Synthesis of *N*-(Dialkylaminoalkyl)alcohols by Homogeneously Catalyzed Hydrogenolysis of Cyclic *N*,*O*-Acetals

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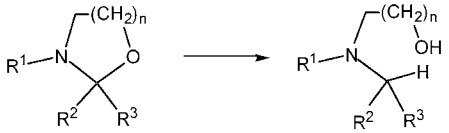
Received 6 November 2001; revised 14 December 2001

Abstract: The homogeneously catalyzed hydrogenation of 1,3-oxazolidines affording unsymmetrically substituted 2-*N*-(dialkylamino)ethanols is reported showing for the first time that Rh(I) catalysts based on chelating diphosphines can be advantageous for this reaction.

Key words: hydrogenation, transition metals, catalysis, amino alcohols

The selective cleavage of cyclic *N*,*O*-acetals under the formation of substituted ω-aminoalkyl alcohols has great synthetic potential since it offers an interesting alternative for the alkylation of secondary amines with functionalized alkylhalogenides (Scheme 1). In addition, the facile preparation of cyclic *N*,*O*-acetals which serve as substrates in this reaction makes them attractive precursors for unsymmetrically substituted tertiary amines.

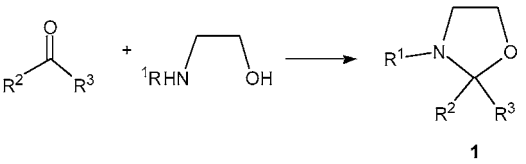


Scheme 1

In general, the chemoselective cleavage of cyclic *N*,*O*-acetals can be achieved by means of hydride reagents.[1] Recently, the use of alkali metals was suggested for this reaction.[2] However, the scope of this transformation is limited to 2-aryl substituted cyclic *N*,*O*-acetals. In contrast, less is known about the chemoselective reductive cleavage of cyclic *N*,*O*-acetals with molecular hydrogen, although this methodology represents an ecologically benign approach for the synthesis of ω-hydroxyalkylamines. Hitherto, the only example described in the literature is concerned with the reduction of cyclic *N*,*O*-acetals under the conditions of a heterogeneous catalysis.[3]

In the course of our mechanistic studies on the reductive amination of aldehydes and ketones with secondary amines, we found that acyclic *N*,*O*-acetals can be cleanly and quantitatively transformed into the corresponding amines by the assistance of homogeneous Rh(I) catalysts bearing diphosphines as ancillary ligands.[4]

Herein, we report that this finding is of considerable synthetic value. Thus, 1,3-oxazolidines can also be cleaved under these conditions affording *N*-trisubstituted 2-amino ethanol derivatives in excellent yields. 1,3-Oxazolidines of the general structure **1** serving as starting materials are easily prepared from a broad variety of commercially available 1,2-aminoalcohols and the corresponding aldehydes and ketones, respectively (Scheme 2).[5]



Scheme 2

As listed in Table 1 these 1,3-oxazolidines are well suited substrates for the reductive cleavage with [Rh(dppb)COD]BF_4 [dppb = 1,4-bis(diphenylphosphino)butane] as homogeneous precatalysts in methanol as solvent. Under mild conditions (50 bar H_2 pressure, ambient temperature) 1,3-oxazolidines were quantitatively converted into the desired 2-dialkylamino ethanols (**2**).

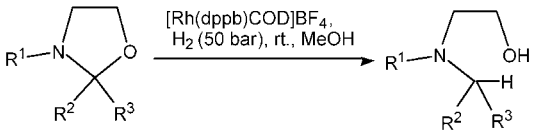
The generality of this hydrogenation reaction is shown by employing different types of 1,3-oxazolidines (**1a**–**j**) as substrates. Thus, relevant 1,3-oxazolidines can be derived from *N*-alkylaminoethanols and formaldehyde (**1a**), aromatic aldehydes (**1b**–**e**), aliphatic aldehydes (**1f**–**g**) and acetophenone (**1h**). In all trials fast transformations were observed and only amines **2a**,**c**–**h** were produced. Particular noteworthy is the clean reaction of a substrate with a phenolic group (**1d**). The nitro group in oxazolidine (**1i**) did not survive under these conditions. Unfortunately, the 1,3-oxazolidine (**1j**) derived from substituted aniline was not cleaved.

The hydrogenation can be even conducted under solvent free conditions as shown using substrates **1b** and **1f** as examples. In the presence of 0.1 mol% of the precatalyst, H_2 uptake ceased after 10 and 20 hours, respectively. After distillation, analytically pure amines **2a** and **2f** were obtained in 86% and 92% yield (For NMR data see Tables 4 and 5).

