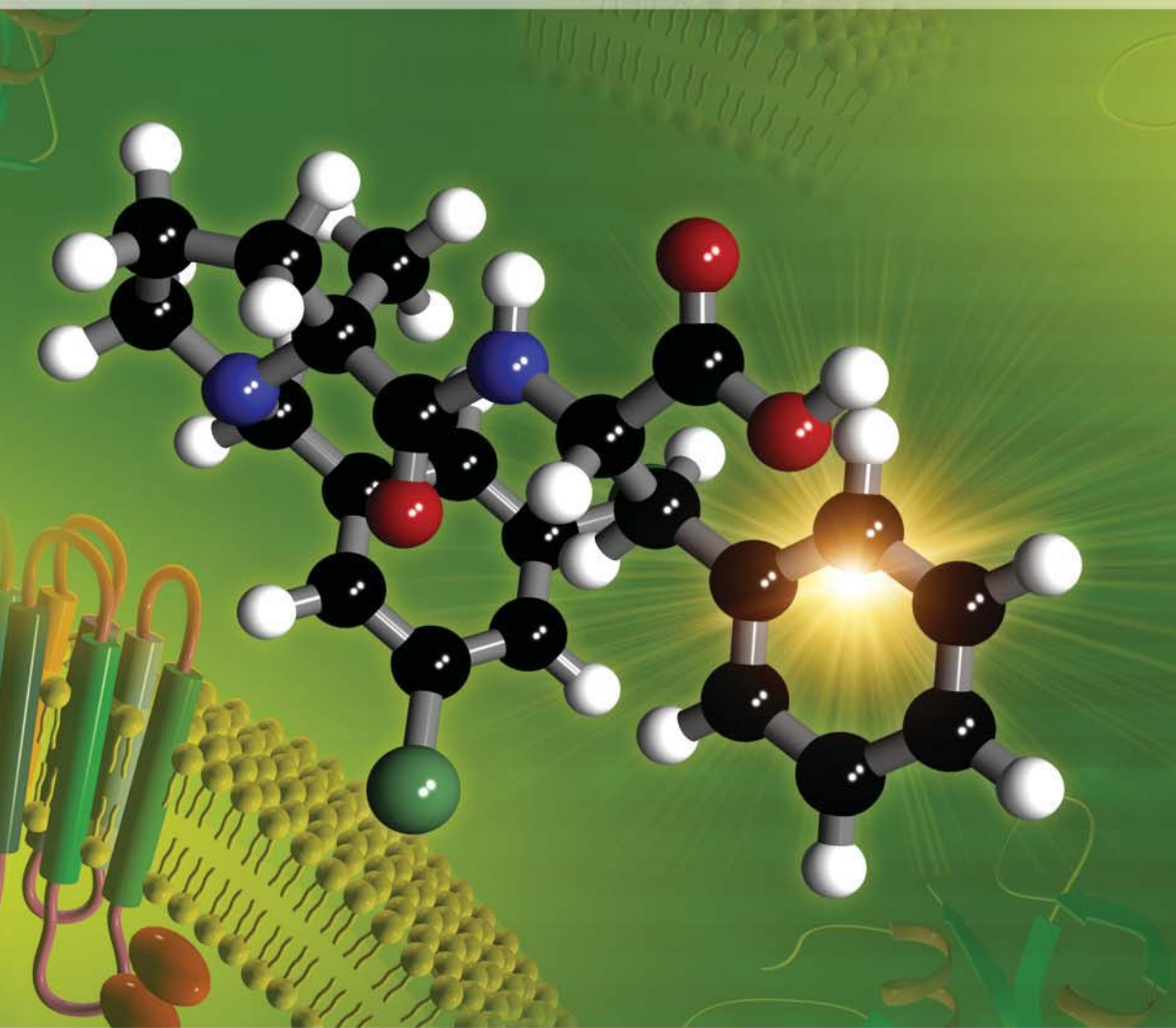


Innovations in Peptide Synthesis and Conjugation
Tools for Drug Discovery



Proline Derivatives
and Analogs

New Amino Acid
Building Blocks

New Guanidinylation
Reagents

New Monoprotected
Bifunctional Linkers

Functionalized
Polyethylene Glycols

Fluorescent Labeling
of Peptides

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1. Proline Derivatives and Analogs

