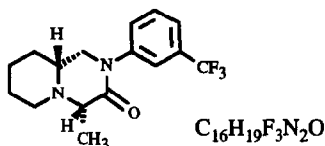


STEREOCHEMISTRY ABSTRACTS

Leandro Baiocchi e Giuseppe Picconi

Tetrahedron: Asymmetry 1991, 2, 231



2-(3-trifluoromethylphenyl)-4-methyl-3-oxo,octahydro-2H-pyrido[1,2-a]pyrazine

E.e.=100 (by nmr with $Eu(hfc)_3$)

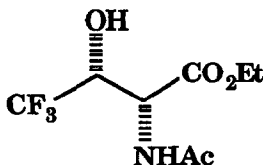
$[\alpha]_D = +33.0$ (c 4.5, EtOH)

Source of chirality : asymm. synth.

Absolute configuration 4R,9aS
(assigned by mechanistic considerations)

T. Kitazume, J. T. Lin and T. Yamazaki

Tetrahedron: Asymmetry 1991, 2, 235



Ethyl 2-acetylamino-3-hydroxy-4,4,4-trifluorobutyrate

E.e = >97% [by GLC with (-)-MTPA ester]

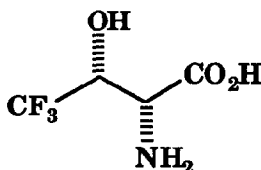
$[\alpha]_D^{23} = +18.7$ (c = 1.13, MeOH)

Source of chirality: enzymatic kinetic resolution

Absolute configuration *syn*-2R, 3R (assigned by comparison with *syn*-2S, 3S enantiomer)

T. Kitazume, J. T. Lin and T. Yamazaki

Tetrahedron: Asymmetry 1991, 2, 235



4,4,4-Trifluorothreonine

E.e = >97% : mp 209 - 211 °C

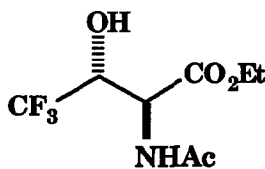
$[\alpha]_D^{23} = +12.5$ (c = 1, H₂O)

Source of chirality: enzymatic kinetic resolution

Absolute configuration *syn*-2R, 3R

T. Kitazume, J. T. Lin and T. Yamazaki

Tetrahedron: Asymmetry 1991, 2, 235



Ethyl 2-acetylamino-3-hydroxy-4,4,4-trifluorobutyrate

E.e = 95% [by GLC with (-)-MTPA ester]

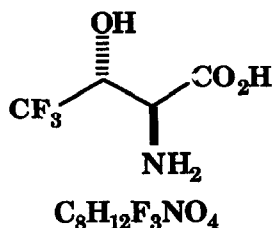
$[\alpha]_D^{23} = +26.9$ (c = 1.15, MeOH)

Source of chirality: enzymatic kinetic resolution

Absolute configuration *syn*-2S, 3R (assigned by chem correlation with (R)-(+)-3-hydroxy-4,4,4-trifluorobutanol)

T. Kitazume, J. T. Lin and T. Yamazaki

Tetrahedron: Asymmetry 1991, 2, 235



4,4,4-Trifluoro-*allo*-threonine

E.e = >95% : mp 190 - 193 °C

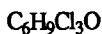
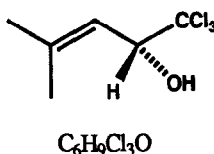
[α]_D²³ = -11.9 (c = 1, H₂O)

Source of chirality: enzymatic kinetic resolution

Absolute configuration *syn*-2S, 3R

Z. Muljiani, S.R. Gadre, S.R. Modak, N. Pathan and R.B. Mitra

Tetrahedron: Asymmetry 1991, 2, 239



(-)-1,1,1-trichloro-2-hydroxy-4-methyl-3-pentene

E.e ≥ 98% [by nmr of acetate with Eu (tfc)₃]

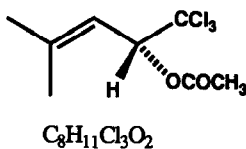
[α]_D²⁵ = -12 (c 2, CHCl₃) m.p. = 109°

Source of chirality : Microbial kinetic resolution

Absolute configuration : R (Lit. assignment)

