Synthesis of Optically Active 1-(1-Phenylethyl)-1*H*-imidazoles Derived from 1-Phenylethylamine[1])

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The three-component reaction of (*R*)- or (*S*)-1-phenylethylamine (**6**), formaldehyde, and an α-(hydroxyimino) ketone **5**, *i.e.*, 3-(hydroxyimino)butan-2-one (**5a**) or 2-(hydroxyimino)-1,2-diphenylethanone (**5b**), yields the corresponding enantiomerically pure 1-(1-phenylethyl)-1*H*-imidazole 3-oxide **7** in high yield (*Schemes 2* and *3*). The reactions are carried out either in MeOH or in AcOH. Smooth transformations of the *N*-oxides into optically active 1-(1-phenylethyl)-1*H*-imidazoles **10** and 2,3-dihydro-1-(1-phenylethyl)-1*H*-imidazole-2-thiones **11** are achieved by treatment of **7** with *Raney*-Ni and 2,2,4,4-tetramethyl-3-thioxocyclobutanone (**12**), respectively (*Scheme 4*).

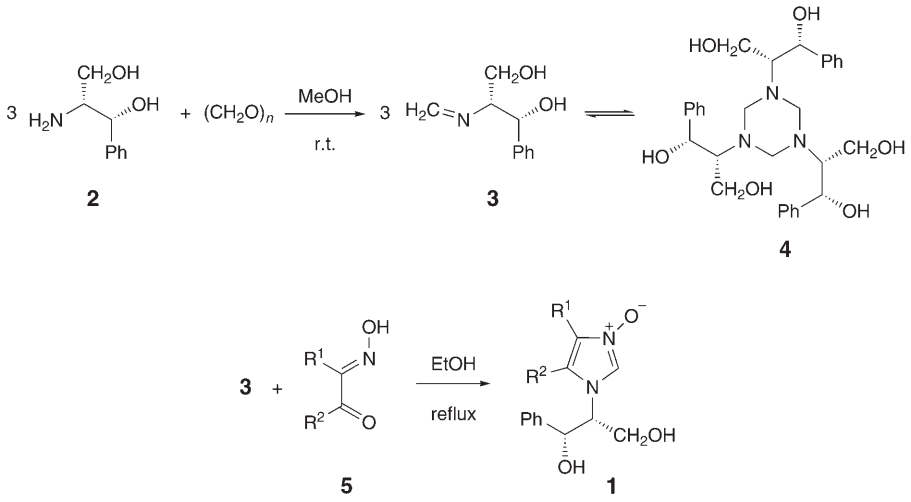
1. Introduction. – Imidazole derivatives are widely applied as versatile building blocks for the preparation of more complex molecules [1]. Several compounds containing the imidazole moiety display biological activities (*e.g.*, [2–5]). Recently, some new methods for the synthesis of differently substituted imidazoles were described [6–8]. Furthermore, in a series of papers, 2-unsubstituted imidazole 3-oxides were shown to be useful starting materials for the preparation of imidazole derivatives such as imidazole-2-thiones [9], imidazol-2-ones [10], 2-cyanoimidazoles [10], 2-(perfluoroethyl)imidazoles [11], 2-(dicyanomethylidene)imidazoles [12], as well as the parent imidazoles [12][13].

Relatively little is known about optically active imidazole derivatives bearing substituents with stereogenic centers [14]. In the first paper on optically active imidazole 3-oxides, a three-component reaction with α-(hydroxyimino) ketones, formaldehyde, and optically active α-amino acids was reported [15]. By using a modified procedure, we succeeded in the preparation of some optically active 1*H*-imidazole 3-oxides **1** starting from enantiomerically pure β-amino alcohols, *e.g.*, **2** [13] (*Scheme 1*). The corresponding formaldimine **3**, which is in equilibrium with its trimer **4**, reacts with α-(hydroxyimino) ketones **5** in refluxing EtOH to give **1**. Analogous

[1]) Presented in preliminary form at the 50th Annual Meeting of the Polish Chemical Society, Toruń, Poland, September 2007.

amino components were exploited in a multicomponent reaction with an aldehyde, a diketone, and NH_3 to yield imidazoles with a chiral 2-hydroxyethyl group attached to N(1) [16]. In a very recent paper, we reported the synthesis of a new type of optically active bis-imidazoles by using enantiomerically pure *trans*-1,2-diaminocyclohexane [17].