1.1 Introduction

Proline is a non-polar proteinogenic amino acid that forms a tertiary amide when incorporated into peptides. It does not have a hydrogen on the amide group and therefore cannot act as a hydrogen bond donor. Proline is known as a classical breaker of both the α -helical and β -sheet structures in proteins and peptides. Nevertheless, it is widely distributed in the putative transmembrane domains of many protein transporters and channels, regions believed to be α -helical.¹

Among the proteinogenic amino acids, proline plays a special role. In protein structures the planar peptide bond occurs predominantly in the *trans* conformation.² The proline residue restricts the conformational space of the peptide chain. However, due to the small free enthalpy difference between the *cis*

and *trans* Xaa-Pro bond isomers of 2.0 kJ·mol⁻¹ (compared to 10.0 kJ·mol⁻¹ for other Xaa-non-Pro peptide bonds), there is a relatively high intrinsic probability of 30% *cis* conformation at RT and both *cis* and *trans* isomers are present in solution.^{3,4}

The *cis/trans*-isomerization of peptide bonds on the N-terminal side of Pro residues plays a key role in the folding process of a protein because the rotational barrier of the *cis/trans*-isomerization is quite high (85,0 ± 10,0 kJ·mol⁻¹). Therefore, this interconversion is described to be one of the limiting steps of protein folding *in vitro* and *in vivo*.⁵ In nature there is a class of enzymes, the peptidyl-prolyl-*cis/trans*-isomerases (**PIases**). They are able to catalyze protein folding by accelerating the isomerization of the Xaa-Pro-bond.⁶⁻⁸

Table 1. Proline Analog or Homolog Structures for the Restriction of the Xaa-Pro Imide Conformation

Chapter	Structure	Name/(IUPAC Nomenclature)
1.4		α -methyl-L-proline (2 <i>R</i>)-2-methyl-pyrrolidine-2-carboxylic acid
1.4		α -benzyl-L-proline (2 <i>R</i>)-2-benzyl-pyrrolidine-2-carboxylic acid
1.5		<i>trans</i> -4-hydroxy-L-proline (2 <i>S</i> ,4 <i>R</i>)-4-hydroxypyrrolidine-2-carboxylic acid
1.5		<i>cis</i> -4-hydroxy-L-proline (2 <i>S</i> ,4 <i>S</i>)-4-hydroxypyrrolidine-2-carboxylic acid
1.5		<i>trans</i> -3-hydroxy-L-proline (2 <i>S</i> ,3 <i>R</i>)-3-hydroxypyrrolidine-2-carboxylic acid
1.5		<i>cis</i> -3-hydroxy-L-proline (2 <i>S</i> ,3 <i>S</i>)-3-hydroxypyrrolidine-2-carboxylic acid
1.6		<i>trans</i> -4-amino-L-proline (2 <i>S</i> ,4 <i>R</i>)-4-aminopyrrolidine-2-carboxylic acid
1.7		3,4-dehydro-DL-proline (±)-3-pyrrolin-2-carboxylic acid
1.8		(2 <i>S</i>)-aziridine-2-carboxylic acid
1.8		(2 <i>S</i>)-azetidine-2-carboxylic acid
1.9		L-pipecolic acid (2 <i>S</i>)-piperidine-2-carboxylic acid
1.10		4-oxa-L-proline (4 <i>S</i>)-oxazolidine-4-carboxylic acid
1.10		3-thia-DL-proline thiazolidine-2-carboxylic acid
1.10		4-thia-L-proline (4 <i>R</i>)-thiazolidine-4-carboxylic acid



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1. Proline Derivatives and Analogs—Cont'd

Comparative studies performed with proline analogues revealed that the key step in the catalysis of the *cis/trans*-isomerization of a peptidyl-prolyl bond is a reduction of the double bond character of the planar, conjugated C–N amide bond. Any factor that can weaken the double bond character of the amide bond by destabilizing the planar peptide bond, or shifting the hybridization of the prolyl nitrogen from sp^2 to sp^3 , is expected to accelerate the isomerization.^{9,10}

In order to understand the relationship between imide bond geometry and bioactivity of peptides,^{11,12} synthetic proline analogues have been developed that provide restrictions of the Xaa-Pro imide conformation. Such proline mimetics are based on ring substitutions with alkyl and aromatic groups, incorporation of heteroatoms into the ring, or the expansion or contraction of the proline ring (Table 1). Those analogues are promising candidates for conformational studies and for tuning the biological, pharmaceutical, or physicochemical properties of naturally occurring, as well as *de novo* designed, linear, and cyclic peptides.

