Remarkably Efficient Charcoal-Promoted Ring-Closing Carbonylations

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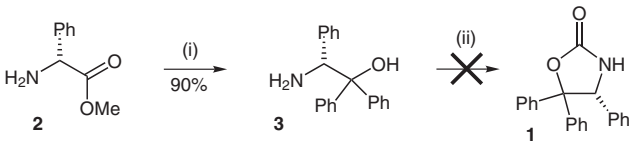
Abstract: An efficient, versatile and practical gram-scale preparation of oxazolidinone, imidazolidinone and dioxolanone is achieved.

Key words: oxazolidinones, imidazolidinones, dioxolanones, phosgenes

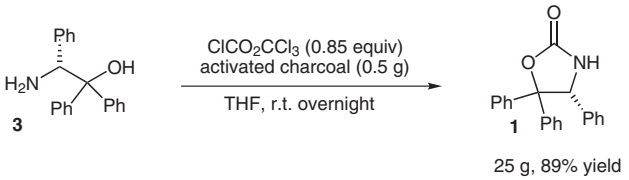
Ring-closing carbonylation of vicinal diamines, aminoalcohols, and diols is a very useful class of reaction, leading to highly versatile imidazolidinones, oxazolidinones and dioxolanones. However, despite the development of many methodologies, these transformations remain sometimes challenging.

In the course of our research program directed towards the study of new allenyl copper reagents, we focused on these reactions to obtain analytical samples suitable for structure determinations as well as multi-gram amounts of chiral inductors such as enantiopure oxazolidinone **1**. A practical synthesis of oxazolidinone **1** reported by Davies began with the addition of excess PhMgBr to α-amino methyl ester **2** to afford the corresponding aminoalcohol **3**, followed by a ring-closing carbonylation using 1,1′-carbodiimidazole (CDI).[1] However, we were unable to achieve this last transformation on the gram scale. Other attempts using diphosgene with triethylamine in dichloromethane[2] gave poorly reproducible results (Scheme 1).



Scheme 1 *Reagents and conditions:* (i) PhMgBr (6.5 equiv), Et_2O; (ii) CDI, CH_2Cl_2 or diphosgene, Et_3N, CH_2Cl_2.

An alternate protocol of ring-closing carbonylation has been reported for the synthesis of *N*-carboxy anhydrides from α-aminoacids.[3,4] This two-step reaction implies an in situ decomposition of diphosgene promoted by a catalytic amount of activated charcoal[3] and the subsequent cyclization of the amino acid by the resulting phosgene. This procedure allows an easy and safe access to phosgene solutions. We decided to transpose it to the ring-closing carbonylation of aminoalcohols. A solution of the aminoalcohol **3** (2 mmol, 578 mg) in THF was thus added to a suspension of diphosgene (0.85 equiv) and activated charcoal (10 mg) in THF and the resulting mixture was stirred overnight at room temperature. A simple filtration through Celite followed by standard workup cleanly affords oxazolidinone **1** in 96% yield (602 mg). Scale-up of the reaction to 89 mmol of the starting material gave the desired product with roughly the same yield (89%, 25 g) (Scheme 2). Noteworthy, the crude product required no further purification.



Scheme 2

Encouraged by this result, we checked the generality of this reaction with aminoalcohols **4**–**8** according to Scheme 2 (Table 1). In all cases, the reaction proceeded cleanly and the expected oxazolidinones **9**–**13** were obtained with good to excellent yields. Noteworthy, no racemization was observed during the process,[5] giving a smooth access to enantiopure oxazolidinones. In that way, the Evans oxazolidinone **10** was obtained, without any purification, quantitatively and in a suitable analytical purity. In selected cases a smooth heating to 50 °C was needed to ensure a good conversion (entries 1 and 4) and we observed that the presence of triethylamine as additive had a beneficial effect (entries 2 and 5). The reaction seemed to be insensitive to the nature of the starting aminoalcohol and particularly to the steric hindrance of the nitrogen substituent. Indeed, even aminoalcohol **8** where nitrogen bears a bulky *tert*-butyl group was converted into the corresponding oxazolidinone **13** with high yield. In this case the presence of triethylamine was required.

