Supporting information for:

A new Method for the Synthesis of Chiral β-Branched α-Amino Acids

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General Experimental

All reagents were used as purchased from commercial suppliers without further purification. The reactions were carried out in oven dried or flamed vessels and performed under argon. Solvents were dried and purified by conventional methods prior use. Et_2O and THF were freshly distilled from sodium/benzophenone and dichloromethane was distilled from CaH_2. **Caution**: the generation and handling of diazomethane requires special precautions. Flash column chromatography was performed with Merck silica gel 60, 0.040-0.063 mm (230-400 mesh).[1] Merck aluminum backed plates pre-coated with silica gel 60 (UV_{254}) were used for thin layer chromatography and were visualized by staining with $KMnO_4$.

^1H, ^{13}C, ^{31}P NMR spectra were recorded on Brüker 400/300/200 spectrometers. Conditions are specified for each spectrum (temperature 25 °C unless specified). Chemical shifts (δ) are given in ppm relative to the resonance of their respective solvent peak. Infrared spectra were taken with a Nicolet 380 FT-IR. GC analysis were conducted with a 6890N chromatograph from Agilent Technologies™ using a SUPLECOWAX™-10 FUSED SILICA Capillary Column 30m x 0.25 mm x 0.25 μm film thickness. The inlet temperature was set at 200 °C, gas carrier He (53.6 mL/min). Pressure 12.04 psi, flow 1mL/min. Detector FID 250 °C, H_2 flow 30 mL/min, air flow 406 mL/min, He 1 mL/min (see table below for retention times and gradients). Elemental analyses were performed at the *Institut für Organische Chemie*, Freiburg in Breisgau. High and low resolution mass spectroscopy analyses were conducted by the *Institut Fédératif de Recherche 85* at the Louis Pasteur University, Strasbourg. Melting points were determined on a Gallenkamp melting point apparatus. Specific rotations were measured with a Perkin-Elmer apparatus: values are given in 10^{-1} deg.cm^2.g^{-1}.

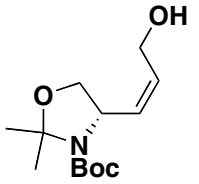
[1] W. Clark Still, M. Kahn, A. Mitra, *J. Org. Chem.*, **1978**, *43* (14), 2923-2925

Experimental Procedures and spectroscopic data for compounds

***tert*-butyl-(4*S*)-4-[(1*E*)-3-ethoxy-3-oxoprop-1-enyl]-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (5)** was exactly synthesized as described by Dondoni et *al*.[2]

***tert*-butyl-(4*S*)-4-[(1*Z*)-3-methoxy-3-oxoprop-1-enyl]-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (6)** was exactly synthesized as described by L. Devel et *al*.[3]

***tert*-butyl-(4*S*)–4-[(1*E*)-3-hydroxyprop-1-enyl]-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (3)** was exactly synthesized as described by L. Devel et *al*.[3]



$C_{13}H_{23}NO_4$
Mol. Wt.: 257,33

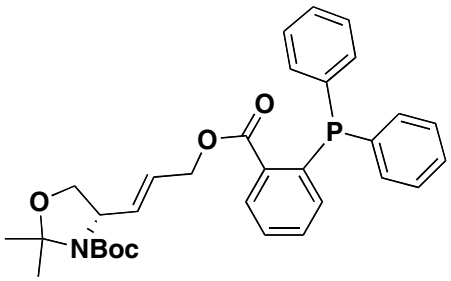
***tert*-butyl-(4*S*)-4-[(1*Z*)-3-hydroxyprop-1-enyl]-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (4).** In a dry round bottomed flask under argon was introduced the unsaturated ester **6** (300 mg, 1.05 mmol) and anhydrous THF (3 mL). The solution was cooled at 0 °C using an ice bath and a solution of DIBAL-H (2.48 mL, 1.0 M in THF) was added dropwise while stirring. After 1.5 h at 0 °C the mixture was poured in MeOH (15 mL) followed after a few minutes by a solution of sodium-potassium tartrate (10 mL), water and EtOAc. The heterogeneous mixture was stirred overnight. The mixture was extracted with EtOAc and the organic layer was washed with brine before being dried over $MgSO_4$. The solvent was removed under reduced pressure. Purification by flash chromatography (1/1 EtOAc/heptane) yielded **4** as a colorless oil (251 mg, 93%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 5.85 (ddd, J = 6.5, 8.5, 10.5 Hz, 1 H), 5.53 (t, J = 10.5 Hz, 1 H), 4.90 (br dd, J = 6,5 10 Hz, 1 H), 4.42 (br dd, J = 8.5, 12 Hz, 1 H), 4.23 (br s, 0.5H), 4.14 (br s, 0.5H), 4.04 (dd, J = 6, 9 Hz, 1 H), 3.88 (m, 1 H), 3.69 (dd, J = 1.5, 9.0 Hz, 1 H), 1.57 (s, 3 H), 1.48 (s, 3

[2] Dondoni, A., Merino, P., Perrone, D. *Tetrahedron* **1993**, *49*, 2939
[3] L. Devel et al. *Carbohydrate Research*, **2003**, *338*, 1591-1601

H), 1.45 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 152.5, 154.0, 130.9, 130.2, 93.5, 81.0, 68.0, 57.6, 53.7, 28.4, 27.6, 24.8.

General procedure for the coupling of the directing group with the unsaturated alcohols **3** and **4** to form the corresponding esters **7** and **8**.

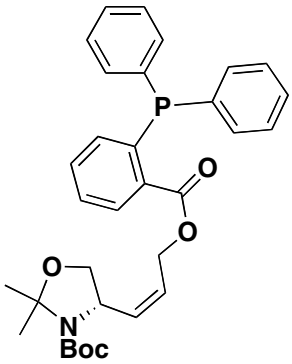
In a dry round bottomed flask under argon was introduced the unsaturated alcohol **3** or **4** (285 mg, 1.11 mmol), o-DPPBA (340 mg, 1.11 mmol), DMAP (136 mg, 1.11 mmol), DCC (229 mg, 1.11 mmol) in dry dichloromethane (5.6 mL, 0.2 M). The cloudy mixture was stirred rt overnight. Brine was added and the mixture was extracted three times with dichloromethane. The organic layer was dried over $MgSO_4$, filtrated and removed under reduced pressure. The residue was purified by flash chromatography (9/1 Heptane/EtOAc).



$C_{32}H_{36}NO_5P$
Mol. Wt.: 545,61

***tert*-butyl(4*S*)-4-((1*E*)-3-{[2-diphenylphosphino)-benzoyl]oxy}prop-1-enyl)-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (7).** 86% yield as a white solid. Mp 102 °C. $[\alpha]_D^{20}$ = + 10.7 ° (c= 1.0, $CHCl_3$). IR (neat): 2981, 1688, 1361, 1248, 1098, 1057, 961, 943, 758, 744, 521, 501 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 8.07 (m, 1 H), 7.44-7.25 (m, 12 H), 6.95 (m, 1H), 5.71 (br s, 2 H), 4.65 (d, J = 4.8 Hz, 2 H), 4.26-4.30 (2 x br s, 1 H), 4.03 (dd, J = 6.3, 9. 0 Hz, 1 H), 3.72 (dd, 1 H, J = 2.6, 8.8 Hz), 1.6 (s, 3 H), 1.51 (s, 3 H), 1.42 (s, 9 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5 (CO), 151.9 (CO), 140.7 (d, $J_{C,P}$ = 25.4 Hz), 138.0 (d, $J_{C,P}$ = 12.4 Hz), 134.4 (br s), 133.99 (d, $J_{C,P}$ = 20.7 Hz) 133.97 (d, $J_{C,P}$ = 20.7 Hz), 132.1 (br s), 130.7 (br s), 128.7, 128.5 (d, $J_{C,P}$ = 7.6 Hz), 128.2, 125.8, 94.1 (Cquat), 79.8 (Cquat), 68.0, 64.9, 58.7, 28.5,

26.7, 23.8. ^{31}P (162 MHz, $CDCl_3$) δ - 4.53. Elemental analysis: Calc. C (70.44%), H (6.65%), N (2.57%), found C (70.6%), H (6.94%), N (2.38%).