Several proline analogs and homologs occur in nature. *Trans*-3-hydroxyproline and *trans*-4-hydroxyproline represent constituents of common proteins as a result of post-translational hydroxylation, especially in collagens.¹³ Various 3- and 4-alkylated derivatives of proline and hydroxyproline as well as analogues with ring restrictions, such as aziridine-2-carboxylic acid and

azetidine-2-carboxylic acid, and ring expansions, i.e. pipercolic acid, are found in natural products.^{14,15} Derivatives such as L-azetidine-2-carboxylic acid, *cis*-4-hydroxy-L-proline, and 3,4-dehydro-DL-proline prevent pro-collagen from folding into a stable triple-helical conformation, thereby reducing excessive deposition of collagen in fibrotic processes and the growth of tumors.¹⁶

Thiazolidine-4-carboxylic acid thiaproline has also been incorporated into collagen model compounds^{17,18} and other bioactive molecules such as thrombin inhibitors,¹⁹ somatostatin,^{20,21} dipeptidyl peptidase IV substrates,²² angiotensin II,²³ HIV inhibitors,²⁴ ACE inhibitors,²⁵ and oxytocin.²⁶

α -Methyl-proline is a bioactive molecule restoring normal levels of bone collagen type I synthesis.²⁷ It can be looked at as a conformationally constrained aminoisobutyric acid analog. The α -methyl-proline residue has been inserted into morphiceptin to perform conformational studies on the bioactivity of the Xaa-Pro *cis/trans*-isomers.²⁸ A α -methyl-proline containing potential dual $\alpha_v\beta_1$ integrin antagonist has been described recently.²⁹

α -Benzyl-proline combines the conformational restrictions of a proline derivative with the electronic properties of phenylalanine. Spirolactams containing an α -benzyl-proline substructure have been synthesized as potential beta-turn mimetics.³⁰

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1.2 Proline Derivatives

Boc-Pro-OH puriss., ≥99.0% T

$C_{10}H_{17}NO_4$	
MW: 215.25	
[15761-39-4]	
15490-5G	5 g
15490-25G	25 g
15490-100G	100 g

Boc-D-Pro-OH puriss., ≥99.0% T

$C_{10}H_{17}NO_4$	
MW: 215.25	
[37784-17-1]	
92517-1G-F	1 g
92517-5G-F	5 g

Boc-Pro-OSu purum, ≥98.0% N

$C_{14}H_{20}N_2O_6$	
MW: 312.32	
[3392-10-7]	
15491-1G	1 g
15491-5G	5 g
15491-25G	25 g

Fmoc-Pro-OH purum, ≥99.0% HPLC

$C_{20}H_{19}NO_4$	
MW: 337.37	
[71989-31-6]	
47636-5G-F	5 g
47636-50G-F	50 g
47636-100G-F	100 g

Fmoc-Pro-OPfp purum, ≥96.0% HPLC

$C_{26}H_{18}F_5NO_4$	
MW: 503.42	
[86060-90-4]	
47475-1G	1 g
47475-5G	5 g
47475-25G	25 g

Fmoc-D-Pro-OH purum, ≥98.0% TLC

$C_{20}H_{19}NO_4$	
MW: 337.37	
[101555-62-8]	
47532-1G	1 g
47532-5G	5 g
47532-25G	25 g

Z-Pro-OH puriss., ≥99.0% T

$C_{13}H_{15}NO_4$	
MW: 249.26	
[1148-11-4]	
97090-10G	10 g
97090-50G	50 g

Z-D-Pro-OH 98%

$C_{13}H_{15}NO_4$	
MW: 249.26	
[6404-31-5]	
860735-100MG	100 mg
860735-500MG	500 mg

Ddz-Pro-OH purum, ≥99.0% HPLC

$C_{17}H_{23}NO_6$	
MW: 337.37	
71215-1G	1 g
71215-5G	5 g

Dansyl-L-proline puriss., ≥99.0% TLC

$C_{17}H_{20}N_2O_4S$	
MW: 348.42	
[1239-94-7]	
87778-250MG-F	250 mg
87778-1G-F	1 g

