DOI 10.1007/s10593-018-2288-9

Chemistry of Heterocyclic Compounds **2018**, *54*(4), 428–436

Use of hypervalent iodine in the synthesis of isomeric dihydrooxazoles

Claudia Carlucci[1]*, Arianna Tota[1], Marco Colella[1], Giuseppe Ronamazzi[2], Guy J. Clarkson[3], Renzo Luisi[1]*, Leonardo Degennaro[1]

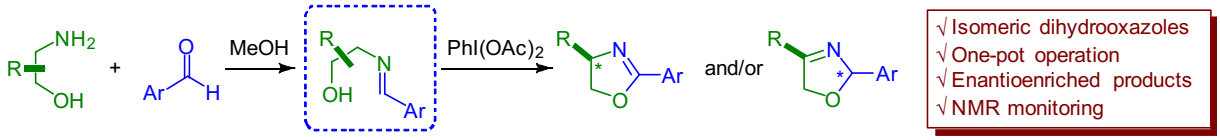
[1] *Flow Chemistry and Microreactor Technology Laboratory (FLAME-Lab), Department of Pharmacy – Pharmaceutical Sciences, University of Bari Aldo Moro, Via E. Orabona 4, Bari 70125, Italy; e-mail: claudia.carlucci@uniba.itt*

[2] *DICATECh, Politecnico di Bari, Via E. Orabona 4, Bari 70125, Italy; e-mail: g.romanazzi@poliba.it*

[3] *Department of Chemistry, University of Warwick, Gibbet Hill Road, Coventry CV4 7AL, UK; e-mail: g.clarkson@warwick.ac.uk*

Published in Khimiya Geterotsiklicheskikh Soedinenii, 2018, *54*(4), 428–436

Submitted January 29, 2018
Accepted after revision May 25, 2018



A convenient synthesis of 2- and/or 3-oxazolines has been developed depending on the structure and stereochemistry of the starting amino alcohol. $PhI(OAc)_2$ acted as oxidant on the intermediate imine, as supported by NMR investigation. The findings demonstrate a new route providing access to unusual 3-oxazolines.

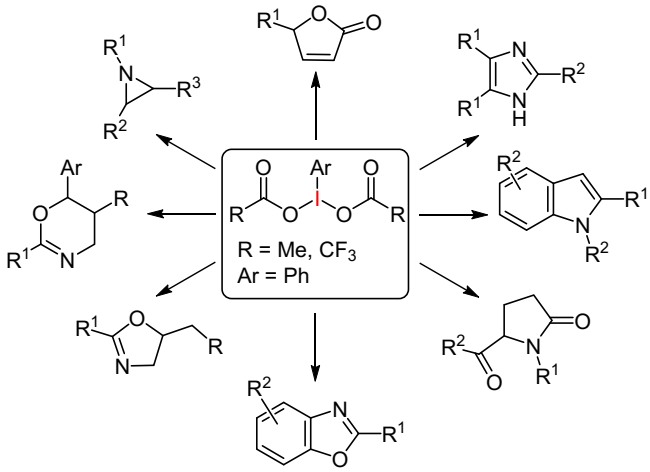
Keywords: hypervalent iodine, 2-oxazolines, 3-oxazolines, NMR studies, stereoselective synthesis.

Compounds of hypervalent iodine have found broad practical applications in organic synthesis due to their diverse reactivity combined with safety and commercial availability.[1] The organic derivatives of 3- and 5-iodanes, commonly known as hypervalent iodine reagents, have proved particularly useful. Compared with classical heavy metal-containing oxidants, hypervalent iodine reagents can be characterized by ready availability, low toxicity, and environmentally benign nature.

Such iodine(III) derivatives include phenyliodine(III) diacetate ($PhI(OAc)_2$, PIDA, or DIB), phenyliodine(III) bis (trifluoroacetate) (PIFA), and iodosobenzene (PhIO), which have been widely applied to form C–C, C–X, and X –Y bonds leading to an extensive collection of new heterocyclic compounds (Scheme 1).[2–7]

In addition to the formation of biologically relevant heterocycles, such transformations represent an attractive alternative to traditional transition metal-catalyzed oxidative heterocyclizations. For example, $PhI(OAc)_2$ has been used as oxidant for the synthesis of such heterocycles as 2-oxazolines.[8–10] Ranjith and coworkers developed a facile synthesis of 2,5-disubstituted 2-oxazolines by using $PhI(OAc)_2$ and pyridinium fluoride (HF·Py) in THF/CH_2Cl_2 at room temperature.[8] The same author reported

Scheme 1



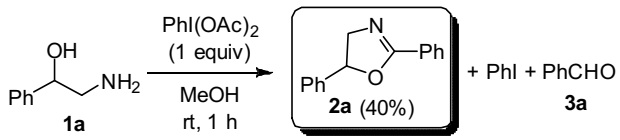
that *N*-allylamides bearing a ring at the distal position of the olefin provided an oxazine, instead of the expected 2-oxazoline. A mechanism involving two different routes, *exo* or *endo* cyclization, has been proposed, where HF·Py acted as a promoter and $PhI(OAc)_2$ was consumed as oxidant.[8] Liu and coworkers reported a similar synthetic

pathway where the reaction was carried out using *N*-allyl carboxamide as starting material, $PhI(OAc)_2$ as oxidant, and trimethyl silyl iodide as promoter.[9b]

The reactivity of the oxygen atom of the carboxamide functional group might be increased after the formation of *N*-TMS intermediate. Karade and coworkers reported a straightforward synthesis of 2-oxazolines starting from amino alcohols and aldehydes in the presence of $PhI(OAc)_2$. The authors proposed an oxazolidine as intermediate, based on GC-MS analysis.[11] Fujioka and coworkers reported the synthesis of 3-oxazoline-4-carboxylates as precursors of oxazoles, starting from amino alcohols and aliphatic aldehydes.[12] Xiao and coworkers demonstrated the use of 3-oxazolines as intermediates in photochemical syntheses of oxazoles.[13] As the synthesis of 3-oxazolines has been seldom documented in the literature, compared to 2-oxazolines, we believe that this approach could also provide useful preparative routes to 3-oxazolines.

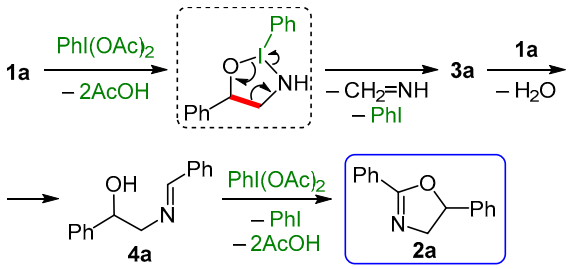
In the present work we revisited the Karade's protocol for the synthesis of oxazolines by investigating the reaction through ^{1}H NMR spectroscopy and highlighting an unusual reaction pathway leading to 3-oxazolines. Our investigation started from the observation that (±)-2-amino-1-phenylethanol (**1a**) reacted with $PhI(OAc)_2$, furnishing 2,4-diphenyl-2-oxazoline (**2a**) in moderate yield. The reaction was carried out in MeOH at room temperature (Scheme 2).

Scheme 2



It was observed that the reaction leading to 2-oxazoline **2a** and iodobenzene was accompanied by the formation of benzaldehyde (**3a**). On the basis of these preliminary observations, we considered a $PhI(OAc)_2$-promoted cleavage of the amino alcohol producing benzaldehyde (**3a**), methanimine, and iodobenzene as a plausible mechanism (Scheme 3).

