

Chemoselectivity in the Reaction of 2-Diazo-3-oxo-3-phenylpropanal with Aldehydes and Ketones

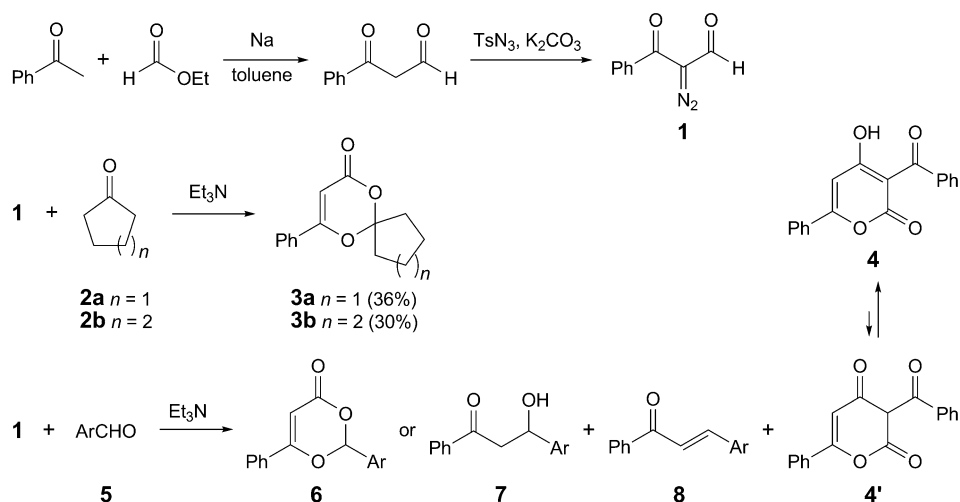
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The chemoselectivity in the reaction of 2-diazo-3-oxo-3-phenylpropanal (**1**) with aldehydes and ketones in the presence of Et₃N was investigated. The results indicate that **1** reacts with aromatic aldehydes with weak electron-donating substituents and cyclic ketones under formation of 6-phenyl-4*H*-1,3-dioxin-4-one derivatives. However, it reacts with aromatic aldehydes with electron-withdrawing substituents to yield 1,3-diaryl-3-hydroxypropan-1-ones, accompanied by chalcone derivatives in some cases. It did not react with linear ketones, aliphatic aldehydes, and aromatic aldehydes with strong electron-donating substituents. A mechanism for the formation of 1,3-diaryl-3-hydroxypropan-1-ones and chalcone derivatives is proposed. We also tried to react **1** with other unsaturated compounds, including various olefins and nitriles, and cumulated unsaturated compounds, such as *N,N'*-dialkylcarbodiimines, phenyl isocyanate, isothiocyanate, and CS₂. Only with *N,N'*-dialkylcarbodiimines, the expected cycloaddition took place.

1. Introduction. – α -Diazo- β -dicarbonyl compounds react with various unsaturated bonds yielding different heterocyclic compounds [1–9]. Recently, we reported on the synthesis of a series of 2,3,6-trisubstituted 2*H*-1,3-oxazin-4(3*H*)-one derivatives in satisfactory-to-good yields by reaction of imines and 2-diazo-3-oxo-3-phenylpropanal (**1**) in the presence of the catalytic amount of Et₃N under mild conditions [9][10]. The reaction is metal-free, mild, and highly regioselective. To extend the application of 2-diazo-3-oxo-3-phenylpropanal (**1**) to cycloaddition reactions with unsaturated compounds, we explored its reaction with aldehydes and ketones and found that the cycloaddition reaction shows a different chemoselectivity depending on the structural feature of aldehydes and ketones. The mechanism of the formation of 1,3-diaryl-3-hydroxypropan-1-ones and chalcone derivatives is also discussed.

2. Results and Discussion. – 2-Diazo-3-oxo-3-phenylpropanal (**1**) was previously prepared from 2-diazoacetophenone and the *Vilsmeier–Haack* reagent [9a], which was synthesized from DMF and (COCl)₂ [11]. However, the overall yield was low. The diazo transformation is a general method for the preparation of α -diazo- β -diketones and α -diazo- β -oxo alkanoates from the corresponding β -diketones and β -oxo alkanoates under basic conditions [2]. We attempted to synthesize 2-diazo-3-oxo-3-phenylpropanal (**1**) by this method. 3-Oxo-3-phenylpropanal was prepared from acetophenone and HCOOEt by *Claisen* condensation. It was converted to 2-diazo-3-

Scheme 1. Reactions of 2-Diazo-3-oxo-3-phenylpropanal (**1**) with Ketones and Aldehydes

oxo-3-phenylpropanal (**1**) by the treatment with TsN_3 in the presence of K_2CO_3 in a high yield (Scheme 1).

