SHORT PAPER

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Synthesis of chiral isoxazoline derivatives by highly diastereoface-selective 1,3-dipolar cycloaddition of nitrile oxides mediated by magnesium bromide and ytterbium trifluoromethanesulfonate†

Hidetoshi Yamamoto[a]*, Sadaka Watanabe[a], Masayuki Hasegawa[a], Michihiko Noguchi[a] and Shuji Kanemasa[b]

[a]*Department of Applied Chemistry and Chemical Engineering, Yamaguchi University, Tokiwadai, Ube 755-8611, Japan*

[b]*Institute of Advanced Material Study, Kyushu University, Kasugakoen, Kasuga 816-8580, Japan*

In the presence of an equimolar amount of Lewis acid such as magnesium bromide and ytterbium trifluoromethanesulfonate, 1,3-dipolar cycloaddition reactions of aromatic nitrile oxides to a chiral 3-acryloyl-2-oxazolidinone gave the corresponding chiral 2-isoxazolines in a diastereoselective manner.

Keywords: nitrile oxide, cycloaddition, heterocycles, diastereoselectivity,

We have recently reported[1] that, in nitrile oxide cycloadditions[2] to the electron-deficient alkene dipolarophiles bearing a chiral 2-oxazolidinone chiral auxiliary, diastereoselectivity is highly improved in the presence of an equimolar amount of magnesium(II) bromide.[3] Combined use of appropriate reaction solvent and relatively high concentration was important to attain high selectivity. Reactions using the benzonitrile oxides having an electron donating substituent at the *p*-position only provided high selectivity; diastereoselectivity disappeared in the reactions of benzonitrile oxides having an electron-withdrawing *p*-substituent. In this paper,[4] we report a much more effective stereocontrol method for asymmetric cycloadditions of aromatic nitrile oxides, both for electron rich and deficient types to afford chiral 2-isoxazoline derivatives. Not only magnesium (II) bromide but also ytterbium (III) trifluoromethanesulfonate can be effectively utilised when the reaction solvent is appropriately chosen.

In the previous work, magnesium ion was found to be the only effective metal ion, which improved diastereoselectivity of nitrile oxide cycloadditions to a 3-acryloyl-2-oxazolidinone dipolarophile. [1] Selectivity was highly dependent upon the reaction solvent, dichloromethane and acetonitrile being especially effective. Kinetic study by competitive nitrile oxide cycloadditions between dipolarophile **1** and norbornene in the presence of a Lewis acid (1 equiv.)[5] suggested that reactions using magnesium bromide in dichloromethane or acetonitrile and ytterbium trifluoromethanesulfonate in dichloromethane were highly diastereoselective, *l*-**3a** being the major stereoisomeric cycloadduct. Such effective reaction conditions with respect to Lewis acid and reaction solvent were applied to the cycloadditions of a variety of *p*-substituted benzonitrile oxides to **1** (Table 1). As mentioned above, the reaction of benzonitrile oxides having an electron-withdrawing *p*-substituent such as *p*-fluoro moiety did not show high diastereoselectivity in the presence of magnesium bromide in dichloromethane (*l*-**3d**:*u*-**3d** = 76:24). However, diastereoselectivity was fortunately much improved (96:4) simply by changing the reaction solvent from dichloromethane to acetonitrile. Similarly, *p*-chloro- and *p*-nitrobenzonitrile oxide provided cycloadducts in the selectivities of 98/2 and 97/3 in acetonitrile (64/36 and 73/27 in dichloromethane, respectively). The best selectivity was observed in the reaction of benzonitrile oxide. In the preliminary study, cycloaddition reaction between **1** and benzonitrile oxide in acetonitrile in the presence of $MgBr_2$ was carried out with 0.25 M of **1** to afford **3a** in the selectivity of 93/7. The reaction performed in a higher concentration (1.0 M) gave *l*-**3a** almost as a single isomer (99:1).