Z. Muljiani, S.R. Gadre, S.R. Modak, N. Pathan and R.B. Mitra

Tetrahedron: Asymmetry 1991, 2, 239



(+)-1,1,1-trichloro-2-acetoxy-4-methyl-3-pentene

E.e ≥ 98% [by nmr with Eu (tfc)₃]

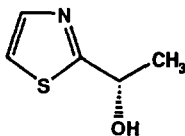
[α]_D²³ = +0.93 (c 7.5, CHCl₃)

Source of chirality : Microbial kinetic resolution

Absolute configuration : R [Prepared from R-(-)-alcohol]

G. Fantin, M. Fogagnolo, A. Medici, P. Pedrini, S. Poli,
M. E. Guerzoni and F. Gardini

Tetrahedron: Asymmetry 1991, 2, 243



1-(2-Thiazolyl)ethanol

ee => 95% [by GLC analysis on a 25 m permethylated β-cyclodextrine
in OV 1701]

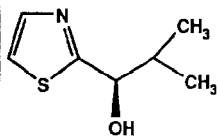
[α]_D²⁵ = - 19.2 (c = 1.16, CHCl₃)

Source of chirality: microbial reduction

Absolute configuration: S

G. Fantin, M. Fogagnolo, A. Medici, P. Pedrini, S. Poli,
M. E. Guerzoni and F. Gardini

Tetrahedron: Asymmetry 1991, 2, 243



$C_7H_{11}NOS$

2-Methyl-1-(2-thiazolyl)propanol

ee => 95% [by GLC analysis on a 25 m permethylated β -cyclodextrine
in OV 1701]

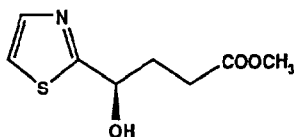
$[\alpha]_D^{25} = -16.7$ (c = 1.6, $CHCl_3$)

Source of chirality: microbial reduction

Absolute configuration: R

G. Fantin, M. Fogagnolo, A. Medici, P. Pedrini, S. Poli,
M. E. Guerzoni and F. Gardini

Tetrahedron: Asymmetry 1991, 2, 243



$C_8H_{11}NO_3S$

Methyl 4-Hydroxy-4-(2-thiazolyl)butanoate

ee => 95% [by GLC analysis on a 25 m permethylated β -cyclodextrine
in OV 1701]

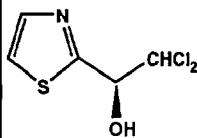
$[\alpha]_D^{25} = +15.8$ (c = 3.0, $CHCl_3$)

Source of chirality: microbial reduction

Absolute configuration: R

G. Fantin, M. Fogagnolo, A. Medici, P. Pedrini, S. Poli,
M. E. Guerzoni and F. Gardini

Tetrahedron: Asymmetry 1991, 2, 243



$C_5H_5Cl_2NOS$

2,2-Dichloro-1-(2-thiazolyl)ethanol

ee => 95% [by GLC analysis on a 25 m permethylated β -cyclodextrine
in OV 1701]

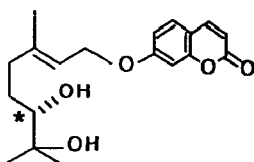
$[\alpha]_D^{25} = +2.9$ (c = 1.85, $CHCl_3$)

Source of chirality: microbial reduction

Absolute configuration: S

X.M. Zhang, A. Archelas, A. Méou and R. Furstoss*

Tetrahedron: Asymmetry 1991, 2, 247



$C_{19}H_{24}O_5$

Marmin: 7-[(6',7'-dihydroxy-3',7'-
dimethyl-2'-octenyl)oxy]coumarin

E.e. = 100% [by HPLC analysis of the (-)-camphanic
ester]

$[\alpha]_D^{26} = -17.1$ (c 0.6, $CHCl_3$)

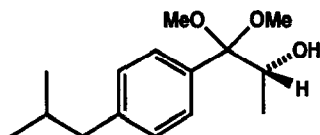
Source of chirality: microbiological oxygenation

Absolute configuration: 6S

(assigned by comparison with literature values)