Next, we decided to expand the scope of the method. The reaction conditions were examined with various vicinal diols. The results are summarized in Table 2. As expected for the use of these less-nucleophilic reagents, longer reaction times were often required to ensure good levels of conversion. For reaction times above 72 hours (entries 4 and 5), a significant amount of 4-chlorobutyl chloroformate was formed, resulting from the ring opening of the

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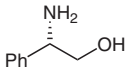
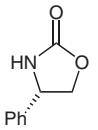
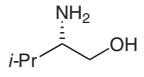
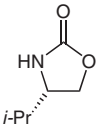
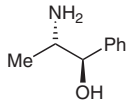
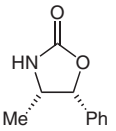
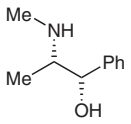
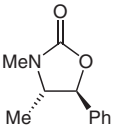
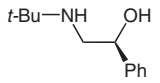
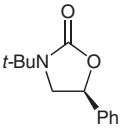
Table 1 Ring-Closing Carbonylations of Aminoalcohols **4–8**

Entry	Aminoalcohol	Product	Conditions	Yield (%)
1	 **4**	 **9**	50 °C, 48 h	96[a]
2	 **5**	 **10**	Et_3N (7 equiv) 50 °C, 48 h	97[b]
3	 **6**	 **11**	r.t., 16 h	93[a]
4	 **7**	 **12**	50 °C, 16 h	86[a]
5	 **8**	 **13**	Et_3N (7 equiv) 50 °C, 72 h	91[a]

[a] Isolated yield.
[b] The analytical purity of the crude compound was satisfactory.

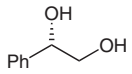
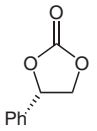
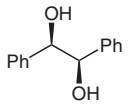
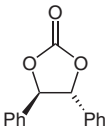
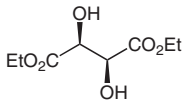
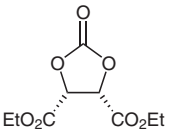
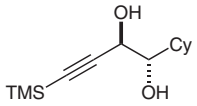
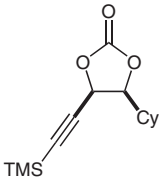
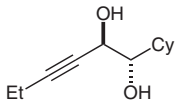
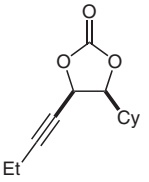
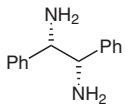
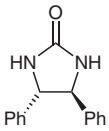
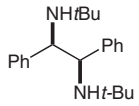
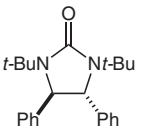
THF by phosgene. This side product was quantitatively converted into 4-chlorobutanol after standard workup and easily removed under reduced pressure (0.1 mm Hg). However, due to this side reaction, further addition of diphosgene until the completion of the reaction was needed. Nevertheless, the expected dioxolanones **21–25** were cleanly provided in high yield, ranging from 80% to 93%. Interestingly, the presence of sensitive groups such as esters, propargylic alcohols or (trimethylsilyl)acetylenic moiety is compatible with our methodology since no degradation or epimerization products were detected in the crude mixture (entries 3–5).

Finally, the observation of an easy reaction with the hindered aminoalcohol **8** prompted us to examine the limits of the method. The reactivity of diamines was thus addressed. As expected, imidazolidinone **26** was easily obtained from the Corey diamine **19** (Table 2, entry 6). More challenging was the case of the very sterically hindered D,L-diamine **20**. Even in this case, the reaction proceeded efficiently leading to the formation of the corresponding oxazolidinone **27** in a good yield (78%). The completion of the reaction required a longer reaction time (4 h) and then these conditions resulted in the formation of high quantities of 4-chlorobutyl chloroformate, which necessitated the portionwise addition of 1.7 equivalents of diphosgene. To avoid the formation of this side product and thus reduce the quantities of diphosgene used, the reaction was conducted in THP with 0.85 equivalent of diphosgene and we were pleased to note that under these conditions the desired product **27** was obtained after only 96 hours with an excellent yield (98%, entry 7). Only trace amount of 5-chloropentanol was detected in the crude mixture and was easily removed under high vacuum to give the analytically pure imidazolidinone **27**. On the contrary, almost no reaction occurred in dichloromethane.

In conclusion, we have developed a practical, safe and versatile methodology for the preparation of oxazolidinones, dioxolanones and imidazolidinones in high yield. In many cases, no further purifications are needed. Furthermore, this methodology still works even with very bulky substituents.