Ytterbium trifluoromethanesulfonate was also effective although the reactions were performed in dichloromethane. In all cases, the diastereoselectivities were better than 93/7 as shown in Table 1. Only in the reaction of *p*-methoxybenzonitrile oxide, yield of **3b** was decreased. The cycloadduct **3** decomposes slowly in the presence of strong Lewis acid such as ytterbium trifluoromethanesulfonate, overreaction having to be avoided.

In conclusion, our Lewis acid coordination methodology is extremely effective for the diastereocontrol in nitrile cycload-

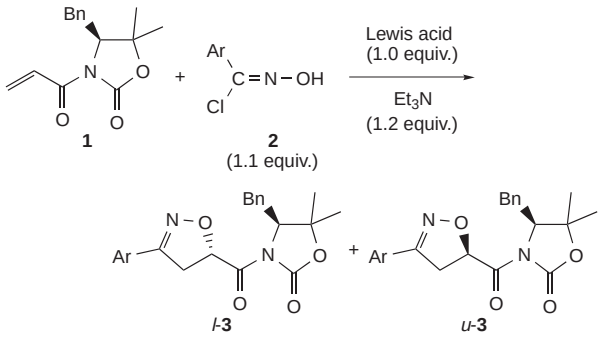

Table 1 Lewis acid-mediated 1,3-dipolar cycloadditions of nitrile oxides to **1**

Ar	Product	*l*-**3**/*u*-**3**[a]		
		no L.A. in CH_2Cl_2[b]	$MgBr_2$ in CH_3CN[c]	$Yb(OTf)_3$ in CH_2Cl_2[d]
Ph	**3a**	43/57	99/1 (74)	96/4 (71)
p-$MeOC_6H_4$	**3b**	52/48	97/3 (62)	95/5 (39)
p-FC_6H_4	**3c**	44/56	96/4 (72)	95/5 (70)
p-ClC_6H_4	**3d**	43/57	98/2 (85)	95/5 (80)
p-$O_2NC_6H_4$	**3e**	39/61	97/3 (63)	93/7 (76)

[a]Isolated yield is in parentheses. [b]1, 0.17 M; 0 °C, 6 h. [c]1, 1.00 M, 0 °C, 6 h. [d]1, 0.08 M; r.t., 3 h.

*To receive any correspondence. E-mail: h-ymmt@yamaguchi-u.ac.jp

† This is a Short Paper, there is therefore no corresponding material in *J Chem. Research (M).*

dition reactions to an electron-deficient dipolarophile bearing an oxazolidinone chiral auxiliary.[6] Combined use of appropriate reaction solvent and Lewis acid was important to attain chiral 2-isoxazoline derivatives with high selectivity for the diastereoface-selective cycloadditions of nitrile oxides bearing an electron-deficient substituent as well as ones have an electron-donating substituent.

Experimental

Commercial reagents were used without further purification unless stated. Solvents were distilled over calcium hydride. Magnesium bromide and ytterbium trifluoromethanesulfonate were purchased from Aldrich and Tokyo Kasei Kogyo, respectively, and dried at 140 °C for 24 h under a reduced pressure each time just before its use. Hydroximoyl chlorides **2a**–**e** were prepared from the corresponding oximes.[7] The IR spectra were taken with a JASCO IR-Report-100 spectrophotometer. The ^{1}H and ^{13}C NMR spectra were recorded on a JEOL EX-270 (270 MHz for ^{1}H and 67.94 MHz for ^{13}C NMR) instruments. Chemical shifts are expressed in ppm downfield from tetramethylsilane as an internal standard.

*General procedure for the cycloaddition between dipolarophile **1** and benzonitrile oxides in the presence of Lewis acid*: To a Lewis acid (0.250 mmol) were added a solution of the dipolarophile **1** (64.8 mg, 0.250 mmol) and hydroximoyl chloride **2** (0.275 mmol) in dry solvent (see Table 1) and triethylamine (42 μl, 0.300 mmol) at 0 °C under N_2. The mixture was stirred at 0 °C for 6 h, quenched with brine, and extracted with ethyl acetate (10 ml × 4). The combined extracts were dried over anhydrous $MgSO_4$ and concentrated under a reduced pressure. The residue was chromatographed on silica gel with hexane-ethyl acetate (5/1 v/v) as an eluent to afford the cycloadducts as a mixture of diastereo-isomers. The major isomers *l*-**3a** and *l*-**3c** were isolated by recrystallisation from EtOH.