2-Diazo-3-oxo-3-phenylpropanal (**1**) was reacted with various ketones, however, only with cycloalkanones **2** (cyclopentanone (**2a**) and cyclohexanone (**2b**)), the corresponding spiro 6-phenyl-4*H*-1,3-dioxin-4-one derivatives **3a** and **3b** were formed in the presence of Et_3N (Scheme 1). On the other hand, aromatic ketones (acetophenone and benzophenone) and aliphatic linear ketones (acetone, heptan-2-one, and heptan-4-one) did not undergo the desired cycloaddition. Instead, the benzoylketene generated from **1** dimerized to 3-benzoyl-6-phenyl-2*H*-pyran-2,4(3*H*)-dione (**4'**), which tautomerized to give 3-benzoyl-4-hydroxy-6-phenyl-2*H*-pyran-2-one (**4**) [12].

The reaction of **1** with various aldehydes was attempted as well. No desired cycloaddition was observed with aliphatic aldehydes. However, diverse products were obtained with aromatic aldehydes **5**, depending on the electronic characters of the aromatic aldehydes (Table). We found that **1** reacted with aromatic aldehydes with weak electron-donating substituents to generate 6-phenyl-4*H*-1,3-dioxin-4-one derivatives **6**. However, it reacted with aromatic aldehydes with electron-withdrawing substituents to yield 1,3-diaryl-3-hydroxypropan-1-ones **7**, accompanied by chalcone derivatives **8** in some cases.

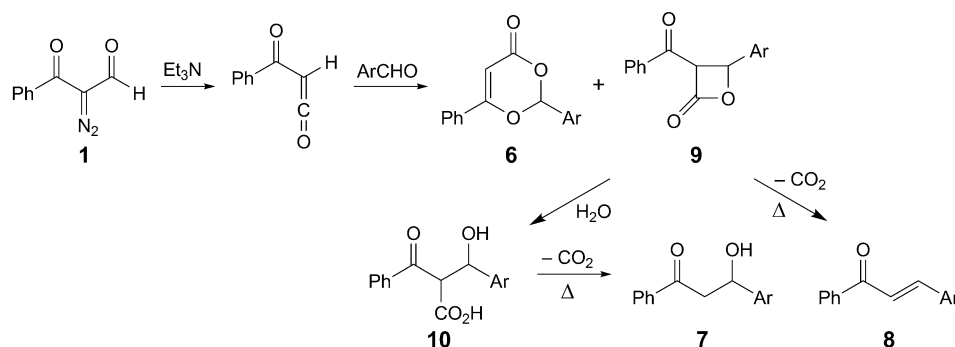
On the basis of the above results, we propose the mechanism of the chemoselective reaction of **1** with aromatic aldehydes as depicted in Scheme 2. Diazo compound **1** is converted in the presence of Et_3N to benzoylketene, which undergoes [4 + 2] or [2 + 2] cycloaddition chemoselectively with different aromatic aldehydes to give rise to 6-phenyl-4*H*-1,3-dioxin-4-one derivatives **6** or 3-benzoyl- β -lactones **9**, respectively. However, 3-benzoyl- β -lactones **9** are unstable under the reaction conditions and undergo elimination of CO_2 to afford chalcone derivatives **8**. Lactones **9** are hydrolyzed to β -oxo alkanolic acids **10** during workup, which undergo a decarboxylation to form 3-hydroxy-1-phenylalkan-1-ones **7**. The different cycloadditions result in the chemo-

Table. Reactions of 2-Diazo-3-oxo-3-phenylpropanal (**1**) with Aromatic Aldehydes

Entry	ArCHO	Ar	Yield [%]			
			6	7	8	4
1	5a	4-MeO-C ₆ H ₄	–	–	–	46
2	5b	4-Me ₂ N-C ₆ H ₄	–	–	–	45
3	5c	4-Me-C ₆ H ₄	80	–	–	n.d. ^{a)}
4	5d	Ph	71	–	–	n.d.
5	5e	4-F-C ₆ H ₄	21	–	–	37
6	5f	2-Cl-C ₆ H ₄	–	10	14	36
7	5g	4-Cl-C ₆ H ₄	–	11	–	44
8	5h	2,6-Cl ₂ -C ₆ H ₃	–	10	–	40
9	5i	2-O ₂ N-C ₆ H ₄	–	16	–	33
10	5j	4-O ₂ N-C ₆ H ₄	–	27	–	35
11	5k	2-Cl-5-O ₂ N-C ₆ H ₃	–	16	46	19
12	5l	Pyridin-4-yl	–	19	33	20

^{a)} n.d. = Not determined.