Experiments were carried out under dry argon. All glassware was dried at 120 °C and assembled while hot under a stream of dry nitrogen. All moisture-sensitive reactants were handled under argon. The flask was equipped with an internal thermometer, an argon inlet, and a septum cap. THF and tetrahydropyran (THP) were distilled from sodium/benzophenone ketyl. Column chromatography was performed on silica gel Si 60 (0.015–0.040 mesh). Melting points were measured with a Stuart Scientific 7SMP3 and are un-

Table 2 Synthesis of Dioxolanones **21**–**25**

Entry	Reactant	Product	Conditions	Yield (%)
1	 **14**	 **21**	50 °C, 48 h	93[a]
2	 **15**	 **22**	50 °C, 72 h	88[a]
3	 **16**	 **23**	50 °C, 120 h	90[b]
4	 **17**	 **24**	Et_3N (7 equiv) 50 °C, 120 h	80[b]
5	 **18**	 **25**	Et_3N (7 equiv) 50 °C, 120 h	87[b]
6	 **19**	 **26**	Et_3N (7 equiv) 50 °C, 16 h	88[b]
7	 **20**	 **27**	Et_3N (7 equiv) 50 °C, 96 h	98[a,c]

[a] The analytical purity of the crude compound was satisfactory.
[b] Isolated yield.
[c] THP was used as solvent instead of THF.

corrected. Optical rotations were measured at 20 °C with a Perkin-Elmer 343 polarimeter. IR data were recorded on a Bruker Tensor 27 instrument. ^{1}H NMR spectra were recorded at 400 MHz, and ^{13}C NMR spectra were recorded at 100 MHz, in $CDCl_3$ as solvent on a Bruker Ultra Shield 400 spectrometer. Chemical shifts are reported in ppm (reference $CDCl_3$ for ^{1}H NMR and ^{13}C NMR). Microanalyses were performed by ICSN-CNRS, Gif-sur-Yvette, France.

General Procedure
Method A

To a suspension of diphosgene (0.20 mL, 1.7 mmol), and activated charcoal (10 mg) in anhyd THF (10 mL), was added aminoalcohol or diol or diamine (2 mmol) in THF (10 mL). The reaction mixture was stirred at 50 °C until no starting material could be detected by NMR. The solution was then filtered through Celite, quenched with sat. $NaHCO_3$ (30 mL) and extracted with Et_2O. The combined organic layers were washed with brine, dried with $MgSO_4$ and filtered off. The solvents were removed under reduced pressure to afford the corresponding oxazolidinone or dioxolanone or imidazolidinone.

When needed, the crude product was purified by flash chromatography (FC) on silica gel.

Method B

To a suspension of diphosgene (0.20 mL, 1.7 mmol), Et_3N (2 mL) and activated charcoal (10 mg) in anhyd THF (10 mL), was added aminoalcohol or diol or diamine (2 mmol) in THF (10 mL). The reaction mixture was stirred at 50 °C until no starting material could be detected by NMR. Workup procedure as for method A was followed.

(4*R*)-4,5,5-Triphenyloxazolidin-2-one (1)

This compound was prepared according to method A, using (*R*)-2-amino-1,2,2-triphenylethanol (25.9 g, 89 mmol). Oxazolidin-2-one **1** (25.0 g, 89%) was obtained as a white solid without further purification; mp 245 °C; $[\alpha]_D^{20}$ +198 (*c* = 1.00, $CHCl_3$) {Lit.[1b] $[\alpha]_D^{25}$ +218 (*c* = 1.00, $CHCl_3$)}.

IR (neat): 274, 1755, 1727 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 5.54 (s, 1 H, CH), 5.64 (s, 1 H, NH), 7.08–7.71 (m, 15 H, Ph).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 65.8, 90.7, 126.2, 126.5, 127.3, 127.5, 127.8, 128.3, 128.4, 128.5, 128.7, 137.1, 138.8, 142.8, 158.2.

Anal. Calcd for $C_{21}H_{17}NO_2$: C, 79.98; H, 5.43; N, 4.44; O, 10.15. Found: C, 79.95; H, 5.43; N, 4.42.

(4*S*)-4-Phenyloxazolidin-2-one (9)

This compound was prepared according to method A, using (*S*)-(+)-2-phenylglycinol (274 mg, 2 mmol). Purification by FC (pentane–Et_2O, 50:50) gave oxazolidin-2-one **9** (313 mg, 96%) as a white solid; mp 114 °C; $[\alpha]_D^{20}$ +55 (*c* = 1.00, $CHCl_3$) {Lit.[6] $[\alpha]_D^{25}$ +59 (*c* = 1.00, $CHCl_3$)}.