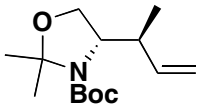
$C_{32}H_{36}NO_5P$
Mol. Wt.: 545,61

***tert*-butyl(4*S*)-4-((1*Z*)-3-{[2-(diphenylphosphino)benzoyl]oxy}prop-1-enyl)-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (8).** 94% to 97% yield as a white solid. Mp 96 °C. $[\alpha]_D^{20}$ = -70.0 ° (c= 1.0, $CHCl_3$). IR (neat): 2970, 1712, 1681, 1388, 1377, 1268, 1253, 1105, 1065, 757, 744, 696, 526, 400 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 8.07 (m, 1 H), 7.44-7.25 (m, 12 H), 6.95 (m, 1H), 5.58 (m, 2 H), 4.89-5.00 (2 x br s, 1H), 4.68 (m, 2H), 3.98 (dd, 2 H, J = 6.3, 9 Hz), 3.55 (br s, 1 H), 1.60 (s, 3H), 1.52 (s, 3H), 1.43 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 151.8, 140.6 (d, $J_{C,P}$ = 27.0 Hz), 138.0 (d, $J_{C,P}$ = 10.4 Hz), 132.1 (br s), 134.4, 134.0 (d, $J_{C,P}$ = 20.8 Hz) 133.8 (d, $J_{C,P}$ = 20.6 Hz), 132.1 (br s), 130.7 (br s), 128.7 (d, $J_{C,P}$ = 4.1 Hz), 128.50 (d, $J_{C,P}$ = 6.5 Hz), 128.48 (d, $J_{C,P}$ = 7.1 Hz), 128.2, 125.2, 123.6, 94.2/93.5 (rotamers, Cquat), 80.1/79.9 (rotamers, Cquat), 68.6/68.3 (rotamers), 61.0/60.6 (rotamers), 54.5/54.3 (rotamers), 28.5 (tBu), 27.5/26.6 (rotamers), 25.0/23.9 (rotamers).^{31}P NMR (162 MHz, $CDCl_3$) δ -4.53. Elemental analysis: Calc. C (70.44%), H (6.65%), N (2.57%), found C (70.21%), H (6.67%), N (2.53%).

General procedure for allylic substitutions

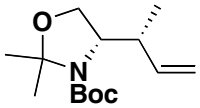
In a dry round bottomed flask under argon was introduced **7** or **8** (80 mg, 0.146 mmol) in anhydrous Et_2O (14.6 mL, 0.01 M) followed by CuBr-Me_2S (15 mg, 0.073 mmol). The mixture was stirred at rt until the copper salt was completely dissolved (ca 10 min) to yield a clear light yellow solution. Then the alkylmagnesium bromide in ether at 0.1 M (3 mL) was added via a

syringe pump (rate: 3 mL/h) at room temperature. The mixture was stirred for an additional 1 hour and NH_4Cl_{sat} was added and the extraction with EtOAc was performed. The organic layer was dried over $MgSO_4$, filtrated and the solvent removed under reduced pressure. The residue was purified by flash chromatography (9/1 Heptane/EtOAc).



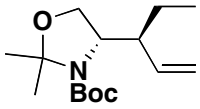
$C_{14}H_{25}NO_3$
Mol. Wt.: 255,35

***tert*-butyl-(4*S*)-2,2-dimethyl-4-[(1*R*)-1-methylprop-2-enyl]-1,3-oxazolidine-3-carboxylate (major diastereomer) (1a)**. IR (neat): 2926, 1695, 1374, 1364, 1254, 1175, 1084, 1054, 849 cm^{-1} . ^1H NMR (300 MHz, 50 °C, $CDCl_3$): δ 5.82 (m, 1 H), 5.08-5.02 (m, 2 H), 3.94-3.81 (m, 3 H), 2.75 (br m, 1 H), 1.60 (s, 3 H), 1.49 (s, 9 H, tBu), 1.46 (s, 3 H), 1.01 (d, J = 7.18 Hz, 3 H).^{13}C NMR (75 MHz, 50 °C, $CDCl_3$): δ 152.6 (CO), 141.1, 114.7, 94.1, 79.8, 68.7, 61.1, 39.9, 28.6 (tBu), 26.7 (br s), 24.0 (br s), 14.2. HRMS (ESI positive): calculated 255.1834, 256.1868, 257.1902, found (M+1) 256.1903, 257.1935, 258.1925.



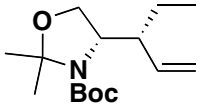
$C_{14}H_{25}NO_3$
Mol. Wt.: 255,35

***tert*-butyl-(4*S*)-2,2-dimethyl-4-[(1*S*)-1-methylprop-2-enyl]-1,3-oxazolidine-3-carboxylate (major diastereomer) (2a).** IR (neat): 2926, 1694, 1384, 1364, 1254, 1175, 1085, 1056, 543 cm^{-1}.^1H NMR (300 MHz, 50 °C, $CDCl_3$) δ 5.78 (m, 1 H), 5.04 (m, 2 H), 3.87 (m, 3H), 2.75 (br m, 1 H), 1.61 (s, 3 H), 1.55 (s, 3 H), 1.49 (s, 9 H), 1.01 (d, J = 7. 18 Hz, 3 H).^{13}C NMR (50 MHz, $CDCl_3$) δ 152.9 (CO), 140.0, 116.5, 94.5, 80.3, 65.4, 65.0, 57.1, 39.4, 28.7 (tBu), 17.2. HRMS (ESI positive): calculated 255.1834, 256.1868, 257.1902, found (M+1) 256.1901, 257.1928, 258.1736.



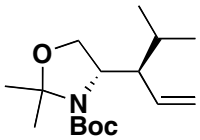
$C_{15}H_{27}NO_3$
Mol. Wt.: 269,38

***tert*-butyl-(4*S*)-4-[(1*S*)-1-ethylprop-2-enyl]-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (major diastereomer) (1b).** IR (neat): 2930, 1694, 1384, 1363, 1255, 1174, 1089, 847, 767 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 5.65 (m, 1 H), 5.14-5.00 (m, 2 H), 3.92-3.81 (m, 3 H), 2.39 (m, 1 H), 1.59 (s, 3 H), 1.53 (s, 9 H), 1.46 (s, 3 H), 1.18 (m, 2 H), 0.88 (t, J= 7.49 Hz, 3 H).^{13}C NMR (75 MHz, $CDCl_3$) δ 152.9/152.4 (rotamers) (CO), 139.5, 117.2/116.7 (rotamers), 94.2/93.6 (rotamers) (Cquat), 80.0/79.7 (rotamers) (Cquat), 65.7, 61.0/60.9 (rotamers), 49.73/49.66 (rotamers), 29.6 (tBu), 27.2/26.4 (rotamers), 24.6, 23.1/22.7 (rotamers), 12.2. HRMS (ESI positive): calculated 269.1991 found (M+1): 270.2064



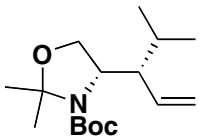
$C_{15}H_{27}NO_3$
Mol. Wt.: 269,38

***tert*-butyl-(4*S*)-4-[(1*R*)-1-ethylprop-2-enyl]-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (major diastereomer) (2b).** IR (neat): 2928, 1698, 1387, 1365, 1259, 1176, 1091 cm^{-1}.^1H NMR (300 MHz, 50 °C, $CDCl_3$) δ 5.67-5.55 (m, 1 H), 5.13-5.00 (m, 2 H), 3.87-3.80 (m, 3 H), 2.46 (br m, 1 H), 1.55 (s, 3 H), 1.48 (s, 9 H), 1.46 (s, 3 H), 1.26 (m, 2 H), 0.89 (t, J= 7.49 Hz, 3 H).^{13}C NMR (75 MHz, $CDCl_3$) δ 152.8/152.4 (rotamers) (CO), 138.7, 117.7, 94.2/93.2 (rotamers) (Cquat), 80.0/79.7 (rotamers) (Cquat), 64.7/64.3 (rotamers), 61.1/60.5 (rotamers), 49.0/47.9 (rotamers), 28.6 (tBu), 26.9/26.3 (rotamers), 24.8, 24.5/24.3, 12.3. HRMS (ESI positive): calculated 269.1991 (100.0%), 270.2024 (16.7%), 271.2058 (1.3%) found (M+1) 270.2064, 271.2096, 272.2122.



