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Alkylated and bicyclic sugar amino acids : synthesis and applications

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ALKYLATED AND BICYCLIC SUGAR AMINO ACIDS

Synthesis and Applications

Proefschrift

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de graad van doctor aan de Universiteit Leiden
op gezag van Rector Magnificus prof. Mr. P.F. van der Heijden
volgens besluit van het College voor Promoties
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door

Martijn Daniël Pieter Risseeuw

Geboren te Oostburg in 1981

Promotiecommissie

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Voor mijn broer Jeroen

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List of abbreviations

δ	chemical shift
Ac	acetyl
AcOH	acetic acid
Ada	adamantyl
Ala	alanine
All	allyl
APT	attached proton test
aq.	aqueous
Ar.	aromatic
BAIB	[bis(acetoxy)iodo]benzene
BF_3•OEt_2	borontrifluoride diethyletherate
Bn	benzyl
Boc	*tert*-butoxycarbonyl
Boc-ON	2-(tert-butoxycarbonyloxyimino)-2-phenylacetonitrile
Bu	butyl
BuLi	*n*-butyllithium
Bz	benzoyl
c	concentration
calc.	calculated
CAN	ceric ammonium nitrate
cat.	catalytic
CBz	benzyloxycarbonyl
COSY	correlation spectroscopy
C_q	quarternary carbon atom
CSA	camphor sulphonic acid
d	doublet
DABCO	1,4-diazabicyclo[2.2.2]octane
DCM	dichloromethane
dd	double doublet
ddd	double double doublet
DDQ	2,3-dichloro-5,6-dicyano benzoquinone
DEAD	diethyl azodicarboxylate
DEMS	diethylmethylsilyl
DIPEA	*N*,*N*-di-isopropyl-*N*-ethylamine
DMAP	4-(*N*,*N*-dimethylamino)pyridine
DMF	*N*,*N*-dimethylformamide
DMSO	dimethylsulfoxide
DPPA	diphenylphosphoryl azide
dt	double triplet
e.g.	*exempli gratia* (for example)
EDC	*N*-(3-dimethylaminopropyl)-*N*′-ethylcarbodiimide hydrochloride
eq.	molar equivalents
ESI	electron spray ionization
Et	ethyl
et al.	*et alii* (and others)
Et_2O	diethylether
Et_3N	triethylamine
EtOAc	ethyl acetate
Fmoc	9H-fluoren-9-ylmethoxycarbonyl
g	gram
GBA1	glucocerebrosidase
GBA2	β-glucosidase 2
Glc	glucose
Gly	glycine
h	hour
HATU	O-(7-azabenzotriazol-1-yl)-*N*,*N*,*N*',*N*'-tetramethyluronium hexafluorophosphate
HCTU	(2-(6-chloro-1H-benzotriazole-1-yl)-1,1,3,3-tetramethylaminium hexafluorophosphate)
HMPB	4-(4-hydroxymethyl-3-methyoxy-phenoxy)-butyric acid
HOAt	1-hydroxy-7-azabenzotriazole
HOBt	*N*-hydroxybenzotriazole
HPLC	high performance liquid chromatography
HRMS	high resolution mass spectrometry
HSQC	heteronuclear single quantum coherence spectroscopy
Hz	Herz
hv	irradiation with light

*i*Bu	isobutyl
IC_{50}	inhibitor concentration resulting in 50% inhibition of enzyme activity
Ile	isoleucine
*i*Pr	isopropyl
IR	infrared
J	coupling constant
LCMS	liquid chromatography mass spectrometry
Leu	leucine
m	multiplet
M	molar
m/z	mass over charge ratio
Me	methyl
MeOH	methanol
mg	milligram(s)
MHz	megaherz
min.	minute(s)
mL	milliliter(s)
mmol	millimole (s)
MS	mass spectrometry
Ms	methanesulfonyl (mesyl)
NaH	sodium hydride
NBS	*N*-bromosuccinimide
NMR	nuclear magnetic resonance
p	*para*
Pd/C	palladium on activated charcoal
PE	petroleum ether
Ph	phenyl
PMB	*para*-methoxybenzyl
ppm	parts per million
Pr	propyl
pyr.	pyridine
q	quartet
quant.	quantitative
RCM	ring-closing metathesis
ref.	reference
R_f	retardation factor
RP	reversed phase
rt	room temperature
s	singlet
SAA(s)	sugar amino acid(s)
sat.	saturated
SPPS	solid phase peptide synthesis
t	tertiary
t	triplet
TBAF	tetra-*n*-butylammonium fluoride
TBAI	tetra-*n*-butylammonium iodide
TBDMS	*tert*-butyldimethylsilyl
TBDPS	*tert*-butyldiphenylsilyl
*t*Bu	*tert*-butyl
TEMPO	2,2,6,6-tetramethyl-1-piperidinyloxy (free radical)
Thr	threonine
Tf	trifluoromethanesulfonate
TFA	trifluoroacetic acid
THF	tetrahydrofuran
TLC	thin layer chromatography
TMS	trimethylsilyl
tol	toluene
Tr	triphenylmethylene (trityl)
TrisCl	2,4,6-triisopropylbenzenesulfonyl chloride
Ts	*para*-toluenesulfonyl (tosyl)
Tyr	tyrosine
UV	ultraviolet
v	volume
Val	valine
wt.	weight
Z	benzyloxycarbonyl