Synthesis 2002, No. 3, 18 02 2002. Article Identifier:
1437-210X,E;2002,0,03,0375,0380,ftx,en;T10501SS.pdf.

ISSN 0039-7881

Table 1 Hydrogenolytic Cleavage of 1,3-Oxazolidines[a]



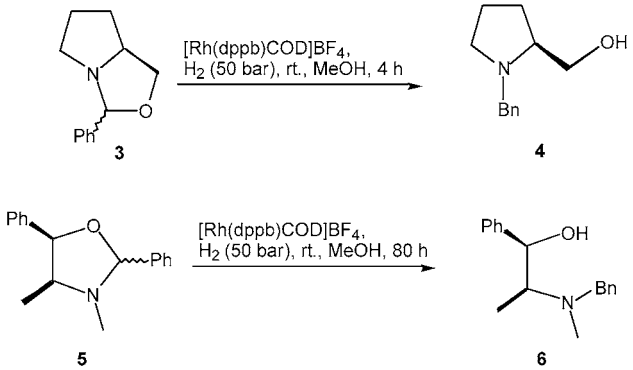
1 2

Substrate	R^1	R^2	R^3	Product	Solvent	Time (h)[b]
1a	$PhCH_2$	H	H	**2a**	MeOH	3
1b	Me	Ph	H	**2a**	MeOH	0.3
1c	Me	2-MeC_6H_4	H	**2c**	MeOH	0.5
1d	Me	4-HOC_6H_4	H	**2d**	MeOH	1.5
1e	Me	2-furyl	H	**2e**	MeOH	2
1f	Me	Ph(Me)CH	H	**2f**	MeOH	0.4
1g	Me	C_7H_{15}	H	**2g**	MeOH	0.2
1h	Me	Ph	Me	**2h**	MeOH	4
1i	Me	4-$NO_2C_6H_4$	H	mixture	MeOH	20
1j	Ph	Ph	H	no reduction	MeOH	24
1b	Me	Ph	H	**2a**	–	10
1f	Me	Ph(Me)CH	H	**2f**	–	20

[a] Reaction conditions: substrate (5 mmol), [Rh(dppp)COD]BF_4 (0.01 mmol), MeOH (10 ml), 50−52 bar initial H_2 pressure, r.t.
[b] Time required for quantitative conversion.

Diastereomeric oxazolidines such as **3** and **5** can also be successfully subjected to hydrogenation (Scheme 3). The examples depicted emphasize the importance of the new method for the selective N-benzylation of aminoalcohols, which can be useful for N-protection strategies in multistep syntheses. In each case the conversion proceeded quantitatively and both diastereomers were reduced affording a single stereoisomer.

It should be noted that the usage of our Rh (I) phosphine catalyst proved superior to the application of heterogeneous Pd based catalysts since benzylic group in the products remained untouched.



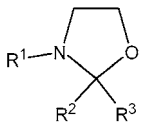
Scheme 3

All solvents and liquids used in hydrogenations were distilled and kept under Ar. Other commercial reagents were used without additional purification. NMR spectra were recorded with a Bruker ARX 400 spectrometer. Chemical shifts (δ, in ppm) are given for ^1H relative to TMS as internal standard and for ^{13}C relative to the residual $CDCl_3$ peak (77.36 ppm). Spin-spin coupling constants (*J*) are given in Hz. The optical rotation was measured on a 'gyromat-HP' instrument (Fa. Dr. Kernchen). Melting points are corrected.

3-Benzyl-1,3-oxazolidine (1a)
Prepared according to the protocol given in ref.[6]; bp 89 °C/0.05 mbar. NMR data are given in Tables 2 and 3.

1,3-Oxazolidines (1b−j, 3, 5)
Prepared by azeotropic removal of H_2O using a Dean−Stark trap by refluxing a 1:1 mixture of amine and carbonyl compound in benzene as solvent in the presence of cat. amounts of *p*-TsOH·H_2O. In case of **1h**, toluene was used as solvent. When separation of H_2O was complete, the solvent was evaporated and the residue was distilled in vacuum. 1,3-Oxazolidines had the following bp: **1b**, 96−97 °C/10 mbar (Lit.[7]135 °C, 14 torr); **1c**, 103−104 °C, 5 mbar; **1e**, 70−71 °C, 5 mbar; **1f**, 60−62 °C, 0.3 mbar; **1g**, 99−100 °C, 10 mbar; **1h**, 89−90 °C, 5 mbar (Lit.[8] 99 °C, 3 torr); **1i**, 118−119 °C, 0.06 mbar (solidified after several days). Solid substrates were recrystallised and had the following mp: **1d**, 108−110 °C, **1j**, 85−86 °C (MeOH) [Lit.[9] 84−85 °C (MeOH)]. NMR data for substrates **1a−j** are given in Tables 2 and 3.