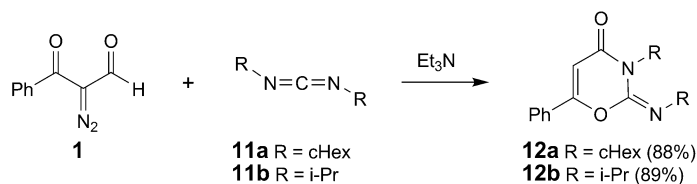
selective formation of various products in the reaction. However, it is not completely clear so far why different aromatic aldehydes undergo [4 + 2] or [2 + 2] cycloaddition chemoselectively with benzoylketene.

Scheme 2. Proposed Reaction Mechanism of the Chemoselective Reaction of 2-Diazo-3-oxo-3-phenylpropanal (**1a**) and Aromatic Aldehydes

To investigate the generality of the cycloaddition of **1** and other unsaturated compounds, we attempted to react **1** with olefins (such as styrene, cyclohexene, 1-phenylpropene, cyclopentadiene, 1,1-dichloroethene, vinyl acetate, and butyl vinyl ether) and nitriles (including benzonitrile, 2-chlorobenzonitrile, acetonitrile, and ethyl cyanoacetate). In all cases, no desired reaction occurred, only 3-benzoyl-6-phenyl-2H-pyran-2,4(3H)-dione, dimer of benzoylketene, was obtained in each of the reactions. The reaction of **1** with cumulated unsaturated compounds, including *N,N'*-dicyclohexylcarbodiimide (DCC; **11a**), *N,N'*-diisopropylcarbodiimide (DIC; **11b**), phenyl isocyanate, phenyl isothiocyanate, and CS_2 , was also attempted. With **11a** and **11b**, the desired cycloadducts, 3-cyclohexyl-2-(cyclohexylimino)-6-phenyl-2H-1,3-oxazin-

4(3*H*)-one (**12a**) and 3-isopropyl-2-(isopropylimino)-6-phenyl-2*H*-1,3-oxazin-4(3*H*)-one (**12b**), respectively, were formed (*Scheme 3*). However, no desired reaction occurred with phenyl isocyanate, phenyl isothiocyanate, and CS₂.

Scheme 3. Reaction of 2-Diazo-3-oxo-3-phenylpropanal (**1**) and *N,N'*-Dialkylcarbodiimides



3. Conclusions. – The chemoselectivity of the reaction of 2-diazo-3-oxo-3-phenylpropanal (**1**) with aldehydes and ketones in the presence of Et₃N was investigated. The results indicate that 2-diazo-3-oxo-3-phenylpropanal (**1**) reacted with aromatic aldehydes with weak electron-donating substituents and cyclic ketones to generate 6-phenyl-4*H*-1,3-dioxin-4-one derivatives. With aromatic aldehydes with electron-withdrawing substituents, however, **1** furnished 1,3-diaryl-3-hydroxypropan-1-ones, accompanied by chalcone derivatives in some cases. For linear aliphatic and aromatic ketones, aliphatic aldehydes, and aromatic aldehydes with strong electron-donating substituents, no cycloaddition reaction was observed. A mechanism of the formation of 1,3-diaryl-3-hydroxypropan-1-ones and chalcone derivatives has been proposed (*Scheme 2*). 2-Diazo-3-oxo-3-phenylpropanal (**1**) was also attempted to react with other unsaturated compounds, such as different olefins and nitriles, and cumulated unsaturated compounds, including *N,N'*-dialkylcarbodiimides, phenyl isocyanate, phenyl isothiocyanate, and CS₂. Only *N,N'*-dialkylcarbodiimides undergo the expected cycloaddition reaction.