IR (neat): 3229, 1701 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 4.14 (dd, *J* = 6.8, 8.6 Hz, 1 H, H-5), 4.71 (t, *J* = 8.6 Hz, 1 H, H-4), 4.95 (t, *J* = 8.6 Hz, 1 H, H-5), 6.78 (s, 1 H, NH), 7.28–7.41 (m, 5 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 56.4, 72.5, 126.0, 128.7, 129.1, 139.7, 160.3.

Anal. Calcd for $C_9H_9NO_2$: C, 66.25; H, 5.56; N, 8.58; O, 19.61. Found: C, 66.38; H, 5.65; N, 8.52.

(4*S*)-4-Isopropyloxazolidin-2-one (10)

This compound was prepared according to method B, using (*S*)-(+)-2-amino-3-methyl-1-butanol (0.22 mL, 2 mmol). Oxazolidin-2-one **10** (250 mg, 97%) was obtained as a pale yellow solid without further purification; mp 59 °C; $[\alpha]_D^{20}$ –18 (*c* = 1.00, EtOH) {Lit.[6] $[\alpha]_D^{25}$ –17 (*c* = 1.02, EtOH)}.

IR (neat): 3263, 1720 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 0.87 (d, *J* = 6.6 Hz, 3 H, CH_3), 0.93 (d, *J* = 6.6 Hz, 3 H, CH_3), 1.63 (m, 1 H, H-4), 3.59 (q, *J* = 6.6, 8.3 Hz, 1 H, CH), 4.06 (dd, *J* = 6.3, 8.6 Hz, 1 H, H-5), 4.41 (t, *J* = 8.6 Hz, 1 H, H-5), 7.22 (s, 1 H, NH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 17.63, 17.94, 32.69, 58.42, 68.62, 160.8.

Anal. Calcd for $C_6H_{11}NO_2$: C, 55.80; H, 8.58; N, 10.84; O, 24.78. Found: C, 55.81; H, 8.64; N, 10.73.

(4*R*,5*S*)-4-Methyl-5-phenyloxazolidin-2-one (11)

This compound was prepared according to method A, using (1*R*,2*S*)-(–)-norephedrin (300 mg, 2 mmol). Oxazolidin-2-one **11** (329 mg, 93%) was obtained as a pale yellow solid without further purification; mp 89 °C; $[\alpha]_D^{20}$ –166 (*c* = 1.00, $CHCl_3$) {Lit.[7] $[\alpha]_D^{20}$ –170 (*c* = 1.20, $CHCl_3$)}.

IR (neat): 3244, 1710 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 0.79 (d, *J* = 6.6 Hz, 3 H, CH_3), 4.19 (m, 1 H, H-4), 5.68 (d, *J* = 8.1 Hz, 1 H, H-5), 7.04 (s, 1 H, NH), 7.26–7.39 (m, 5 H, Ph).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 17.5, 52.5, 81.1, 125.8, 128.2, 128.5, 135.1, 160.1.

Anal. Calcd for $C_{10}H_{11}NO_2$: C, 67.78; H, 6.26; N, 7.90; O, 18.06. Found: C, 67.78; H, 6.26; N, 7.65.

(4*S*,5*S*)-3,4-Dimethyl-5-oxazolidin-2-one (12)

This compound was prepared according method A, using (1*S*,2*S*)-(+)-pseudoephedrine (330 mg, 2 mmol). Oxazolidin-2-one **12** (328 mg, 86%) was obtained as a pale yellow solid without further purification; mp 47 °C; $[\alpha]_D^{20}$ +37 (*c* = 1.00, $CHCl_3$) {Lit.[8] $[\alpha]_D^{25}$ +36.7 (*c* = 0.98, $CHCl_3$)}.

IR (neat): 3479, 1737 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.19 (d, *J* = 6.3 Hz, 3 H, CH_3), 2.70 (s, 1 H, H-4), 3.39 (m, 1 H, H-5), 4.74 (d, *J* = 6.3 Hz, 3 H, NCH_3), 7.19–7.33 (m, 5 H, Ph).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 17.1, 28.6, 61.1, 82.3, 125.9, 128.8, 128.9, 137.6, 157.7.