H.R. Sonawane, B.S. Nanjundiah, D.G. Kulkarni and J.R. Ahuja

Tetrahedron: Asymmetry 1991, 2, 251



$C_{15}H_{24}O_3$

(R)-1,1-Dimethoxy-2-hydroxy-1-[4-(2-methylpropyl)phenyl]propane

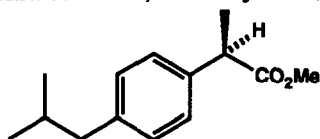
E.e = 82% by $^1\text{H-NMR}$ using chiral shift reagent $\text{Eu}(\text{hfc})_3$
 $[\alpha]_D^{25} = -1.79$ (C1, CHCl_3)

Source of chirality: S(-)-Ethyl Lactate

Absolute configuration: R

H.R. Sonawane, B.S. Nanjundiah, D.G. Kulkarni and J.R. Ahuja

Tetrahedron: Asymmetry 1991, 2, 251



$C_{14}H_{20}O_2$

Methyl (R)-2-[4-(2-methylpropyl)phenyl]propanoate

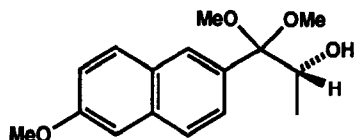
E.e = 82% by $^1\text{H-NMR}$ using chiral shift reagent $\text{Eu}(\text{hfc})_3$
 $[\alpha]_D^{25} = -46.8$ (C1, CHCl_3)

Source of chirality: S(-)-Ethyl Lactate and stereospecific rearrangement of the hydroxy acetal

Absolute configuration: R

H.R. Sonawane, B.S. Nanjundiah, D.G. Kulkarni and J.R. Ahuja

Tetrahedron: Asymmetry 1991, 2, 251



$C_{16}H_{20}O_4$

(R)-1,1-Dimethoxy-2-hydroxy-1-(6-methoxy-2-naphthyl)propane

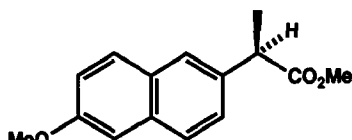
E.e = 70% by $^1\text{H-NMR}$ using chiral shift reagent $\text{Eu}(\text{hfc})_3$
 $[\alpha]_D^{25} = -9.7$ (C 0.92, MeOH)

Source of chirality: S(-)-Ethyl Lactate

Absolute configuration: R

H.R. Sonawane, B.S. Nanjundiah, D.G. Kulkarni and J.R. Ahuja

Tetrahedron: Asymmetry 1991, 2, 251



$C_{15}H_{16}O_3$

Methyl (R)-2-(6-methoxy-2-naphthyl) propanoate

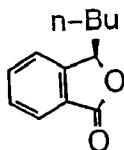
E.e = 70% by $^1\text{H-NMR}$ using chiral shift reagent $\text{Eu}(\text{hfc})_3$
 $[\alpha]_D^{25} = -53.7$ (C1, CHCl_3)

Source of chirality: S(-)-Ethyl Lactate and stereospecific rearrangement of the hydroxy acetal

Absolute configuration: R

K. Soai, H. Hori, and M. Kawahara

Tetrahedron: Asymmetry **1991**, 2, 253



$C_{12}H_{13}O_2$
3-*n*-Butylphthalide

E.e. = 86% (by HPLC using a chiral column)

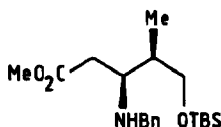
$[\alpha]_D^{24} +29.0$ (c 4.3, $CHCl_3$)

Source of chirality: *asymm. synth.* (alkylation)

Absolute configuration *R*

A V Rama Rao, M K Gurjar and B Ashok

Tetrahedron: Asymmetry **1991**, 2, 255



$C_{20}H_{35}NO_3Si$

Methyl-3-benzylamino-5-tert.butyltrimethylsilyloxy-4-methylpentanoate.

E.e = 100%

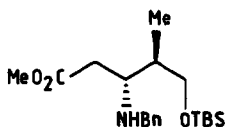
$[\alpha]_D = +1.73$ (c 1.01, $CHCl_3$)

Source of Chirality: *S*-methyl-3-hydroxy-2-methylpropionate and resolution of diastereomers.

Absolute configuration - 3*S*,4*R*.

A V Rama Rao, M K Gurjar and B Ashok

Tetrahedron: Asymmetry **1991**, 2, 255



$C_{20}H_{35}NO_3Si$

Methyl-3-benzylamino-5-tert.butyltrimethylsilyloxy-4-methylpentanoate.