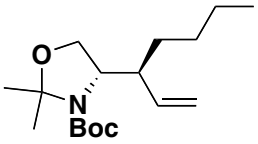
$C_{16}H_{29}NO_3$
Mol. Wt.: 283,41

***tert*-butyl-(4*S*)-4-[(1*S*)-1-isopropylprop-2-enyl]-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (major diastereomer) (1c).** IR (neat): 2929, 1694, 1384, 1364, 1253, 1173, 1089, 912, 848, 767, 545 cm^{-1}. ^1H NMR (300 MHz, 50 °C $CDCl_3$) δ 5.81 (m, 1 H), 5.14-4.87 (m, 2 H), 4.14-3.70 (m, 3 H), 2.11 (m, 1 H), 1.69 (m, 1 H), 1.55 (s, 3 H), 1.50 (s, 3 H), 1.49 (s, 9 H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.1 (CO), 136.3/135.6 (rotamers), 118.7/117.6 (rotamers), 94.0/93.4 (Cquat) (rotamers), 79.8 (Cquat), 67.6/67.1 (rotamers), 59.0/58.9 (rotamers), 55.8/55.2 (rotamers), 29.8, 29.0/28.6 (rotamers) (tBu), 27.5/26.8 (rotamers), 24.8, 23.3, 22.3, 21.3, 17.0, 16.6. HRMS (ESI positive): calculated 283.2147 (100.0%), 284.2181 (17.8%), 285.2215 (1.5%), found (M+1) 284.2220, 285.2253, 286.2279.



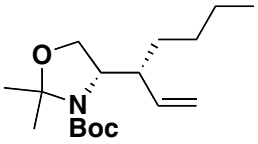
$C_{16}H_{29}NO_3$
Mol. Wt.: 283,41

***tert*-butyl-(4*S*)-4-[(1*R*)-1-isopropylprop-2-enyl]-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (major diastereomer) (2c).** IR (neat): 2930, 1695, 1382, 1253, 1175, 1098, 1054, 963, 850, 768 cm^{-1}. ^1H NMR (300 MHz, 50 °C, $CDCl_3$) δ 5.64 (ddd, J = 10.1, 10.3, 16.8 Hz, 1 H), 5.12 (dd, J = 10.3, 2.4 Hz, 1 H), 5.00 Hz (dd, J = 16.8, 2.2 Hz), 4.10 (br m, 1 H), 3.92 (m, 2 H), 2.34 (br m, 1 H), 1.56 (s, 3 H), 1.48 (s, 9 H), 1.45 (s, 3 H), 0.94 (d, J= 6.8 Hz, 3 H), 0.89 (d, J= 6.8 Hz, 3 H). ^{13}C NMR (50 MHz, $CDCl_3$) δ 152.2 (CO), 137.5/137.2 (rotamers), 118.4, 94.2/93.7 (Cquat) (rotamers), 79.9/79.7 (Cquat) (rotamers), 64.3/64.2 (rotamers), 59.3/59.1 (rotamers), 53.5/52.3 (rotamers), 52.0, 29.8, 29.2/28.8 (rotamers) (tBu), 26.7/26.0 (rotamers), 24.8, 23.2, 22.3, 21.2. HRMS (ESI positive): calculated 283.2147 (100.0%), 284.2181 (17.8%), 285.2215 (1.5%), found (M+Na) 306.2040, 307.2073, 308.2099.



$C_{17}H_{31}NO_3$
Mol. Wt.: 297,43

***tert*-butyl-(4*S*)-4-[(1*S*)-1-butylprop-2-enyl]-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (major diastereomer) (1d).** IR (neat): 2928, 1694, 1385, 1363, 1252, 1175, 1087, 912, 848, 767, 544 cm^{-1}.^{1}H NMR (300 MHz, 50 °C, $CDCl_3$) δ 5.64-5.55 (m, 1 H), 5.10-4.98 (m, 2 H), 3.91-3.79 (m, 3 H), 2.42 (m, 1 H), 1.58 (s, 3 H), 1.48 (s, 12 H), 1.34-1.26 (m, 6 H), 0.87 (t, J= 6.6 Hz, 3 H).^{13}C NMR (50 MHz, $CDCl_3$) δ 152.4 (CO), 139.9, 117.0/116.4 (rotamers), 94.2/93.6 (rotamers) (Cquat), 80.0/79.7 (Cquat) (rotamers), 65.6, 61.1, 47.5, 30.8, 29.8/29.7 (rotamers), 28.6 (tBu), 27.2, 26.4, 24.7, 23.1, 22.8, 14.1. HRMS (ESI positive): calculated 297.2304 (100.0%), 298.2337 (18.9%), 299.2371 (1.7%), found (M+Na) 320.2196, 321.2229, 322.2256.

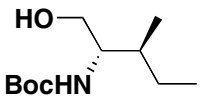


$C_{17}H_{31}NO_3$
Mol. Wt.: 297,43

***tert*-butyl-(4*S*)-4-[(1*R*)-1-butylprop-2-enyl]-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (major diastereomer) (2d).** IR (neat): 2929, 1694, 1384, 1363, 1254, 1174, 1090, 913, 847, 767 cm^{-1}. ^{1}H NMR (300 MHz, 50 °C $CDCl_3$) δ 5.64 (ddd, J = 16.8, 10.3, 9.6 Hz, 1 H), 5.11 (dd, J = 10.3, 2.1 Hz, 1 H), 4.98 (dd, J = 16.8, 2.1 Hz, 1 H), 3.87 (m, 3 H), 2.55 (br m, 1 H), 1.54 (s, 3 H), 1.48 (s, 9 H), 1.46 (s, 3 H), 1.33-1.26 (m, 6 H), 0.90 (t, J= 6.8 Hz, 3 H).^{13}C NMR (50 MHz, $CDCl_3$) δ 152.8/152.3 (CO) (rotamers), 139.1, 117.5, 94.2/93.7 (Cquat) (rotamers), 80.0/79.7 (Cquat) (rotamers), 64.7/64.3 (rotamers), 61.2/60.8 (rotamers), 46.9/46.1 (rotamers), 31.3/31.1 (rotamers), 30.9, 28.6 (tBu), 26.9, 26.3, 24.8, 23.2, 22.8, 14.1. HRMS (ESI positive): calculated 297.2304 (100.0%), 298.2337 (18.9%), 299.2371 (1.7%), found (M+1) 298.2377, 299.2410, 300.2436.

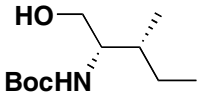
General procedure for the reduction of N-Boc protected isoleucines to the corresponding alcohol.

To a solution of the N-Boc protected isoleucine (1.39 g, 6.01 mmol) in THF (6 mL) cooled at -15 °C was successively added N-methyl morpholine (0.661 mL, 6.01 mmol) and iBuO-CO-Cl (0.780 mL, 6.01 mmol). After 1 min the precipitate was filtrated off and washed with THF (5 x 1 mL). The filtrates were combined in an erlenmeyer flask placed at -15 °C. A solution of $NaBH_4$ (342 mg, 9.02 mmol) in water (3 mL) was added in one portion and an evolution of CO_2 was observed. After 30 sec. additional water was added followed by an extraction with EtOAc. The organic layer was dried over $MgSO_4$, filtrated and the solvents were removed under reduced pressure. Purification by flash chromatography (1/1 Heptane/EtOAc).



$C_{11}H_{23}NO_3$
Mol. Wt.: 217,31

***tert*-butyl-(1*S*,2*S*)-1-(hydroxymethyl)-2-methylbutylcarbamate.** 84% yield as a colorless oil. IR (neat): 3338, 2964, 1682, 1505, 1365, 1167, 1048, 570 cm^{-1}. ^1H NMR (300 MHz, 50 °C, $CDCl_3$): δ 4.68 (br s, 1 H), 3.71 (dd, J = 3.43, 10.92 Hz, 1 H), 3.60 (dd, J = 6.24, 10.92 Hz, 1 H), 3.48 (m, 1 H), 2.23 (br s, 1 H), 1.60-1.46 (m, 2 H), 1.44 (s, 9 H, tBu), 1.15 (m, 1 H), 0.91 (d, J= 6.86 Hz, 3 H), 0.90 (t, J= 7.3 Hz, 3 H).^{13}C NMR (50 MHz, $CDCl_3$): δ 156.9 (CO), 79.6, 63.8, 57.0, 36.2, 28.5 (tBu), 25.5, 15.6, 11.6. HRMS (ESI positive): calculated 217.1678, 218.1711 found (M+1) 218.1751, 219.1783.