L-Proline ≥99.5% NT

$C_5H_9NO_2$	
MW: 115.13	
[147-85-3]	
81709-25G	25 g
81709-100G	100 g
81709-500G	500 g

L-Proline ≥99.0% NT

$C_5H_9NO_2$	
MW: 115.13	
[147-85-3]	
81710-10G	10 g
81710-50G	50 g
81710-250G	250 g

D-Proline puriss., ≥99.0% NT

$C_5H_9NO_2$	
MW: 115.13	
[344-25-2]	
81705-1G	1 g
81705-5G	5 g
81705-25G	25 g

DL-Proline purum, ≥98.0% NT

$C_5H_9NO_2$	
MW: 115.13	
[609-36-9]	
81720-5G	5 g
81720-25G	25 g



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1.3 β^3 - Homoprolines

Boc- β^3 -Homopro-OH purum, $\geq 98.0\%$ TLC

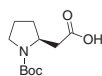
$C_{11}H_{19}NO_4$

MW: 229.27

[56502-01-3]

14982-250MG

14982-1G



250 mg

1 g

Fmoc-L- β^3 -Homopro-OH purum, $\geq 98.0\%$ HPLC

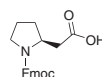
$C_{21}H_{21}NO_4$

MW: 351.4

[193693-60-6]

47912-250MG

47912-1G



250 mg

1 g

1.4 Prolines with Substituents in α -Position

Boc- α -Me-DL-Pro-OH purum, $\geq 96.0\%$ HPLC

$C_{11}H_{19}NO_4$

MW: 229.27

[203869-80-1]

68691-500MG



500 mg

α -Methyl-L-proline purum, $\geq 98.0\%$ TLC

$C_6H_{11}NO_2$

MW: 129.16

[42856-71-3]

17249-250MG

17249-1G



250 mg

1 g

Boc- α -propyl-DL-Pro-OH tech., $\geq 90\%$ HPLC

$C_{13}H_{23}NO_4$

MW: 257.33

[351002-88-5]

95566-500MG



500 mg

Boc- α -allyl-DL-Pro-OH purum, $\geq 96.0\%$ HPLC

$C_{13}H_{21}NO_4$

MW: 255.31

[315234-49-2]

58147-500MG



500 mg

Boc-(R)- α -allyl-Pro-OH purum, $\geq 98.0\%$ HPLC

$C_{13}H_{21}NO_4$

MW: 255.31

[144085-23-4]

06538-500MG-F



500 mg

Boc-(S)- α -allyl-Pro-OH purum, $\geq 98.0\%$ HPLC

$C_{13}H_{21}NO_4$

MW: 255.31

[706806-59-9]

06486-500MG-F



500 mg

L- β^3 -Homopro-OH HCl purum, $\geq 98.0\%$ TLC

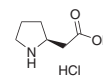
$C_6H_{11}NO_2 \cdot HCl$

MW: 165.62

[53912-85-9]

03768-250MG

03768-1G



250 mg

1 g

(R)- α -Allyl-proline hydrochloride purum, $\geq 98.0\%$ HPLC

$C_8H_{13}NO_2 \cdot HCl$

MW: 191.66

[177206-69-8]

06541-500MG-F



500 mg

(S)- α -Allyl-proline hydrochloride purum, $\geq 98.0\%$ HPLC

$C_8H_{13}NO_2$

MW: 155.19

[129704-91-2]

06594-500MG-F



500 mg

Boc- α -benzyl-DL-Pro-OH purum, $\geq 96.0\%$ HPLC

$C_{17}H_{23}NO_4$

MW: 305.37

[351002-72-7]

52969-500MG



500 mg

Boc-(R)- α -benzyl-Pro-OH purum, $\geq 99.0\%$ HPLC

$C_{17}H_{23}NO_4$

MW: 305.37

[706806-60-2]

47079-500MG-F



500 mg

Boc-(S)- α -benzyl-Pro-OH purum, $\geq 97.0\%$ HPLC

$C_{17}H_{23}NO_4$

MW: 305.37

[706806-61-3]

76896-500MG-F



500 mg

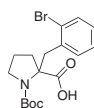
1.4 Prolines with Substituents in α -Position—Cont'd