Scheme 1



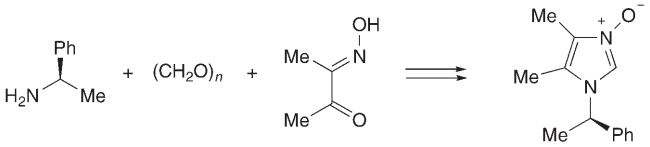
It is well-known that, along with *trans*-1,2-diaminocyclohexane, the commercially available and cheap enantiomerically pure 1-phenylethylamine (α-methylbenzylamine) is one of the most frequently used chiral amine in organic synthesis (see, *e.g.*, [18]). Here, we report the preparation of new optically active imidazole derivatives based on 1-phenylethylamine[2]) as the key reagent used in the reaction with formaldehyde and α-(hydroxyimino) ketones.

2. Results and Discussion. – The three-component reaction carried out with equimolar amounts of (+)-(*R*)- and (−)-(*S*)-1-phenylethylamine (**6**), respectively, formaldehyde, and 3-(hydroxyimino)butan-2-one (**5a**) in boiling EtOH gave the expected 1*H*-imidazole 3-oxide, albeit in low yield. Therefore, the alternative method presented in *Scheme 1* was applied. Thus, the amine **6** and HCHO reacted in MeOH at room temperature to yield the corresponding 1,3,5-triazinane of type **4** as an oily product, which, without purification, was treated with **5a** leading to the desired product **7a** in good yield (*Scheme 2*).

The spectroscopic data confirm the structure of the product, *e.g.*, the ^{1}H-NMR spectrum revealed the typical signal of H−C(2) at 8.16 ppm. Furthermore, the presence of the 1-phenylethyl residue was established by the *doublet* of the Me group (1.82 ppm, $J = 7.2$ Hz) and the *quadruplet* of H−C(1′) at 5.27 ppm. The product (*R*)-**7a**,

[2]) Recently, another strategy for the preparation of imidazole derivatives based on the exploitation of the racemic α-methylbenzylamine was reported [19].

Scheme 2

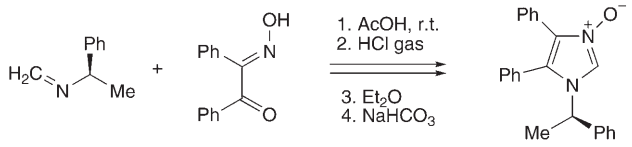


(*R*)-**6** **5a** (*R*)-**7a**

obtained from the reaction with (+)-(*R*)-**6**, showed $[\alpha]_D^{20} = -138.5$ ($c = 1.00$, MeOH). On the other hand, the enantiomer (*S*)-**7a**, which was formed in the reaction with (−)-(*S*)-**6**, displayed the same spectroscopic data and $[\alpha]_D^{20} = +131.5$ ($c = 1.02$, MeOH).

The 4,5-diphenyl analogues **7b** were prepared by using 2-(hydroxyimino)-1,2-diphenylethanone (**5b**). In this case, however, the highest yield of the product was obtained when the reaction of the imine (*R*)-**8** with **5b** was carried out in AcOH instead of MeOH, according to the procedure described earlier [13][17]. After 24 h at room temperature, the product formed was converted into the corresponding hydrochloride, which, after crystallization, was neutralized by treatment with solid $NaHCO_3$ to give the free *N*-oxide **7b** in 75–78% yield (*Scheme 3*).

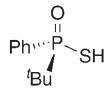
Scheme 3



(*R*)-**8** **5b** (*R*)-**7b**

The successful preparation of **7a** and **7b** prompted us to carry out the analogous reaction with optically active α-cyanobenzylamine (=2-amino-2-phenylacetonitrile). However, the experiment with **5a** and HCHO under the described conditions failed to give the desired product of type **7**.