*(S)-4-Benzyl-5,5-dimethyl-3-[(S)-3-phenyl-2-isoxazoline-5-carbonyl]-1,3-oxazolidin-2-one (l-**3a**) and (S)-4-benzyl-5,5-dimethyl-3-[(R)-3-phenyl-2-isoxazoline-5-carbonyl]-1,3-oxazolidin-2-one (u-**3a**)*: *l*-**3a:** colourless prisms (form EtOH); m.p. 132.2 – 133.2 °C; $[\alpha]_D^{25}$ = 109.9 ° (*c* 1.01, $CHCl_3$); IR (KBr) 600, 620, 680, 700, 740, 760, 850, 860, 910, 938, 960, 980, 1080, 1098, 1160, 1202, 1230, 1242, 1268, 1290, 1348, 1380, 1438, 1480, 1598, 1710, 1760, 2960 and 3000 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ = 1.36 and 1.43 (each 3H, s, Me-5), 2.90 (1H, dd, J_{gem}=14.3 and J_{vic}=9.6 Hz, one of CH_2-4), 3.28 (1H, dd, J_{gem}=14.3 and J_{vic}=3.6 Hz, the other of CH_2-4), 3.54 (1H, dd, J_{gem}=17.2 and $J_{4'\text{-}5'}$=6.1 Hz, one of H-4'), 3.78 (1H, dd, J_{gem}=17.2 and $J_{4'\text{-}5'}$=11.6 Hz, the other of H-4'), 4.50 (1H, dd, J_{vic}=9.6 and 3.6 Hz, H–4), 6.12 (1H, dd, $J_{5'\text{-}4'}$=11.6 and 6.1 Hz, H-5'), 7.18 – 7.30, 7.38 – 7.42 and 7.64 – 7.70 (each 5H, 3H, 2H, m, Ph); ^{13}C NMR ($CDCl_3$) δ = 22.37, 28.72, 34.77, 38.74, 63.90, 78.22, 83.40, 126.86, 128.61, 128.68, 128.73, 128.97, 130.35, 136.44, 152.27, 155.72 and 169.63. *u*-**3a:** ^{1}H NMR ($CDCl_3$) δ = 1.45 (6H, s, Me-5), 2.96 (1H, dd, J_{gem}=14.4 and J_{vic}=8.9 Hz, one of CH_2-4), 3.04 (1H, dd, J_{gem}=11.9 and 5.9 Hz, H-5'). Other signals overlap those of *l*-**3a**.

