Angewandte Chemie
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Supporting Information

69451 Weinheim, Germany

Organic solvent-free, highly diastereo- and enantio-selective direct aldol reaction in the presence of water

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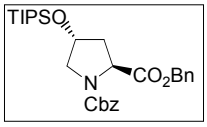
Typical procedure for the synthesis of (2*S*, 4*R*)-4-triisopropylsiloxy-pyrrolidine-2-carboxylic acid (1b).

To a CH_2Cl_2 solution (10 mL) of (2*S*, 4*R*)-*N*-benzyloxycarbonyl-4-hydroxypyrrolidine-2-carboxylic acid benzyl ester (3.55 g, 10.0 mmol) was added 2,6-lutidine (1.90 mL, 13.0 mmol) and TIPSOTf (2.96 mL, 13.0 mmol) at 0 °C. The reaction mixture was stirred for 30 minutes at room temperature, then the reaction was quenched by addition of phosphate buffer (pH 7.0). Organic materials were extracted three times with AcOEt, and the combined organic phases were dried (Na_2SO_4), concentrated, and purified by column chromatography (AcOEt:hexane = 1:5) to afford silyl ether (4.8 g, 9.54 mmol, 95%) as a clear viscous oil.

To a MeOH solution (10 mL) of (2*S*, 4*R*)-N-benzyloxycarbonyl-4-triisopropylsiloxy-pyrrolidine-2-carboxylic acid benzyl ester (4.8 g, 9.54 mmol) was added Pd/C (480 mg, 10 wt%) at room temperature and the reaction mixture was stirred for 20 h at that temperature. The filtration of the inorganic materials and concentration afforded (2*S*, 4*R*)-4-triisopropylsiloxy-pyrrolidine-2-carboxylic acid (1b) in 96% yield (2.8 g) as a white solid.

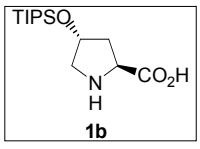
(2*S*, 4*R*)-4-*tert*-Butyldimethylsiloxy-pyrrolidine-2-carboxylic acid (1a)[1]
(2*S*, 4*R*)-*N*-Benzyloxycarbonyl-4-hydroxypyrrolidine-2-carboxylic acid benzyl ester[2]
(2*S*, 4*R*)-4-*tert*-Butyldiphenylsiloxy-pyrrolidine-2-carboxylic acid (1c)[1]
are known compounds.

(2*S*, 4*R*)-N-Benzyloxycarbonyl-4-triisopropylsiloxy-pyrrolidine-2-carboxylic acid benzyl ester



Data are shown as a mixture of two conformers.
^{1}H NMR ($CDCl_3$): δ 1.01 (21H, d, *J*=4.5 Hz), 2.00-2.12 (1H, m), 2.17-2.31 (1H, m), 3.42-3.59 (1H, m), 3.62-3.76 (1H, m), 4.45-4.60 (2H, m), 4.91-5.26 (4H, m), 7.17-7.37 (10H, m);
^{13}C NMR ($CDCl_3$): δ 12.4, 18.3, 39.5, 40.4, 55.3, 55.7, 58.5, 58.7, 67.1, 67.3, 67.5, 70.3, 71.0, 128.2, 128.3, 128.4, 128.55, 128.61, 128.65, 128.75, 128.8, 128.9, 129.0, 135.9, 136.1, 136.9, 137.1, 154.8, 155.5, 172.8, 173.0;
IR (neat): ν 2943, 2866, 1749, 1712, 1458, 1415, 1117, 1022, 883, 696 cm^{-1};
HRMS (FAB): [M+H] calcd for [$C_{29}H_{42}NO_5Si$]: 512.2832, found: 512.2809;
$[\alpha]_D^{22}$-35.1 (*c* = 1.00, $CHCl_3$).

(2*S*, 4*R*)-4-Triisopropylsiloxy-pyrrolidine-2-carboxylic acid (1b)



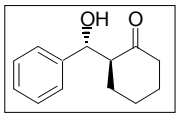
^{1}H NMR ($CDCl_3$):δ 0.97-1.05 (21H, m), 2.13 (1H, ddd, *J*=12.9, 7.8, 5.4 Hz), 2.27 (1H, ddd, *J*=12.9, 7.8, 4.1 Hz), 3.20 (1H, br-d, *J*=9.0 Hz), 3.46 (1H, br-s), 4.15(1H, t, *J*=7.8 Hz), 4.51(1H, quintet, *J*=4.1 Hz);
^{13}C NMR ($CDCl_3$): δ 11.9, 17.8, 39.2, 52.5, 59.8, 71.0, 173.7;
IR (KBr): ν 3438, 2942, 1624, 1464, 1400, 1389, 1101, 999, 883, 685 cm^{-1};
HRMS (FAB): [M+H] calcd for [$C_{14}H_{29}NO_3Si$]: 288.1995, found: 288.2010;
$[\alpha]_D^{22}$-15.9 (*c* = 1.01, $CHCl_3$).

Typical procedure for the synthesis of 2-(hydroxyphenylmethyl)cyclohexan-1-one (Table 1, entry 7).

Catalyst **1c** (14.8 mg, 0.04 mmol) was added to a suspension of benzaldehyde (40.6 μl, 0.4 mmol) and cyclohexanone (207 μl, 2.0 mmol) in water (0.13 ml) at room temperature. The reaction mixture was stirred for 18 h at this temperature, then the reaction was quenched by addition of phosphate buffer (pH 7.0). Organic materials were extracted three times with ethyl acetate, and the combined organic phases were dried over anhydrous Na_2SO_4, and concentrated in vacuo after filtration. Purification by silica gel column chromatography (ethyl acetate:hexane=1:10 ~ 1:3) gave 2-(hydroxyphenylmethyl)-cyclohexanone (63.7 mg, 78%) as a clear oil: *anti*:*syn* = 13:1 (by ^{1}H NMR spectroscopy of the crude mixture), >99% ee (by HPLC on a chiralcel OD-H column, λ=213 nm, *i*PrOH/hexane 1/100, 1.0 ml min^{-1}; t_R=19.4 min (major), 25.9 min (minor)).