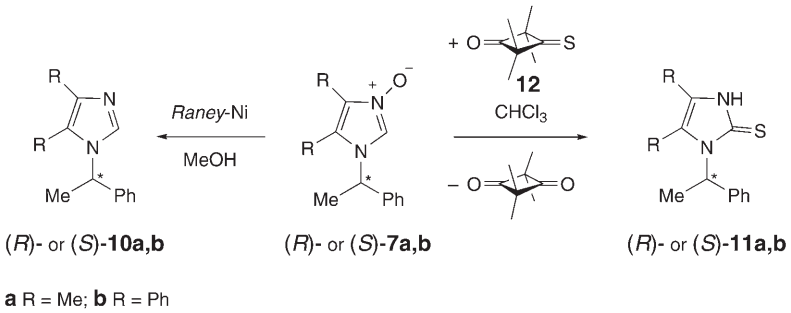
The optical purity of (*R*)-**7b** and (*S*)-**7b** was tested by using (+)-(*R*)-(*tert*-butyl)(phenyl)thiophosphonic acid ((*R*)-**9**) as a chiral solvating agent [20]. Preliminary ^{1}H-NMR experiments with racemic **7b** and equimolar amounts of (*R*)-**9** evidenced that, in $CDCl_3$ at room temperature, two well separated signals of H–C(2) appeared at 9.25 and 9.10 ppm. Under similar conditions, the optically active (*R*)-**7b** and (*S*)-**7b** showed only one single signal at 9.42 and 9.30 ppm, respectively. This result confirms that (*R*)-**7b** and (*S*)-**7b** are optically pure according to ^{1}H-NMR accuracy, *i.e.*, the conversion of **6** to the corresponding 1*H*-imidazole 3-oxides **7b** occurred with complete retention of the configuration of the 1-phenylethylamine fragment.



(*R*)-**9**

Freshly prepared *Raney*-Ni has been shown to be an excellent deoxygenating agent for 1*H*-imidazole 3-oxides [12][13]. Treatment of the (*R*)- and (*S*)-enantiomer of **7a** and **7b** with this reagent in MeOH at room temperature led to the corresponding 1*H*-imidazoles **10** within 30 min (TLC) in almost quantitative yield (*Scheme 4*).

Scheme 4



The optically active 1*H*-imidazole-2-thiones of type **11** were easily prepared by using the S-transfer reaction described in [9]. For example, the color of the red solution of 2,2,4,4-tetramethyl-3-thioxocyclobutanone (**12**) and (*S*)-**7a** in $CHCl_3$ vanished after 1 h stirring at room temperature. The colorless crystalline product (*S*)-**11a** was isolated in 84% yield (*Scheme 4*). The mechanism of this conversion *via* an intermediate 1,4,2-oxathiazolidine, formed as an unstable [2 + 3] cycloadduct, was described earlier [9].

3. Conclusions. – The results described show that optically active 1-phenylethylamine can be used for the synthesis of optically active 2-unsubstituted 1*H*-imidazole 3-oxides of type **7** *via* condensation with formaldehyde and α-(hydroxyimino) ketones. The reaction occurs without loss of optical purity. The enantiomerically pure thiophosphonic acid (*R*)-**9** is a convenient chiral solvating agent for the determination of the optical purity of compounds **7**. According to the known protocols, 1*H*-imidazole 3-oxides **7** can easily be converted into the corresponding 1*H*-imidazoles **10** and 2,3-dihydro-1*H*-imidazole-2-thiones **11**. All of the described optically active imidazole derivatives can be considered as attractive ligands in coordination chemistry and precursors of carbenes derived from imidazole [21].

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Experimental Part

1. *General.* M.p.: *Melt-Temp. II* apparatus (*Aldrich*); in capillary, uncorrected. IR Spectra (KBr): *NEXUS FT-IR* spectrophotometer. ^{1}H- and ^{13}C-NMR spectra: *Tesla BS567A* (80 and 20 MHz, resp.) or *Bruker AC 300* instrument (300 and 75.5 MHz, resp.); in $CDCl_3$, TMS as an internal standard. The multiplicity of the ^{13}C signals was deduced from the DEPT spectra. MS (EI or CI): *Finnigan MAT-90* or *Finnigan SSQ-700* instruments. Elemental analyses were performed in the Analytical Laboratory of the University of Zürich.