Scheme 3



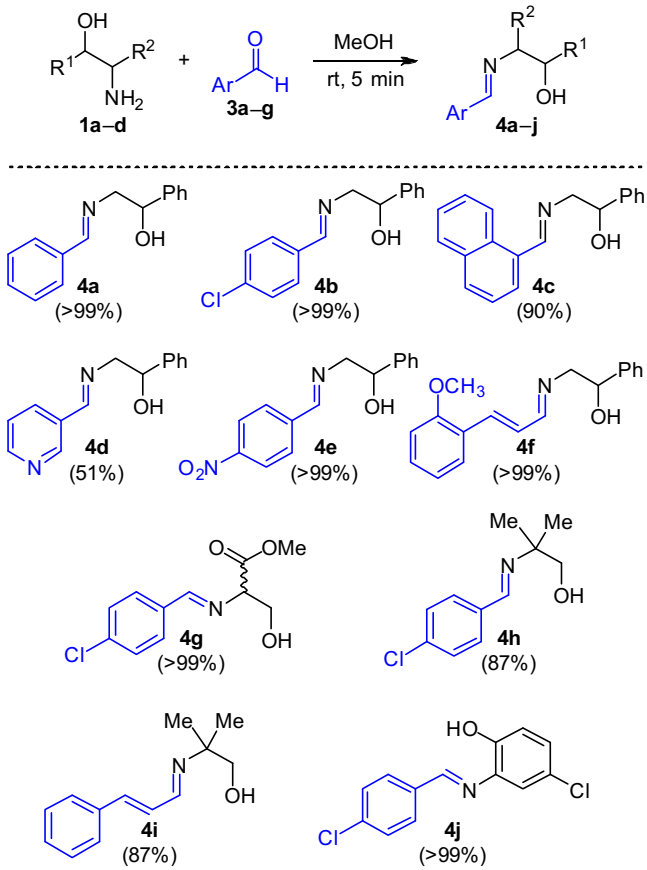
We anticipated that the aldehyde formed *in situ* could react with the amino alcohol **1a**, producing the respective imine **4a** that underwent cyclization promoted by $PhI(OAc)_2$, resulting in the 2-oxazoline **2a**. Nevertheless, our hypothesis did not consider the role of oxazolidine intermediate, as reported by Karade.[11] It is worth pointing out that both the expected oxazolidine intermediate and imine **4a** have the same molecular weight.

In order to shed light on this novel and direct conversion of 1,2-amino alcohols into 2-oxazolines, we decided to monitor the reaction by NMR. Similarly to the report by Karade,[11] we considered that a mixture of aldehyde and amino alcohol upon treatment with $PhI(OAc)_2$ could be a converted to 2-oxazolines. In fact, when 2-amino-1-phenylethanol (**1a**) was mixed to an equimolar amount of *p*-chlorobenzaldehyde (**3b**) in an NMR tube, using CD_3OD as solvent, a quick and clean formation of the imine **4b** was observed (Fig. 1). ^{1}H NMR analysis revealed the characteristic singlet peak at approximately 8 ppm, belonging to the =C–H proton of the imine intermediate **4b**. After the addition of $PhI(OAc)_2$ and immediate recording of ^{1}H NMR spectrum only the formation of oxazoline **2b** was observed (Fig. 1).

According to our NMR evidence, we deduced that the imine acted as intermediate in the synthesis of 2-oxazolines. However, even though NMR analysis did not provide any evidence for the formation of oxazolidine (i.e., a signal around 5 ppm for the 2-CH proton), we cannot rule out its involvement in the reaction. To further support the proposed route to 2-oxazolines, imines **4b–j** were prepared simply by mixing equimolar amounts of amino alcohols **1a–d** and aldehydes **3a–g** (Scheme 4). The reaction occurred quickly in methanol solution.

The imines **4a–j** thus obtained were subsequently converted into the respective 2-oxazolines **2a–i** and

Scheme 4



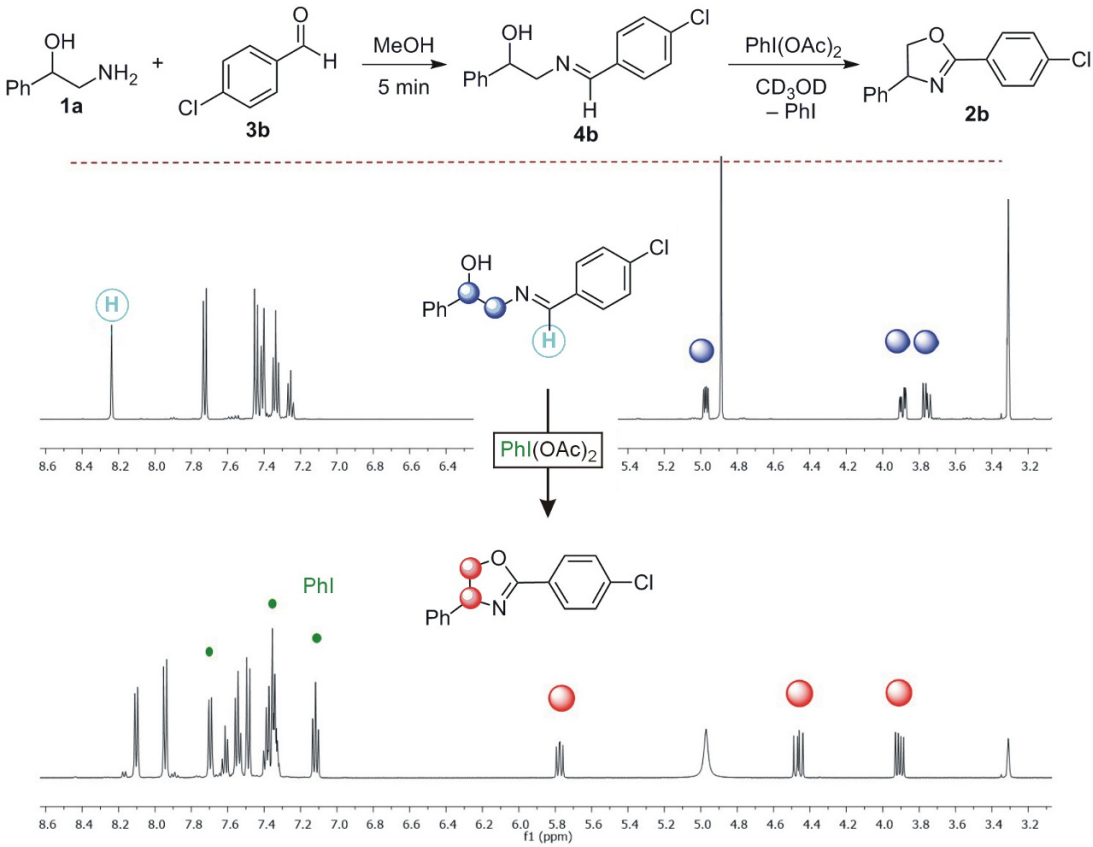


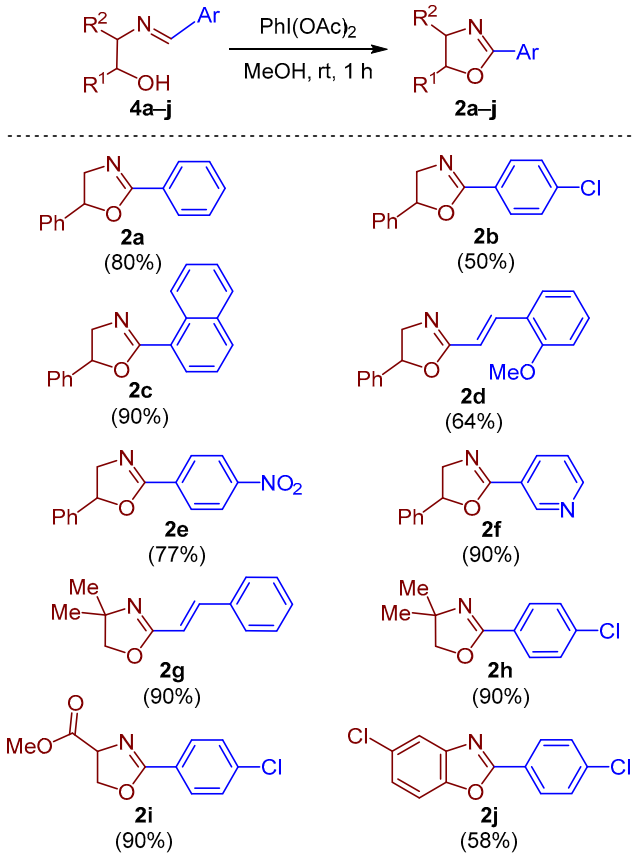
Figure 1. NMR monitoring of the conversion of imine **4b** into oxazoline **2b**.