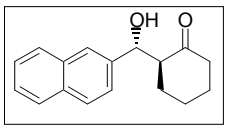
(2*S*, 1'*R*)-2-(Hydroxy-*p*-nitrophenylmethyl)cyclohexan-1-one[3]
(2*S*, 1'*R*)-2-(Hydroxy-*p*-bromophenylmethyl)cyclohexan-1-one[4]
(2*S*, 1'*R*)-2-(Hydroxy-*p*-methoxyphenylmethyl)cyclohexan-1-one[4]
(2*S*, 1'*R*)-2-(Hydroxy-2-furylmethyl)cyclohexan-1-one[5]
(2*S*, 1'*R*)-2-(1'-Hydroxy-3'-methylbutyl)cyclohexan-1-one[5]
(2*S*, 1'*R*)-2-(Cyclohexylhydroxymethyl)cyclohexan-1-one[6]
(2*S*, 1'*R*)-2-(Hydroxyphenylmethyl)cyclopentan-1-one[7]
(4*S*, 1'*S*)-4-(Hydroxyphenylmethyl)-2,2-dimethyl-1,3-dioxane-5-one[8]
(2*S*)-2-(Hydroxymethyl)cyclohexan-1-one[9]
(4*R*)-4-Hydroxy-*p*-nitrophenylbutan-2-one[10]
(3*S*, 4*S*)-3,4-Dihydroxy-*o*-chlorophenylbutan-2-one[11]
are known compounds.

(2*S*, 1'*R*)-2-(Hydroxyphenylmethyl)cyclohexan-1-one[6]



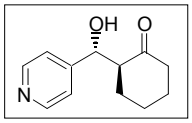
Absolute stereochemistry is determined by the comparison with the literature data[6].
$[\alpha]_D^{14}$+27.7 (*c* = 0.85, $CHCl_3$), >99% ee.
Lit. $[\alpha]_D^{24}$-24.2 (*c* = 1.03, $CHCl_3$). (93% ee, (2*R*, 1'*S*)-2-(Hydroxyphenylmethyl)-cyclohexanone).
Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (100:1 hexane:2-propanol), 1.0 mL/min; major enantiomer tr =19.4 min, minor enantiomer tr =25.9 min.

(2*S*, 1'*R*)-2-(Hydroxynaphthalen-2-ylmethyl)cyclohexan-1-one



^{1}H NMR ($CDCl_3$): δ 1.23-1.40 (1H, m), 1.42-1.61 (2H, m), 1.62-1.79 (2H, m), 2.07 (1H, ddd, *J*=13.2, 6.6, 3.2 Hz), 2.36 (1H, td, *J*=13.2, 5.8 Hz), 2.49 (1H, br-d, *J*= 13.8 Hz), 2.64-2.74 (1H, m), 4.02 (1H, br-s), 4.95 (1H, d, *J*=8.6 Hz), 7.41-7.50 (3H, m), 7.73 (1H, s), 7.77-7.86 (3H, m);
^{13}C NMR ($CDCl_3$): δ 24.6, 27.7, 30.8, 42.6, 57.3, 74.8, 124.6, 125.9, 126.1, 126.2, 127.6, 127.9, 128.2, 133.0, 133.1, 138.2, 215.5;
IR (KBr): ν 3354, 3055, 2933, 2854, 1695, 1444, 1309, 1122, 1057, 833 cm^{-1};
HRMS (FAB): calcd for [$C_{17}H_{18}O_2$]: 254.1307, found: 254.1311;
$[\alpha]_D^{22}$+7.4 (*c* = 1.07, $CHCl_3$). (mixture of diastereomers, *anti*:*syn*=19:1, 97% ee for *anti*-isomer.)
Enantiomeric excess was determined by HPLC with a Chiralpak AS-H column (50:1 hexane:2-propanol), 1.0 mL/min; major enantiomer tr =17.6 min, minor enantiomer tr =20.5 min.

(2*S*, 1'*R*)-2-(Hydroxypyridin-4-ylmethyl)cyclohexan-1-one



^{1}H NMR ($CDCl_3$): δ 1.38 (1H, qd, *J*=12.8, 3.8 Hz), 1.47-1.73 (3H, m), 1.77-1.86 (1H, m), 2.04-2.14 (1H, m), 2.34 (1H, td, *J*=13.3, 6.2 Hz), 2.42-2.50 (1H, m), 2.56 (1H, ddd, *J*=13.5, 8.2, 3.5 Hz), 3.97 (1H, br-s), 4.75 (1H, d, *J*=8.2 Hz), 7.23 (2H, d, *J*=5.7 Hz), 8.56(2H, d, *J*=5.7 Hz);
^{13}C NMR ($CDCl_3$): δ 24.6, 27.7, 30.7, 42.6, 56.8, 73.5, 122.0, 149.7, 149.8, 214.7;
IR (KBr): ν 3140, 2860, 2738, 1711, 1606, 1415, 1300, 1128, 1047, 835 cm^{-1};

HRMS (FAB): [M+H] calcd for [$C_{12}H_{16}NO_2$]: 206.1181, found: 206.1177;
$[\alpha]_D^{21}$+15.8 (*c* = 1.02, $CHCl_3$). (mixture of diastereomers, *anti*:*syn*=12:1, 95% ee for *anti*-isomer .)
Enantiomeric excess was determined by HPLC with a Chiralpak AD-H column (10:1 hexane:2-propanol), 1.0 mL/min; major enantiomer tr = 22.5 min, minor enantiomer tr =20.7 min.