Table 2 ^{1}H NMR Characterization of 1,3-Oxazolidines **1a–j** in $CDCl_3$[a]



Compound	1,3-oxazolidine ring position			R1	R2	R3
	C-2	C-4	C-5			
1a	4.28 (s, 2 H)	2.94 (t, 3 H, *J* = 6.8)	3.78 (t, 3 H, *J* = 6.8)	3.68 (s, 2 H), 7.2–7.4 (m, 5 H_{arom})		
1b	4.56 (s, 1 H)	2.55–2.63 (m, 1 H); 3.18–3.25 (m, 1 H)	3.59–4.04 (m, 2 H)	2.19 (s, 3 H)	7.18–7.42 (m, 5 H_{rom})	
1c	4.82 (s, 1 H)	2.54–2.63 (m, 1 H), 3.11–3.19 (m, 1 H)	3.80–3.96(m, 2 H)	2.14 (s, 3 H)	2.31 (s, 3 H), 6.98–7.10 (m, 3 H_{arom}), 7.38–7.44 (m, 2 H_{arom})	
1d	4.46 (s, 1 H)	2.54–268 (m, 1 H), 3.18–3.29 (m, 1 H)	3.87–4.03 (m, 2 H)	2.15 (s, 3 H)	6.71 (d, 2 H_{arom}, *J* = 8.3 Hz), 7.19 (d, 2 H_{arom}, *J* = 8.3 Hz)	
1e	4.80 (s, 1 H)	2.60–2.68 (m, 1 H), 3.18–3.26 (m, 1 H)	3.87–4.00 (m, 2 H)	2.28 (s, 3 H)	6.23–6.27 (m, 1 H_{arom}), 6.31–6.35 (m, 1 H_{arom}), 7.32–7.35 (m, 1 H_{arom})	
1f[b]	3.88 (d, 1 H, *J* = 4.2) 4.01 (d, 1 H, *J* = 4.9)			2.14 (s, 3 H) 2.23 (s, 3 H)	1.22 (d, 3 H, *J* = 7.1) 1.26 (d, 3 H, *J* = 7.1)	
1g	3.77–3.90 (m, 1 H)	2.54–2.63 (m, 1 H), 3.13–3.22 (m, 1 H)	3.77–3.90 (m, 2 H)	2.34 (s, 3 H)	0.86 (t, 3 H, *J* = 7.0), 1.18–1.67 (m, 12 H, 6 CH_2)	
1h		2.72–2.88 (m, 2 H)	3.59–3.68 (m, 1 H), 3.82–3.90 (m, 1 H)	2.29 (s, 3 H)	7.09–7.26 (m, 3 H_{arom}), 7.38–7.46 (m, 2 H_{arom})	1.45 (s, 3 H)
1i	4.72 (s, 1 H)	2.61–2.71 (m, 1 H), 3.12–3.21 (m, 1 H)	3.90–3.98 (m, 2 H)	2.23 (s, 3 H)	7.54 (d, 2 H, *J* = 8.6), 8.09 (d, 2 H, *J* = 8.6)	
1j	5.87 (s, 1 H)	3.49–3.59 (m, 1 H), 3.71–3.78 (m, 1 H)	4.05–4.16 (m, 2 H)	6.47–6.53 (m, 2 H_{arom}), 6.69–6.76 (m, 1 H_{arom}), 7.12–7.20 (m, 2 H_{arom}), 7.28–7.38 (m, 3 H_{arom}), 7.42–7.48 (m, 2 H_{arom})		

[a] Coupling constants *J* in Hz.
[b] Mixture of 2 diastereomers in 1.5:1 ratio; only selected resonances are given. Anal. Calcd for $C_{12}H_{17}NO$: C, 75.35; H 8.96; N, 7.32. Found: C, 75.15; H, 8.99; N, 7.44.

(2*rac*,4*S*)-2-Phenyl-3-oxa-1-azabicyclo[3.3.0]octane (3)
Mixture of the two diastereomers in ca. 3:1 ratio. Bp 116–117 °C, 5 mbar.