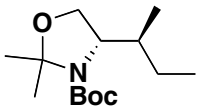
$C_{11}H_{23}NO_3$
Mol. Wt.: 217,31

***tert*-butyl-(1*S*,2*R*)-1-(hydroxymethyl)-2-methylbutylcarbamate.** 82% yield as a waxy solid. IR (neat): 3219, 2965, 1678, 1558, 1287, 1171, 1090, 647 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 4.75 (br s, 1 H), 3.58 (br m, 3 H), 3.01 (br s, 1 H), 1.59 (m, 1 H), 1.41 (s, 9 H), 1.20 (m, 1 H), 0.88 (t,

J= 7.2 Hz), 0.85 (d, J= 6.8 Hz). ^{13}C NMR (50 MHz, $CDCl_3$) δ 157.0 (CO), 79.6 (C_{quat}), 64.4, 56.2, 35.7, 28.5, 26.4, 14.6, 11.6. HRMS (ESI positive): calculated 217.1678, 218.1711 found (M+1) 218.1751, 219.1783.

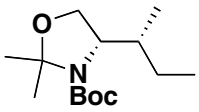
General procedure for the formation of the oxazolidine moiety from N-Boc isoleucinols

N-Boc-L-isoleucinol or N-Boc-L-alloisoleucinol (310 mg, 1.43 mmol) was dissolved in dry acetone (6 mL) and 2,2 dimethoxypropane (2.14 mL, 17.40 mmol) was added followed by the addition of BF_3-Et_2O (0.023 mL, 0.128 mmol). The solution became yellow and was stirred for 2 h at rt. NEt_3 was added and the solvent was removed under reduced pressure. Purification by flash chromatography (9/1 Heptane/EtOAc).



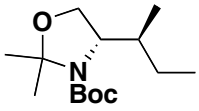
$C_{14}H_{27}NO_3$
Mol. Wt.: 257,37

***tert*-butyl-(4*S*)-2,2-dimethyl-4-[(1*S*)-1-methylpropyl]-1,3-oxazolidine-3-carboxylate (9a).** 85% yield as a colorless oil. $[\alpha]_D^{20}$= + 11.1 ° (c= 1.0, $CHCl_3$). IR (neat): 2964, 1693, 1374, 1174, 1084, 846 cm^{-1}. ^{1}H NMR (300 MHz, 50 °C, $CDCl_3$) δ 4.40-4.27 (m, 3 H), 2.08 (br m, 1 H), 1.63 (s, 3 H), 1.49 (s, 12 H), 1.33-1.24 (m, 1 H), 1.14-1.05 (m, 1 H), 0.83 (t, J= 8.9 Hz, 3 H), 0.75 (d, J= 8.3 Hz, 3 H).^{13}C NMR (200 MHz, $CDCl_3$) δ 152.8 (CO), 94.0/93.5 (Cquat) (rotamers), 79.9/79.6 (Cquat) (rotamers), 63.9, 61.6/60.8 (rotamers), 37.0/35.9 (rotamers), 28.6 (tBu), 26.8, 26.2, 24.5, 14.2/13.8 (rotamers), 12.3. HRMS (ESI positive): calculated 257.1991, 258.2024 found (M+1) 258.2037, 259.2059.



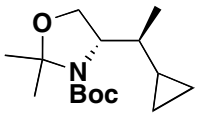
$C_{14}H_{27}NO_3$
Mol. Wt.: 257,37

***tert*-butyl (4*S*)-2,2-dimethyl-4-[(1*R*)-1-methylpropyl]-1,3-oxazolidine-3-carboxylate (9b).** 60% yield as a waxy solid. $[\alpha]_D^{20}$= + 11.4 ° (c= 0.7, $CHCl_3$). IR (neat): 2964, 1693, 1374, 1174, 1084, 846 cm^{-1}. ^1H NMR (300 MHz, 50 °C, $CDCl_3$) δ 3.91-3.77 (m, 3 H), 1.85 (m, 1 H), 1.60 (s, 3 H), 1.50 (s, 3 H), 1.49 (s, 9 H, tBu), 1.08 (m, 1 H), 0.92 (t, J = 7.2 Hz, 3 H), 0.9 (d, J = 6.9 Hz, 3 H).^{13}C NMR (75 MHz, $CDCl_3$) δ 153.2/152.8 (rotamers, CO), 94.1/93.7 (rotamers, Cquat), 80.0/79.6 (rotamers, Cquat), 65.4/64.9 (rotamers), 62.3/62.0 (rotamers), 37.7/37.1 (rotamers), 28.6 (tBu), 27.1/26.4 (rotamers), 24.5, 23.1, 15.9/15.7 (rotamers), 12.0. HRMS (ESI positive): calculated 257.1991, 258.2024 found (M+1) 258.2037, 259.2059.



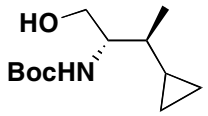
$C_{14}H_{27}NO_3$
Mol. Wt.: 257,37

***tert*-butyl-(4*S*)-2,2-dimethyl-4-[(1*S*)-1-methylpropyl]-1,3-oxazolidine-3-carboxylate (major diastereomer) (9a).** In a suitable flask was introduced **8** (80 mg, 0.31 mmol) and 9 mL of EtOAC/MeOH (1/1). Then $Pd(OH)_2/C$ 20% (62 mg) was added. The flask was purged with H_2 before setting a pressure of 5 bars for 2 h at RT. The mixture was filtrated over a celite plug. The solvent was removed under reduced pressure and the residue was purified over a silica gel column (9/1 heptane/EtOAc) to yield a colorless oil (75 mg, 95%). ^1H NMR (300 MHz, 50 °C $CDCl_3$): δ 4.40-4.27 (m, 3 H), 2.08 (br m, 1 H), 1.63 (s, 3 H), 1.49 (s, 12 H), 1.33-1.24 (m, 1 H), 1.14-1.05 (m, 1 H), 0.83 (t, J= 8.9 Hz, 3 H), 0.75 (d, J= 8.3 Hz, 3 H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 153.0 (CO), 94.2/93.7 (rotamers), 80.1/79.8 (rotamers), 64.0, 61.8/61.0 (rotamers), 37.2/36.1 (rotamers), 28.8 (tBu), 27.0, 24.7/23.1 (rotamers), 14.4/14.0 (rotamers), 12.48.



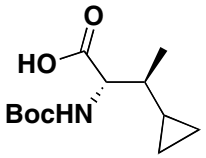
$C_{15}H_{27}NO_3$
Mol. Wt.: 269,38

***tert*-butyl-(4*S*)-4-[(1*S*)-1-cyclopropylethyl]-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (major diastereomer) (12).** In a dry flask containing **1a** (118 mg, 0.462 mmol) was added at 0 °C a dry solution of diazomethane (2.7 mmol, 12 mL) in ether. Then $Pd(OAc)_2$ (1.5 mg) was added at 0 °C in one portion and gas evolution was observed. After 10 min at 0 °C the solvent was removed under reduced pressure and the residue was purified over a silica gel column (9/1 heptane/EtOAc) to yield the desired product (124 mg, quant). ^{1}H NMR (300 MHz, 50 °C, $CDCl_3$) δ 4.07-3.88 (m, 3 H), 1.70 (m, 1 H), 1.61 (d, rotamers, 3 H), 1.49 (rotamers, 12 H), 0.98 (d, J = 7 Hz, 3 H), 0.87 (m, 1 H), 0.55-0.38 (m, 2 H), 0.24 (m, 1 H), 0.06 (m, 1 H).^{13}C NMR (50 MHz, $CDCl_3$) δ 152.8/152.6 (CO) (rotamers), 94.1/93.6 (Cquat) (rotamers), 79.9/79.5 (Cquat) (rotamers), 64.2, 61.5/61.0 (rotamers), 41.0/40.0 (rotamers), 28.6 (tBu), 26.9/26.1 (rotamers), 24.6/22.9 (rotamers), 15.2/14.9 (rotamers), 14.4, 4.2, 3.7.HRMS (ESI positive): calculated: 269.1991 (100.0%), 270.2024 (16.7%), 271.2058 (1.3%), found (M+1) 270.2064, 271.2096, 272.2122.