References

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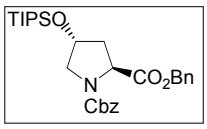

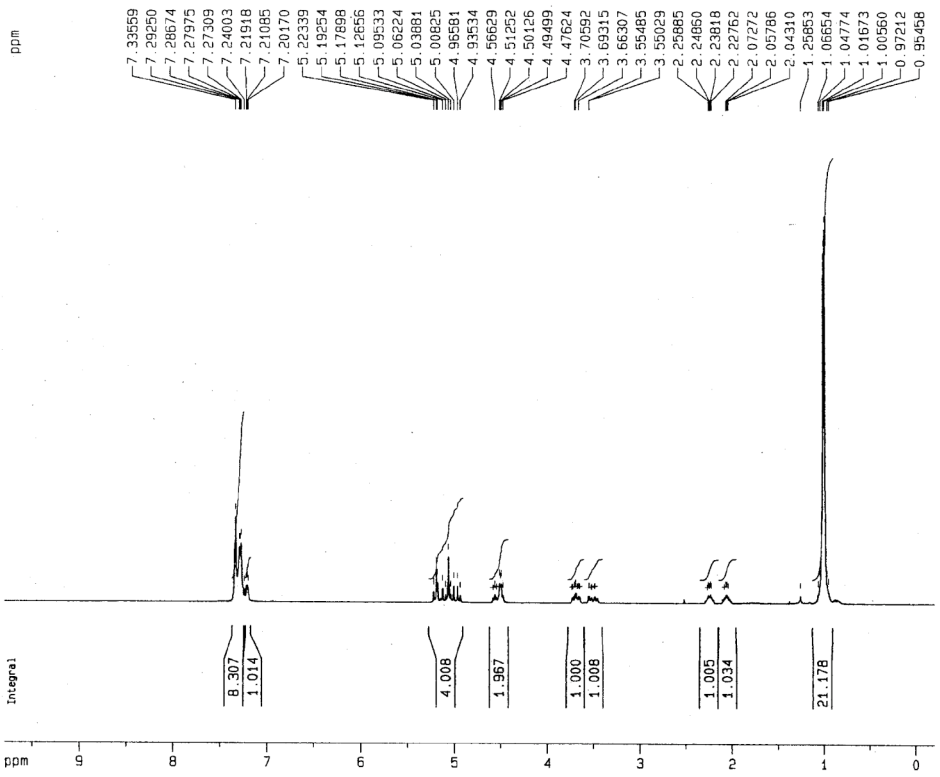


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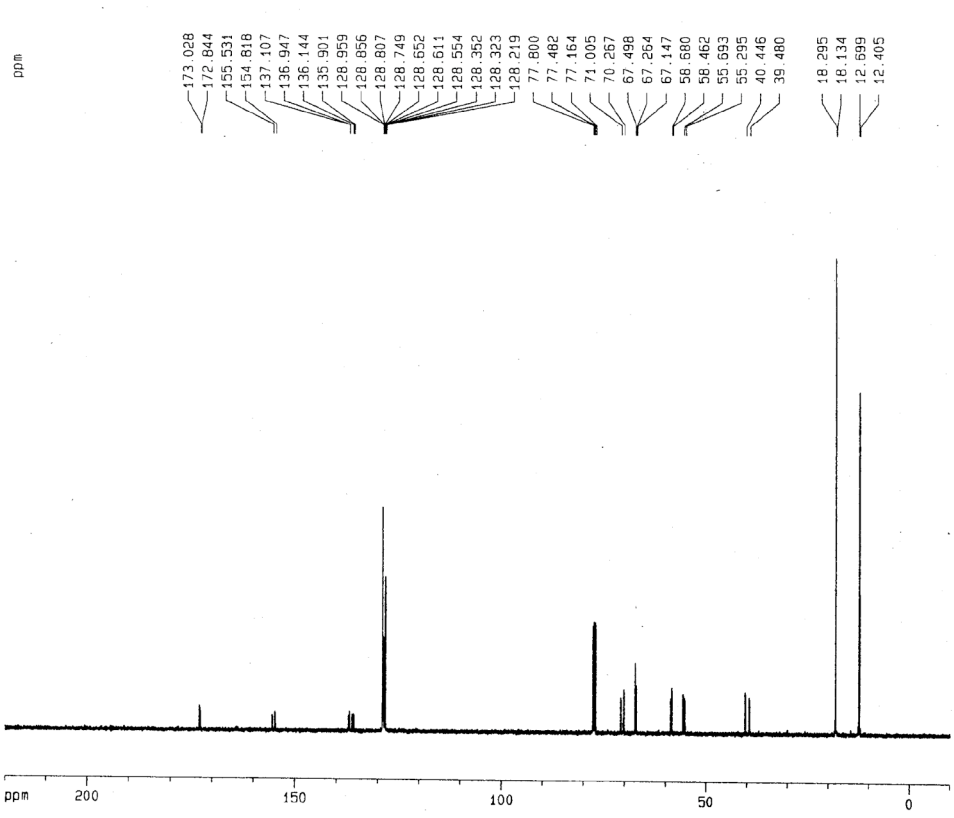