Experimental Part

General. Column chromatography (CC): silica gel (200–300 mesh) with petroleum ether (PE) and AcOEt as the eluent. M.p.: Yanaco MP-500; uncorrected. IR Spectra: Nicolet Avatar 330 FT-IR spectrometer; in KBr; $\tilde{\nu}$ in cm^{−1}. ¹H- and ¹³C-NMR spectra: Bruker AV 400 at 400 MHz in CDCl₃; δ in ppm rel. to Me₄Si as internal standard, *J* in Hz. HR-ESI-MS: LC/MSD TOF mass spectrometer; in *m/z*.

Reaction of 2-Diazo-3-oxo-3-phenylpropanal (1) with Unsaturated Compounds in the Presence of Et₃N: General Procedure. To a soln. of **1** (87 mg, 0.5 mmol) and an aldehyde, ketone, or carbodiimide (0.5 mmol) in CH₂Cl₂ (1 ml) was added a drop of Et₃N (30 mg, 0.3 mmol) under stirring in an ice-water bath or at r.t. N₂ was liberated immediately, and TLC monitoring indicated that the reaction was complete. After stirring for 2 to 5 s, the residue was separated by CC (AcOEt/PE(60–90°) 1:10 to 1:20) to afford product(s).

9-Phenyl-6,10-dioxaspiro[4.5]dec-8-en-7-one (3a). Colorless oil. M.p. 54–56° ([13]). Yield: 42 mg (36%). IR: 1722 (C=O). ¹H-NMR: 1.51–2.31 (*m*, 4 CH₂); 5.93 (*s*, CH); 7.47–7.50 (*m*, 2 arom. H); 7.55 (*m*, arom. H); 7.71–7.73 (*m*, 2 arom. H). ¹³C-NMR: 18.4; 23.2; 36.7; 58.5; 67.3; 71.0; 88.4; 92.3; 116.3; 126.4; 128.9; 132.2.

4-Phenyl-1,5-dioxaspiro[5.5]undec-3-en-2-one (3b). Colorless oil. M.p. 54–55° ([13]). Yield: 37 mg (30%). IR: 1721 (C=O). ¹H-NMR: 1.49–3.51 (*m*, 5 CH₂); 5.91 (*s*, CH); 7.47–7.55 (*m*, 2 arom. H); 7.56

(*m*, arom. H); 7.74–7.76 (*m*, 2 arom. H). ^{13}C -NMR: 22.5; 24.7; 33.8; 91.5; 107.3; 126.4; 128.9; 132.1; 162.0; 164.6.

3-Benzoyl-4-hydroxy-6-phenyl-2H-pyran-2-one (4). Yellow crystals. M.p. 169–170° ([12b]: 169–170°). Yield: 67 mg (46%). ^1H -NMR: 6.66 (*s*, CH); 7.43–7.92 (*m*, 10 arom. H); 15.94 (*s*, C=COH).

2-(4-Methylphenyl)-6-phenyl-4H-1,3-dioxin-4-one (6c). Yield: 106 mg (80%). Colorless crystals. M.p. 109–110°. IR: 1724 (C=O). ^1H -NMR: 2.45 (*s*, Me); 6.09 (*s*, C=CH); 6.56 (*s*, CH); 7.31–7.79 (*m*, 9 arom. H). ^{13}C -NMR: 21.4; 93.2; 100.5; 126.7; 128.9; 129.4; 130.2; 130.9; 132.5; 140.6; 163.2; 168.3. HR-ESI-MS: 267.1014 ($[M+H]^+$, $\text{C}_{17}\text{H}_{15}\text{O}_3^+$; calc. 267.1016).

2,6-Diphenyl-4H-1,3-dioxin-4-one (6d). Yield: 90 mg (71%). Colorless crystals. M.p. 103–105° ([13]: 103–105°). IR: 1680 (C=O). ^1H -NMR: 6.11 (*s*, C=CH); 6.60 (*s*, CH); 7.47–7.81 (*m*, 10 arom. H). ^{13}C -NMR: 93.3; 100.4; 126.7; 126.7; 128.7; 129.0; 130.5; 132.6; 163.0; 168.2.

2-(4-Fluorophenyl)-6-phenyl-4H-1,3-dioxin-4-one (6e). Yield: 28 mg (21%). Colorless crystals. M.p. 152–153°. IR: 1733 (C=O). ^1H -NMR: 6.08 (*s*, C=CH); 6.55 (*s*, CH); 7.16–7.77 (*m*, 9 arom. H). ^{13}C -NMR: 93.3; 99.7; 115.7; 116.0; 126.7; 128.8; 128.9; 129.0; 132.6; 162.8; 168.2; 172.1. HR-ESI-MS: 271.0771 ($[M+H]^+$, $\text{C}_{16}\text{H}_{12}\text{FO}_3^+$; calc. 271.0765).