E.e = 100%

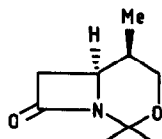
$[\alpha]_D = +3.32$ (c 1.03, $CHCl_3$)

Source of Chirality: *S*-methyl-3-hydroxy-2-methylpropionate and resolution of diastereomers.

Absolute configuration - 3*R*,4*R*.

A V Rama Rao, M K Gurjar and B Ashok

Tetrahedron: Asymmetry **1991**, 2, 255



$C_9H_{15}NO_2$

1-Aza-3-oxa-2,2,5-trimethylbicyclo[4.2.0]octan-8-one.

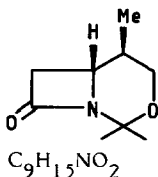
E.e = 100%

$[\alpha]_D = +37$ (c 1.18, $CHCl_3$)

Source of Chirality: *S*-methyl-3-hydroxy-2-methylpropionate and resolution of diastereomers.

Absolute configuration - 5*R*,6*S*.

A V Rama Rao, M K Gurjar and B Ashok



1-Aza-3-oxa-2,2,5-trimethylbicyclo[4.2.0]octan-8-one.

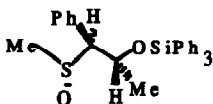
E.e = 100%

 $[\alpha]_D = +22$ (c 0.8, $CHCl_3$)

Source of Chirality: S-methyl-3-hydroxy-2-methylpropionate and resolution of diastereomers.

Absolute configuration - 5R,6R.

V. Conte, F. Di Furia*, G. Licini, G. Sbampato, G. Modena* and G. Valle

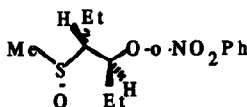
 $C_{28}H_{28}O_2SSi$

1-Methyl-2-(methylsulfinyl)-2-phenyl-1-(triphenylsilyloxy)ethane

e.e. > 98% [by 1H NMR in the presence of (S)-(+)-2,2,2-trifluoro-1-(9-anthryl)-ethanol] $[\alpha]_D^{25} = +19.6$ (c=1.1, chloroform)Source of chirality: oxidation with $Ti(i-PrO)_4/(+)$ -DET/alkylhydroperoxide

Absolute configuration 1-R, 2-S, S-R: (determined by correlation of its X-ray struct. with known abs. config. of derivatives)

V. Conte, F. Di Furia*, G. Licini, G. Sbampato, G. Modena* and G. Valle

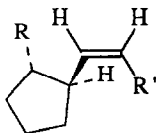
 $C_{14}H_{19}NO_5S$

3-(methylsulfinyl)-4-hexyl-o-nitrobenzoate

e.e. > 98% [by 1H NMR in the presence of (S)-(+)-2,2,2-trifluoro-1-(9-anthryl)-ethanol] $[\alpha]_D^{25} = -104.8$ (c=1.1, chloroform)Source of chirality: oxidation with $Ti(i-PrO)_4/(+)$ -DET/alkylhydroperoxide

Proposed absolute configuration: 1-R, 2-S, S-R (by correlation of its X-ray struct. with gc elution order of derivatives determined on a Ni(II)-bis-[1-S-(+)-heptafluorobutanoyl-10-ethylidencamphorate]-coated capillary column)

H. C. Brown, R. R. Iyer, V. K. Mahindroo, N. G. Bhat

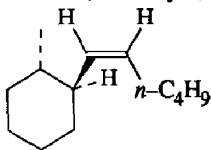
 $\geq 99\%$ ee.1) $[\alpha]^{23}D + 25.1$ (c 0.45, MeOH)2) $[\alpha]^{23}D + 86.0$ (c 2.50, MeOH)Source of chirality: (R)-(+)- α -pinene

Absolute configuration: 1) 1'S, 2'S

2) 1'S, 2'S

1) R = CH_3 , R' = $n-C_4H_9$ (Z)-1-(1'S, 2'S-trans-methylcyclopentyl)-2-butylethylene2) R = Ph, R' = $-C(CH_3)_3$ (Z)-1-(t-butyl)-2-(1'S, 2'S-trans-phenylcyclopentyl)ethylene

H. C. Brown, R. R. Iyer, V. K. Mahindroo, N. G. Bhat



≥ 99% ee.