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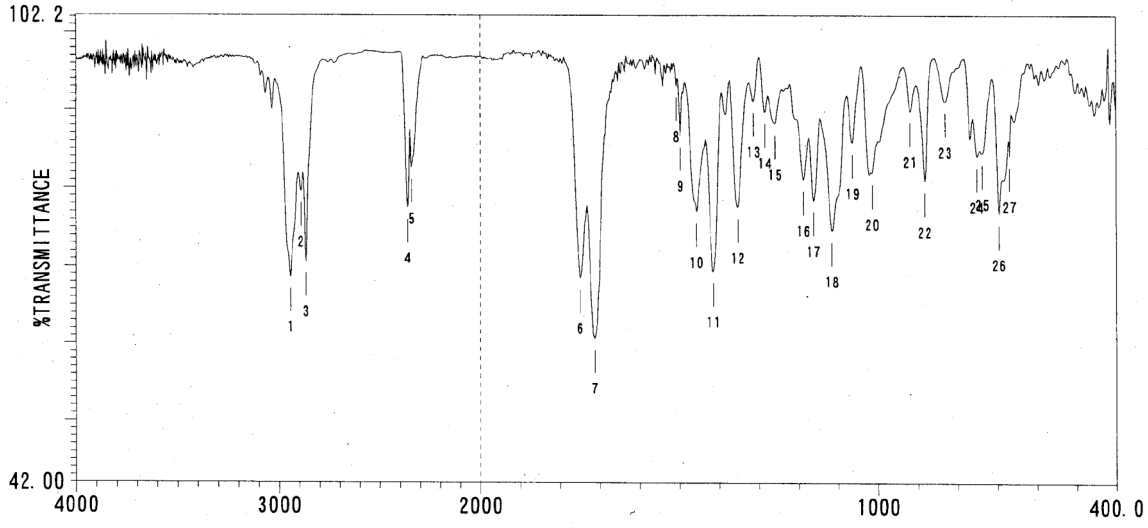
FTIR system for Windows



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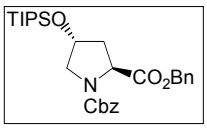
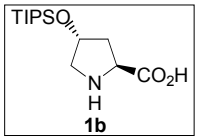
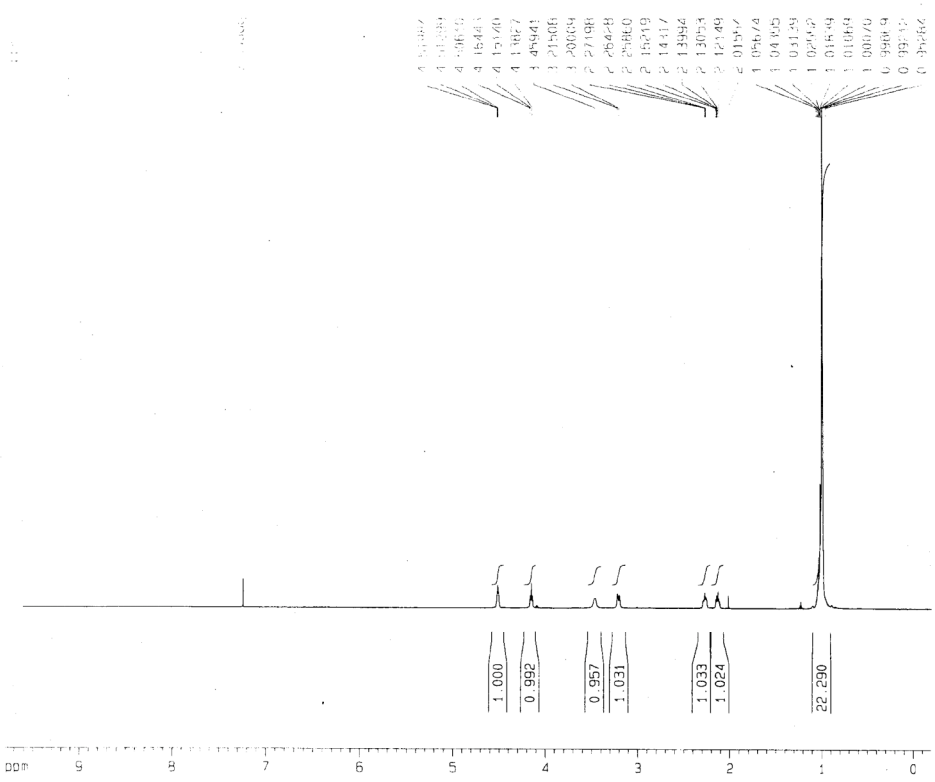




1b

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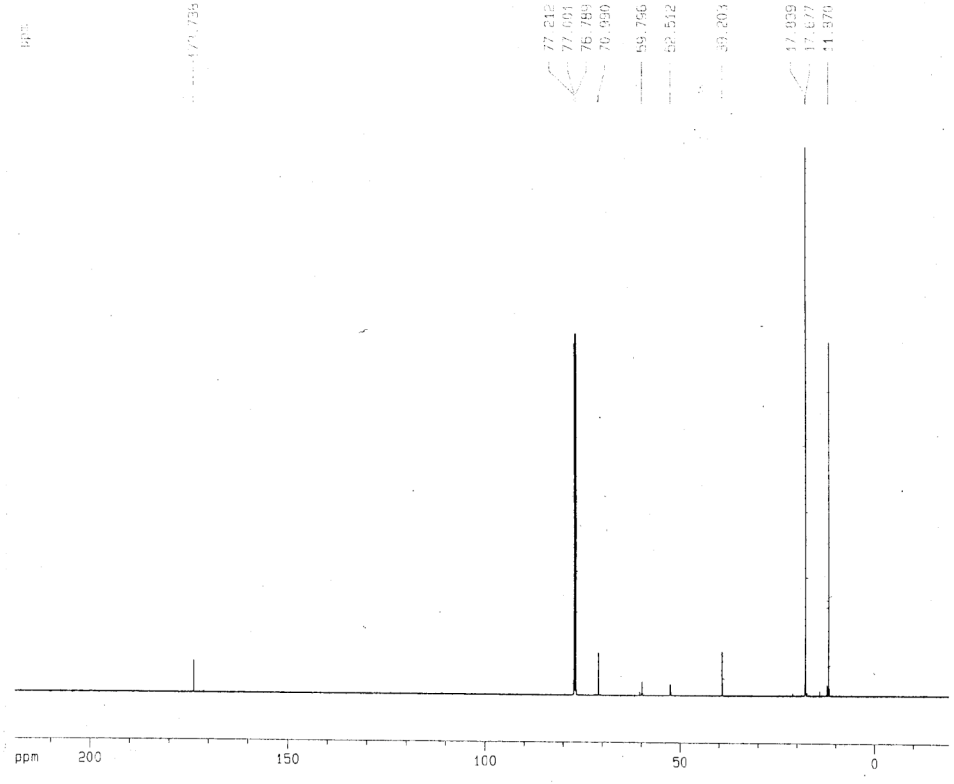


