Synthesis and Reactions of Fluorous Carbobenzyloxy (^FCbz) Derivatives of α-Amino Acids

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Supporting Information

The following pages contain tables of HRMS data, optical rotations and melting points of all the ^FCbz derivatives along with copies of all ¹H and ¹³C NMR spectra and two representative IR spectra.

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MS, MP and Rotation Data for “a” Series (L)-Amino Acids with the C₈F₁₇^FCBz Group.

Compound	HRMS -Cal	HRMS - Fnd	MP	Opt Rot	c ^a
Bis Cys	1423.0798	1423.0815 (M + Na) ⁺	188.0 d	-39.2	0.0075
Pro	695.0964	695.0936	105.0-106.0	-13.2	0.0075
Gly	655.0651	655.0619	138.8-140.0	0.0	0.0075
Ala	669.0808	669.0784	108.2-109.8	-1.0	0.0075
Val	697.1121	697.1155	91.0-92.2	+4.4	0.0075
Leu	711.1277	711.1255	78.4-80.4	-2.4	0.0042
Ile	711.1277	711.1239	79.1-80.2	+4.9	0.00515
Ser	685.0757	685.0791	145.0 d	+4.4	0.0075
Phe	745.1121	745.1092	115-118	-0.25	0.0075
Asn	694.0760	694.0721 M - H ₂ O	149.0 d	+9.1	0.0047
Gln	708.0917	708.0920 M - H ₂ O	146.0 d	+0.1	0.0031
Thr	699.0914	699.0912	138.3-139	+2.3	0.0075
Met	729.0842	729.0827	144.2-144.5	-4.5	0.0075
Asp	713.0706	713.0644	143.4 d	-2.4	0.0075
Glu	727.0863	727.0859	185.0 d	-3.6	0.0075
Trp	784.1230	784.1227	139.0 d	-4.3	0.0075
Tyr	554.0538	554.0515 M - C ₁₀ H ₉ NO ₄	140.0 d	-1.0	0.0029
N(E) BOC Lys	865.1548	865.1596 (M + K) ⁺	122.8-124.2	-0.1	0.0035
Double Lys	1329.1615	1329.1618	140.0 d	-0.4	0.0028
S-Benzyl Cys	814.0896	814.0872 (M + Na) ⁺	150.8-151.7	-18.9	0.0075

a) in methanol

MS, MP and Rotation Data for “b” Series (D)-Amino Acids with the C₆F₁₃ FCBz Group.

Compound	HRMS -Cal	HRMS - Fnd	MP	Opt Rot	c ^a
Bis Cys	1223.0926	1223.0827(M +Na) ⁺	192.0 d	+46.6	0.0075
Pro	595.1028	595.1018	59.0-60.0	+14.3	0.0044
Gly	555.0715	555.0743	119.5-120.5	0	0.0075
Ala	569.0872	569.0867	93.4-94.2	+2.8	0.0075
Val	597.1185	597.1162	64.6-65.0	-3.4	0.0075
Leu	537.061	537.0595M - C ₄ H ₉ OH	72.6-74.5	+3.1	0.0075
Ile	611.1341	611.1327	59.5-60.3	-6.5	0.0075
Ser	585.0821	585.0836	154.0 d	-4.8	0.0075
Phe	555.0715	555.0712M - C ₇ H ₇	112-115	+0.1	0.0075
Asn	595.0902	595.0881M - H ₂ O	147.0 d	-10.3	0.0029
Gln	608.0981	608.0975M - H ₂ O	147.5 d	+0.8	0.0075
Thr	599.0977	599.0958	152.1-153.0	-4.0	0.0075
Met	629.0905	629.0940	93.2-94.5	+7.1	0.0075
Asp	613.077	613.0763	155.0 d	+5.3	0.0075
Glu	627.1927	627.1947	181.0 d	+6.4	0.0075
Trp	707.1191	707.1179(M +Na) ⁺	130.0 d	+5.6	0.0075
Tyr	661.1134	661.1163	163.0-164.0	+2.8	0.0075
N(E) BOC Lys	765.1612	765.1656(M+K) ⁺	118-120	+1.5	0.0075

a) in methanol

Asn

ma26 acetone 100mg 300MHz

CSF13CBZ (D) glutamine Acetone-d6 300MHz 1H RT 11/11/02

C₆F₁₃ C62 N(c) Boc (b) 45 300MHz 1H AT Acetone-d6

13F Cbz-Met-11B in Acetone-d6 300 MHz 7-18-02

13F Cbz-Pro-166 in Acetone-d6 300 MHz 7-18-02

¹³Cbz-Thr-22B in Acetone-d₆ 300 MHz 7-22-02

ppm

7.45548
7.40590
7.33301
7.25035
7.20695
7.08295
6.41339
6.38430
6.01222
5.29554
5.05292
4.80521
4.19058
4.17292
4.16068
4.14313
2.98242
2.95572
2.94495
2.92741
2.63400
2.60534
2.57080
2.54007
2.51294
2.47779
2.45140
2.25889
2.23596
2.21307
2.19483
2.17227
2.14969
2.05472
2.04740
2.04007
2.03274
2.02542
1.39890
1.00450
0.98160
0.97532
0.95233
0.79315
0.76990
0.12249