2. *Starting Materials.* α-(Hydroxyimino) ketones **5** were obtained according to known protocols: *3-(hydroxyimino)butan-2-one* (**5a**) by nitrosation of butan-2-one [22a] and *2-(hydroxyimino)-1,2-*

diphenylethanone (**5b**, 'benzil monooxime') from dibenzoyl and hydroxylamine hydrochloride [22b]. *(+)-(*R*)-*(tert-*Butyl)(phenyl)thiophosphonic acid* ((*R*)-**9**) was obtained by resolution of the racemic mixture according to a known protocol [20b].

3. *Synthesis of Enantiomerically Pure 1*H-*Imidazole 3-Oxides. Method A*. To a stirred soln. of (*R*)- or (*S*)-*1-phenylethylamine* ((*R*)-**6** or (*S*)-**6**; 242 mg, 2.00 mmol) in MeOH (5 ml), solid HCHO (63.0 g, 2.1 mmol) was added, and stirring was continued overnight. Then, the mixture was concentrated, **5a** (258 mg, 2.56 mmol) was added, and the mixture was heated to reflux for 3 h. The solvent was removed under reduced pressure, and the resulting solid was washed with acetone to give anal. pure samples.

3.1. *(*R*)-1-(1-Phenylethyl)-4,5-dimethyl-1*H-*imidazole 3-Oxide* ((*R*)-**7a**). Yield: 355 mg (82%). Colorless crystals. M.p. (dec.) 224°. $[\alpha]_D^{20} = -138.5$ ($c = 1.00$, MeOH). IR: 3059*m*, 2993*s*, 2939*m*, 1633*m*, 1602*w*, 1490*m*, 1445*m*, 1409*m*, 1346*s*, 1335v*s*, 1236*m*, 1198*s*, 1047*m*, 850*m*, 807*m*, 752*s*, 699v*s*, 595*s*, 581*m*. ^{1}H-NMR: 1.82 (*d*, $J = 7.2$, *Me*CH); 2.00, 2.18 (2*s*, 2 Me); 5.27 (*q*, $J = 7.0$, MeC*H*); 7.08–7.40 (*m*, 5 arom. H); 8.16 (*s*, H–C(2)). ^{13}C-NMR: 7.2, 9.0 (2 Me); 22.0 (*Me*CH); 55.7 (Me*C*H); 125.6, 128.4, 129.2 (5 arom. CH); 122.8 (C(2)); 121.3, 127.3, 140.1 (3 C_q). EI-MS: 216 (6, M^+), 200 (10, $[M-16]^+$), 105 (100), 96 (27). CI-MS (NH_3): 218 (15), 217 (100, $[M+1]^+$), 201 (19), 105 (9). Anal. calc. for $C_{13}H_{16}N_2O$ (216.29): C 72.19, H 7.46; found: C 72.24, H 7.38.

3.2. *(*S*)-1-(1-Phenylethyl)-4,5-dimethyl-1*H-*imidazole 3-Oxide* ((*S*)-**7a**). Yield: 380 mg (88%). Colorless crystals. M.p. (dec.) 230°. $[\alpha]_D^{20} = +131.5$ ($c = 1.02$, MeOH). IR: 3060*m*, 2982*s*, 2939*m*, 1633*m*, 1602*w*, 1490*m*, 1445*m*, 1408*m*, 1345*s*, 1334v*s*, 1236*m*, 1197*s*, 1047*m*, 849*m*, 807*m*, 752*s*, 700v*s*, 595*s*, 580*m*. CI-MS (NH_3): 218 (15), 217 (100, $[M+1]^+$), 201 (8), 105 (100).

Method B. To a stirred soln. of (*R*)-**6** or (*S*)-**6** (242 mg, 2.00 mmol) in MeOH (5 ml), solid HCHO (63.0 g, 2.1 mmol) was added, and stirring was continued overnight. The solvent was evaporated, the resulting oil and **5b** (304 mg, 2.48 mmol) in AcOH (10 ml) was stirred overnight at r.t. Then, HCl gas was bubbled through the soln. for 1.5 h. The solvent was evaporated, the obtained oil was washed with Et_2O, and the colorless hydrochloride was filtered and dried *in vacuo*. The crude hydrochloride was dissolved in MeOH (30 ml), solid $NaHCO_3$ (1.0 g) was added, and stirring was continued for 1 h. The inorg. salts were filtered off, the solvent was evaporated, and the solid residue washed with $CHCl_3$/MeOH 2:1 and then triturated with acetone. The colorless crystals obtained were anal. pure.