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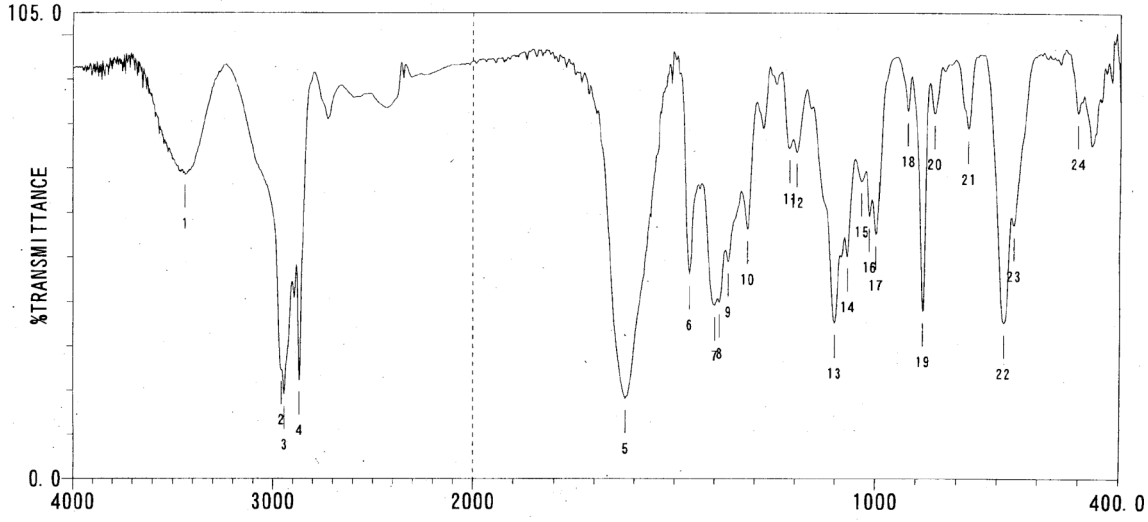
FTIR system for Windows



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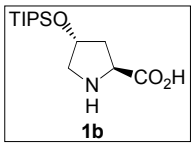
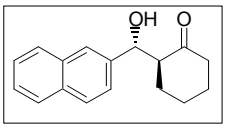
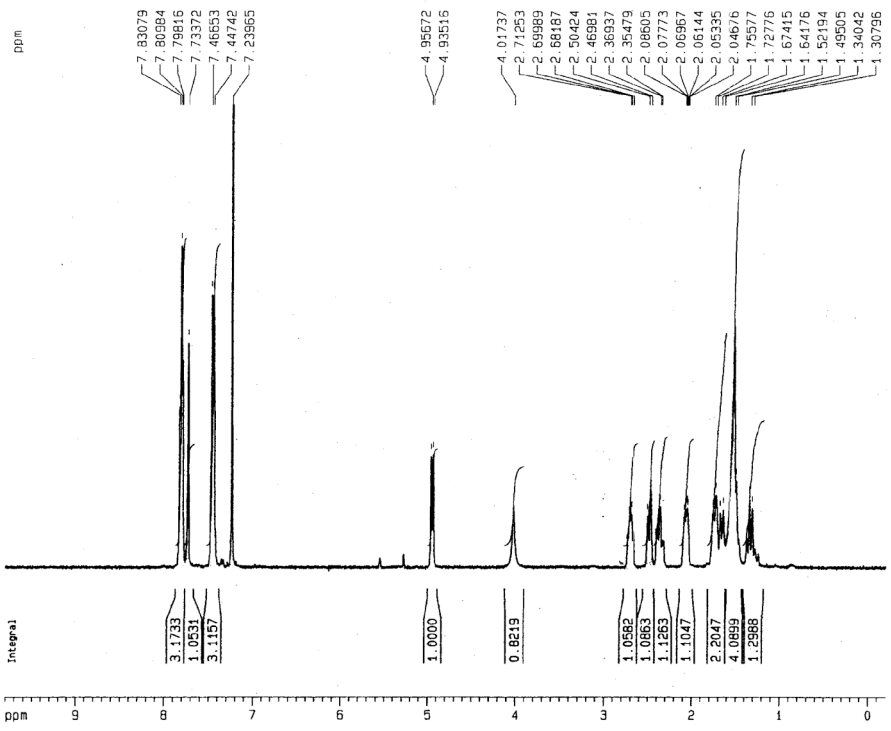
測定日時	: 2005年06月07日 16時55	02	2958. 27	24. 6801	12	1195. 65	73. 6684	22	684. 606	35. 3187
測定分解能	: 4 cm-1	03	2942. 84	19. 0443	13	1101. 15	35. 2986	23	659. 536	57. 2460
スキャン回数	: 10 回	04	2867. 63	22. 3003	14	1068. 37	50. 2335	24	503. 330	82. 5278
測定ゲイン	: 1	05	1623. 77	18. 1691	15	1033. 66	67. 1317			
		06	1463. 71	46. 1917	16	1014. 37	59. 1628			
		07	1400. 07	39. 1751	17	998. 946	55. 1418			
		08	1388. 50	39. 7735	18	919. 879	82. 9669			
		09	1367. 28	49. 0090	19	883. 238	37. 9544			
		10	1319. 07	56. 3434	20	854. 311	82. 3684			