3-(2-Chlorophenyl)-3-hydroxy-1-phenylpropan-1-one (7f). Yield: 13 mg (10%). Colorless crystals. M.p.: 78–80° ([14]: 80°). IR: 1677 (C=O), 3466 (OH). ^1H -NMR: 3.18 (*dd*, $J=9.6$, 17.6, 1 H of CH_2); 3.59 (*dd*, $J=2.4$, 17.6, 1 H of CH_2); 3.87 (*s*, OH); 5.72 (*dd*, $J=2.4$, 9.6, CH); 7.25–8.00 (*m*, 9 arom. H). ^{13}C -NMR: 45.4; 66.9; 127.3; 128.2; 128.6; 128.8; 129.4; 131.2; 133.8; 136.5; 140.4; 200.3.

3-(4-Chlorophenyl)-3-hydroxy-1-phenylpropan-1-one (7g). Yield: 15 mg (11%). Colorless crystals. M.p. 99–100° ([15]: 99.4–100.4°). IR (KBr): 1724 (C=O), 3446 (OH). ^1H -NMR: 3.37 (*dd*, $J=2.8$, 17.2, 1 H of CH_2); 4.04 (*dd*, $J=9.6$, 17.2, 1 H of CH_2); 3.92 (*s*, OH); 6.19 (*dd*, $J=2.8$, 9.6, CH); 7.45–8.00 (*m*, 8 arom. H). ^{13}C -NMR: 44.8; 66.5; 123.1; 123.4; 128.2; 128.9; 130.3; 134.1; 142.6; 199.6.

3-(2,6-Dichlorophenyl)-3-hydroxy-1-phenylpropan-1-one (7h). Yield: 15 mg (10%). Colorless crystals. M.p. 150–151°. IR: 1643 (C=O), 3243 (OH). ^1H -NMR: 7.71 (*d*, $J=18.8$, ArCH); 7.66 (*d*, $J=18.8$, COCH); 7.47–7.65 (*m*, 5 arom. H); 8.02–8.70 (*m*, 4 arom. H). HR-ESI-MS: 295.0288 ($[M+H]^+$, $\text{C}_{15}\text{H}_{13}\text{Cl}_2\text{O}_2^+$; calc. 295.0287).

3-Hydroxy-3-(2-nitrophenyl)-1-phenylpropan-1-one (7i). Yield: 22 mg (16%). Colorless crystals. M.p. 106–107° ([16]: 106–107°). IR: 1678 (C=O), 3509 (OH). ^1H -NMR: 3.21 (*dd*, $J=9.2$, 17.6, 1 H of CH_2); 3.74 (*dd*, $J=2.0$, 17.6, 1 H of CH_2); 4.01 (*s*, OH); 5.88 (*dd*, $J=2.0$, 9.2, CH); 7.45–8.00 (*m*, 9 arom. H). ^{13}C -NMR: 45.4; 66.9; 127.3; 128.2; 128.6; 128.8; 129.4; 131.2; 133.8; 136.5; 140.4; 200.3.

3-Hydroxy-3-(4-nitrophenyl)-1-phenylpropan-1-one (7j). Yield: 37 mg (27%). Colorless crystals. M.p. 113–114° ([15]: 114–114.7°). IR: 1670 (C=O), 3499 (OH). ^1H -NMR: 3.38 (*dd*, $J=9.6$, 17.6, 1 H of CH_2); 3.58 (*dd*, $J=4.0$, 17.6, 1 H of CH_2); 3.90 (*s*, OH); 5.71 (*dd*, $J=4.0$, 9.6, CH); 7.48–8.26 (*m*, 8 arom. H). ^{13}C -NMR: 47.0; 69.2; 123.8; 126.6; 128.2; 134.0; 136.2; 147.4; 150.3; 199.5.

