 83°–85° C.

EXAMPLE226

10,11-Dihydro-5-(2-propenyl)-N-propyl-5H-dibenzo-[a,d]cycloheptene-5-carboxamide

MS (CI-NH_3, +ions) m/e 320 (M+H). Anal. Calcd for $C_{22}H_{25}NO$: C, 81.98; H, 7.92; N, 4.35. Found: C, 82.01; H, 7.91; N, 4.32. mp 76°–79° C.

EXAMPLE 227

9-(3-Phenylpropyl)-N-propyl-9H-thioxanthene-9-carboxamide

MS (CI-NH_3, +ions) m/e 402 (M+H). Anal. Calcd for $C_{26}H_{27}NOS$: C, 77.77; H, 6.78; N, 3.49. Found: C, 77.60; H, 6.83; N, 3.42. mp 130°–131° C.

EXAMPLE 228

N,9-Dipropyl-9H-thioxanthene-9-carboxamide

MS (CI-NH_3, +ions) m/e 326 (M+H). Anal. Calcd for $C_{20}H_{23}NOS$: C, 73.81; H, 7.12; N, 4.30. Found: C, 73.84; H, 7.36; N, 4.24. mp 132°–133° C.

EXAMPLE 229

10,11-Dihydro-5-(3-phenylpropyl)-N-propyl-5H-dibenzo-[a,d]cycloheptane-5-carboxamide

MS (CI, NH_3, +ions) m/z 398 (M+H). Anal. Calcd for $C_{28}H_{31}NO$+0.4H_2O: C, 82.90; H, 7.93; N, 3.45. Found: C, 82.99; H, 7.95; N, 3.36. mp 109°–112° C.

EXAMPLE 230

(E)-2,7-Difluoro-9-(3-phenyl-2-propenyl)-N-propyl-9H-fluorene-9-carboxamide

MS (CI, M+H)$^+$ m/z 404. Anal. Calcd for $C_{26}H_{23}NF_2O$: C, 77.40; H, 5.75; N, 3.47. Found: C, 77.32; H, 5.70; N, 3.33. mp 124°–126° C.

EXAMPLE 231

9-(3-Phenylpropyl)-N-(2-pyridinylmethyl)-9H-fluorene-9-carboxamide

CI-Mass Spec. (M+H)=419. Anal. Calcd for $C_{29}H_{26}N_2O$: C, 83.22; H, 6.26; N, 6.70. Found: C, 83.42; H, 6.31; N, 6.62. mp 115°–116° C.

EXAMPLE 232

2,7-Difluoro-9-(3-phenylpropyl)-N-propyl-9H-fluorene-9-carboxamide

MS (CI, M+H)$^+$ m/z 406. Anal. Calcd for $C_{26}H_{25}F_2NO$.0.12H_2O: C, 76.62; H, 6.24; N, 3.44; F, 9.32. Found: C, 76.64; H, 6.33; N, 3.42; F, 9.12. Mp 99°–100.5° C.

EXAMPLE 233

2,7-Difluoro-9-(3-phenylpropyl)-N-(4-pyridinylmethyl)-9H-fluorene-9-carboxamide

MS (electrospray, M+H)$^+$ m/z 455$^+$. Anal. Calcd for $C_{29}H_{24}N_2F_2O.0.25H_2O$: C, 75.88; H, 5.38; N, 6.10. Found: C, 75.93; H, 5.15; N, 6.04. mp 60°–62° C.

EXAMPLE 234

9-(Butylthio)-9-propyl-9H-fluorene

MS (CI-NH_3, +ions) m/e 297 (M+H), 207 (M+H—$C_4H_{10}S$). Anal. Calcd for $C_{20}H_{24}S$: C, 81.03; H, 8.16; N, 10.81. Found: C, 81.40; H, 8.47; N, 10.85.

EXAMPLE 235

9-(Butylsulfinyl)-9-propyl-9H-fluorene

MS (ES, +ions) m/e 625 (2M+H), 313 (M+H). Anal. Calcd for $C_{20}H_{24}SO$: C, 76.88; H, 7.74; N, 10.26. Found: C, 77.12; H, 7.78; N, 9.93. mp 57°–59° C.

EXAMPLE 236

9-(4-Hydroxybutyl)-N-propyl-9H-fluorene-9-carboxamide

MS (CI-NH_3, +ions) m/e 324 (M+H). Anal. Calcd for $C_{21}H_{25}NO_2$: C, 77.99; H, 7.79; N, 4.33. Found: C, 77.89; H, 7.92; N, 4.35. mp 73°–75° C.