*(S)-4-Benzyl-3-[(S)-3-(p-methoxyphenyl)-2-isoxazoline-5-carbonyl]-5,5-dimethyl-1,3-oxazolidin-2-one (l-**3b**) and (S)-4-benzyl-3-[(R)-3-(p-methoxyphenyl)-2-isoxazoline-5-carbonyl]-5,5-dimethyl-1,3-oxazolidin-2-one* (*u*-**3b**): A 78 / 22 mixture of *l*-**3b** and *u*-**3b**: colourless plates (from EtOH); IR (KBr) 600, 635, 700, 730, 830, 990, 1020, 1040, 1100, 1178, 1260, 1302, 1344, 1360, 1420, 1450, 1510, 1600, 1700, 1770 and 2330 cm^{-1}; ^{1}H NMR ($CDCl_3$) *l*-**3b**: δ = 1.36 and 1.43 (each 3H, s, Me-5), 2.90 (1H, dd, J_{gem}=14.2 and J_{vic}=9.7 Hz, one of CH_2-4), 3.28 (1H, dd, J_{gem}=14.2 and J_{vic}=3.6 Hz, the other of CH_2-4), 3.50 (1H, dd, J_{gem}=17.2 and $J_{4'\text{-}5'}$=5.9 Hz, one of H-4'), 3.76 (1H, dd, J_{gem}=17.2 and $J_{4'\text{-}5'}$=11.6 Hz, the other of H-4'), 3.84 (3H, s, MeO), 4.50 (1H, dd, J_{vic}=9.7 and 3.6 Hz, H-4), 6.08 (1H, dd, $J_{5'\text{-}4'}$=11.6 and 5.9 Hz, H-5'), 6.92 (2H, d, J_{vic}=9.8 Hz, Ar), 7.21 – 7.30 (5H, m, Ph), and 7.62 (2H, d, J_{vic}=8.9 Hz, Ar). *u*-**3b**: δ = 1.44 (3H, s, Me-5), 2.96 (1H, dd, J_{gem}=14.5 and J_{vic}=9.0 Hz, one of CH2-4), 3.11 (1H, dd, J_{gem}=14.5 and J_{vic}=4.5 Hz, the other of CH2-4), 3.85 (3H, s, MeO), 4.52 (1H, dd, J_{vic}=9.0 and 4.5 Hz, H-4), 6.03 (1H, dd, $J_{5'\text{-}4'}$=11.5 and 5.9 Hz, H-5'). Other signals are overlapping with those of *l*-**3b**.; ^{13}C NMR ($CDCl_3$) *l*-**3b**: δ = 22.37, 28.74, 34.74, 39.03, 55.29, 63.90, 78.01, 83.36, 114.09, 121.13, 126.85, 128.43, 128.72, 128.97, 136.48, 152.27, 155.24, 161.22 and 169.83. *u*-**3b**: δ = 22.19, 28.43, 35.20, 39.34, 63.33, 77.70, 83.74, 126.94, 128.63, 129.06, 136.12, 152.42 and 169.27. Other signals overlap those of *l*-**3b**.

(S)-4-Benzyl-3-[(S)-3-(p-fluorophenyl)-2-isoxazoline-5-carbonyl]-5,5-dimethyl-1,3-oxazolidin-2-one (*l*-**3c**) *and (S)-4-benzyl-3-[(R)-3-(p-fluorophenyl)-2-isoxazoline-5-carbonyl]-5,5-dimethyl-1,3-oxazolidin-2-one (u-**3c**)*: *l*-**3c:** colourless prisms (from EtOH); $[\alpha]_D^{25}$ = 123.9 ° (*c* 1.01, $CHCl_3$); m.p. 132.9 – 133.7 °C; IR (KBr) 620, 696, 724, 820, 860, 884, 950, 1098, 1140, 1162, 1200, 1220, 1240, 1302, 1340, 1380, 1446, 1500, 1688, 1700 and 1760 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ = 1.36 and 1.43 (each 3H, s, Me-5), 2.90 (1H, dd, J_{gem}=14.5 and J_{vic}=9.6 Hz, one of CH_2-4), 3.27 (1H, dd, J_{gem}=14.5 and J_{vic}=4.0 Hz, the other of CH_2-4), 3.52 (1H, dd, J_{gem}=16.8 and $J_{4'\text{-}5'}$=6.3 Hz, one of H-4'), 3.75 (1H, dd, J_{gem}=16.8 and $J_{4'\text{-}5'}$=11.6 Hz, the other of H-4'), 4.50 (1H, dd, J_{vic}=9.6 and 4.0 Hz, H-4), 6.12 (1H, dd, $J_{5'\text{-}4'}$=11.6 and 6.3 Hz, H-5'), 7.05 – 7.14, 7.18 – 7.33, and 7.64 – 7.71 (each 2H, 5H, and 2H, m, Ar); ^{13}C NMR ($CDCl_3$) δ = 22.39, 28.77, 34.81, 38.76, 63.95, 78.33, 83.45, 115.90 (d, $J_{C\text{-}F}$=22.0 Hz), 124.92 (d, $J_{C\text{-}F}$=3.7 Hz), 126.92, 128.77, 128.89 (d, $J_{C\text{-}F}$=8.5 Hz), 128.99, 136.42, 125.29, 154.81, 163.91 (d, $J_{C\text{-}F}$=250.2 Hz) and 169.56. *u*-**3c**: ^{1}H NMR ($CDCl_3$) δ = 1.45 and 1.46 (each 3H, s, Me-5), 2.97 (1H, dd, J_{gem}=14.2 and J_{vic}=8.7 Hz, one of CH_2-4), 3.11 (1H, dd, J_{gem}=14.2 and J_{vic}=4.6 Hz, the other of CH_2-4), 3.30 (1H, dd, J_{gem}=17.2 and $J_{4'\text{-}5'}$=5.9 Hz, one of H-4'), 3.73 (1H, dd, J_{gem}=17.2 and $J_{4'\text{-}5'}$=11.7 Hz, the other of H-4'), 4.52 (1H, dd, J_{vic}=8.7 and 4.6 Hz, H-4), 6.07 (1H, dd, $J_{5'\text{-}4'}$=11.7 and 5.9 Hz, H-5'). Other signals overlap those of *l*-**3c**.