^{1}H NMR ($CDCl_3$): δ = 5.44 (s, O–CH–N, major isomer), 5.51 (s, O–CH–N, minor isomer). Other resonances could not be assigned.

^{13}C NMR ($CDCl_3$): major isomer, δ = 25.6 (CH_2), 30.4 (CH_2), 54.0 (CH_2N), 71.5 (CH_2O), 98.9 (OCHN), 126.7 (CH), 128.0 (CH), 128.4 (CH), 141.5 (C); minor isomer, δ = 25.8 (CH_2), 31.3 (CH_2), 47.5 (CH_2N), 64.2 (CHN), 71.3 (CH_2O), 94.9 (OCHN), 126.9 (CH), 128.1 (CH), 128.3 (CH), 137.4 (C).

(2*rac*,4*S*,5*R*)-3,4-Dimethyl-2.5-diphenyl-1,3-oxazolidine (5)
Mixture of two distereomers in ca. 12:1 ratio. Mp.72–73 °C (EtOH) (Lit.[10] 73–74 °C). Chemical shifts are given only for major isomer.

^{1}H NMR ($CDCl_3$): δ = 0.77 (d, 3 H, *J* = 6.3, Me–C), 2.17 (s, 3 H, Me–N), 2.95 (dq, 1 H, *J* = 6.3, 8.2, CH–Me), 4.68 (s, 1 H, N–CH–O), 5.13 (d, 1 H, *J* = 8.2, O–CH–Ph), 7.2–7.7 (m, 10 H_{arom}).

^{13}C NMR ($CDCl_3$): δ = 15.3 CH_3–C), 36.0 (CH_3–N), 64.3 (CH–N), 82.7 (CH–O), 99.1 (N–CH–O), 127.9 (CH), 128.2 (CH), 128.3 (CH), 128.7 (CH), 128.8 (CH), 129.5 (CH).

Hydrogenation of 1,3-Oxazolidines; General procedure
A glass beaker the precatalyst [Rh(dppb)COD]BF_4 (7.2 mg, 0.01 mmol) and a stirring bar was placed in a standard stainless autoclave (25 ml inner volume) equipped with the valve and connected with a vacuum pump, Ar and H_2 lines. The air was evacuated with a pump and the autoclave was filled with Ar. This cycle was repeated 2–3 times. In the flow of Ar through the open valve MeOH (10 mL) and liquid substrate (5 mmol) were added with a syringe (solid sub-

Table 3 ^{13}C NMR Characterization of 1,3-Oxazolidines **1a–j** in $CDCl_3$

Compound	1,3-oxozolidine ring position			R^1	R^2	R^3
	C-2	C-4	C-5			
1a	86.7 (CH_2)	52.1 (CH_2)	58.1 (CH_2)	63.34 (CH_2), 127.3 (CH), 128.5 (CH), 128.8 (CH), 139.1 (C)		
1b	98.5 (CH)	54.9 (CH_2)	65.5 (CH_2)	38.4 (CH_3)	127.8 (CH), 128.5 (CH), 128.9 (CH), 139.3 (C)	
1c	96.0 (CH)	54.7 (CH_2)	64.8 (CH_2)	38.7 (CH_3)	19.1 (CH_3), 125.8 (CH), 127.4 (CH), 128.4 (CH), 130.6 (CH), 136.5 C), 137.2 (C)	
1d	98.1 (CH)	54.4 (CH_2)	64.9 (CH_2)	37.5 (CH_3)	115.2 (CH), 128.5 (C), 128.9 (CH) 157.8 (C)	
1e	91.5 (CH)	53.9 (CH_2)	64.9 (CH_2)	38.8 (CH_3)	108.9 (CH), 110.0 (CH), 142.9 (CH), 151.6 (C)	
1f[a]	101.2, 102.0 (CH)	55.0, 55.3 (CH_2)	64.6, 65.1 (CH_2)	40.2, 41.2 (CH_3)	15.5, 17.5 (CH_3), 43.3, 43.4 (CH); 126.4, 126.6, 128.2, 128.3, 128.5, 1288.6 (CH); 143.3, 144.2 (C)	
1g	97.5 (CH)	54.9 (CH_2)	64.1 (CH_2)	39.0 (CH_3)	14.2 (CH_3), 22.8 (CH_2), 25.1 (CH_2), 29.4 (CH_2), 29.9 (CH_2), 31.9 (CH_2), 33,4 (CH_2)	
1h	98.8 (C)	53.7 (CH_2)	62.9 (CH_2)	37.8 (CH_3)	126.0 (CH), 127.4 (CH), 128.1 (CH); 145.2 (C)	24.2 (CH_3)
1i	97.1 (CH)	54.5 (CH_2)	65.6 (CH_2)	38.8 (CH_3)	123.5 (CH), 128.6 (CH), 147.1 (C), 148.3 (C)	
1j	91.7 (CH)	48.1 (CH_2)	65.1 (CH_2)	113.1 (CH), 117.6 (CH), 127.0 (CH), 128.6 (CH), 128.8 (CH), 129.1 (CH), 139.8 (C), 145.7 (C)		