1b



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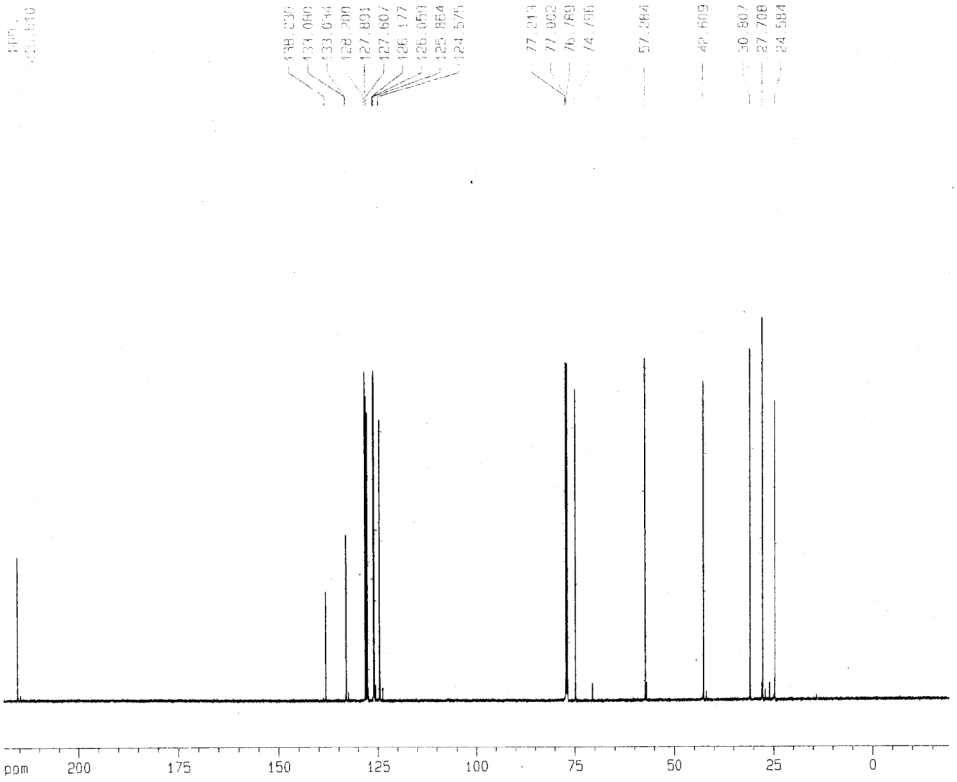
Current Data Parameters
NAME        Jun01-2005-haya
EXPNO              175
PROCNO               1

F2 - Acquisition Parameters
Date_          20050602
Time               0.31
INSTRUM          dpx400
PROBHD   5 mm QNP 1H/29
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG                456.1
DW               60.800 usec
DE                6.00 usec
TE                303.2 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                7.90 usec
PL1               3.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                16384
SF          400.1300177 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00

1D NMR plot parameters
CX               20.00 cm
CY               12.50 cm
F1P               9.800 ppm
F1             3921.27 Hz
F2P              -0.200 ppm
F2               -80.03 Hz
PPMCM          0.50000 ppm/cm
HZCM         200.06500 Hz/cm
```

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Current Data Parameters
NAME          Jun01-2005
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_          20050601
Time               22.19
INSTRUM          av600
PROBHD   5 mm CPDUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   37
DS                    4
SWH          35971.223 Hz
FIDRES        0.548877 Hz
AQ            0.9110004 sec
RG                9195.2
DW               13.900 usec
DE                50.00 usec
TE                297.9 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1               10.00 usec
PL1               -4.50 dB
SFO1        150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2               -5.80 dB
PL12               8.74 dB
PL13               8.74 dB
SFO2        600.1324005 MHz

F2 - Processing parameters
SI                32768
SF          150.9028312 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40

1D NMR plot parameters
CX               20.00 cm
CY                6.00 cm
F1P             219.184 ppm
F1            33075.43 Hz
F2P             -19.190 ppm
F2            -2895.80 Hz
PPMCM         11.91667 ppm/cm
HZCM        1798.56140 Hz/cm
```