3-(2-Chloro-5-nitrophenyl)-3-hydroxy-1-phenylpropan-1-one (7k). Yield: 25 mg (16%). Colorless crystals. M.p. 120–122°. IR: 1681 (C=O), 3466 (OH). ^1H -NMR: 3.17 (*dd*, $J=9.6$, 17.6, 1 H of CH_2); 3.58 (*dd*, $J=2.0$, 17.6, 1 H of CH_2); 4.01 (*s*, OH); 5.71 (*dd*, $J=2.0$, 9.6, CH); 7.26–8.64 (*m*, 8 arom. H). ^{13}C -NMR: 42.8; 67.7; 128.3; 128.7; 129.32; 129.5; 133.6; 134.8; 136.3; 136.6; 198.9. HR-ESI-MS: 306.0528 ($[M+H]^+$, $\text{C}_{15}\text{H}_{13}\text{ClNO}_4^+$; calc. 306.0528).

3-Hydroxy-1-phenyl-3-(pyridin-4-yl)propan-1-one (7l) [17]. Yield: 22 mg (19%). Colorless oil. ^1H -NMR: 3.35 (*d*, $J=6.0$, CH_2); 4.21 (*s*, OH); 5.36 (*t*, $J=6.0$, CH); 7.37 (*d*, $J=6.0$, 2 arom. H); 7.47 (*t*, $J=7.6$, 2 arom. H); 7.60 (*t*, $J=7.2$, arom. H); 7.95 (*dd*, $J=7.2$, 1.2, 2 arom. H); 8.57 (*dd*, $J=1.2$, 4.4, 2 arom. H). ^{13}C -NMR: 46.7; 68.6; 120.6; 128.1; 128.7; 133.7; 136.3; 149.7; 152.0; 199.4.

(2E)-3-(2-Chlorophenyl)-1-phenylprop-2-en-1-one (8f) [18]. Yield: 17 mg (14%). Colorless oil. M.p. 50–52°. IR: 1663 (C=O). ^1H -NMR: 8.21 (*d*, $J=16.0$, ArCH); 7.52 (*d*, $J=16.0$, COCH); 7.34–8.06 (*m*, 9 arom. H).

(2E)-3-(2-Chloro-5-nitrophenyl)-1-phenylprop-2-en-1-one (8k). Yield: 66 mg (46%). Colorless crystals. M.p. 152–155° ([19]: 151–152°). IR: 1670 (C=O). ^1H -NMR: 8.18 (*d*, $J=17.2$, ArCH); 7.70 (*d*, $J=17.2$, COCH); 7.65–8.65 (*m*, 8 arom. H).

(2E)-1-Phenyl-3-(pyridin-4-yl)prop-2-en-1-one (8l). Yield: 35 mg (33%). Colorless crystals. M.p. 74–75° ([20]: 73–74.5°). IR: 1667 (C=O). ^1H -NMR: 7.71 (*d*, $J=18.8$, ArCH); 7.66 (*d*, $J=18.8$, COCH);

7.47–7.65 (*m*, 5 arom. H); 8.02–8.70 (*m*, 4 arom. H). ¹³C-NMR: 122.0; 125.9; 128.5; 128.6; 133.1; 137.2; 141.3; 141.9; 150.4; 189.6.

3-Cyclohexyl-2-(cyclohexylimino)-6-phenyl-2,3-dihydro-4H-1,3-oxazin-4-one (**12a**). Yield: 155 mg (88%). Colorless crystals. M.p. 123–125° ([21]; 119–120°). IR: 1670 (C=O). ¹H-NMR: 1.24–2.63 (*m*, 10 CH₂); 3.85 (*m*, CHN); 4.77 (*m*, CHN); 6.08 (*s*, CH); 7.48–7.54 (*m*, 3 arom. H); 7.70–7.73 (*m*, arom. H). ¹³C-NMR: 22.4; 25.5; 26.1; 26.4; 28.0; 33.9; 53.6; 54.5; 97.1; 125.6; 129.0; 131.6; 159.5; 162.1.

2,3-Dihydro-3-(1-methylethyl)-2-[(1-methylethyl)imino]-6-phenyl-4H-1,3-oxazin-4-one (**12b**). Yield: 122 mg (89%). Colorless crystals. M.p. 47–48°. IR: 1670 (C=O). ¹H-NMR: 1.21 (*d*, *J* = 6.4, NCHMe₂); 1.48 (*d*, *J* = 6.4, NCHMe₂); 4.12 (*sept.*, *J* = 6.4, NCHMe₂); 5.15 (*sept.*, *J* = 6.4, NCHMe₂); 6.05 (*s*, CH); 7.45–7.71 (*m*, 5 arom. H). HR-ESI-MS: 272.1525 ([*M* + H]⁺, C₁₆H₂₀N₂O₂⁺; calc. 272.1525).

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