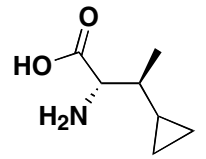
$C_{12}H_{23}NO_3$
Mol. Wt.: 229,32

***tert*-butyl-(1*S*,2*S*)-2-cyclopropyl-1-(hydroxymethyl)propylcarbamate (major diastereomer) (13).** Compound **12** (111 mg, 0.412 mmol) was dissolved in 2 mL of dry MeOH and TFA (0.4 mL, 5.15 mmol) was added at 0 °C. The mixture was stirred for 3 h at rt and DCM and Na_2CO_3sat were added to the mixture. The aqueous layer was extracted with DCM (3 times). The organic layer was dried over $MgSO_4$ and the solvent was removed under reduced pressure. The desired product was obtained as a white solid (90 mg, 95%) and needed no further purification. ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.94 (br d, J = 6.9 Hz, 1 H), 3.70-3.43 (m, 3 H), 2.90 (br s, 1 H), 1.41 (s, 9 H), 0.97 (br s, 3 H), 0.60-0.30 (m, 3 H), 0.20-0.16 (m, 1 H), 0.03- -0.04 (m, 1 H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.9 (CO), 79.5 (Cquat), 64.1, 58.0, 39.9, 28.5, 17.1, 14.4, 5.3, 2.8. HRMS (ESI positive): calculated 229.1678, found (M+Na) 252.1557



$C_{12}H_{21}NO_4$
Mol. Wt.: 243,30

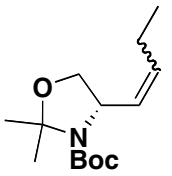
(2*S*,3*S*)-2-[(*tert*-butoxycarbonyl)amino]-3-cyclopropylbutanoic acid (major diastereomer) (14). To a solution of **13** (50 mg, 0.218 mmol) in MeCN/H_2O 0.75% (2.75 mL) was added at 0 °C in a dropwise manner 1.25 mL of a stock solution of H_5IO_6/CrO_3 (H_5IO_6, 5g, 0.22 mmol, CrO_3, 10 mg, 0.1 mmol, MeCN 49.6 mL, H_2O, 0.4 mL). After 2 h no starting material could be detected anymore by TLC. The crude mixture was diluted in toluene and a phosphate buffer (pH 5.8) was added. The organic phase was separated and dried over $MgSO_4$. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (100% EtOAc) to yield the desired compound (90 mg, 85%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (br s, 1 H), 5.22 (br d, J= 8.7 hz), 4.40 (m, 1 H), 1.46 (s, 9 H), 1.07 (d, J= 6.9 Hz, 3 H), 0.67 (m, 1 H), 0.46 (m, 2 H), 0.25 (m, 1 H), 0.08 (m, 1 H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 177.1 (CO), 155.9 (CO), 80.1, 58.1, 41.6, 16.9, 13.4, 4.4, 3.2. HRMS (ESI positive): calculated 243.1471, found (M+Na) 266.1439



$C_7H_{13}NO_2$
Mol. Wt.: 143,18

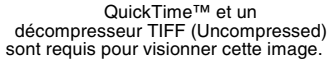
(2*S*,3*S*)-2-amino-3-cyclopropylbutanoic acid (major diastereomer) (15). In a dry flask under argon was introduced **14** (33 mg, 0.136 mmol) and anhydrous DCM (2.5 mL). Then trifluoroacetic acid (188 μL, 2.448 mmol) was added at rt. The mixture was stirred overnight and the mixture was evaporated under reduced pressure to yield the desired product as a TFA salt in quantitative yield (*Syn*/*Anti* 10/90).^{1}H NMR (400 MHz, D_2O-DMSO) δ 3.87 (d, J = 4.7 Hz, 1 H), 1.28 (m, 1 H), 0.97 (d, J = 6.7 Hz, 3 H), 0.55 (m, 1 H), 0.36 (m, 2 H), 0.08 (m, 1 H), 0.00 (m, 1

H). ^{13}C NMR (100 MHz, D_2O-DMSO) δ 173.3 (CO), 59.7, 41.0, 17.3, 14.4, 5.6, 4.4 ; LRMS (ESI positive): calculated 143.1, 144.1 found (M+1) 144.1, 145.0.



$C_{14}H_{25}NO_3$
Mol. Wt.: 255,35

***tert*-butyl-(4*S*)-4-[(1*Z*)-but-1-enyl]-2,2-dimethyl-1,3-oxazolidine-3-carboxylate (10/11).** In a flamed round-bottomed flask under argon was introduced n-propyltriphenylphosphonium bromide (472 mg, 1.195 mmol) and dry THF (2.00 mL). The suspension was cooled at -78 °C and a solution of nBuLi (1.6 M in hexane, 0.490 mL, 0.794 mmol) was added dropwise (orange color). The solution was stirred at RT for 1 h and was again cooled at -78 °C. A solution of Garner's aldehyde (R) (200 mg, 0.872 mmol) in dry THF (0.2 mL) was added dropwise and the mixture was allowed to rise to RT over a period of 30 min and was stirred for an additional 2 h. NH_4Cl_{sat} was added and the mixture was extracted with ether. The organic layer was dried over $MgSO_4$ and the solvent was removed under reduced pressure. The residue was purified over a silica gel column (9/1 Heptane/EtOAc) to yield 182 mg (90%, E/Z:7/93) of a colorless oil. ^1H NMR (300 MHz, 50 °C $CDCl_3$): 5.36 (m, 2 H), 4.56 (bs, 1 H), 4.01 (dd, J = 8.7, 6.2 Hz, 1 H), 3.58 (dd, J = 8.4, 3.4 Hz, 1 H), 2.09 (m, 2 H), 1.54 (s, 3 H, Me), 1.48 (s, 3 H, Me), 1.41 (s, 9 H, tBu), 0.95 (t, J = 7.8 Hz, 3 H, Me); ^{13}C NMR (75 MHz, 50°C $CDCl_3$): 152.3, 132.8 (bs), 129.8 (bs), 94.0 (Cquat), 79.9 (Cquat), 69.2 (bs), 54.9, 28.8/28.7 (rotamers), 27.1 (bs), 24.8 (bs), 21.0, 14.6/14.5 (rotamers).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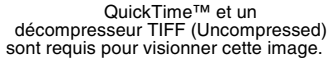
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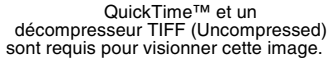
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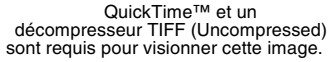
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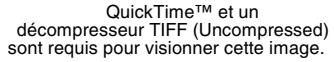
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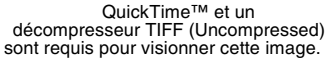
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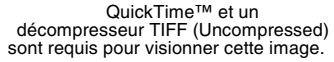
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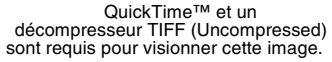
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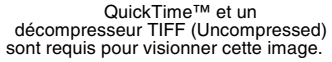
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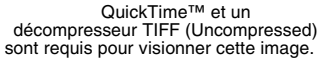
QuickTime™ et un
décompresseur TIFF (Uncompressed)
sont requis pour visionner cette image.


QuickTime™ et un
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sont requis pour visionner cette image.


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décompresseur TIFF (Uncompressed)
sont requis pour visionner cette image.


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QuickTime™ et un
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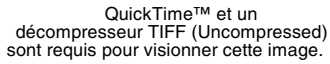
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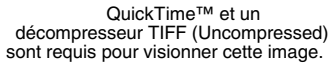
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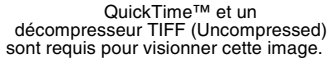
QuickTime™ et un
décompresseur TIFF (Uncompressed)
sont requis pour visionner cette image.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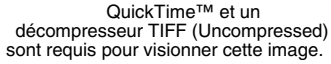
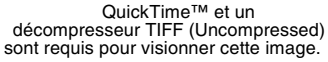

QuickTime™ et un
décompresseur TIFF (Uncompressed)
sont requis pour visionner cette image.


QuickTime™ et un
décompresseur TIFF (Uncompressed)
sont requis pour visionner cette image.


QuickTime™ et un
décompresseur TIFF (Uncompressed)
sont requis pour visionner cette image.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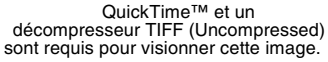
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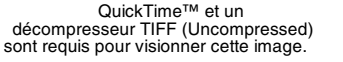
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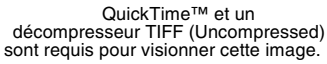
sont requis pour visionner cette image.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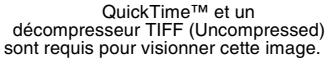
QuickTime™ et un
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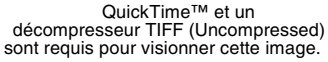

QuickTime™ et un
décompresseur TIFF (Uncompressed)
sont requis pour visionner cette image.