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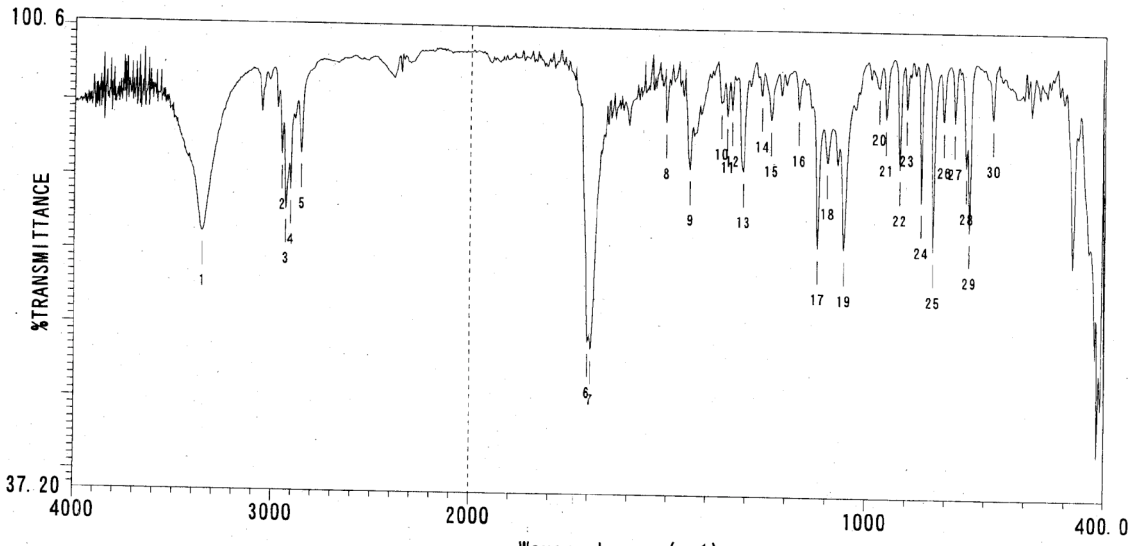
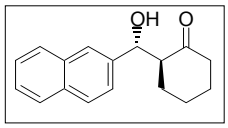
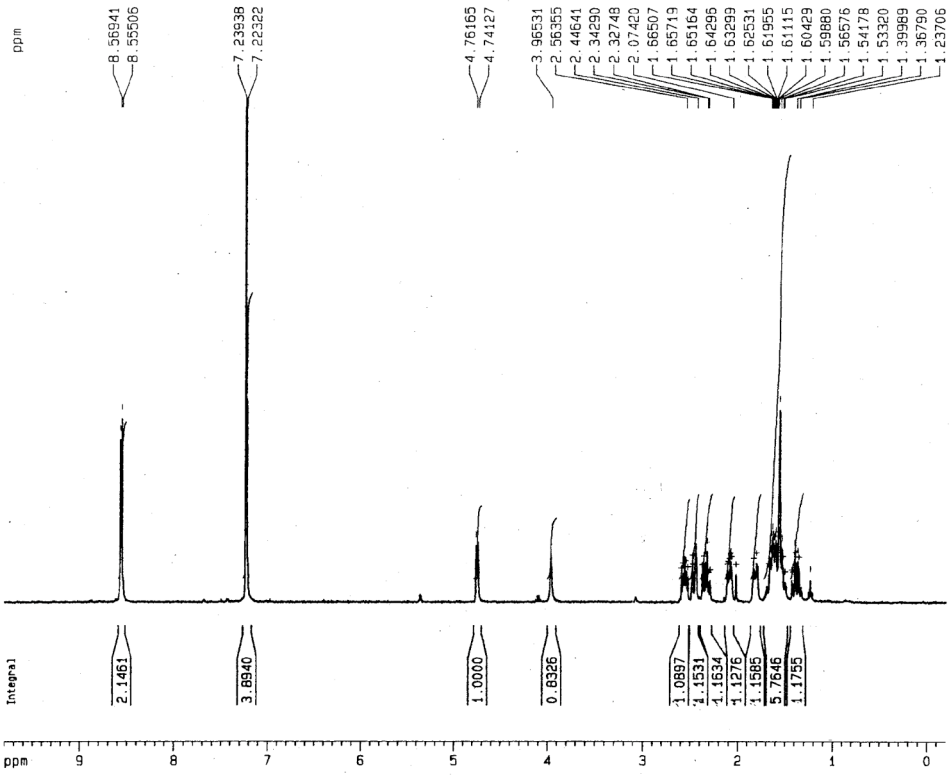
FTIR system for Windows

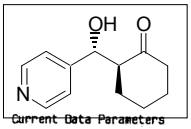


ファイル名 : nap
タイトル :
測定日時 : 2005年06月02日 00時55
測定分解能 : 4 cm-1
スキャン回数 : 10 回
測定ゲイン : 1

ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)
01	3353.60	72.3737	11	1349.93	88.9809	21	952.663	88.9250
02	2952.48	82.9774	12	1338.36	89.8489	22	917.950	82.1675
03	2933.20	75.6302	13	1309.43	81.5954	23	900.594	90.3201
04	2910.06	78.2256	14	1263.15	91.8489	24	863.953	77.7364
05	2854.13	83.1631	15	1240.00	88.5705	25	833.098	71.0403
06	1702.84	58.0542	16	1170.58	90.0428	26	806.099	88.7699
07	1695.12	57.1644	17	1122.37	71.2485	27	779.101	88.5727
08	1504.20	88.0486	18	1099.23	82.9274	28	750.174	82.5413
09	1444.42	81.7616	19	1056.80	71.0831	29	742.460	73.7252
10	1365.35	90.7609	20	970.019	93.0966	30	682.677	89.0485