benzoxazole **2j** by reaction with $PhI(OAc)_2$ (Scheme 5). The process also can be conducted by one-pot procedure, with essentially the same yields as over two separate steps, as long as the oxidant is added only after complete formation of the imine (NMR or GCMS control). The reaction did not proceed smoothly with an aliphatic aldehyde, likely because the formation of the respective imine proved problematic. However, several 2,5-disubstituted 2-oxazolines **2a**–**f** were prepared using aromatic, heteroaromatic, and unsaturated aldehydes. Moreover, the use of 2-methyl-2-aminopropanol provided 2,4,4-trisubstituted 2-oxazolines **2g**,**h**, while the use of racemic serine methyl ester gave access to 2-aryl-substituted 2-oxazoline-4-carboxylate **2i** in a very good yield (Scheme 5).

With the aim of preparing enantioenriched oxazolines, a surprising and almost unexpected result was obtained when the readily available chiral enantioenriched (*er* 98:2) amino alcohol (*R*)-**1e** was employed for this reaction. In fact, when the imine **4k** was reacted with $PhI(OAc)_2$ under optimized conditions, two different products were isolated in almost 1:1 ratio. After isolation of the two products, one was identified as the expected 2-oxazoline **2k**, while the other product was found to be the isomeric 3-oxazoline **5a**.

In an attempt to optimize the reaction, NMR monitoring was performed using imine **4k** as the starting material. After the addition of $PhI(OAc)_2$, rapid formation of compounds **2k** and **5a** was observed by NMR (Fig. 2). In accordance to what was reported above for imine **4b** (Fig. 1), the reaction was fast even with imine **4k**, giving

Scheme 5



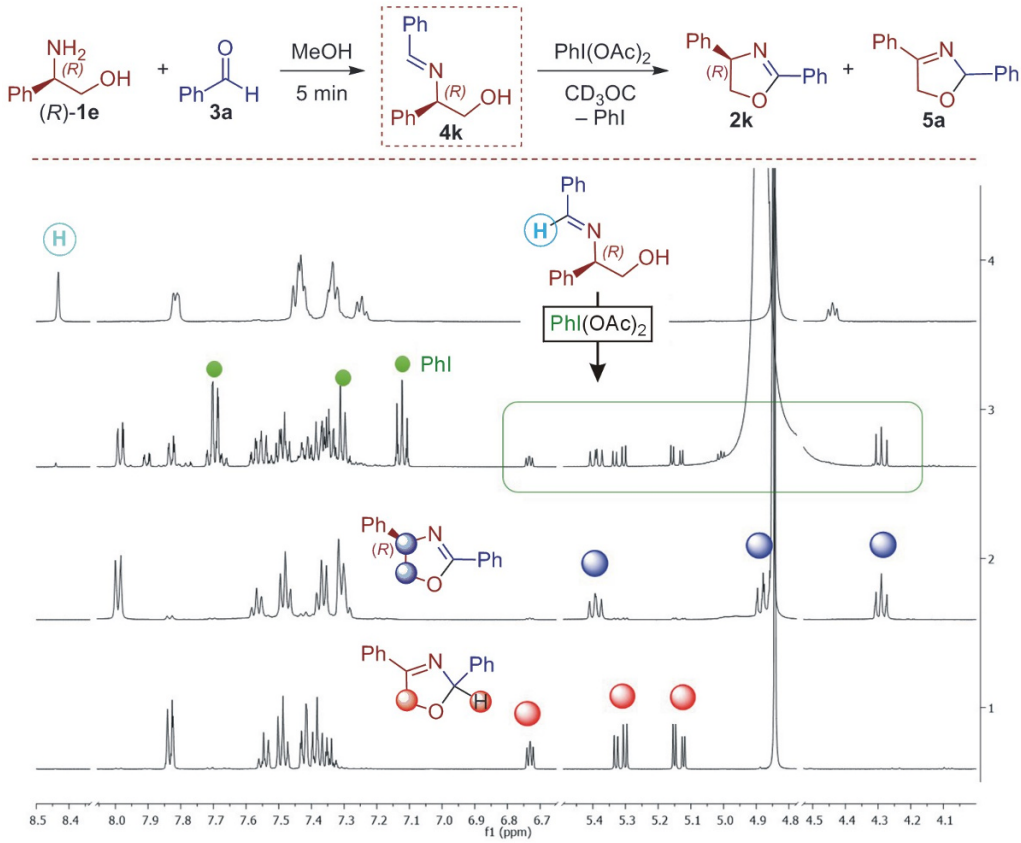


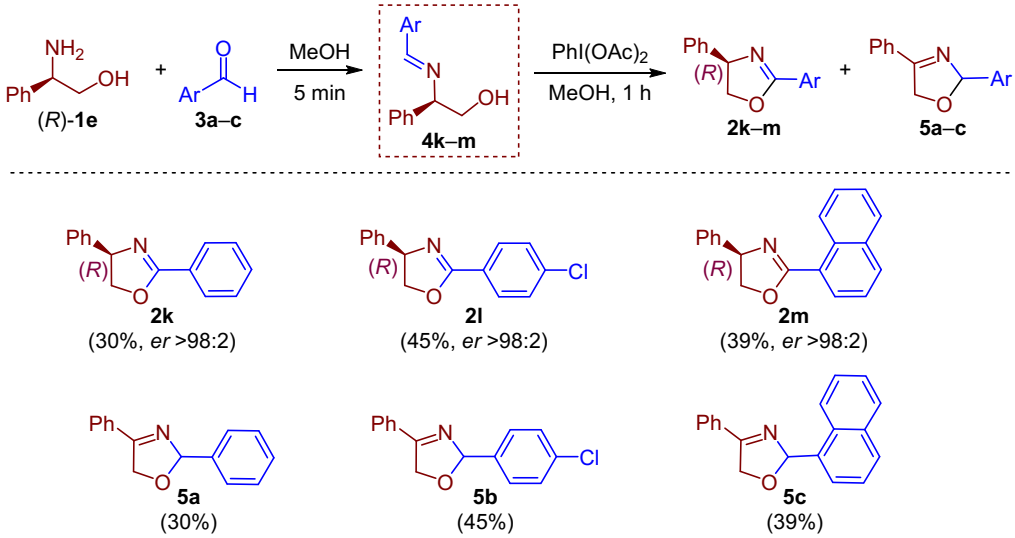
Figure 2. NMR monitoring of the conversion of imine **4k** into the isomeric oxazolines **2k** and **5a**.

the expected 2-oxazoline **2k**. Nevertheless, the presence of a phenyl substituent at a position relative to the nitrogen atom affected the selectivity of the reaction, leading to a mixture of two isomeric oxazolines.