[a] Mixture of 2 diastereoisomers in 1.5:1 ratio.

strates **1d**,**j** and **3** were placed in the reaction vessel together with the catalyst). The valve was closed and the autoclave was pressurized with H_2. The contents of the autoclave were stirred with a magnetic stirrer. The consumption of H_2 was monitored by the decrease of pressure with a pressure detector. When the hydrogen consumption ceased the autoclave was opened, the solution evaporated in vacuum and the residue analyzed by NMR spectroscopy. The data for amines **2a**,**c**–**h** are given in Tables 4 and 5.

(*S*)-*N*-Benzylprolinol (4)

$[\alpha]_D^{23}$ −58 (*c* 2, $CHCl_3$) {Lit.[11] $[\alpha]_D^{25}$ −59.9 (*c* 2, $CHCl_3$)}.

^1H NMR ($CDCl_3$): δ = 1.62–1.77 (m, 2 H), 1.77–2.0 (m, 2 H), 2.24–2.35 (m, 1 H), 2.68–2.80 (m, 1 H), 2.93–3.04 (m, 1 H), 3.36 (d, 1 H, *J* = 13.1, $PhCH_a$), 3.41–3.51 (m, 1 H), 3.61–3.69 (m, 1 H), 3.98 (d, 1 H, *J* = 13.1, $PhCH_b$), 7.2–7.4 (m, 5 H_{arom}).

^{13}C NMR ($CDCl_3$): δ = 23.7 (CH_2), 28.0 (CH_2), 54.7 (CH_2), 58.8 (CH_2), 62.1 (CH_2), 64.6 (CH), 127.4 (CH), 128.6 (CH), 127.4 (CH), 139.3 (C).

(1*R*, 2*S*)-*N*-Benzylephedrine (6)

Mp 48–49 °C (hexane) (Lit.[12] 49–50 °C). $[\alpha]_D^{23}$ −29.6 (*c* 2.4, $CHCl_3$) {Lit.[11] $[\alpha]_D^{23}$ −29.5 (*c* 2.35, $CHCl_3$)}.

^1H NMR ($CDCl_3$): δ = 0.98 (d, 3 H, *J* = 6.7, CH_3–C), 2.17 (s, 3 H, CH_3N), 2.91 (dq, 1 H, *J* = 6.7, 4.9, CH–N), 3.57 (d, 1 H, *J* = 13.5, $PhCHH_a$), 3.62 (d, 1 H, *J* = 13.5, $PhCHH_b$), 4.85 (d, *J* = 4.9, CH–O), 7.18–7.35 (m, 10 H_{arom}).

^{13}C NMR ($CDCl_3$): δ = 10.2 (CH_3–C), 38.9 (CH_3–N), 59.4 CH_2Ph), 63.7 (CH–N), 73.9 (CH–O), 121.7 (CH), 126.5 (CH), 128.3 (CH), 128.5 (CH), 128.9 (CH), 139.7 (C), 142.8 (C).

Acknowledgment

Financial support from Degussa AG, Project House Catalysis (Frankfurt, Germany) and the Fonds der Chemischen Industrie is gratefully acknowledged.

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Table 4 ^{1}H NMR Characterization of 2-(dialkylamino)ethanols, $R^1R^4NCH_2CH_2OH$ ($R^4 = R^2R^3CH$), **2a**,**c**−**h** (positions of OH resonance are not indicated) in $CDCl_3$[a]