EXAMPLE 237

9-[4-(Phenylthio)butyl]-N-propyl-9H-fluorene-9-carboxamide

MS (CI-NH_3, +ions) m/e 416 (M+H). Anal. Calcd for $C_{27}H_{29}NOS$: C, 78.03; H, 7.03; N, 3.37; S, 7.71. Found: C, 77.70; H, 7.26; N, 3.35; S, 7.51. mp 50°–53° C.

EXAMPLE 238–240

cis-N,9-Dipropyl-1H-thioxanthene-9-carboxamide, 10-oxide

MS (CI-NH_3, +ions) m/e 342 (M+H). Anal. Calcd for $C_{20}H_{23}NO_2S$: C, 70.35; H, 6.79; N, 4.10. Found: C, 70.25; H, 6.86; N, 4.10. mp 201°–204° C.

EXAMPLE 241

5-(2-Propenyl)-N-propyl-5H-indeno[1,2-b]pyridine-5-carboxamide

MS (CI, M+H)$^+$ m/z 293$^+$. Anal. Calcd for $C_{19}H_{20}N_2O.0.1H_2O$: C, 77.58; H, 6.92; N, 9.52. Found: C, 77.50; H, 6.84; N, 9.57. mp 131°–133.5° C.

EXAMPLE 242

(E)-5-(3-Phenyl-2-propenyl)-N-propyl-5H-indeno[1,2-b]pyridine-5-carboxamide

mp 153–154.5. MS (CI, M+H)$^+$ m/z 369$^+$. Anal. Calcd for $C_{25}H_{24}N_2O$: C, 80.32; H, 6.63; N, 7.49. Found: C, 80.26; H, 6.51; N, 7.55.

EXAMPLE 243

N-Ethyl-N-methyl-9-(2-propenyl)-9H-fluorene-9-carboxamide

MS (CI, M+H)$^+$ m/z 292. Anal. Calcd for $C_{20}H_{21}NO.0.06$ dioxane: C, 81.94; H, 7.30; N, 4.72. Found: C, 81.76; H, 7.39; N, 4.68.

EXAMPLE 244

N,9-Dipropyl-9H-thioxanthene-9-carboxamide, 10,10-dioxide

MS (CI-NH_3, +ions) m/z 380 (M+Na) 375 (M+NH_4), 358 (M+H). Anal. Calcd for $C_{20}H_{23}NO_3S+0.6CH_2Cl_2$: C, 60.58; H, 5.97; N, 3.43. Found: C, 60.58; H, 5.79; N, 3.39. mp 264°–266° C.

EXAMPLE 245

trans-N,9-Dipropyl-9H-thioxanthene-9-carboxamide, 10-oxide

MS (CI-NH_3, +ions) m/z 342 (M+H). Anal. Calcd for $C_{20}H_{23}NO_2S+0.4H_2O$: C, 68.92; H, 6.88; N, 4.02. Found: C, 68.96; H, 7.18; N, 3.98. mp 147°–150° C.

EXAMPLE 246

9-[3-(Dibutoxyphosphinyl)propyl]-N-(2-pyridinylmethyl)-9H-fluorene-9-carboxamide

CI-Mass Spec. (M+H)=535. Anal. Calcd for $C_{31}H_{39}N_2PO_4.0.5H_2O$: C, 68.48; H, 7.42; N, 5.15; P, 5.70. Found: C, 68.28; H, 7.23; N, 5.28; P, 5.50.

EXAMPLE 247

1-(9-Propyl-9H-fluorene-9-yl)-2-(1-piperidinyl) ethanone, monohydrochloride

MS (ES) 334 (M+H). Anal. Calcd for $C_{23}H_{28}ClNO.H_2O$: C, 71.21; H, 7.79; N, 3.61. Found: C, 71.01; H, 7.75; N, 3.93.

EXAMPLE 248

N-(5-Hydroxypentyl)-9-propyl-9H-fluorene-9-carboxamide

MS (CI, +ions) m/z 338 (M+H). Anal. Calcd for $C_{22}H_{27}NO_2+0.3H_2O$: C, 77.13; H, 8.11; N, 4.09/ Found: C, 77.10; H, 8.23; N, 4.00. mp 48.51° C.

EXAMPLE 249

9-(3-Cyanopropyl)-N-propyl-9H-fluorene-9-carboxamide

MS (ES, +ions) m/z 319 (M+H) Anal. Calcd for $C_{21}H_{22}N_2O$: C, 79.21; H, 6.96; N, 8.80. Found: C, 78.98; H, 6.89; N, 8.68. mp 80°–83° C.