(S)-4-Benzyl-3-[(S)-3-(p-chlorophenyl)-2-isoxazoline-5-carbonyl]-5,5-dimethyl-1,3-oxazolidin-2-one (*l*-**3d**) *and (S)-4-benzyl-3-[(R)-3-(p-chlorophenyl)-2-isoxazoline-5-carbonyl]-5,5-dimethyl-1,3-oxazolidin-2-one* (*u*-**3d**): A 81 / 19 mixture of *l*-**3d** and *u*-**3d**: colourless solid (from EtOH); IR (KBr) 600, 620, 696, 720, 740, 820, 894, 960, 1000, 1098, 1150, 1170, 1200, 1250, 1340, 1364, 1380, 1420, 1440, 1488, 1550, 1590, 1690, 1708 and 1762 cm^{-1}; ^{1}H NMR ($CDCl_3$) *l*-**3d:** δ = 1.36 and 1.43 (each 3H, s, Me-5), 2.90 (1H, dd, J_{gem}=14.5 and J_{vic}=9.6 Hz, one of CH_2-4), 3.26 (1H, dd, J_{gem}=14.5 and J_{vic}=4.0 Hz, the other of CH_2-4), 3.51 (1H, dd, J_{gem}=16.8 and $J_{4'\text{-}5'}$=6.3 Hz, one of H-4'), 3.74 (1H, dd, J_{gem}=16.8 and $J_{4'\text{-}5'}$=11.6 Hz, the other of H-4'), 4.50 (1H, dd, J_{vic}=9.6 and 4.0 Hz, H-4), 6.13 (1H, dd, $J_{5'\text{-}4'}$=11.6 and 6.3 Hz, H-5'), 7.16 - 7.33 (5H, m, Ph), 7.38 and 7.62 (each 2H, d, J_{vic}=8.9 Hz, Ar). *u*-**3d:** δ = 1.45 and 1.46 (each 3H, s, Me-5), 2.97 (1H, dd, J_{gem}=14.5 and J_{vic}=8.9 Hz, one of CH_2-4), 3.11 (1H, dd, J_{gem}=14.5 and J_{vic}=4.6 Hz, the other of CH_2-4), 3.28 (1H, dd, J_{gem}=17.2 and $J_{4'\text{-}5'}$=6.1 Hz, one of H-4'), 3.72 (1H, dd, J_{gem}=17.2 and $J_{4'\text{-}5'}$=11.7 Hz, the other of H-4'), 4.52 (1H, dd, J_{vic}=8.9 and 4.6 Hz, H-4), 6.07 (1H, dd, $J_{5'\text{-}4'}$=11.7 and 6.1 Hz, H-5'), 7.16 - 7.33 (5H, m, Ph), 7.39 and 7.59 (each 2H, d, J_{vic}=8.6 Hz, Ar); ^{13}C NMR ($CDCl_3$) *l*-**3d:** δ = 22.39, 28.77, 34.83, 38.55, 63.94, 78.46, 83.47, 126.92, 127.15, 128.12, 128.77, 128.99, 129.02, 129.11, 136.41, 152.27, 154.88 and 169.47. *u*-**3d:** δ = 22.22, 28.47, 35.26, 38.92, 63.34, 78.13, 83.88, 127.03, 128.66, 129.22, 136.06 and 168.91. Other signals overlap those of *l*-**3d**.