HPLC analysis: Chiralpak AD-H, nHeptan/EtOH 97/3, UV absorption at 230 nm: Retention Times: compound **7** = 10.25 min., compound **8** = 8.19 min. Both compounds were free of each other's isomer.

D-7000 HSM: Spangenberg Series: 0016 Report: original System: Sys 2

Universität Freiburg
Inst. für Organische Chemie und Biochemie

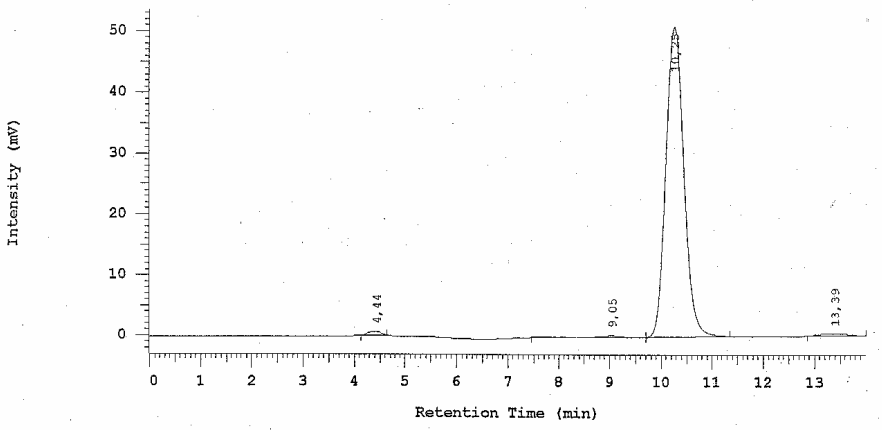
Analyzed: 25.01.06 15:18
Reported: 25.01.06 15:33
Processed: 25.01.06 15:33

Data Path: D:\Spangenberg\DATA\0016\
Processing Method: AD-H nHeptan/EtOH 97/3 230nm

System(acquisition): Sys 2
Application: Spangenberg
Sample Name: E isomer
Injection from this vial: 1 of 1
Sample Description:

Series:0016
Vial Number: 1
Vial Type: UNK
Volume: 1,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: AD-H nHeptan/EtOH 97/3 230nm
Column Type: Chiralpak AD-H Developed by: fehrenbach
Pump A Type: L-7100
Solvent A: Heptan Solvent B: i-Propanol
Solvent C: Äthanol Solvent D: Äthanol
Method Description: n-Heptan 97%, EtOH 3 %, 230nm, 0.8ml/min

Chrom Type: HPLC Channel : 1

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area %	Area	BC
1	4,44	0,864	10802	BB
2	9,05	0,711	8887	BB
3	10,25	97,216	1215292	BB
4	13,39	1,209	15118	BB
		100,000	1250099	

Peak rejection level: 0

Page Indicator 1

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Inst. für Organische Chemie und Biochemie

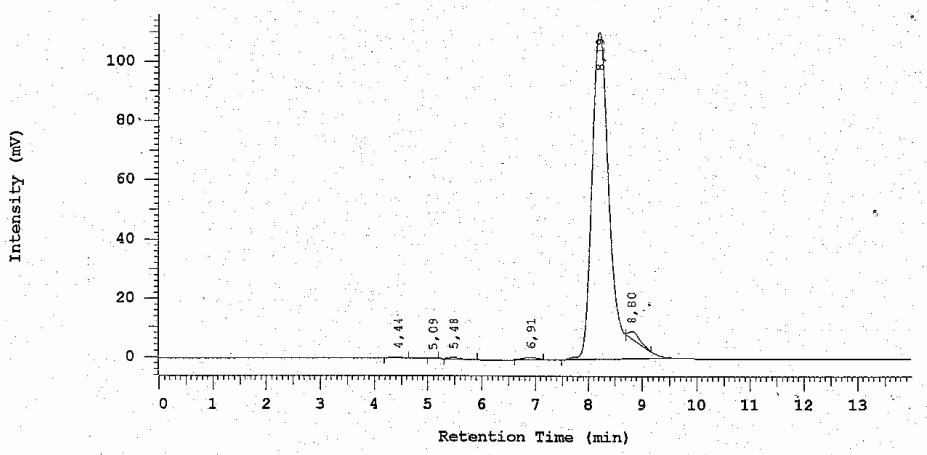
Analyzed: 25.01.06 15:02
Reported: 25.01.06 15:17
Processed: 25.01.06 15:17

Data Path: D:\Spangenberg\DATA\0015\
Processing Method: AD-H nHeptan/EtOH 97/3 230nm

System(acquisition): Sys 2
Application: Spangenberg
Sample Name: Z isomer
Injection from this vial: 1 of 1
Sample Description:

Series:0015
Vial Number: 1
Vial Type: UNK
Volume: 1,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: AD-H nHeptan/EtOH 97/3 230nm
Column Type: Chiralpak AD-H Developed by: fehrenbach
Pump A Type: L-7100
Solvent A: Heptan Solvent B: i-Propanol
Solvent C: Äthanol Solvent D: Äthanol
Method Description: n-Heptan 97%, EtOH 3 %, 230nm, 0.8ml/min

Chrom Type: HPLC Channel : 1

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area %	Area	BC
1	4,44	0,193	4936	BB
2	5,09	0,032	821	BB
3	5,48	0,322	8222	BB
4	6,91	0,451	11515	BB
5	8,19	97,686	2494130	BV
6	8,80	1,316	33589	TBB
		100,000	2553213	

Peak rejection level: 0

Page Indicator 1

GC Chromatography

Column temperature gradients:

Method 1: Column: initial 50 °C for 3 min, 15 °C/min until 200°C, isotherm for 5 min, 20 °C/min until 250 °C, isotherm for 10 min.

Method 2: Column: initial 140 °C for 10 min, 10 °C/min until 180°C then 20 °C/min until 250 °C, isotherm for 5 min.

Method 3: Column: initial 150 °C for 25 min then 20 °C/min until 250 °C, isotherm for 5 min.

Method 4: Column: initial 100 °C for 25 min, 20 °C/min until 250°C, isotherm for 5 min.

Method 5: Column: initial 120 °C for 3 min, 10 °C/min until 200°C, isotherm for 5 min, 20 °C/min until 250 °C, isotherm for 5 min.

GC retention times of 1a and 2a correlated with 9a, 9b, 10 and 11

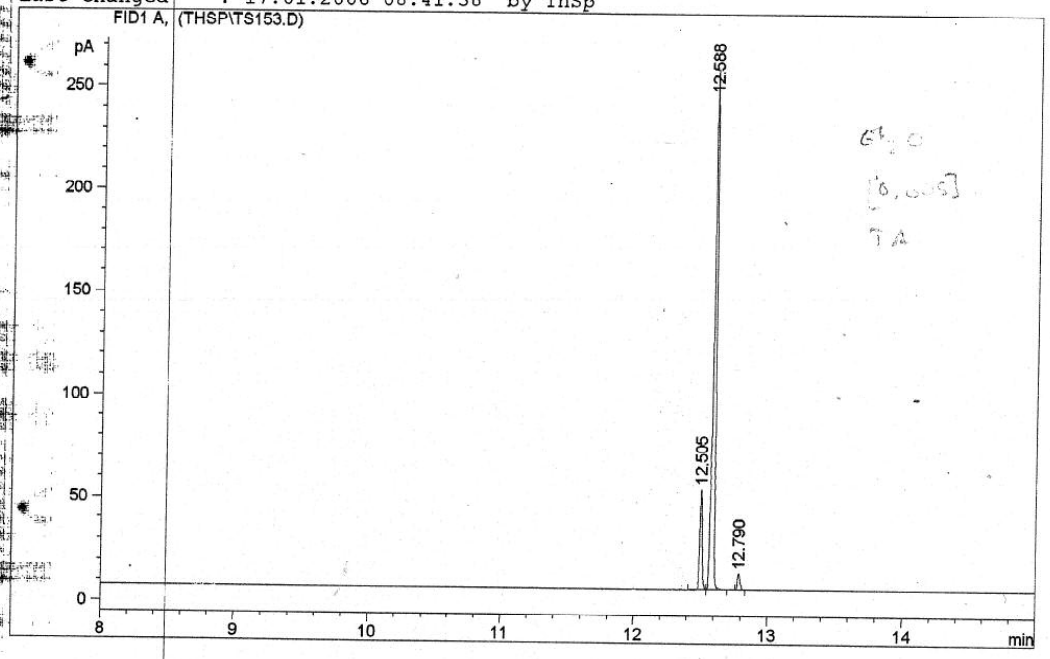
Product(s)	**Method**	**RT(min) *Syn***	**RT(min) *Anti***	**RT (min) S_N2**
From iLeu	1	-	12.484 (**9a**)	-
From *allo*-iLeu	1	12.403 (**9b**)	-	-
Wittig S_N2	1	-	-	12.547 (**10E**) 12.747 (**11Z**)
10 / 11	5	-	-	8.272 (**10E**) 8.492 (**11Z**)
Hydrogenation of entry 3	1	12.399 (**9b**)	12.483 (**9a**)	12.654
Entry 3, table 1	1	12.505 (**2a**)	12.588 (**1a**)	12.790