EXAMPLE 250

N-[[4-[[(9-Propyl-9H-fluoren-9-yl)carbonyl]amino]-phenyl]methyl]-9-propyl-9H-fluorene-9-carboxamide

MS (CI, +ions) 591 (M+H). Anal. Calcd for $C_{41}H_{38}N_2O_2.0.3H_2O$: C, 82.60; H, 6.53; N, 4.70. Found: C, 82.62; H, 6.44; N, 4.64. mp 188°–190° C.

EXAMPLE 251

N-[4-(4-Aminophenyl)methyl]-9-propyl-9H-fluorene-9-carboxamide

MS (ES, +ions) 357 (M+H). Anal. Calcd for $C_{24}H_{24}N_2O.0.7H_2O$: C, 78.10; H, 6.94; N, 7.59. Found: C, 78.26; H, 6.70; N, 7.48. mp 96°–99° C.

EXAMPLE 252

9-[3-(Dibutoxyphosphinyl)propyl]-N-propyl-9H-fluorene-9-carboxamide

MS (CI-NH_3, +ions) m/e 486 (M+H). Anal. Calcd for $C_{28}H_{40}NO_4P$+0.75 mol H_2O: C, 67.37; H, 8.38; N, 2.81; P, 6.21. Found: C, 67.49; H, 8.28; N, 2.69; P, 6.45.

EXAMPLES 253–254

N-Methyl-9-(3-phenylpropyl)-9H-fluorene-9-carboxamide

MS (CI, +ions) m/z 342 (M+H). Anal. Calcd for $C_{24}H_{23}NO$+0.2H_2O: C, 83.51; H, 6.84; N, 4.06. Found: C, 83.55; H, 6.69; N, 4.02. mp 101°–102° C.

EXAMPLE 255

2-(Dimethylamino)-9-(3-phenylpropyl)-N-propyl-9H-fluorene-9-carboxamide

MS (CI, M+H)$^+$ m/z 413$^+$. Anal. Calcd for $C_{28}H_{32}N_2O$.0.34H_2O: C, 80.32; H, 7.87; N, 6.69. Found: C, 80.30; H, 7.74; N, 6.71.

EXAMPLE 256

9-[4-(Dibutoxyphosphinyl)-2-butenyl]-N-propyl-9H-fluorene-9-carboxamide

MS (ES) 498 (M+H). Anal. Calcd for $C_{29}H_{40}NO_4P$: C, 70.00; H, 8.10; N, 2.81; P, 6.22. Found: C, 69.85; H, 8.15; N, 3.13; P, 6.19.

EXAMPLE 257

9-[4-(4-Nitrophenyl)butyl]-N-propyl-9H-fluorene-9-carboxamide

MS (ES) 429 (M+H). Anal. Calcd for $C_{27}H_{28}N_2O_3$: C, 75.68; H, 6.59; N, 6.54. Found: C, 75.70; H, 6.58; N, 6.57. mp 109°–110° C.

EXAMPLE 258

9-[3-(4-Nitrophenyl)-2-propenyl]-N-propyl-9H-fluorene-9-carboxamide

MS (CI, +ions) 413 (M+H). Anal. Calcd for $C_{26}H_{24}N_2O_3$.0.3H_2O: C, 74.73; H, 5.93; N, 6.70. Found: C, 74.54; H, 5.75; N, 6.67. mp 143°–146° C.

EXAMPLE 259

5-(3-Phenylpropyl)-N-propyl-5H-indeno[1,2-b]pyridine-5-carboxamide

MS (CI, M+H)$^+$ m/z 371$^+$. Anal. Calcd for $C_{25}H_{26}N_2O$: C, 81.05; H, 7.07; N, 7.56. Found: C, 80.97; H, 7.12; N, 7.51. mp 124.5°–126° C.

EXAMPLE 260

9-[4-(4-Aminophenyl)butyl]-N-propyl-9H-fluorene-9-carboxamide

MS (CI) 399 (M+H). Anal. Calcd for $C_{27}H_{30}N_2O$.0.3H_2O: C, 80.28; H, 7.64; N, 6.93. Found: C, 80.37; H, 7.53; N, 7.34.

EXAMPLE 261

9-[3-(4-Aminophenyl)propyl]-N-propyl-9H-fluorene-9-carboxamide

MS (CI, +ions) 385 (M+H). Anal. Calcd for $C_{26}H_{28}N_2O$.0.3H_2O: C, 80.09; H, 7.39; N, 7.18. Found: C, 80.01; H, 7.31; N, 7.17. mp 138°–140° C.