The scope of the reaction was explored using three different aromatic aldehydes **3a**–**c**, which gave similar outcomes (Scheme 6). Two isomeric oxazolines could be isolated in each case. The structure of the 3-oxazolines **5a**,**c** was unequivocally established by single crystal X-ray analysis (Fig. 3). As expected, after HPLC analysis using chiral stationary phase only the 2-oxazolines **2k**–**m** were optically active (see the Supplementary material), whereas the 3-oxazolines **5a**,**b** were found to be racemic.

The mechanistic rationale for the competing formation of 2- and 3-oxazolines is reported in Scheme 7. Even if NMR analysis did not provide evidence on the involvement of the oxazolidine, it is likely that the chiral imine reacted with the oxidant $PhI(OAc)_2$, giving complex **I** by ligand exchange. The latter underwent a fast intramolecular cyclization producing two diastereomeric complexes

Scheme 6



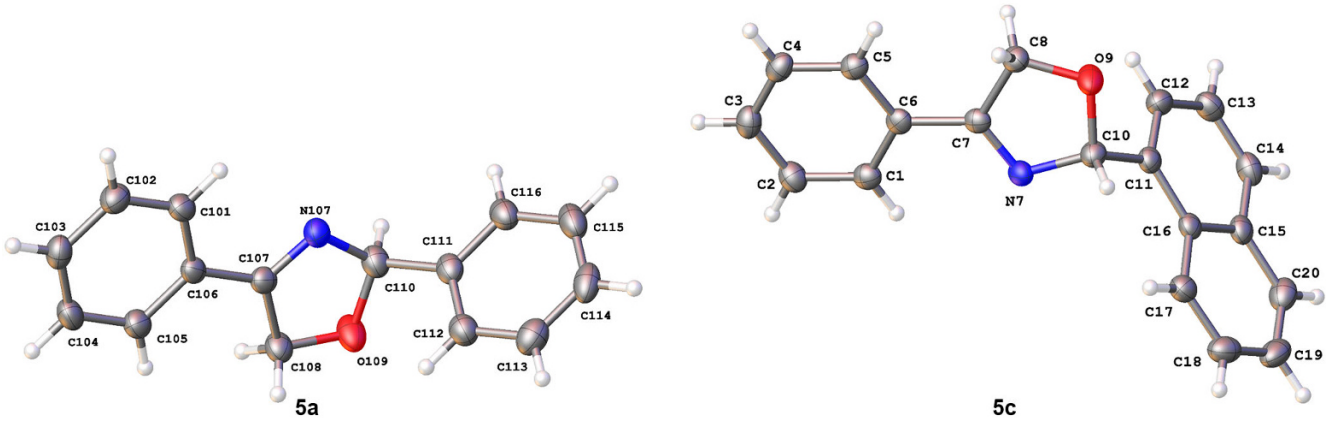
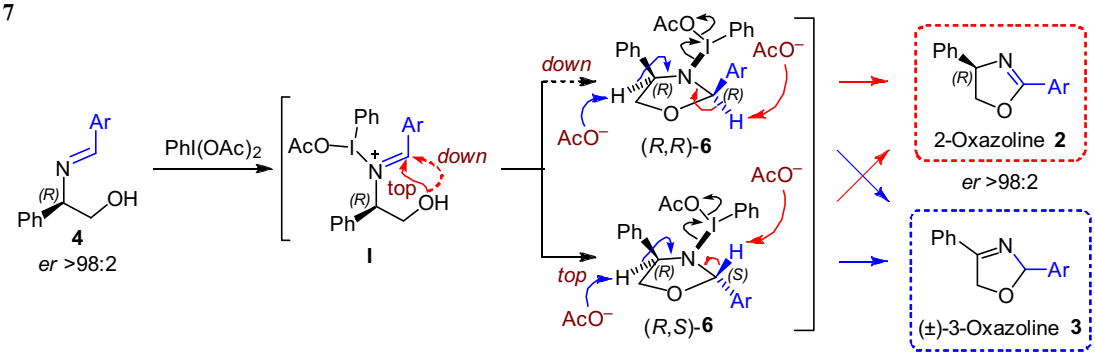


Figure 3. The molecular structure of 3-oxazolines **5a**,**c** with atoms represented by thermal vibration ellipsoids of 50% probability.

Scheme 7



(*R*,*R*)-**6** and (*R*,*S*)-**6**,[14] following two pathways (*top* and *down* in Scheme 7). Under the reaction conditions, such diastereomeric complexes can undergo a very fast b-elimination involving the proton at C-2 atom of both epimers (*R*,*R*)-**6**, and (*R*,*S*)-**6**, leading to enantioenriched 2-oxazolines. The removal of proton at the C-4 atom of both epimers (*R*,*R*)-**6**, and (*R*,*S*)-**6** led to a racemic 3-oxazoline (Scheme 6). Nevertheless, alternative pathways cannot be ruled out.[15]

In conclusion, this work demonstrated that hypervalent iodine can be used as a convenient reagent for the preparation of heterocycles such as 2-oxazolines and/or 3-oxazolines depending on the structure and stereochemistry of the starting 1,2-amino alcohol. NMR investigation supported the hypothesis that $PhI(OAc)_2$ acted as oxidant towards imine intermediates. In addition, a new route was developed that opened access to unusual 3-oxazolines.

Experimental

IR spectra were recorded on a PerkinElmer 283 spectrophotometer in thin film. ^1H (300 and 500 MHz) and ^{13}C (126 MHz) NMR spectra were recorded on Varian Inova-300 and Agilent VNMRS 500 spectrometers, using CD_3OD or $CDCl_3$ as solvents. The chemical shifts were reported in ppm using the residual protonated solvent as internal standard for the scale ($CDCl_3$: 7.26 ppm for ^1H nuclei and 77.0 ppm for ^{13}C nuclei; CD_3OD: 4.87 ppm for ^1H nuclei and 49.0 ppm for ^{13}C nuclei). The NMR yields were calculated using an internal standard. High-resolution mass spectra were recorded using a Bruker micrOTOF-Q II mass spectrometer equipped with an electrospray ion source operating in positive ion mode. Column chromatography was performed on neutral alumina (150 mesh, Brockmann I) with the composition of eluent reported separately for each experiment. The TLC analyses were performed using Merck F-254 silica gel plates with fluorescent indicator and the visualization was performed by UV light (254 nm). The optical rotation values ($[\alpha]_D^{20}$) were measured by using a PerkinElmer 341 polarimeter with 1 dm cell path length and the concentration (*c*) was expressed in g/100 ml.

Synthesis of oxazoline 2a starting from amino alcohol 1a. Amino alcohol **1a** (0.14 g, 1.0 mmol) and methanol (3.6 ml) were placed in a round-bottom flask. Then $PhI(OAc)_2$ (0.32 g, 1.0 mmol) was added. The mixture was stirred for 1 h and the reaction progress was monitored by TLC (hexane–EtOAc, 1:1). The solvent was removed under vacuum, the crude mixture was purified on neutral alumina (hexane–EtOAc, 8:2), yielding oxazoline **2a** as yellow oil. Yield 89 mg (40%).

Synthesis of imines 4a–m from amino alcohols and aldehydes (General method). Benzaldehyde (**3a**) (0.11 g, 1.0 mmol) and (±)-2-amino-1-phenylethanol (**1a**) (0.14 g, 1.0 mmol) were dissolved in MeOH (3.6 ml) in a round-bottom flask and stirred for 5 min. After monitoring the imine formation by GC or TLC the solvent was removed under vacuum. The product **4a** was characterized and used without further purification. Compounds **4b–m** were synthesized analogously.