GC retention times of products 1a-d and 2a-d associated with Table 2

Product(s)	**Method**	**RT(min) *Syn***	**RT(min) *Anti***	**RT (min) S_N2**
Me	1	12.505 (**2a**)	12.588 (**1a**)	12.790 (**10E**) 12.851 (**11Z**)
	5	8.242 (**2a**)	8.361 (**1a**)	8.720 (**10E**) 8.921 (**11Z**)
Et	2	10.775 (**2b**)	10.033 (**1b**)	9.777
iPr	4	27.996 (**2c**)	28.115 (**1c**)	28.691
nBu	3	7.204 (**2d**)	6.921 (**1d**)	9.966

Data File D:\HPCHEM\1\DATA\THSP\TS153.D Sample Name: ts153

Injection Date : 16.01.2006 22:49:51 Seq. Line : 5
Sample Name : ts153 Location : Vial 105
Acq. Operator : ThSp Inj : 1
Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\OADOMIN2.M
Last changed : 10.01.2006 15:13:02 by Oa
Analysis Method : C:\HPCHEM\1\METHODS\THSPINTE.M
Last changed : 17.01.2006 08:41:38 by ThSp



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	12.505	BV	0.0199	61.40646	48.16062	15.16595
2	12.588	VB	0.0209	333.34268	252.27948	82.32780
3	12.790	PB	0.0208	10.14771	7.50659	2.50625
Totals :				404.89686	307.94669	

Results obtained with enhanced integrator!

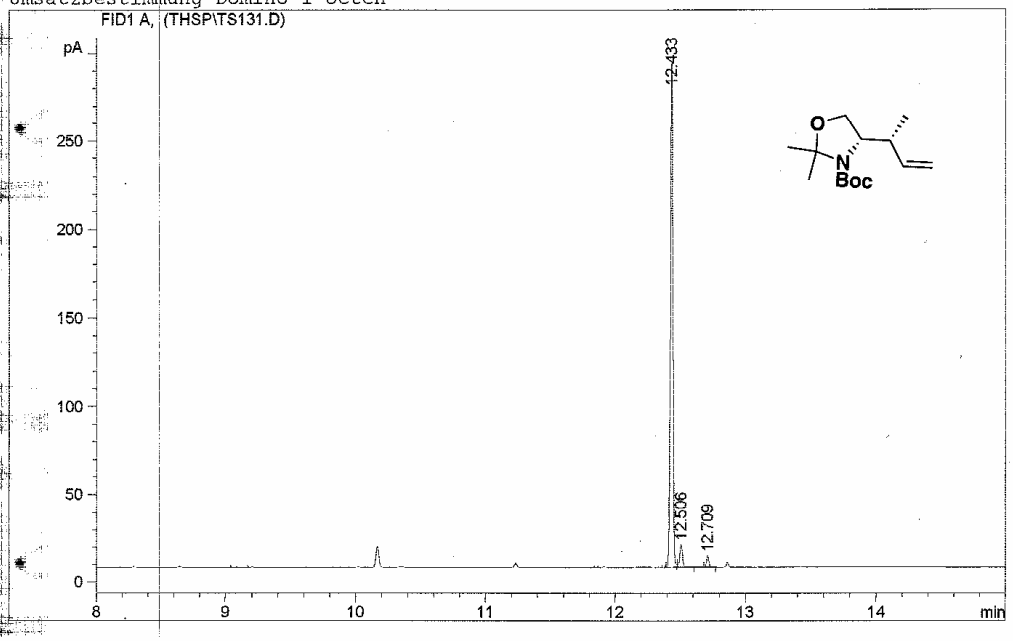
*** End of Report ***

Instrument 1 17.01.2006 08:45:57 ThSp Page 1 of 1

Data File D:\HPCHEM\1\DATA\THSP\TS131.D Sample Name: ts131

Injection Date	: 16.01.2006 20:53:46	Seq. Line	: 2
Sample Name	: ts131	Location	: Vial 102
Acq. Operator	: ThSp	Inj	: 1
		Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\OADOMIN2.M		
Last changed	: 10.01.2006 15:13:02 by Oa		
Analysis Method	: C:\HPCHEM\1\METHODS\OADOMIN2.M		
Last changed	: 17.01.2006 08:39:54 by ThSp		
	(modified after loading)		

Umsatzbestimmung Domino-1-octen



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	12.433	VV	0.0205	379.88031	285.70157	93.25518
2	12.506	VB	0.0225	19.53173	13.42040	4.79476
3	12.709	BB	0.0202	7.94369	6.32434	1.95006
Totals :				407.35572	305.44630	

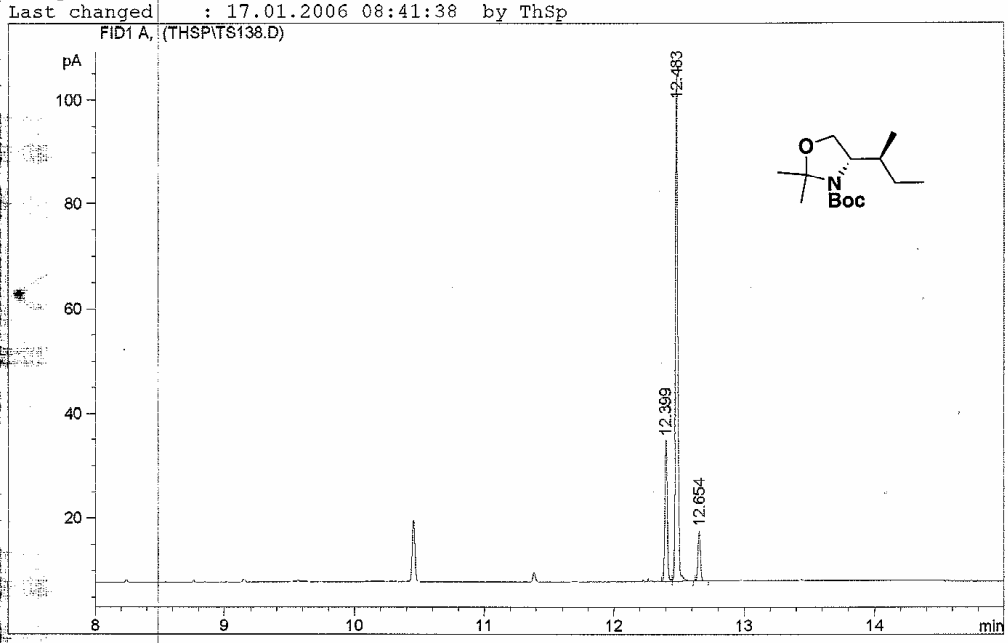
Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 17.01.2006 08:39:57 ThSp Page 1 of 1

Data File D:\HPCHEM\1\DATA\THSP\TS138.D Sample Name: ts138

Injection Date : 16.01.2006 21:32:31 Seq. Line : 3
Sample Name : ts138 Location : Vial 103
Acq. Operator : ThSp Inj : 1
Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\OADOMIN2.M
Last changed : 10.01.2006 15:13:02 by Oa
Analysis Method : C:\HPCHEM\1\METHODS\THSPINTE.M
Last changed : 17.01.2006 08:41:38 by ThSp



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	12.399	BV	0.0198	34.07732	26.91788	20.14777
2	12.483	VB	0.0194	122.61285	96.14343	72.49322
3	12.654	BP	0.0208	12.44681	9.47331	7.35901
Totals :				169.13698	132.53462	

Results obtained with enhanced integrator!