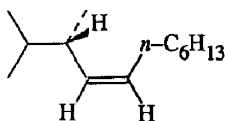
$[\alpha]^{23}_D + 35.7$ (c 3.5, MeOH)

Source of chirality : (*R*)-(+)- α -pinene

Absolute configuration : 1'*S*, 2'*S*

(*Z*)-1-(1'*S*, 2'*S*-*trans*-methylcyclohexyl)-2-butylethylene

H. C. Brown, R. R. Iyer, V. K. Mahindroo, N. G. Bhat



≥ 99% ee.

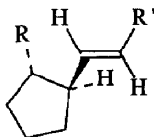
$[\alpha]^{23}_D + 28.5$ (c 3.05, MeOH)

Source of chirality : (*R*)-(+)- α -pinene

Absolute configuration : *S*

(*S*)-(*Z*)-2,3-dimethyl-4-undecene

H. C. Brown, R. R. Iyer, V. K. Mahindroo, N. G. Bhat



≥ 99% ee.

1) $[\alpha]^{23}_D + 25.5$ (c 4.90, MeOH)

2) $[\alpha]^{23}_D + 86.8$ (c 4.65, MeOH)

Source of chirality : (*R*)-(+)- α -pinene

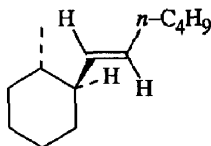
Absolute configuration : 1) 1'*S*, 2'*S*

2) 1'*S*, 2'*S*

1) R = CH₃, R' = *n*-C₄H₉ (*E*)-1-(1'*S*, 2'*S*-*trans*-methylcyclopentyl)-2-butylethylene

2) R = Ph, R' = -C(CH₃)₃ (*E*)-1-(*t*-butyl)-2-(1'*S*, 2'*S*-*trans*-phenylcyclopentyl)ethylene

H. C. Brown, R. R. Iyer, V. K. Mahindroo, N. G. Bhat



≥ 99% ee.

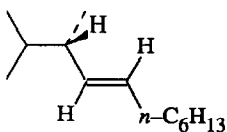
$[\alpha]^{23}_D + 35.8$ (c 3.05, MeOH)

Source of chirality : (*R*)-(+)- α -pinene

Absolute configuration : 1'*S*, 2'*S*

(*E*)-1-(1'*S*, 2'*S*-*trans*-methylcyclohexyl)-2-butylethylene

H. C. Brown, R. R. Iyer, V. K. Mahindroo, N. G. Bhat



(*S*)-(*E*)-2,3-dimethyl-4-undecene

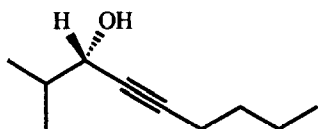
≥ 99% ee.

$[\alpha]_D^{23} + 28.5$ (*c* 2.0, MeOH)

Source of chirality: (*R*)-(+)- α -pinene

Absolute configuration: *S*

M. Falorni, G. Giacomelli, M. Marchetti, N. Culeddu and L. Lardicci



$C_{10}H_{18}O$

2-Methyl-4-nonyn-3-ol

E.e. = 82% [by nmr with (*R*)-2-methoxy-2-tri-fluoromethyl-2-phenylacetic ester]

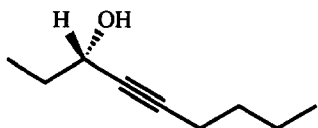
$[\alpha]_D^{25} = -6.70$ (neat)

b.p. 90°C/25 Torr

Source of chirality: asymmetric reduction of 2-methyl-4-nonyn-3-one

Absolute configuration: 2*S*

M. Falorni, G. Giacomelli, M. Marchetti, N. Culeddu and L. Lardicci



$C_9H_{16}O$

4-Nonyn-3-ol

E.e. = 66% [by nmr with (*R*)-2-methoxy-2-tri-fluoromethyl-2-phenylacetic ester]

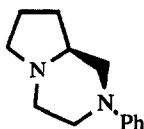
$[\alpha]_D^{25} = -10.30$ (*c* 2, hexane)

b.p. 78°C/25 Torr

Source of chirality: asymmetric reduction of 4-nonyn-3-one

Absolute configuration: 2*S*

M. Falorni, G. Giacomelli, M. Marchetti, N. Culeddu and L. Lardicci



$C_{13}H_{18}N_2$

4-Phenyl-1,4-diaza[4.3.0]bicyclononane

E.e. = about 99%

$[\alpha]_D^{25} = +17.7$ (*c* 1.5, Et₂O)

b.p. 118-125°C/0.07 Torr

Source of chirality: natural (*S*)- proline

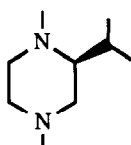
Absolute configuration: 2*S*

Use: chiral ligand in asymmetric synthesis

See: M. Falorni et. al. *Tetrahedron Lett.*, 1989, 30, 3551.