EXAMPLE 262

9-[4-(Dibutoxyphosphinyl)butyl]-9H-fluorene-9-carboxylic acid, methyl ester

MS (CI, +ions) m/z 473 (M+H). Anal. Calcd for $C_{27}H_{37}O_5P$: C, 68.63; H, 7.89; N, 6.55. Found: C, 68.37; H, 7.96; N, 6.21.

EXAMPLE 263

N,N-Dibutyl-9-[(propylamino)carbonyl]-9H-fluorene-9-butanamide

MS (CI-NH_3, +ions) m/e 449 (M+H). Anal. Calcd for $C_{29}H_{40}N_2O_2$+0.29 mol H_2O: C, 76.75; H, 9.01; N, 6.17. Found: C, 76.71; H, 8.92; N, 6.21. mp 109°–111° C.

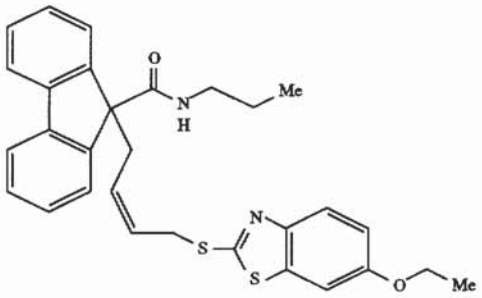
EXAMPLE 264

9-(5-Cyanopentyl)-N-propyl-9H-fluorene-9-carboxamide

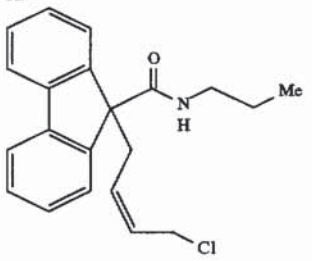
MS (ES, +ions) m/e 347 (M+H). Anal. Calcd for $C_{23}H_{26}N_2O$: C, 79.73; H, 7.56; N, 8.09. Found: C, 79.25; H, 7.55; N, 7.76. mp 92°–94° C.

EXAMPLES 265–266

(Z)-9-[4-[(6-Ethoxy-2-benzothiazolyl)thio]-2-butenyl]-N-propyl-9H-fluorene-9-carboxamide



A.



Butyllithium (8.4 mL, 2.5M in hexane, 21 mmol) was added dropwise over 10 min to a solution of 9-fluorenecarboxylic acid (2.10 g, 10 mmol) in THF (50 mL) at 0° C. under argon. During addition of the first equivalent of BuLi, the reaction became thick with a white precipitate which became yellow and cleared after addition of the second equivalent. The reaction was stirred at 0° C. for 20 min, then cis-1,4-dichloro-2-butene (1.2 mL, 11 mmol) was added dropwise over 5 min. The reaction lightened in color during addition and was stirred at 0° C. for 3 h, then poured into 1N HCl (50 mL) and extracted with CH_2Cl_2 (3×50 mL). The combined organic layers were washed with brine (30 mL) then dried over $MgSO_4$. Evaporation provided 3.5 g of

DOCKET ALARM

Explore Litigation Insights

Docket Alarm provides insights to develop a more informed litigation strategy and the peace of mind of knowing you're on top of things.

Real-Time Litigation Alerts

Keep your litigation team up-to-date with **real-time alerts** and advanced team management tools built for the enterprise, all while greatly reducing PACER spend.

Our comprehensive service means we can handle Federal, State, and Administrative courts across the country.

Advanced Docket Research

With over 230 million records, Docket Alarm's cloud-native docket research platform finds what other services can't. Coverage includes Federal, State, plus PTAB, TTAB, ITC and NLRB decisions, all in one place.

Identify arguments that have been successful in the past with full text, pinpoint searching. Link to case law cited within any court document via Fastcase.

Analytics At Your Fingertips

Learn what happened the last time a particular judge, opposing counsel or company faced cases similar to yours.

Advanced out-of-the-box PTAB and TTAB analytics are always at your fingertips.

API

Docket Alarm offers a powerful API (application programming interface) to developers that want to integrate case filings into their apps.

LAW FIRMS
Build custom dashboards for your attorneys and clients with live data direct from the court.

Automate many repetitive legal tasks like conflict checks, document management, and marketing.

FINANCIAL INSTITUTIONS
Litigation and bankruptcy checks for companies and debtors.

E-DISCOVERY AND LEGAL VENDORS
Sync your system to PACER to automate legal marketing.