Anal. Calcd for $C_{11}H_{13}NO_2$: C, 69.09; H, 6.86; N, 7.32; O, 16.73. Found: C, 67.69; H, 6.86; N, 6.95.

(5*S*)-3-*tert*-Butyl-5-oxazolidin-2-one (13)

This compound was prepared according to method B, using (*S*)-2-(*tert*-butylamino)-1-phenylethanol (386 mg, 2 mmol). Purification by FC (pentane–Et_2O, 50:50) gave oxazolidin-2-one **13** (400 mg, 91%) as a pale yellow oil; $[\alpha]_D^{20}$ +59 (*c* = 1.20, $CHCl_3$) {Lit.[9] $[\alpha]_D^{25}$ +49 (*c* = 1.00, $CHCl_3$)}.

IR (neat): 2974, 1736 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.39 [s, 9 H, $(CH_3)_3$], 3.44 (dd, *J* = 7.8, 8.6 Hz, 1 H, H-4), 3.96 (t, *J* = 8.6 Hz, 1 H, H-4), 5.35 (t, *J* = 7.8 Hz, 1 H, H-5), 7.28–7.55 (m, 5 H, Ph).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 27.4, 51.1, 53.5, 73.5, 125.5, 128.7, 128.8, 138.9, 156.8.

Anal. Calcd for $C_{13}H_{17}NO_2$: C, 71.21; H, 7.81; N, 6.39; O, 14.59. Found: C, 69.29; H, 7.75; N, 6.55.

(4*S*)-4-Phenyl[1,3]dioxolan-2-one (21)

This compound was prepared according to method A, using (*S*)-1-phenylethane-1,2-diol (276 mg, 2 mmol). Dioxolan-2-one **21** (306 mg, 93%) was obtained as a white solid without further purification; mp 64 °C; $[\alpha]_D^{20}$ –44 (*c* = 1.00, $CHCl_3$) {Lit.[10] $[\alpha]_D^{20}$ –31 (*c* = 1.00, $CHCl_3$)}.

IR (neat): 3354, 2926, 1773 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 4.34 (t, *J* = 8.3 Hz, 1 H, H-5), 4.81 (t, *J* = 8.3 Hz, 1 H, H-5), 5.68 (t, *J* = 8.3 Hz, 1 H, H-4), 7.22–7.55 (m, 5 H, Ph).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 71.3, 78.1, 125.9, 129.3, 129.7, 135.9, 155.0.

Anal. Calcd for $C_9H_8O_3$: C, 65.85; H, 4.91; O, 29.24. Found: C, 65.82; H, 4.97.

(4*R*,5*R*)-4,5-Diphenyl[1,3]dioxolan-2-one (22)

This compound was prepared according to method A, using (1*R*,2*R*)-1,2-diphenylethane-1,2-diol (363 mg, 1.7 mmol). Dioxolan-2-one **22** (460 mg, 96%) was obtained as a pale yellow solid without further purification; mp 122 °C; $[\alpha]_D^{20}$ +66 (*c* = 1.00, $CHCl_3$) {Lit.[11] $[\alpha]_D^{20}$ +66 (*c* = 1.05, $CHCl_3$)}.

IR (neat): 2945, 2857, 1813 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.46 (s, 2 H, H-4, H-5), 7.17–7.64 (m, 10 H, Ph).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 85.4, 126.2, 129.3, 129.8, 134.8, 154.1.

Anal. Calcd for $C_{15}H_{12}O_3$: C, 74.99; H, 5.03; O, 19.98. Found: C, 71.94; H, 6.07.

(4*S*,5*S*)-2-Oxo[1,3]dioxolane-4,5-dicarboxylic Acid Diethyl Ester (23)

This compound was prepared according to method A, using (–)-diethyl D-tartrate (0.34 mL, 2 mmol) and adding further diphosgene (0.03 mL, 0.13 equiv) after 72 h. Purification by FC (pentane–Et_2O, 60:40) gave dioxolan-2-one **23** (420 mg, 90%) as a pale yellow oil; $[\alpha]_D^{20}$ +23 (*c* = 1.30, $CHCl_3$).

IR (neat): 2855, 1751, 1840 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.37 (t, *J* = 7.1 Hz, 6 H, CH_3), 4.37 (q, *J* = 7.1 Hz, 4 H, $COOCH_2$), 5.12 (s, 2 H, H-4, H-5).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 63.4, 74.8, 167.9, 166.1.