13FChz-Va1-116 in Acetone-d6 300 MHz 7-18-02

ASN - m26 acetone 100mg 300MHz

U

F13CBZ-(D)-Asp in Acetone-d6

13F-Cbz-11e-15B 7-19-02 75 MHz in Acetone-d6

ma24 acetone 100mg 300Mhz 13cdh

ppm
7.61327
7.38640
7.34486
7.33006
7.30341
7.25268
7.20275
7.08215
7.05255
6.56744
6.54739
6.40108
5.29102
5.26859
5.15615
5.08358
5.06707
5.04816
5.00449
4.59503
4.28915
4.26473
4.24871
4.23981
4.22344
4.21432
4.19906
3.89430
3.87911
3.29316
3.15730
2.98138
2.96152
2.95420
2.94355
2.92627
2.85934
2.63634
2.60272
2.57283
2.54559
2.51558
2.47897
2.45302
2.07658
2.07338
2.06417
2.05496
2.04762
2.04028
2.03295
2.02561
1.82851
1.81384
1.41090
1.38654
1.27899
0.41246
0.38702
0.11798

17Fcbz-Ala-66 in Acetone-d6 300 MHz 6-19-02

ppm

7.37672
7.34925
7.33684
7.31067
7.29502
7.26999
7.24552
7.22302
7.20062
6.62601
6.59804

17F Cbz-Cys-25B

in Acetone-d6 300 MHz B-1-02

5.13959
5.09779
5.08729
5.04521
4.51851
4.50258
4.49160
4.47548
4.44849
3.80659
3.00453
2.98770
2.97736
2.95764
2.95097
2.94185
2.92234
2.85930
2.83246
2.81313
2.78625
2.62489
2.59557
2.56185
2.53299
2.50368
2.46936
2.05460
2.04748
2.04032
2.03311
2.02593

C8F17CBZ Bis (L) Cysteine 300MHz 1H Acetone-d6 RT

17FCbz-61U-126 in Acetone-d₆ 300 MHz 7-25-02

ppm

CBF17CBZ (L) Glutamine 300MHz Acetone-d6 RT 1H 12/04/02

ppm

07/23/02 Acetone-D6 300MHz RT 1H F17CBZ-Isoleucine

new batch

CBF17CBZ (L) Leucine Acetone-d6 300MHz 1H RT 11/20/02

N(A) C8F17CBZ-N(E) BOC-(L) Lysine Acetone-d6 300MHz RT 1H

(A) (E)-N-CBFL7CBZ (L) Lysine Acetone-d₆ 300MHz 1H RT 11/20/02

CBF17CBZ (L) Tyrosine Acetone-d6 300MHz 1H RT 12/04/02

ppm

7.32650
7.29971
7.27745
7.25046
7.11400
7.08586
6.75673
6.72888
6.40859
6.38089

5.04887
5.00423
4.45682
4.44050
4.42779
4.41165
4.39925
4.38254
3.15225
3.13613
3.10580
3.08979
2.97305
2.94511
2.91708
2.89737
2.86696
2.63145
2.59991
2.56880
2.53762
2.51028
2.47675
2.05499
2.04787
2.04067
2.03358
2.02651

F17-L-Alanine

15.11
made acetone 50 mg 300 MHz

F17CBZ-(L)-S-Benzyl-Cys

CBF17CBZ-(L) Glutamine RT 75MHz Acetone-d6 C13 1/7/03

CBF17CBZ (L) Glycine C13 Acetone-d6 75MHz RT 11/19/02

CBF17CBZ (L) Leucine Acetone-d6 75MHz C13 RT 11/20/02

N(E) BOC-N(A) C8F17CBZ (L) Lysine Acetone-d6 75MHz C13 RT 11/11/02

F17 CBZ - (L)-double Lys

ma25 acetone 100mg 300MHz

CBF17CBZ (L) Proline 75MHz Acetone-d6 C13 RT 12/6/02

CBF 17CBZ (L) Tyrosine Acetone-d6 75MHz RT C13 11/21/02

Matthew Campbell C8F17CBZ-(L) Valine 300MHz 100mg C13 Acetone-d6 RT

%Transmittance

%Transmittance