Boc- α -(2-bromobenzyl)-DL-Pro-OH purum, $\geq 96.0\%$ HPLC

$C_{17}H_{22}BrNO_4$

MW: 384.26

[351002-85-2]



90682-500MG

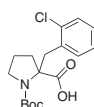
500 mg

Boc- α -(2-chlorobenzyl)-DL-Pro-OH purum, $\geq 96.0\%$ HPLC

$C_{17}H_{22}ClNO_4$

MW: 339.81

[351002-86-3]



90683-500MG

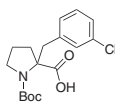
500 mg

Boc- α -(3-chlorobenzyl)-DL-Pro-OH purum, $\geq 96.0\%$ HPLC

$C_{17}H_{22}ClNO_4$

MW: 339.81

[351002-87-4]



90684-500MG

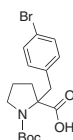
500 mg

Boc- α -(4-bromobenzyl)-DL-Pro-OH purum, $\geq 96.0\%$ HPLC

$C_{17}H_{22}BrNO_4$

MW: 384.26

[336817-91-5]



94866-500MG

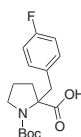
500 mg

Boc- α -(4-fluorobenzyl)-DL-Pro-OH purum, $\geq 96.0\%$ HPLC

$C_{17}H_{22}FNO_4$

MW: 323.36

[351002-78-3]



74082-500MG

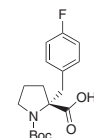
500 mg

Boc-(R)- α -(4-fluorobenzyl)-Pro-OH purum, $\geq 98.0\%$ HPLC NEW

$C_{17}H_{22}FNO_4$

MW: 323.36

[706806-64-6]



67420-500MG-F

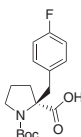
500 mg

Boc-(S)- α -(4-fluorobenzyl)-Pro-OH purum, $\geq 98.0\%$ HPLC NEW

$C_{17}H_{22}FNO_4$

MW: 323.36

[706806-65-7]



14931-500MG-F

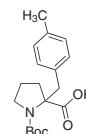
500 mg

Boc- α -(4-methylbenzyl)-DL-Pro-OH purum, $\geq 96.0\%$ HPLC

$C_{18}H_{25}NO_4$

MW: 319.4

[351002-82-9]



76501-500MG

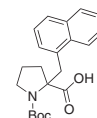
500 mg

Boc- α -(1-naphthylmethyl)-DL-Pro-OH purum, $\geq 96.0\%$ HPLC

$C_{21}H_{25}NO_4$

MW: 355.43

[351002-65-8]



36748-500MG

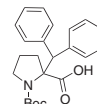
500 mg

Boc- α -(diphenylmethyl)-DL-Pro-OH technical, $\geq 90\%$ HPLC

$C_{23}H_{27}NO_4$

MW: 381.46

[351002-64-7]



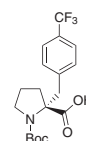
30763-500MG

500 mg

Boc-(R)- α -(4-trifluoromethylbenzyl)-Pro-OH purum, $\geq 98.0\%$ HPLC NEW

$C_{18}H_{22}F_3NO_4$

MW: 373.37



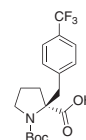
42004-500MG-F

500 mg

Boc-(S)- α -(4-trifluoromethylbenzyl)-Pro-OH purum, $\geq 98.0\%$ HPLC NEW

$C_{18}H_{22}F_3NO_4$

MW: 373.37



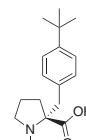
05199-500MG-F

500 mg

Boc-(R)- α -(4-tert-butylbenzyl)-Pro-OH purum, $\geq 97.0\%$ HPLC NEW

$C_{21}H_{31}NO_4$

MW: 361.48



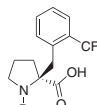
39793-500MG-F

500 mg

Boc-(S)- α -(4-tert-butylbenzyl)-Pro-OH purum, $\geq 98.0\%$ HPLC NEW

$C_{21}H_{31}NO_4$

MW: 361.48



39166-500MG-F

500 mg

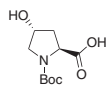


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1.5 Hydroxyproline Derivatives