Anal. Calcd for $C_{19}H_{12}O_7$: C, 46.56; H, 5.21; O, 48.24. Found: C, 46.65; H, 5.21.

4-Cyclohexyl-5-trimethylsilanylethynyl[1,3]dioxolan-2-one (24)

This compound was prepared according to method B, using 1-cyclohexyl-4-trimethylsilanyl-but-3-yne-1,2-diol (72 mg, 0.3 mmol) and adding further diphosgene (0.08 mL, 0.3 equiv) after 72 h. Purification by FC (pentane–Et_2O, 50:50) gave dioxolan-2-one **24** (64 mg, 80%) as a pale yellow oil.

IR (neat): 2923, 2852, 1813 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.23 [s, 9 H, $(CH_3)_3$], 0.88–2.19 (m, 11 H, Cy), 4.32 (dd, *J* = 7.3, 8.6 Hz, 1 H, H-5), 5.27 (d, *J* = 7.3 Hz, 1 H, H-4).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 0.00, 25.6, 25.8, 26.5, 28.7, 29.1, 39.6, 70.6, 83.2, 95.9, 98.2, 162.7.

Anal. Calcd for $C_{14}H_{22}O_3Si$: C, 63.12; H, 8.32; O, 18.02; Si, 10.54. Found: C, 63.17; H, 8.38.

4-But-1-ynyl-5-cyclohexyl[1,3]dioxolan-2-one (25)

This compound was prepared according to method B, using 1-cyclohexylhex-3-yne-1,2-diol (135 mg, 0.69 mmol). Purification by FC (pentane) gave dioxolan-2-one **25** (135 mg, 87%) as a pale yellow oil.

IR (neat): 2927, 2854, 1802 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.18 (t, *J* = 7.6 Hz, 3 H, CH_3), 1.94–2.17 (m, 11 H, Cy), 2.29 (dq, *J* = 2.0, 7.3 Hz, 2 H, CH_2), 4.29 (dd, *J* = 7.3, 8.8 Hz, 1 H, H-5), 5.26 (dt, *J* = 2.0, 7.3 Hz, 1 H, H-4).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.3, 14.5, 25.1, 25.3, 25.6, 28.1, 28.6, 38.8, 70.4, 70.7, 82.9, 93.9, 154.2.

Anal. Calcd for $C_{13}H_{18}O_3$: C, 73.34; H, 9.41; O, 17.24. Found: C, 73.45; H, 9.40.

(4*S*,5*S*)-Diphenylimidazolidin-2-one (26)

This compound was prepared according to method B, using (1*S*,2*S*)-1,2-diphenylethane-1,2-diamine (424 mg, 2 mmol). Purification by FC (pentane–Et_2O, 20:80) gave imidazolidin-2-one **26** (420 mg, 88%) as a pale yellow solid; mp 156 °C; $[\alpha]_D^{20}$ –54 (*c* = 1.00, $CHCl_3$) {Lit.[12] $[\alpha]_D^{20}$ +58.6 (*c* = 1.06, $CHCl_3$) for the *R*,*R* derivative}.

IR (neat): 3210, 1698 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 4.54 (s, 2 H, H-4, H-5), 6.08 (s, 2 H, NH), 7.38–7.25 (m, 10 H, Ph).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 65.9, 126.5, 128.3, 128.8, 140.4, 163.5.

Anal. Calcd for $C_{15}H_{14}N_2O$: C, 75.61; H, 5.92; N, 11.76; O, 6.71. Found: C, 72.07; H, 5.92; N, 10.83.

1,3-Di-*tert*-butyldiphenylimidazolidin-2-one (27)

This compound was prepared according to method B, using *N,N*-di-*tert*-butyl-1,2-diphenylethane-1,2-diamine (649 mg, 2 mmol) and freshly distilled THP instead of THF as solvent. Imidazolidin-2-one **27** (686 mg, 98%) was obtained as a white solid without further purification; mp 140 °C.

IR (neat): 3205, 1742 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.29 [s, 18 H, $(CH_3)_3$], 4.21 (s, 2 H, H-4, H-5), 7.13–7.51 (m, 10 H, Ph).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 28.8, 53.7, 65.5, 125.5, 127.8, 129.0, 145.0, 159.7.

Anal. Calcd for $C_{24}H_{33}N_2O$: C, 78.82; H, 8.63; N, 7.99; O, 4.56. Found: C, 78.80; H, 8.64; N, 7.99.

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