3.3. *(*R*)-1-(1-Phenylethyl)-4,5-diphenyl-1*H-*imidazole 3-Oxide* ((*R*)-**7b**). Yield: 510 mg (75%). Colorless crystals. M.p. (dec.) 217°. $[\alpha]_D^{20} = +30.0$ ($c = 1.00$, MeOH). IR: 3046*s*, 2990*m*, 2964*m*, 2949*m*, 1636*m*, 1602*m*, 1585*m*, 1497*m*, 1444*m*, 1340*s*, 1237*m*, 767*m*, 761*m*, 703*s*.

3.4. *(*S*)-1-(1-Phenylethyl)-4,5-diphenyl-1*H-*imidazole 3-Oxide* ((*S*)-**7b**). Yield: 530 mg (78%). Colorless crystals. M.p. (dec.) 218°. $[\alpha]_D^{20} = -27.0$ ($c = 0.98$, MeOH). IR: 3046*s*, 2990*m*, 2961*m*, 2949*m*, 1636*m*, 1602*m*, 1584*m*, 1497*m*, 1444*m*, 1340*s*, 1236*m*, 767*m*, 760*m*, 703*s*. ^{1}H-NMR: 1.78 (*d*, $J = 7.1$, *Me*CH); 5.24 (*q*, $J = 7.1$, MeC*H*); 7.03–7.60 (*m*, 15 arom. H); 8.08 (*s*, H–C(2)). ^{13}C-NMR: 21.8 (*Me*CH); 55.5 (Me*C*H); 124.1, 125.9, 127.9, 128.5, 129.0, 129.1, 129.5, 129.6, 130.9 (15 arom. CH, C(2)); 126.8, 127.1, 127.6, 128.3, 139.7 (5 C_q). CI-MS (isobutane): 342 (24), 341 (100, $[M+1]^+$), 325 (20). Anal. calc. for $C_{23}H_{20}N_2O$ (340.43): C 81.15, H 5.92, N 8.23; found: C 80.97, H 6.02, N 8.25.

4. *Deoxygenation of Enantiomerically Pure 1*H-*Imidazole 3-Oxides*. To a soln. of the corresponding (*R*)- or (*S*)-1*H*-imidazole 3-oxide **7** (1.0 mmol) in MeOH (5 ml), a suspension of freshly prepared *Raney*-Ni in MeOH was added in small portions. When the starting *N*-oxide **7** was completely reduced (TLC), the mixture was filtered, and the filtrate was concentrated under reduced pressure. Crude products were purified by recrystallization from hexane/CH_2Cl_2.

4.1. *(*R*)-1-(1-Phenylethyl)-4,5-dimethyl-1*H-*imidazole* ((*R*)-**10a**). Yield: 153 mg (77%). Colorless crystals. M.p. (dec.) 179° (hexane/CH_2Cl_2). $[\alpha]_D^{20} = -43.0$ ($c = 1.10$, MeOH). IR: 3103*m*, 3083*m*, 3062*m*, 3029*m*, 2988*m*, 2980*m*, 2925*m*, 2865*m*, 1641*m*, 1599*m*, 1493*m*, 1482*m*, 1445*m*, 1389*m*, 1344*m*, 1236*s*, 755*m*, 699*s*. ^{1}H-NMR: 1.83 (*d*, $J = 7.1$, *Me*CH); 1.92, 2.15 (2*s*, 2 Me); 5.17 (*q*, $J = 7.1$, MeC*H*); 7.00–7.38 (*m*, 5 arom. H); 7.56 (*s*, H–C(2)). ^{13}C-NMR: 8.6, 12.6 (2 Me); 22.3 (*Me*CH); 54.8 (Me*C*H); 125.5, 127.5, 128.7 (5 arom. CH); 122.4, 134.2, 142.0 (3 C_q); 132.6 (C(2)). CI-MS (NH_3): 202 (15), 201 (100, $[M+1]^+$), 200 (10), 105 (8). Anal. calc. for $C_{13}H_{16}N_2$ (200.29): C 77.96, H 8.05, N 13.99; found: C 77.94, H 8.12, N 14.01.