Synthesis of oxazolines 2a–m starting from imines 4a–m (General method). Imine **4a** (0.22 g, 1.0 mmol) was

dissolved in MeOH (3.6 ml) in a round-bottom flask. Then $PhI(OAc)_2$ (0.32 g, 1.0 mmol) was added and the reaction mixture was stirred for 1 h at room temperature. After disappearance of the imine (TLC or GC monitoring) the solvent was removed under vacuum and oxazoline **2a** was purified on neutral alumina (hexane–EtOAc, 8:2). Compounds **2b–m** were synthesized analogously.

One-pot synthesis of oxazolines 2b,k–m, 5a–c (General method). The preparation of oxazoline **2b** is described. Compounds **2k**–**m** and **5a–c** were synthesized analogously. *p*-Chlorobenzaldehyde (**3b**) (0.14 g, 1.0 mmol) and (±)-2-amino-1-phenylethanol (**1a**) (0.14 g, 1.0 mmol) were dissolved in MeOH (3.6 ml) in a round-bottom flask and stirred for 5 min. After monitoring the imine formation by GC or TLC, $PhI(OAc)_2$ (0.32 g, 1.0 mmol) was added to the mixture. The reaction was stirred for 1 h at room temperature and the progress of the reaction was monitored by TLC or GC. After full conversion the solvent was removed under vacuum and the crude residue was purified on neutral alumina (hexane–EtOAc, 8:2).

2,5-Diphenyl-4,5-dihydro-1,3-oxazole (2a).[16] Yield 178 mg (80%, from imine), yellow oil. ^{1}H NMR spectrum (500 MHz, $CDCl_3$), δ, ppm: 8.03 (2H, d, *J* = 6.6, H Ar); 7.50–7.30 (8H, m, H Ar); 5.69 (1H, dd, *J* = 10.0, *J* = 7.9, CH); 4.51 (1H, dd, *J* = 14.7, *J* = 10.0, CH_2); 4.03 (1H, dd, *J* = 14.7, *J* = 7.9, CH_2). Found, *m*/*z*: 224.1073 $[M+H]^+$. $C_{15}H_{14}NO$. Calculated, *m*/*z*: 224.1070.

2-(4-Chlorophenyl)-5-phenyl-4,5-dihydro-1,3-oxazole (2b).[15] Yield 128 mg (50%, from imine, 50%, one-pot method), yellow oil. ^{1}H NMR spectrum (500 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 7.95 (2H, d, *J* = 8.7, H Ar); 7.42–7.33 (7H, m, H Ar); 5.66 (1H, dd, *J* = 9.8, *J* = 8.2, CH); 4.48 (1H, dd, *J* = 15.0, *J* = 9.8, CH_2); 4.00 (1H, dd, *J* = 15.0, *J* = 8.0, CH_2). Found, *m*/*z*: 280.0500 $[M+Na]^+$. $C_{15}H_{12}ClNNaO$. Calculated, *m*/*z*: 280.0500.

2-(Naphthalen-1-yl)-5-phenyl-4,5-dihydro-1,3-oxazole (2c). Yield 246 mg (90%, from imine), yellow oil. IR spectrum, ν, cm^{-1}: 2529, 1719, 1643 (C=N), 1449, 1242, 1121, 778. ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.85 (1H, d, *J* = 8.6, H Ar); 8.08 (2H, t, *J* = 9.4, H Ar); 7.96 (1H, d, *J* = 7.7, H Ar); 7.59–7.43 (8H, m, H Ar); 5.86 (1H, dd, *J* = 10.3, *J* = 7.5, CH); 4.63 (1H, dd, *J* = 14.7, *J* = 10.3, CH_2); 4.08 (1H, dd, *J* = 14.7, *J* = 7.5, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 166.6; 142.4; 135.3; 133.2; 132.3; 130.0 (3C); 129.6; 129.5; 128.3; 127.4; 127.0; 126.9 (2C); 125.9; 125.8; 82.2; 63.8. Found, *m*/*z*: 274.1227 $[M+H]^+$. $C_{19}H_{16}NO$. Calculated, *m*/*z*: 274.1226.

2-(2-Methoxystyryl)-5-phenyl-4,5-dihydro-1,3-oxazole (2d). Yield 178 mg (64%, from imine), yellow oil. IR spectrum, ν, cm^{-1}: 1719, 1647 (C=N), 1489, 1249, 1105. ^{1}H NMR spectrum (500 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 7.76 (1H, d, *J* = 16.5, CH=); 7.52 (1H, d, *J* = 7.5, H Ar); 7.42–7.30 (6H, m, H Ar); 6.97 (1H, t, *J* = 7.5, H Ar); 6.91 (1H, d, *J* = 8.4, H Ar); 6.82 (1H, d, *J* = 16.5, CH=); 5.57 (1H, t, *J* = 8.9, CH); 4.41 (1H, dd, *J* = 14.8, *J* = 10.1, CH_2); 3.91 (1H, dd, *J* = 15.2, *J* = 7.8, CH_2); 3.87 (3H, s, OCH_3). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 164.7; 158.0; 141.3; 135.7; 130.8; 128.9 (2C); 128.4 (2C); 125.9 (2C); 124.3; 120.9; 115.7; 111.2; 80.8; 63.3; 55.6. Found, *m*/*z*: 280.1335 $[M+H]^+$. $C_{18}H_{18}NO_2^+$. Calculated, *m*/*z*: 280.1332.

2-(4-Nitrophenyl)-5-phenyl-4,5-dihydro-1,3-oxazole (2e).[17] Yield 206 mg (77%, from imine), white solid, mp 143–145°C (mp 153–156°C[17]). ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.36 (2H, d, *J* = 9.0, H Ar); 8.21 (2H, d, *J* = 9.0, H Ar); 7.41–7.36 (5H, m, H Ar); 5.86 (1H, dd, *J* = 10.4, *J* = 8.2, CH); 4.55 (1H, dd, *J* = 15.0, *J* = 10.2, CH_2); 3.99 (1H, dd, *J* = 15.0, *J* = 8.2, CH_2). Found, *m*/*z*: 269.0922 $[M+H]^+$. $C_{15}H_{13}N_2O_3$. Calculated, *m*/*z*: 269.0921.

5-Phenyl-2-(pyridin-3-yl)-4,5-dihydro-1,3-oxazole (2f). Yield 202 mg (90%, from imine), yellow solid, mp 57–59°C. IR spectrum, ν, cm^{-1}: 2928, 2872, 1655 (C=N), 1592, 1415, 1340, 1259, 1079, 1022, 700. ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 9.10 (1H, s, H Ar); 8.67 (1H, d, *J* = 4.9, H Ar); 8.33 (1H, d, *J* = 8.1, H Ar); 7.52 (1H, dd, *J* = 8.0, *J* = 6.9, H Ar); 7.38–7.32 (5H, m, H Ar); 5.78 (1H, dd, *J* = 10.5, *J* = 8.1, CH); 4.48 (1H, dd, *J* = 15.1, *J* = 10.1, CH_2); 3.93 (1H, dd, *J* = 14.9, *J* = 8.0, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 163.7; 153.0; 149.7; 141.7 (2C); 137.4; 129.9 (2C); 129.6; 126.8 (2C); 125.2; 83.0; 63.4. Found, *m*/*z*: 225.1022 $[M+H]^+$. $C_{14}H_{13}N_2O$. Calculated, *m*/*z*: 225.1022.