Boc-Hyp-OH purum, ≥98.0% TLC

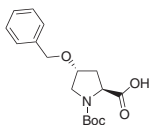
C₁₀H₁₇NO₅
MW: 231.25
[13726-69-7]



15544-5G	5 g
15544-25G	25 g

Boc-Hyp(Bzl)-OH purum, ≥98.0% HPLC

C₁₇H₂₃NO₅
MW: 321.37
[54631-81-1]



15535-1G	1 g
15535-5G	5 g
15535-25G	25 g

Fmoc-Hyp-OH purum, ≥98.0% HPLC, sum of enantiomers

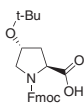
C₂₀H₁₉NO₅
MW: 353.37
[88050-17-3]



47686-1G	1 g
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Fmoc-Hyp(tBu)-OH purum, ≥98.0% HPLC

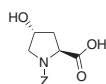
C₂₄H₂₇NO₅
MW: 409.47
[122996-47-8]



47517-1G-F	1 g
47517-5G-F	5 g
47517-25G-F	25 g

Z-Hyp-OH puriss., ≥99.0% T

C₁₃₀H₁₅NO₅
MW: 265.26
[13504-85-3]



96310-5G	5 g
96310-25G	25 g

N-Acetyl-L-hydroxyproline puriss., ≥99.0% T

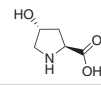
C₇H₁₁NO₄
MW: 173.17
[33996-33-7]



01192-10G-F	10 g
01192-50G-F	50 g

trans-4-Hydroxy-L-proline ≥99.0% NT

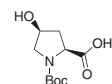
C₅H₉NO₃
MW: 131.13
[51-35-4]



56250-5G	5 g
56250-25G	25 g
56250-100G	100 g

N-Boc-cis-4-Hydroxy-L-proline 97%

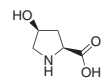
C₁₀H₁₇NO₅
MW: 231.25
[87691-27-8]



654019-1G	1 g
654019-5G	5 g

cis-4-Hydroxy-L-proline puriss., ≥99.0% NT

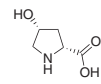
C₅H₉NO₃
MW: 131.13
[618-27-9]



56248-50MG	50 mg
56248-250MG	250 mg
56248-1G	1 g

cis-4-Hydroxy-D-proline ≥99.0% NT

C₅H₉NO₃
MW: 131.13
[2584-71-6]



56246-250MG	250 mg
56246-1G	1 g

trans-3-Hydroxy-L-proline purum, ≥98.0% NT

C₅H₉NO₃
MW: 131.13
[4298-08-2]



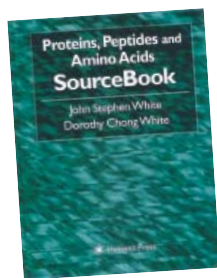
56244-100MG	100 mg
56244-500MG	500 mg

cis-3-Hydroxy-DL-proline purum, ≥97.0% TLC

C₅H₉NO₃
[4298-05-9]



56245-10MG	10 mg
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P0868

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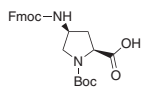
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1.6 4-substituted Proline Derivatives

***N*-Boc-*cis*-4-*N*-Fmoc-amino-L-proline 97%**

$C_{25}H_{28}N_2O_6$

[174148-03-9]



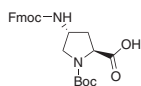
534404-1G

1 g

***N*-Boc-*trans*-4-*N*-Fmoc-amino-L-proline 97%**

$C_{25}H_{28}N_2O_6$

[176486-63-8]



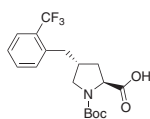
534390-1G

1 g

Boc-(*R*)-4-[2-(trifluoromethyl)benzyl]-Pro-OH purum, ≥98.0% HPLC

NEW

$C_{18}H_{22}F_3NO_4$



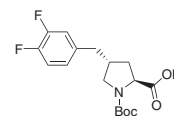
38455-500MG-F

500 mg

Boc-(*R*)-4-(3,4-difluorobenzyl)-Pro-OH purum, ≥97.0% HPLC

NEW

$C_{17}H_{21}F_2NO_4$



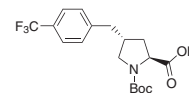
40372-500MG-F

500 mg

Boc-(*R*)-4-[4-(trifluoromethyl)benzyl]-Pro-OH purum, ≥98.0% HPLC

NEW

$C_{18}H_{22}F_3NO_4$



01336-500MG-F

500 mg

1.7 Dehydroprolines

3,4-Dehydro-DL-proline ≥99.0% T

$C_5H_7NO_2$

[3395-35-5]