Compound	R_1 = Me	R_4	NCH_2	CH_2O
2a[b]	2.21 (s, 3 H)	$PhCH_2$; 3.55 (s, 2 H), 7.21−7.34 (m, 5 H_{arom})	2.57 (t, 2 H, *J* = 5.5)	3.61 (t, 2 H, *J* = 5.5)
2c	2.24 (s, 3 H)	2-$MeC_6H_4CH_2$; 2.08 (s, 3 H), 3.39 (s, 2 H), 6.98−7.16 (m, 4 H)	2.47 (t, 2 H, *J* = 5.5)	3.49 (t, 2 H, *J* = 5.5)
2d	2.12 (s, 3 H)	4-$HOC_6H_4CH_2$; 3.37 (s, 2 H), 6.61 (d, 2 H, *J* = 8.5), 6.98 (d, 2 H, *J* = 8.5)	2.48 (t, 2 H, *J* = 5.5)	3.56 (t, 2 H, *J* = 5.5)
2e	2.20 (s, 3 H)	2-furylmethyl; 3.54 (s, 2 H), 6.12 (dd, 1 H, *J* = 0.8 and 3.2), 6.24 (dd, 1 H, *J* = 2.0 and 3.2), 7.29 (dd, 1 H, *J* = 0.8 and 2.0)	2.50 (t, 2 H, *J* = 5.5)	3.54 (t, 2 H, *J* = 5.5)
2f[c]	2.15 (s, 3 H)	$Me(Ph)CHCH_2$; 1.16 (d, 3 H, *J* = 6.9), 2.34−2.52 (m, 2 H), 2.77−2.89 (m, 1 H), 7.06−7.24 (m, 5 H_{arom})	2.34−2.52 (m, 2 H)	3.32−3.41 (m, 2 H)
2g	2.17 (s, 3 H)	C_8H_{18}; 0.81 (t, 3 H, *J* = 7.0, CH_3), 1.15−1.27 (m, 10 H, 5 CH_2), 1.34−1.45 (m, 2 H, CH_2), 2.31 (t, 2 H, *J* = 7.6, CH_2N)	2.44 (t, 2 H, *J* = 5.4)	3.51 (t, 2 H, *J* = 5.4)
2h	2.08 (s, 3 H)	PhMeCH; 1.28 (d, 3 H, *J* = 6.8), 3.57 (q, 1 H, *J* = 6.8), 7.1−7.2 (m, 5 H_{arom})	2.32−2.52 (m, 2 H)	3.38−3.51 (m, 2 H)

[a] Coupling constants *J* in [Hz]. [b] Bp 111 °C, 5 mbar (Lit.[2] 103 °C, 1 torr). Anal. Calcd for $C_{10}H_{15}NO$: C, 72.69; H 9.15; N, 8.48. Found: C, 72.43; H, 9.17; N, 8.49. [c] Bp 115 °C, 3 mbar. Anal. Calcd for $C_{12}H_{19}NO$: C, 74.57; H 9.90; N, 7.25. Found: C, 74.68; H, 9.88; N, 7.32.

Table 5 ^{13}C NMR characterization of 2-(dialkylamino)ethanols, $R^1R^4NCH_2CH_2OH$ ($R^4 = R^2R^3CH$), **2a,c−h** in $CDCl_3$

Compound	R_1 = Me	R_4	NCH_2	CH_2O
2a	41.8	$PhCH_2$; 62.5 (CH_2), 127.4 (CH), 128.6 (CH), 129.3(CH), 138.7 (C)	58.6	58.7
2c	42.1	2-$MeC_6H_4CH_2$; 61.0 (CH_2), 125.7 (CH), 127.4 (CH), 130.1 (CH), 130.5 (CH), 136.7 (C), 137.2 (C)	58.8	59.0
2d	41.7	4-$HOC_6H_4CH_2$; 61.9 (CH_2), 115.8 (CH), 128.3 (C), 131.0 (CH), 156.3 (C)	58.2	58.7
2e	42.0	2-furylmethyl; 54.2 (CH_2), 109.2 (CH), 110.5 (CH), 142.5 (CH), 152.3 (C)	58.4	59.1
2f	38.6	$Me(Ph)CHCH_2$; 20.3 (CH_3), 42.3 (CH), 65.6 (CH_2), 126.7 (CH), 127.5 (CH), 128.8 (CH), 146.2 (C)	58.7	59.7
2g	42.2	C_8H_{18}; 14.5 (CH_3), 23.1 (CH_2), 27.7 (CH_2), 27.9 (CH_2), 29.8 (CH_2), 30.0 (CH_2), 32.3 (CH_2), 58.4 CH_2N)	58.9	59.5
2h	37.9	PhMeCH; 17.8 (CH_3), 63.8 (CH), 127.4 (CH), 128.1 (CH), 128.6 (CH), 143.1 (C)	55.5	58.8

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