4,4-Dimethyl-2-styryl-4,5-dihydro-1,3-oxazole (2g).[18] Yield 181 mg (90%, from imine), yellow oil. ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 7.56 (2H, d, *J* = 6.9, H Ar); 7.44–7.36 (4H, m, H Ar, CH=); 6.59 (1H, d, *J* = 15.6, CH=); 4.12 (2H, s, CH_2); 1.33 (6H, s, CH_3). Found, 202.1230 *m*/*z*: $[M+H]^+$. $[C_{13}H_{16}NO]^+$. Calculated, *m*/*z*: 202.1226

2-(4-Chlorophenyl)-4,4-dimethyl-4,5-dihydro-1,3-oxazole (2h).[19] Yield 188 mg (90%, from imine), white solid, mp 65–67°C (mp 31–33°C[19]). IR spectrum, ν, cm^{-1}: 2926, 1724, 1651 (C=N), 1492, 1312, 1262, 1092, 1015, 731. ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 7.88 (2H, d, *J* = 8.5, H Ar); 7.48 (2H, d, *J* = 8.5, H Ar); 4.21 (2H, s, CH_2); 1.37 (6H, s, CH_3). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 163.7; 138.9; 130.7; 129.8; 127.4; 80.5; 68.5; 28.3. Found, *m*/*z*: 210.0684 $[M+H]^+$. $C_{11}H_{13}ClNO^+$. Calculated, *m*/*z*: 210.0680.

Methyl-2-(4-chlorophenyl)-4,5-dihydro-1,3-oxazole-4-carboxylate (2i).[20] Yield 215 mg (90%, from imine), white solid, mp 85–87°C. IR spectrum, ν, cm^{-1}: 2925, 2254, 1725, 1645 (C=N), 1492, 1405, 1265, 1093, 913. ^{1}H NMR spectrum (500 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 7.92 (2H, d, *J* = 9.2, H Ar); 7.39 (2H, d, *J* = 8.5, H Ar); 4.95 (1H, t, *J* = 9.4, CH); 4.70 (1H, t, *J* = 8.5, CH_2); 4.60–4.58 (1H, m, CH_2); 3.83 (3H, s, CH_3). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 171.6; 138.3; 130.1 (2C); 128.9 (2C); 125.6; 69.9; 68.8; 66.0; 53.0. Found, *m*/*z*: 262.0242 $[M+Na]^+$. $C_{11}H_{10}ClNNaO_3$. Calculated, *m*/*z*: 262.0241.

5-Chloro-2-(4-chlorophenyl)-1,3-benzoxazole (2j).[21] Yield 152 mg (58%, from imine), white solid, mp 148–149°C (mp 114°C[21]). ^{1}H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm (*J*, Hz): 8.18–8.15 (2H, m, H Ar); 7.74–7.73 (1H, d, *J* = 1.9, H Ar); 7.52–7.48 (3H, m, H Ar); 7.35–7.31 (1H, m, H Ar). Found, *m*/*z*: 263.9988 $[M+Na]^+$. $C_{13}H_8Cl_2NO_3$. Calculated, *m*/*z*: 263.9978.

(*R*)-2,4-Diphenyl-4,5-dihydro-1,3-oxazole (2k).[22] Yield 70 mg (30%, one-pot procedure), white solid, mp 32–33°C.

$[\alpha]_D^{20}$ +21° (*c* 1.04, $CHCl_3$) ($[\alpha]_D^{20}$ +36.4° (*c* 1.04, $CHCl_3$)[22]). ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.06–8.02 (2H, m, H Ar); 7.53–7.49 (1H, m, H Ar); 7.42 (2H, m, H Ar); 7.38–7.26 (5H, m, H Ar); 5.39 (1H, dd, *J* = 10.3, *J* = 8.3, CH); 4.80 (1H, dd, *J* = 10.3, *J* = 8.3, CH_2); 4.28 (1H, t, *J* = 8.3, CH_2). Found, *m*/*z*: 224.1077 $[M+H]^+$. $C_{15}H_{14}NO$. Calculated, *m*/*z*: 224.1070.

(*R*)-2-(4-Chlorophenyl)-4-phenyl-4,5-dihydro-1,3-oxazole (2l). Yield 100 mg (45%, one-pot procedure), white solid, mp 58–60°C. $[\alpha]_D^{20}$ +38° (*c* 1, $CHCl_3$). IR spectrum, ν, cm^{-1}: 2918, 1722, 1642 (C=N), 1265, 1095, 728, 665. ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 7.98 (2H, d, *J* = 8.3, H Ar); 7.51 (2H, d, *J* = 8.3, H Ar); 7.38–7.31 (5H, m, H Ar); 5.41 (1H, dd, *J* = 9.9, *J* = 8.1, CH); 4.90 (1H, dd, *J* = 10.2, *J* = 8.6, CH_2); 4.31 (1H, t, *J* = 8.2, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 166.2; 143.3; 139.3; 131.0 (2C); 130.0 (2C); 129.9 (2C); 128.9; 127.9 (2C); 127.0; 76.6; 70.9. Found, *m*/*z*: 258.0683 $[M+H]^+$. $C_{15}H_{13}ClNO$. Calculated, *m*/*z*: 258.0680.

(*R*)-2-(Naphthalen-1-yl)-4-phenyl-4,5-dihydro-1,3-oxazole (2m). Yield 106 mg (39%, one-pot procedure), yellow oil. $[\alpha]_D^{20}$ –10.7° (*c* 1, $CHCl_3$). IR spectrum, ν, cm^{-1}: 2918, 1638, 1510, 1351, 1124, 807, 776. ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.84 (1H, d, *J* = 7.9, H Ar); 8.08–8.06 (2H, m, H Ar); 7.96–7.94 (1H, m, H Ar); 7.61–7.54 (4H, m, H Ar); 7.42–7.41 (3H, m, H Ar); 7.34–7.31 (1H, m, H Ar); 5.54 (1H, dd, *J* = 10.2, *J* = 8.3, CH); 4.96 (1H, dd, *J* = 10.2, *J* = 8.3, CH_2); 4.37 (1H, t, *J* = 8.3, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 167.6; 143.6; 133.3; 130.1 (2C); 129.9 (2C); 129.6 (2C); 128.9; 128.3; 127.8; 127.4 (2C); 126.9 (2C); 125.9; 75.9; 71.4. Found, *m*/*z*: 274.1226 $[M+H]^+$. $C_{19}H_{16}NO$. Calculated, *m*/*z*: 274.1228.

2-(Benzylideneamino)-1-phenylethan-1-ol (4a).[23] Yield 223 mg (99%), white solid, mp 113–115°C (mp 114°C[23]). ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.31 (1H, s, HC=N); 7.80–7.77 (2H, m, H Ar); 7.49–7.41 (5H, m, H Ar); 7.36–7.31 (2H, m, H Ar); 7.26–7.21 (1H, m, H Ar); 5.05–5.03 (1H, m, CH); 3.93 (1H, dd, *J* = 12.0, *J* = 3.3, CH_2); 3.73 (1H, dd, *J* = 12.0, *J* = 9.0, CH_2). Found, *m*/*z*: 226.1237 $[M+H]^+$. $C_{15}H_{16}NO$. Calculated, *m*/*z*: 226.1226.

2-{[(4-Chlorophenyl)methylidene]amino}-1-phenylethan-1-ol (4b).[24] Yield 256 mg (99%), clear crystalline solid, mp 153–156°C (mp 160–163°C (THF)[24]). IR spectrum, ν, cm^{-1}: 3410 (O–H), 2518, 1647 (C=N), 1446, 1116, 1062. ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.24 (1H, s, H–C=N); 7.73 (2H, d, *J* = 9.0, H Ar); 7.45 (2H, d, *J* = 8.4, H Ar); 7.42–7.24 (5H, m, H Ar); 4.97 (1H, dd, *J* = 8.1, *J* = 4.2, CH); 3.89 (1H, dd, *J* = 12.0, *J* = 4.3, CH_2); 3.76 (1H, dd, *J* = 12.0, *J* = 8.2, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 164.4; 144.3; 138.0; 135.9; 130.8 (2C); 129.9 (2C); 129.3 (2C); 128.5; 127.3 (2C); 74.4; 69.8. Found, *m*/*z*: 260.0838 $[M+H]^+$. $C_{15}H_{15}ClNO$. Calculated, *m*/*z*: 260.0837.