M. Falorni, G. Giacomelli, M. Marchetti, N. Culeddu and L. Lardicci

Tetrahedron: Asymmetry **1991**, *2*, 287



2-Isopropyl-1,4-dimethylpiperazine

E.e. = about 99%

$[\alpha]_D^{25} = +60.50$ (c 2, Et₂O)

b.p. 175-180°C

Source of chirality: natural (S)- valine

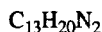
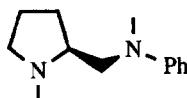
Absolute configuration: 2S

Use: chiral ligand in asymmetric synthesis

See: M. Falorni et. al. *Gazz. Chim. Ital.*, **1990**, *120*, 765.

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Tetrahedron: Asymmetry **1991**, *2*, 287



N,N-Dimethyl-2-(anilinomethyl)pyrrolidine

E.e. = about 99%

$[\alpha]_D^{25} = -115.56$ (c 2, Et₂O)

b.p. 106-110°C/0.01 Torr

Source of chirality: natural (S)- proline

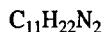
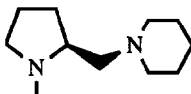
Absolute configuration: 2S

Use: chiral ligand in asymmetric synthesis

See: T. Mukaiyama *Tetrahedron*, **1981**, *37*, 4111.

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Tetrahedron: Asymmetry **1991**, *2*, 287



1-Methyl-2-(piperidinomethyl)pyrrolidine

E.e. = about 99%

$[\alpha]_D^{25} = -46.0$ (c 2, EtOH)

b.p. 67°C/0.02 Torr

Source of chirality: natural (S)- proline

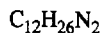
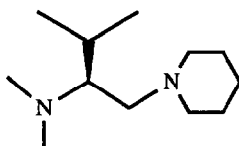
Absolute configuration: 2S

Use: chiral ligand in asymmetric synthesis

See: T. Mukaiyama et al. *Chem. Lett.*, **1984**, 2071.

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Tetrahedron: Asymmetry **1991**, *2*, 287



1-Piperidyl-2-(*N,N*-dimethylamino)-3-methylbutane

E.e. = about 99%

$[\alpha]_D^{25} = +19.27$ (c 2, Et₂O)

b.p. 60-65°C/0.01 Torr

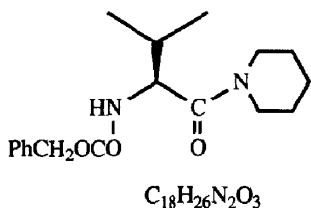
Source of chirality: natural (S)- valine

Absolute configuration: 2S

Use: chiral ligand in asymmetric synthesis

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Tetrahedron: Asymmetry **1991**, *2*, 287

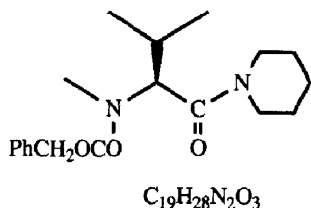


E.e. = about 99%
 $[\alpha]_D^{25} = +22.53$ (c 3, $CHCl_3$)
b.p. 200-205°C/0.01 Torr
Source of chirality: natural (*S*)- valine
Absolute configuration: 2*S*
Intermediate in chiral ligand synthesis

N-(Benzoyloxycarbonyl)valine piperidylamide

M. Falorni, G. Giacomelli, M. Marchetti, N. Culeddu and L. Lardicci

Tetrahedron: Asymmetry **1991**, *2*, 287

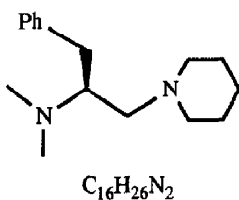


E.e. = about 99% [by nmr with (*R*)-2-methoxy-2-trifluoromethyl-2-phenylacetic amide of the *N*-deprotected derivative]
 $[\alpha]_D^{25} = -78.83$ (c 2, $CHCl_3$)
b.p. 168-175°C/0.01 Torr
Source of chirality: natural (*S*)- valine
Absolute configuration: 2*S*
Intermediate in the chiral ligand synthesis