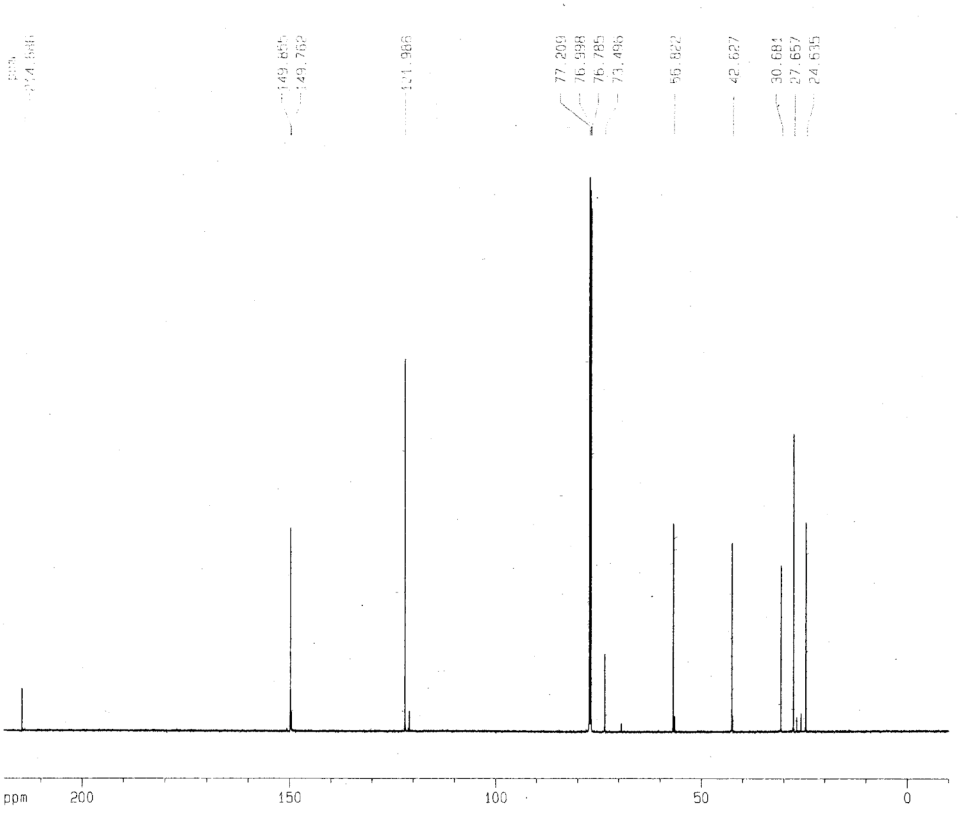
Current Data Parameters
NAME May17-2005-haya
EXPNO 172
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050517
Time 23.56
INSTRUM dpx400
PROBHD 5 mm GNP 1H/29
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 512
DW 60.800 usec
DE 6.00 usec
TE 303.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 16384
SF 400.1300177 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 9.800 ppm
F1 3921.27 Hz
F2P -0.200 ppm
F2 -80.03 Hz
PPMCM 0.50000 ppm/cm
HZCM 200.06500 Hz/cm



Current Data Parameters
NAME May18-2005
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050518
Time 17.22
INSTRUM av600
PROBHD 5 mm CPDUL 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 133
DS 4
SWH 35971.223 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 9195.2
DW 13.900 usec
DE 50.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -4.50 dB
SFO1 150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -5.80 dB
PL12 8.74 dB
PL13 8.74 dB
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 32768
SF 150.9028236 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 220.000 ppm
F1 33198.62 Hz
F2P -10.000 ppm
F2 -1509.03 Hz
PPMCM 11.50000 ppm/cm
HZCM 1735.38232 Hz/cm

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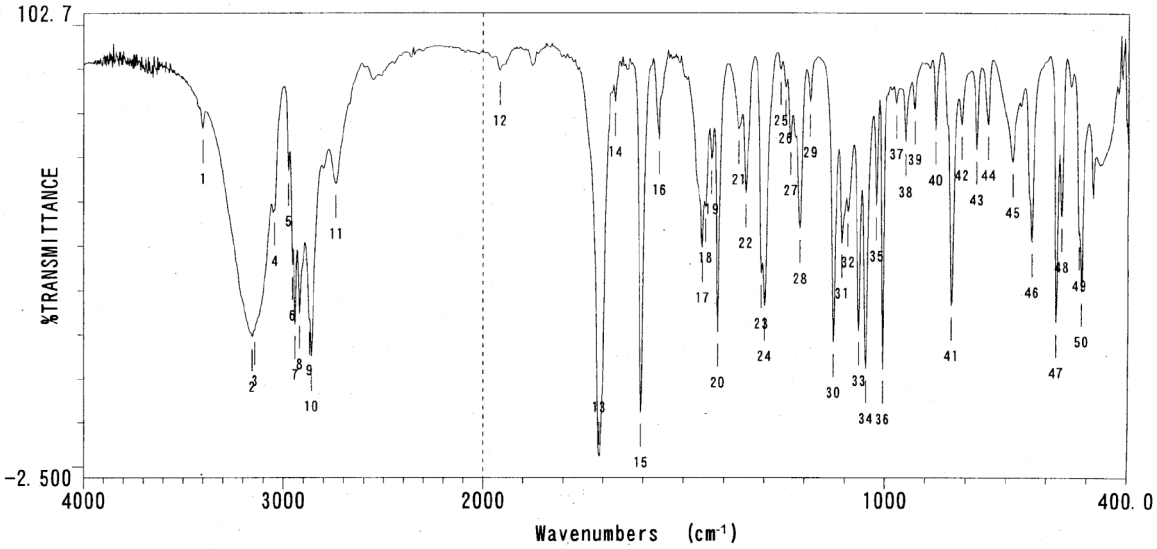
FTIR system for Windows



ファイル名	: N	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号	波数 (cm-1)	透過率 (%)	ピーク番号			
タイトル	:	01	3397.96	76.6211	11	2738.42	64.1743	21	1363.43	76.4560	31			
測定日時	: 2005年06月06日 19時44分	02	3153.04	29.5251	12	1957.39	89.7239	22	1346.07	62.1124	32			
測定分解能	: 4 cm-1	03	3139.54	30.9835	13	1710.55	2.40079	23	1307.50	43.9216	33			
スキャン回数	: 10 回	04	3039.26	58.0053	14	1670.05	82.6898	24	1299.79	36.5504	34			
測定ゲイン	: 1	05	2971.77	66.9675	15	1606.41	12.3853	25	1259.29	89.7885	35			
		06	2952.48	45.7773	16	1560.13	74.1021	26	1247.72	85.8372	36			
		07	2938.98	32.3435	17	1454.06	49.7577	27	1234.22	74.0169	37			
		08	2917.77	34.7175	18	1446.35	58.8190	28	1211.08	54.0681	38			
		09	2869.56	33.3274	19	1430.92	69.6862	29	1186.01	82.5880	39			
		10	2859.92	25.1305	20	1415.49	30.5978	30	1128.15	28.3244	40