4.2. *(*S*)-1-(1-Phenylethyl)-4,5-dimethyl-1*H-*imidazole* ((*S*)-**10a**). Yield: 162 mg (82%). Colorless crystals. M.p. (dec.) 178° (hexane/CH_2Cl_2). $[\alpha]_D^{20} = +33.3$ ($c = 1.00$, MeOH). IR: 3103*m*, 3083*m*, 3062*m*,

3030*m*, 2988*m*, 2980*m*, 2925*m*, 2865*m*, 1646*m*, 1599*m*, 1493*m*, 1482*m*, 1445*m*, 1389*m*, 1344*m*, 1236*s*, 755*m*, 699*s*. CI-MS (NH_3): 202 (15), 201 (100, $[M+1]^+$), 200 (6), 105 (3).

4.3. *(*R*)-1-(1-Phenylethyl)-4,5-diphenyl-1*H*-imidazole* ((*R*)-**10b**). Yield: 227 mg (73%). Colorless crystals. M.p. (dec.) 180° (hexane/CH_2Cl_2). $[\alpha]_D^{20} = +83.0$ ($c = 1.00$, MeOH). IR: 3090*m*, 3063*m*, 3049*m*, 3032*m*, 2983*m*, 2945*m*, 1634*s*, 1601*m*, 1505*m*, 1485*m*, 1474*m*, 1454*m*, 1442*m*, 1374*m*, 768*m*, 695*s*.

4.4. *(*S*)-1-(1-Phenylethyl)-4,5-diphenyl-1*H*-imidazole* ((*S*)-**10b**). Yield: 255 mg (82%). Colorless crystals. M.p. (dec.) 176° (hexane/CH_2Cl_2). $[\alpha]_D^{20} = -80.0$ ($c = 0.60$, MeOH). IR: 3090*m*, 3063*m*, 3049*m*, 3032*m*, 2984*m*, 2946*m*, 1633*s*, 1601*m*, 1505*m*, 1484*m*, 1473*m*, 1454*m*, 1442*m*, 1375*m*, 768*m*, 695*s*. ^{1}H-NMR: 1.81 (*d*, $J = 7.1$, *Me*CH); 5.11 (*q*, $J = 7.1$, MeC*H*); 6.95–7.50 (*m*, 15 arom. H); 7.76 (*s*, H–C(2)). ^{13}C-NMR: 22.4 (*Me*CH); 54.5 (Me*C*H); 125.8, 126.2, 126.4, 127.7, 128.1, 128.7, 128.8, 131.1, 134.4 (15 arom. CH, C(2)); 127.3, 128.3, 130.9, 138.1, 141.8 (5 C_q). CI-MS (isobutane): 326 (22), 325 (100, $[M+1]^+$), 324 (70, $M^{+\bullet}$). Anal. calc. for $C_{23}H_{20}N_2$ (324.43): C 85.15, H 6.21, N 8.63; found: C 84.95, H 6.17, N 8.54.

5. *Transformations of Enantiomerically Pure Imidazole 3-Oxides to 1*H*-Imidazole-2-thiones.* To a cooled $CHCl_3$ soln. (water bath) of *2,2,4,4-tetramethyl-3-thioxocyclobutanone* (**12**; 1 mmol), the respective 1*H*-imidazole 3-oxide **7** (1 mmol) was added in small portions, and the mixture was stirred for *ca.* 1 h until the characteristic red color of the soln. vanished. After concentration under reduced pressure, the mixture was washed with pentane, and the obtained crude solid was recrystallized from MeOH or from petroleum ether/Et_2O.