N-Methyl-*N*-(benzoyloxycarbonyl)valine piperidylamide

M. Falorni, G. Giacomelli, M. Marchetti, N. Culeddu and L. Lardicci

Tetrahedron: Asymmetry **1991**, *2*, 287

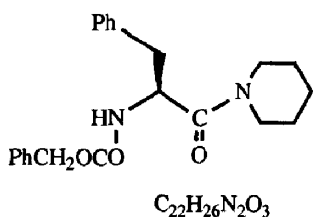


E.e. = about 99% [by nmr with (*R*)-2-methoxy-2-trifluoromethyl-2-phenylacetic amide of the *N*-unmethylated derivative]
 $[\alpha]_D^{25} = -18.69$ (c 2, Et_2O)
b.p. 105°C/0.02 Torr
Source of chirality: natural (*S*)- phenylalanine
Absolute configuration: 2*S*
Use: chiral ligand in asymmetric synthesis

1-Piperidyl-2-(*N,N*-dimethylamino)-3-phenylpropane

M. Falorni, G. Giacomelli, M. Marchetti, N. Culeddu and L. Lardicci

Tetrahedron: Asymmetry **1991**, *2*, 287

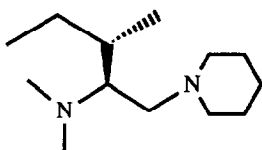


E.e. = about 99%
 $[\alpha]_D^{25} = +21.49$ (c 2, $CHCl_3$)
m.p. 66-68°C
Source of chirality: natural (*S*)- phenylalanine
Absolute configuration: 2*S*
Intermediate in chiral ligand synthesis

N-(Benzoyloxycarbonyl)phenylalanine piperidylamide

M. Falorni, G. Giacomelli, M. Marchetti, N. Culeddu and L. Lardicci

Tetrahedron: Asymmetry **1991**, *2*, 287



$C_{13}H_{28}N_2$

1-Piperidyl-2-(*N,N*-dimethylamino)-3-methylpentane

E.e. = about 99%

$[\alpha]_D^{25} = -5.61$ (*c* 1, Et₂O)

b.p. 60-65°C/0.02 Torr

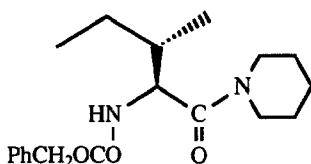
Source of chirality: natural (*2S*, *3S*)- isoleucine

Absolute configuration: *2S*, *3S*

Use: chiral ligand in asymmetric synthesis

M. Falorni, G. Giacomelli, M. Marchetti, N. Culeddu and L. Lardicci

Tetrahedron: Asymmetry **1991**, *2*, 287



$C_{19}H_{28}N_2O_3$

N-(Benzyloxycarbonyl)isoleucine piperidylamide

E.e. = about 99%

$[\alpha]_D^{25} = +11.03$ (*c* 2, CHCl₃)

b.p. 220°C/0.05 Torr

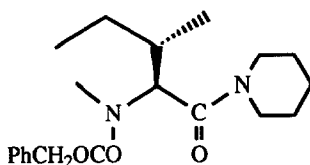
Source of chirality: natural (*2S*, *3S*)- isoleucine

Absolute configuration: *2S*, *3S*

Intermediate in chiral ligand synthesis

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Tetrahedron: Asymmetry **1991**, *2*, 287



$C_{20}H_{30}N_2O_3$

N-Methyl-*N*-(benzyloxycarbonyl)isoleucine piperidylamide

E.e. = about 99% [by nmr with (*R*)-2-methoxy-2-tri-fluoromethyl-2-phenylacetic amide of the *N*-deprotected derivative]

$[\alpha]_D^{25} = -81.36$ (*c* 3, CHCl₃)

b.p. 175°C/0.02 Torr

Source of chirality: natural (*2S*, *3S*)- isoleucine

Absolute configuration: *2S*, *3S*

Intermediate in chiral ligand synthesis