(S)-4-Benzyl-3-[(S)-3-(p-nitrophenyl)-2-isoxazoline-5-carbonyl]-5,5-dimethyl-1,3-oxazolidin-2-one (*l*-**3e**) *and (S)-4-benzyl-3-[(R)-3-(p-nitrophenyl)-2-isoxazoline-5-carbonyl]-5,5-dimethyl-1,3-oxazolidin-2-one* (*u*-**3e**): A 68 / 32 mixture of *l*-**3e** and *u*-**3e**: colourless solid (from EtOH); IR (KBr) 602, 630, 700, 718, 740, 780, 842, 890, 960, 1096, 1140, 1180, 1190, 1204, 1256, 1302, 1324, 1370, 1430, 1450, 1480, 1508, 1576, 1600, 1698, 1710, 1720 and 1768 cm^{-1}; ^{1}H NMR ($CDCl_3$) *l*-**3e:** δ = 1.38 and 1.45 (each 3H, s, Me-5), 2.92 (1H, dd, J_{gem}=14.4 and J_{vic}=9.6 Hz, one of CH_2-4), 3.26 (1H, dd, J_{gem}=14.4 and J_{vic}=4.0 Hz, the other of CH_2-4), 3.60 (1H, dd, J_{gem}=17.2 $J_{4'\text{-}5'}$=6.4 Hz, one of H-4'), 3.78 (1H, dd, J_{gem}=17.2 and $J_{4'\text{-}5'}$=11.6 Hz, the other of H-4'), 4.51 (1H, dd, J_{vic}=9.6 and 4.0 Hz, H-4), 6.22 (1H, dd, $J_{5'\text{-}4'}$=11.6 and 6.4 Hz, H-5'), 7.16 - 7.33 (5H, m, Ph), 7.86 and 8.27 (each 2H, d, J_{vic}=8.9 Hz, Ar). *u*-**3e:** δ = 1.47 and 1.48 (each 3H, s, Me-5), 2.99 (1H, dd, J_{gem}=14.2 and J_{vic}=8.7 Hz, one of CH_2-4), 3.11 (1H, dd, J_{gem}=14.2 and J_{vic}=4.6 Hz, the other of CH_2-4), 3.32 (1H, dd, J_{gem}=17.2 and $J_{4'\text{-}5'}$=5.9 Hz, one of H-4'), 3.76 (1H, dd, J_{gem}=17.2 and $J_{4'\text{-}5'}$=11.9 Hz, the other of H-4'), 4.54 (1H, dd, J_{vic}=8.7 and 4.6 Hz, H-4), 6.16 (1H, dd, $J_{5'\text{-}4'}$=11.9 and 5.9 Hz, H-5'), 7.16 – 7.30 (5H, m, Ph), 7.82 and 8.28 (each 2H, d, J_{vic}=8.9 Hz, Ar); ^{13}C NMR ($CDCl_3$) *l*-**3e:** δ = 22.34, 28.70, 34.79, 37.92, 63.88, 79.03, 83.56, 123.95, 126.92, 127.62, 128.75, 128.95, 134.66, 136.26, 148.59, 152.22, 154.39 and 168.91. *u*-**3e:** δ = 22.16, 28.39, 35.20, 38.42, 63.27, 78.73, 83.99, 126.99, 128.63, 129.09, 135.99, 152.38, 154.30 and 168.45. Other signals overlap those of *l*-**3e**.

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