2-[(Naphthalen-1-ylmethylene)amino]-1-phenylethan-1-ol (4c). Yield 247 mg (90%), straw-yellow solid, mp 126–131°C. IR spectrum, ν, cm^{-1}: 3429 (O–H), 2914, 2376, 1723, 1643 (C=N), 1249, 1063, 704. ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.94 (1H, s, H–C=N); 8.57 (1H, d, *J* = 7.7, H Ar); 7.97–7.86 (3H, m, H Ar); 7.56–7.46 (5H, m, H Ar); 7.37–7.26 (4H, m, H Ar); 5.06 (1H, dd, *J* = 7.6, *J* = 5.4, CH); 4.02 (1H, dd, *J* = 11.5, *J* = 5.0, CH_2); 3.49 (1H, dd, *J* = 11.5, *J* = 7.6, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 165.0; 144.4; 135.2; 132.9; 132.6; 132.2; 129.6; 129.3; 128.8; 128.5; 128.0; 127.4; 127.1; 126.2; 125.0; 74.5; 70.4. Found, *m*/*z*: 276.1386 $[M+H]^+$. $C_{19}H_{18}NO$. Calculated, *m*/*z*: 276.1383.

1-Phenyl-2-[(pyridin-3-ylmethylene)amino]ethanol (4d). Yield 115 mg (51%), yellow oil. IR spectrum, ν, cm^{-1}: 3232 (O–H), 2848, 1706, 1648 (C=N), 1589, 1421, 1246, 1021, 702. ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.84 (1H, s, H Ar); 8.59 (1H, d, *J* = 4.9, H Ar); 8.33 (1H, s, H–C=N); 8.20 (1H, d, *J* = 8.1, H Ar); 7.49 (1H, dd, *J* = 8.1, *J* = 4.9, H Ar); 7.50–7.24 (5H, m, H Ar); 5.01 (1H, dd, *J* = 7.9, *J* = 4.5, CH); 3.94 (1H, dd, *J* = 11.9, *J* = 4.3, CH_2); 3.81 (1H, dd, *J* = 11.9, *J* = 7.8, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 162.3; 151.9; 150.4; 144.2; 136.9; 133.5; 129.3 (2C); 128.5; 127.3 (2C); 125.4; 74.3; 70.0. Found, *m*/*z*: 227.1186 $[M+H]^+$. $C_{14}H_{15}N_2O$. Calculated, *m*/*z*: 227.1184.

2-[(4-Nitrobenzylidene)amino]-1-phenylethan-1-ol (4e).[25] Yield 243 mg (90%), white solid, mp 180–185°C. ^{1}H NMR spectrum (300 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.37 (1H, s, H–C=N); 8.28 (2H, d, *J* = 8.9, H Ar); 7.97 (2H, d, *J* = 8.9, H Ar); 7.30–7.18 (5H, m, H Ar); 5.01 (1H, dd, *J* = 7.8, *J* = 4.3, CH); 3.97 (1H, dd, *J* = 11.8, *J* = 4.3, CH_2); 3.83 (1H, dd, *J* = 11.9, *J* = 7.9, CH_2). Found, *m*/*z*: 271.1074 $[M+H]^+$. $C_{15}H_{15}N_2O_3$. Calculated, *m*/*z*: 271.1077.

2-{[3-(2-Methoxyphenyl)prop-2-en-1-ylidene]amino}-1-phenylethan-1-ol (4f). Yield 252 mg (99%), yellow oil. IR spectrum, ν, cm^{-1}: 3429 (O–H), 2529, 1633 (C=N), 1449, 1244, 1212, 1026, 728. ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 7.98 (1H, d, *J* = 9.1, CH=); 7.54 (1H, d, *J* = 7.7, CH=); 7.40–7.20 (7H, m, H Ar); 7.00 (1H, d, *J* = 8.5, CH=); 6.95 (2H, m, H Ar); 4.90 (1H, dd, *J* = 7.7, *J* = 4.4, CH); 3.88 (3H, s, OCH_3); 3.78 (1H, dd, *J* = 11.9, *J* = 4.4, CH_2); 3.66 (1H, dd, *J* = 11.9, *J* = 7.9, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 168.4; 159.1; 144.2; 140.2; 132.0; 129.3 (2C); 128.9; 128.5; 128.1; 127.2 (2C); 125.4; 121.8; 112.3; 74.5; 69.2; 56.0. Found, *m*/*z*: 282.1490 $[M+H]^+$. $C_{18}H_{20}NO_2$. Calculated, *m*/*z*: 282.1489.

Methyl 2-(4-chlorobenzylideneamino)-3-hydroxypropanoate (4g).[26] Yield 178 mg (74%), yellow oil. ^{1}H NMR spectrum (300 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.38 (1H, s, H–C=N); 7.80 (2H, d, *J* = 8.4, H Ar); 7.43 (2H, d, *J* = 8.6, H Ar); 4.23 (1H, dd, *J* = 7.2, *J* = 4.9, CH); 4.03 (1H, dd, *J* = 11.1, *J* = 4.9, CH_2); 3.83 (1H, dd, *J* = 11.1, *J* = 4.6, CH_2); 3.74 (3H, s, CH_3). Found, *m*/*z*: 242.0590 $[M+H]^+$. $C_{11}H_{13}ClNO_3$. Calculated, *m*/*z*: 242.0579.

2-[(4-Chlorobenzylidene)amino]-2-methylpropan-1-ol (4h).[27] Yield 183 mg (87%), yellow solid, mp 60–63°C (mp 61°C[27]). ^{1}H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.36 (1H, s, H–C=N); 7.77 (2H, d, *J* = 8.3, H Ar); 7.43 (2H, d, *J* = 8.3, H Ar); 3.52 (2H, s, CH_2); 1.26 (6H, s, CH_3). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 158.8;

130.6 (2C); 129.8 (2C); 129.5; 129.0; 71.2; 62.5; 24.2 (2C). Found, *m/z*: 212.0841 $[M+H]^+$. $C_{11}H_{15}ClNO$. Calculated, *m/z*: 212.0837.

2-Methyl-2-[(3-phenylallylidene)amino]propan-1-ol (4i).[28] Yield 177 mg (87%), yellow solid, mp 104–106°C. ^1H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.14 (1H, d, *J* = 8.8, CH=); 7.54 (2H, d, *J* = 7.3, H Ar); 7.35 (3H, m, H Ar); 7.15 (1H, d, *J* = 16.0, CH=); 6.92 (1H, dd, *J* = 16.0, *J* = 8.8, CH=); 3.48 (2H, s, CH_2); 1.23 (6H, s, CH_3). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 162.5; 144.4; 137.0; 130.4; 129.9; 128.4; 71.0; 62.2; 24.2. Found, *m/z*: 204.1392 $[M+H]^+$. $C_{13}H_{18}NO$. Calculated, *m/z*: 204.1383.

4-Chloro-2-{[(4-chlorophenyl)methylidene]amino}-phenol (4j).[29] Yield 263 mg (99%), yellow solid, mp 113–118°C. IR spectrum, ν, cm^{-1}: 3378 (O–H), 3054, 2306, 1626 (C=N), 1492, 1265, 738. ^1H NMR spectrum (300 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.67 (1H, s, H–C=N); 8.01–7.98 (2H, m, H Ar); 7.51–7.48 (2H, m, H Ar); 7.24 (1H, d, *J* = 2.5, H Ar); 7.11–7.07 (1H, m, H Ar); 6.86 (1H, d, *J* = 6.9, H Ar). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 160.7; 131.6 (2C); 130.1; 128.5; 118.6 (2C); 118.0 (2C); 116.3 (2C); 116.1 (2C). Found, *m/z*: 266.0131 $[M+H]^+$. $C_{13}H_{10}Cl_2NO$. Calculated, *m/z*: 266.0134.