30900-250MG

250 mg

30900-1G

1 g

3,4-Dehydro-L-proline ≥99.0% TLC

$C_5H_7NO_2$

[4043-88-3]



30890-10MG

10 mg

30890-50MG

50 mg

1.8 Proline Analogues with Ring Restrictions—Aziridine- and Azetidine-2-Carboxylic Acids

Lithium L-aziridine-2-carboxylate purum, ≥97.0% NT dried material

$C_3H_4LiNO_2$

[67413-27-8]



11558-50MG

50 mg

11558-250MG

250 mg

L-Azetidine-2-carboxylic acid purum, ≥98.0% NT

$C_4H_7NO_2$

[2133-34-8]



11542-500MG

500 mg

11542-2.5G

2.5 g

1-Boc-L-azetidine-2-carboxylic acid purum, ≥98.0% TLC

$C_9H_{15}NO_4$

[51077-14-6]



78324-500MG-F

500 mg

1-Fmoc-(*S*)-azetidine-2-carboxylic acid purum, ≥97.0% HPLC

$C_{19}H_{17}NO_4$

[136552-06-2]



70238-500MG-F

500 mg



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1.9 Proline Analogs with Ring Expansions—Pipelicolic Acids

DL-Pipelicolic acid purum, ≥99.0% NT

C₆H₁₁NO₂
[535-75-1]



80618-25G	25 g
80618-100G	100 g

L-Pipelicolic acid puriss., ≥99.0% NT

C₆H₁₁NO₂
[3105-95-1]



80615-100MG	100 mg
80615-500MG	500 mg

D-Pipelicolic acid puriss., ≥99.0% NT

C₆H₁₁NO₂
[1723-00-8]



80617-100MG	100 mg
80617-500MG	500 mg

Boc-Pip-OH purum, ≥99.0% HPLC

C₁₁H₁₉NO₄
[26250-84-0]



15558-1G	1 g
15558-5G	5 g

Boc-D-Pip-OH purum, ≥99.0% HPLC

C₁₁H₁₉NO₄
[28697-17-8]



75748-250MG	250 mg
75748-1G	1 g

Fmoc-Pip-OH purum, ≥98.0% HPLC

C₂₁H₂₁NO₄
[86069-86-5]



09777-250MG	250 mg
09777-1G	1 g

Fmoc-D-Pip-OH purum, ≥98.0% HPLC

C₂₁H₂₁NO₄
[101555-63-9]



73418-250MG	250 mg
73418-1G	1 g

1.10 Oxa- and Thia-Prolines

(S)-(-)-3-(Boc)-4-oxazolidinecarboxylic acid 98%

C₁₂H₁₃NO₅
[97534-82-2]



469467-1G	1 g
469467-5G	5 g

(R)-(+)-3-(Boc)-4-oxazolidinecarboxylic acid 98%

C₁₂H₁₃NO₅
[97534-84-4]



469475-1G	1 g
469475-5G	5 g

Thiazolidine-2-carboxylic acid 97%

C₄H₇NO₂S
[65126-70-7]



467995-1G	1 g
467995-5G	5 g

L-4-Thiazolidinecarboxylic acid purum, ≥99.0% T

C₄H₇NO₂S
[34592-47-7]



88400-10G	10 g
88400-50G	50 g

(R)-Boc-4-thiazolidinecarboxylic acid puriss. p.a., ≥99.0% HPLC

C₉H₁₅NO₄S
[51077-16-8]



95471-1G-F	1 g
95471-5G-F	5 g

(R)-Fmoc-4-thiazolidinecarboxylic acid puriss., ≥99.0% HPLC

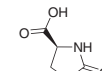
C₁₉H₁₇NO₄S
[133054-21-4]



94703-1G-F	1 g
94703-5G-F	5 g

(R)-(-)-2-Oxothiazolidine-4-carboxylic acid purum, ≥97.0% T

C₄H₅NO₃S
[19771-63-2]



75951-1G	1 g
75951-5G	5 g