5.1. *(*R*)-2,3-Dihydro-4,5-dimethyl-1-(1-phenylethyl)-1*H*-imidazole-2-thione* ((*R*)-**11a**). Yield: 190 mg (82%). Colorless crystals. M.p. 124–126° (petroleum ether/Et_2O). $[\alpha]_D^{20} = +176.0$ ($c = 1.00$, CH_2Cl_2). IR: 3159*m*, 3086*s*, 2979*m*, 2926*s*, 2718*m*, 1655*m*, 1605*m*, 1496*s*, 1448*s*, 1415*s*, 1374*s*, 1332*m*, 1239*m*, 1028*m*, 750*m*, 700*s*.

5.2. *(*S*)-2,3-Dihydro-4,5-dimethyl-1-(1-phenylethyl)-1*H*-imidazole-2-thione* ((*S*)-**11a**). Yield: 194 mg (84%). Colorless crystals. M.p. 126–128° (petroleum ether/Et_2O). $[\alpha]_D^{20} = -170.0$ ($c = 0.90$, CH_2Cl_2). IR: 3162*m*, 3086*s*, 2978*m*, 2926*s*, 2718*m*, 1656*m*, 1605*m*, 1496*s*, 1448*s*, 1415*s*, 1374*s*, 1332*m*, 1239*m*, 1028*m*, 751*m*, 700*s*. ^{1}H-NMR: 1.67, 2.05 (2*s*, 2 Me); 1.81 (*d*, $J = 7.1$, *Me*CH); 6.61 (*q*, $J = 7.1$, MeC*H*); 7.27–7.34 (*m*, 5 arom. H); 11.69 (br. *s*, NH). ^{13}C-NMR: 8.7, 9.8 (2 Me); 17.1 (*Me*CH); 53.3 (Me*C*H); 121.0, 121.3 (2 MeC_q); 126.5, 127.3, 128.5 (5 arom. CH); 139.9 (arom. C_q); 158.5 (C=S). CI-MS (isobutane): 233 (100, $[M+1]^+$), 232 (12). Anal. calc. for $C_{13}H_{16}N_2S$ (232.35): C 67.20, H 6.94, N 12.06, S 13.80; found: C 67.05, H 7.05, N 12.17, S 13.55.

5.3. *(*R*)-2,3-Dihydro-4,5-diphenyl-1-(1-phenylethyl)-1*H*-imidazole-2-thione* ((*R*)-**11b**). Yield: 275 mg (77%). Colorless crystals. M.p. 282–284° (MeOH). $[\alpha]_D^{20} = +104.7$ ($c = 0.57$, CH_2Cl_2). IR: 3088*m*, 3061*m*, 2982*m*, 2936*m*, 2726*m*, 1630*m*, 1603*m*, 1491*s*, 1477*m*, 1446*m*, 1406*m*, 1374*m*, 1345*m*, 1254*m*, 761*m*, 687*s*. ^{1}H-NMR: 1.61 (*d*, $J = 7.1$, *Me*CH); 7.05–7.35 (*m*, 15 arom. H, MeC*H*); 12.15 (*s*, NH). ^{13}C-NMR: 17.4 (*Me*CH); 54.4 (Me*C*H); 126.2, 127.9, 128.7, 140.1 (5 C_q); 126.4, 126.9, 127.2, 127.7, 128.1, 128.2, 128.5, 129.0, 132.0 (15 arom. CH); 160.9 (C=S). CI-MS (isobutane): 358 (24), 357 (100, $[M+1]^+$), 356 (21). Anal. calc. for $C_{23}H_{20}N_2S$ (356.49): C 77.49, H 5.65, N 7.86, S 8.99; found: C 77.28, H 5.73, N 7.74, S 8.78.

5.4. *(*S*)-2,3-Dihydro-4,5-diphenyl-1-(1-phenylethyl)-1*H*-imidazole-2-thione* ((*S*)-**11b**). Yield: 268 mg (75%). Colorless crystals. M.p. 276–278° (MeOH). $[\alpha]_D^{20} = -96.6$ ($c = 0.57$, CH_2Cl_2). IR: 3089*m*, 3061*m*, 2983*m*, 2938*m*, 2729*m*, 1632*m*, 1603*m*, 1491*s*, 1479*m*, 1446*m*, 1407*m*, 1376*m*, 1344*m*, 1254*m*, 759*m*, 697*s*.

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