(*R*)-2-(Benzylideneamino]-2-phenylethan-1-ol (4k).[30] Yield 130 mg (58%), colorless needles, mp 77–79°C ($[\alpha]_D^{20}$ –115° (*c* 0.48, CH_2Cl_2))[30]. ^1H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.40 (1H, s, H–C=N); 7.82–7.24 (10H, m, H Ar); 4.50 (1H, dd, *J* = 7.9, *J* = 4.3, CH); 3.97 (1H, dd, *J* = 11.0, *J* = 4.3, CH_2); 3.90 (1H, dd, *J* = 11.0, *J* = 7.9, CH_2). Found, *m/z*: 226.1230 $[M+H]^+$. $C_{15}H_{16}NO$. Calculated, *m/z*: 226.1226.

(*R*)-2-[(4-Chlorobenzylidene)amino]-2-phenylethan-1-ol (4l).[31] Yield 233 mg (90%), yellow oil, $[\alpha]_D^{20}$ +38° (*c* 1, $CHCl_3$). IR spectrum, ν, cm^{-1}: 3350 (O–H), 2862, 1722, 1640 (C=N), 1489, 1088, 1013, 820, 699. ^1H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.39 (1H, s, H–C=N); 7.78 (2H, d, *J* = 8.2, H Ar); 7.44–7.23 (7H, m, H Ar); 4.42 (1H, t, *J* = 7.5, CH); 3.85–3.83 (2H, m, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 162.3; 142.2; 137.7; 136.2; 130.9 (2C); 129.8 (2C); 129.5 (2C); 128.4; 128.4 (2C); 78.2; 67.8. Found, *m/z*: 260.0840 $[M+H]^+$. $C_{15}H_{15}ClNO$. Calculated, *m/z*: 260.0837.

(*R*)-2-[(Naphthalen-1-ylmethylene)amino]-2-phenylethan-1-ol (4m).[32] Yield 273 mg (99%), yellow oil. $[\alpha]_D^{20}$ +24.2° (*c* 1, $CHCl_3$). IR spectrum, ν, cm^{-1}: 3343 (O–H), 2484, 2215, 2071, 1642 (C=N), 1123, 976, 824. ^1H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 9.09 (1H, s, H–C=N); 8.94 (1H, d, *J* = 7.9, H Ar); 7.95–7.89 (3H, m, H Ar); 7.60–7.50 (5H, m, H Ar); 7.38–7.25 (4H, m, H Ar); 4.56–4.53 (1H, m, CH); 3.94–3.90 (2H, m, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 163.4; 142.5; 135.3 (2C); 133.0; 132.7 (2C); 132.2 (2C); 129.9; 129.6 (3C); 128.1 127.1; 126.2; 125.5; 79.3; 68.1. Found, *m/z*: 276.1380 $[M+H]^+$. $C_{19}H_{18}NO$. Calculated, *m/z*: 276.1383.

2,4-Diphenyl-2,5-dihydro-1,3-oxazole (5a).[33] Yield 70 mg (30%, one-pot procedure), white solid, mp 91–94°C (mp 85–87°C[33]). IR spectrum, ν, cm^{-1}: 3430, 1629 (C=N), 1450, 1266, 1064, 759, 692. ^1H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 7.87–7.85 (2H, m, H Ar); 7.59–7.56 (1H, m, H Ar); 7.53–7.50 (2H, m, H Ar); 7.46–7.44 (2H, m, H Ar); 7.43–7.35 (3H, m, H Ar); 6.76 (1H, dd, *J* = 5.2, *J* = 4.3, CH); 5.34 (1H, dd, *J* = 14.0, *J* = 5.4, CH_2); 5.16 (1H, dd, *J* = 14.0, *J* = 4.1, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 170.7; 141.1; 133.1; 131.7; 130.0 (2C); 129.8; 129.5 (2C); 129.1 (2C); 127.6 (2C); 108.4; 75.4. Found, *m/z*: 224.1069 $[M+H]^+$. $C_{15}H_{14}NO$. Calculated, *m/z*: 224.1070.

2-(4-Chlorophenyl)-4-phenyl-2,5-dihydro-1,3-oxazole (5b). Yield 100 mg (45%, one-pot procedure), white solid, mp 92–97°C. IR spectrum, ν, cm^{-1}: 2851, 1630 (C=N), 1490, 1290, 1067, 759, 691. ^1H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 7.83–7.82 (2H, m, H Ar); 7.56–7.53 (1H, m, H Ar); 7.50–7.47 (2H, m, H Ar); 7.43–7.37 (4H, m, H Ar); 6.74–6.71 (1H, m, CH); 5.33–5.27 (1H, m, CH_2); 5.15–5.11 (1H, m, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 169.5; 138.6; 131.7; 128.6 (2C); 128.2 (2C); 128.1; 127.8 (2C); 127.7 (2C); 126.1; 106.1; 74.1. Found, *m/z*: 280.0504 $[M+Na]^+$. $C_{15}H_{13}ClNNaO$. Calculated, *m/z*: 280.0500.

2-(Naphthalen-1-yl)-4-phenyl-2,5-dihydro-1,3-oxazole (5c). Yield 106 mg (39%, one-pot procedure), white solid, mp 92–97°C. IR spectrum, ν, cm^{-1}: 3052, 2852, 1635 (C=N), 1449, 1265, 1088, 788, 738, 692. ^1H NMR spectrum (500 MHz, CD_3OD), δ, ppm (*J*, Hz): 8.38 (1H, d, *J* = 8.5, H Ar); 7.95–7.90 (4H, m, H Ar); 7.63–7.48 (8H, m, Ar, CH); 5.37 (1H, dd, *J* = 14.0, *J* = 5.6, CH_2); 5.31 (1H, dd, *J* = 14.0, *J* = 4.1, CH_2). ^{13}C NMR spectrum (CD_3OD), δ, ppm: 173.0; 136.6 (2C); 135.4; 133.0 (2C); 132.4; 132.0; 130.2; 129.6 (2C); 129.2; 127.3; 126.8; 126.3 (2C); 125.1; 105.8; 75.1. Found, *m/z:* 274.1225 $[M+H]^+$. $C_{19}H_{16}NO$. Calculated, *m/z*: 274.1226.

X-ray structural study of compounds 5a,c. Single crystals of compound **5a** were grown from CH_2Cl_2 – petrol ether, while single crystals of compound **5c** were grown from diethyl ether. A suitable crystal was selected and mounted on a glass fibre with Fomblin oil and placed on a Rigaku Oxford Diffraction SuperNova diffractometer with a dual source equipped with an Atlas S2 CCD area detector. Cu*K*α radiation (1.54184 Å) was used. The crystal was kept at 150(2) K during data collection. The structure was solved with Olex2[34] and ShelXT[35] software using intrinsic phasing and refined with the ShelXL[36] refinement package using least squares minimization. The crystallographic data sets were deposited at the Cambridge Crystallographic Data Center (for compound **5a** deposit CCDC 1846701, for compound **5c** deposit CCDC 1819791).

Supplementary information file, containing NMR spectra, chiral HPLC data, and X-ray structural study parameters, is available at the journal website at http://link.springer.com/journal/10593.

We thank the University of Bari Aldo Moro, the Polytechnic of Bari, the project Laboratorio SISTEMA (Code PONa300369) financed by Italian Ministry of Education, Universities, and Research.

We are also grateful to Chiara Claudio and Valeria Decaro for their precious contribution.

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