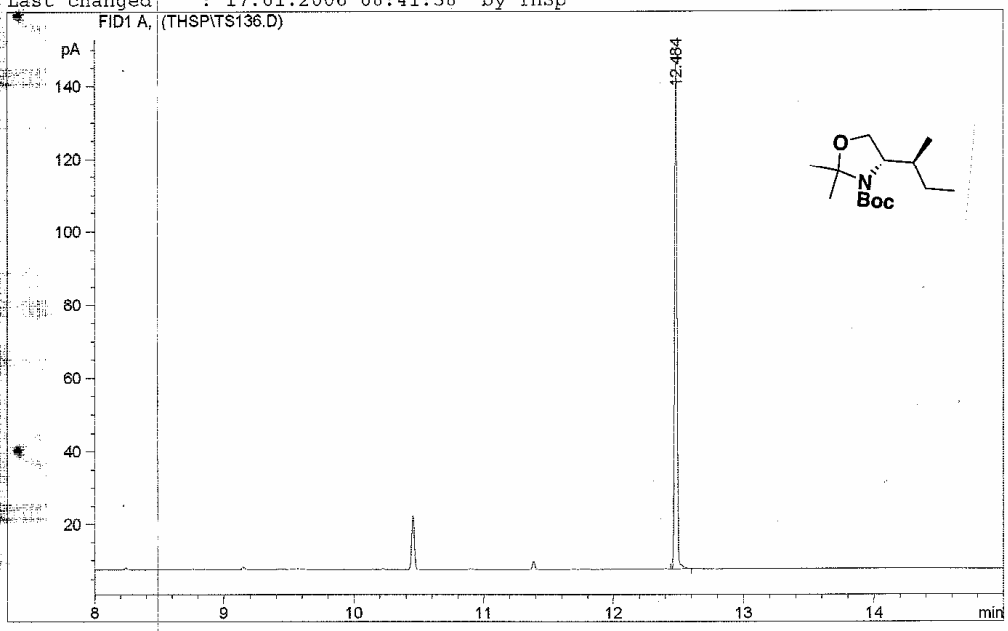
*** End of Report ***

Instrument 1 17.01.2006 08:47:17 ThSp Page 1 of 1

Data File D:\HPCHEM\1\DATA\THSP\TS136.D Sample Name: ts136

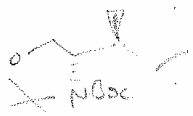
Injection Date : 17.01.2006 00:07:00 Seq. Line : 7
Sample Name : ts136 Location : Vial 107
Acq. Operator : ThSp Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\HPCHEM\1\METHODS\OADOMIN2.M
Last changed : 10.01.2006 15:13:02 by Oa
Analysis Method : C:\HPCHEM\1\METHODS\THSPINTE.M
Last changed : 17.01.2006 08:41:38 by ThSp



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs



Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	12.484	BB	0.0193	175.01312	138.22729	1.000e2
Totals :				175.01312	138.22729	

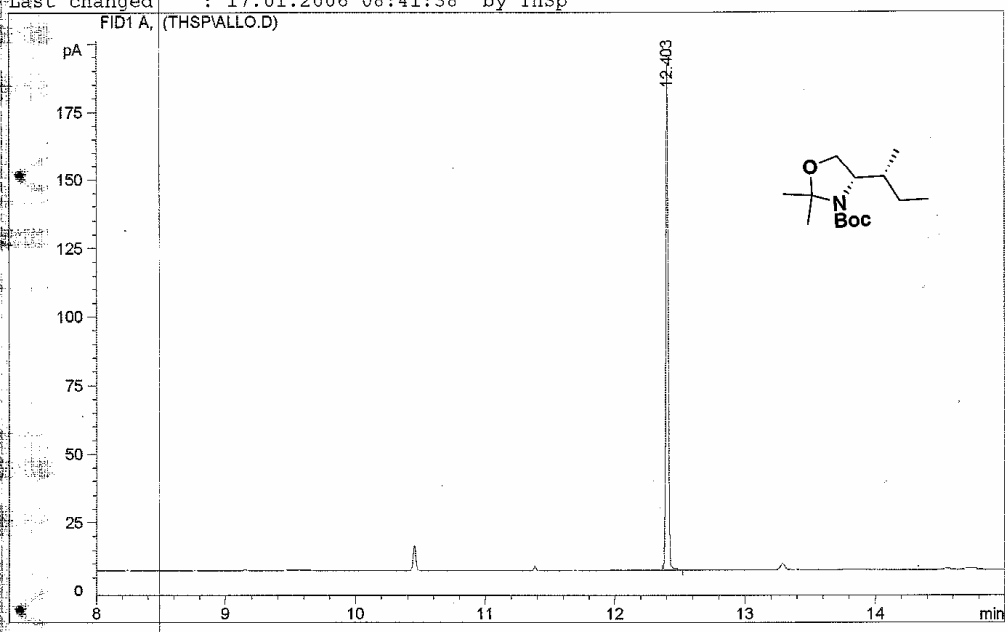
Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 17.01.2006 08:47:51 ThSp Page 1 of 1

Data File D:\HPCHEM\1\DATA\THSP\ALLO.D Sample Name: alloisoleuoxazo

Injection Date	: 16.01.2006 23:28:24	Seq. Line	: 6
Sample Name	: alloisoleuoxazo	Location	: Vial 106
Acq. Operator	: ThSp	Inj	: 1
		Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\OADOMIN2.M		
Last changed	: 10.01.2006 15:13:02 by Oa		
Analysis Method	: C:\HPCHEM\1\METHODS\THSPINTE.M		
Last changed	: 17.01.2006 08:41:38 by ThSp		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	12.403	PB	0.0200	236.16333	183.94034	1.000e2
Totals :				236.16333	183.94034	

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 17.01.2006 08:49:04 ThSp Page 1 of 1

Data File D:\HPCHEM\1\DATA\OA\THSP265A.D Sample Name: thsp265

```
==========================================================================
Injection Date  : 30.06.2006 18:32:49          Seq. Line :   8
Sample Name     : thsp265                      Location : Vial 101
Acq. Operator   : TS                           Inj :   1
                                               Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 0.2 µl
Acq. Method     : C:\HPCHEM\1\METHODS\OADOMIN2.M
Last changed    : 15.06.2006 10:24:28  by CS
Analysis Method : C:\HPCHEM\1\METHODS\THSPOAC3.M
Last changed    : 30.06.2006 21:39:22  by CS
                  (modified after loading)
Acetonid
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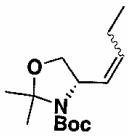
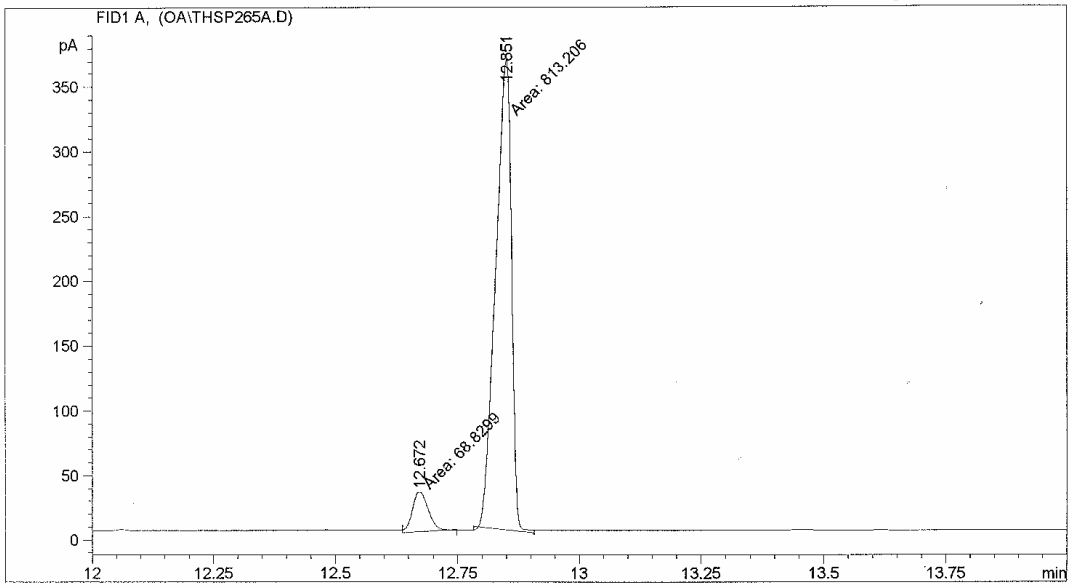


Product SN2




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  12.672 MM    0.0374   68.82988   30.70140  7.80352
   2  12.851 MM    0.0371  813.20593  364.93069 92.19648

Totals :                   882.03581  395.63209

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Instrument 1 30.06.2006 21:39:24 CS Page 1 of 1