Electrochemically Induced *N*-Acryloylation of Chiral Oxazolidin-2-ones

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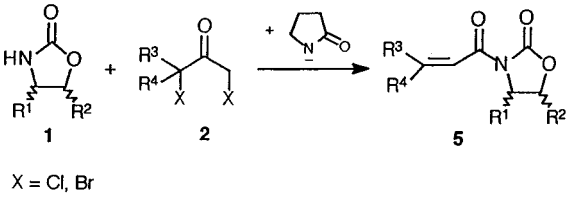
A new method for *N*-acryloylation of Evans' chiral auxiliaries (oxazolidin-2-ones) with α,α′-dichloro ketones in the presence of the electrogenerated base 2-pyrrolidone anion is described. *N*-Enoyloxazolidin-2-ones are obtained, under mild reaction conditions, in good to high yields.

Introduction

In the last two decades, chiral oxazolidin-2-ones have been used as chiral auxiliaries in a wide range of transformations.[1−4] High diastereoselectivity induced by *N*-acyloxazolidin-2-ones in alkylation, acylation, and aldol reactions has frequently been observed.[1,5,6] *N*-Enoyloxazolidin-2-ones have been successfully utilized as dienophiles in the asymmetric Diels−Alder reaction.[7]

The synthesis of acyloxazolidinones,[8] requires a coupling of the lithium salt of the oxazolidinone with the acid chloride or mixed anhydride of the acidic substrate. As the preparation and isolation of acid chlorides or mixed anhydrides requires operations which are undesirable for large-scale preparations, direct *N*-acylation of chiral oxazolidin-2-one auxiliaries with acids has been proposed.[9,10] However, α,β-unsaturated acids, such as 3,3-dimethylacrylic acid, gave *N*-acryloyloxazolidin-2-ones in poor or modest yields (14−57%).

Recently,[11] we have shown that α-halo or α,α′-dihalo ketones, after cathodic reduction of the carbon−halogen bond or in the presence of electrogenerated bases (EGBs), react with amines and phenols to give the corresponding α,β-unsaturated amides and esters according to an electrochemically induced Favorskii rearrangement. We have expanded this method to the *N*-acryloylation of Evans' chiral auxiliaries, in particular, we have studied the reactivity of α,α′-dihalo ketones with oxazolidin-2-ones in aprotic solvents containing the EGB pyrrolidone anion (Scheme 1).[12−15]



Scheme 1

Results and Discussion

The EGB was obtained by electrochemical reduction of the probase (PB) 2-pyrrolidone (Scheme 2, reaction 1). Oxazolidin-2-one **1a** and α,α′-dihalo ketone **2k** were chosen as reference compounds.

In aprotic solvents and in the presence of Et_4N^+ as counter ion, the EGB 2-pyrrolidone anion shows a remarkable deprotonating power toward some N−H acids.[16] In fact, primary and secondary aliphatic and aromatic amines were deprotonated by this EGB to yield the corresponding tetraethylammonium amides and anilides. According to these reactions, the EGB 2-pyrrolidone anion should be able to deprotonate oxazolidinone **1a** as well as dichloro ketone **2k** to yield the anion **3a** and cyclopropanone **4k** intermediates, respectively (Scheme 2, reactions 2 and 3).

Both anion **3a** and EGB react with the chlorocyclopropanone **4k**, according to the Favorskii rearrangement, to yield *N*-enoyloxazolidinone **5a** and *N*-enoylpyrrolidone **6** (Scheme 2, reactions 3′ and 3′′). Since the reduction of the probase (reaction 1) is monoelectronic, the flow of 1.0 $F \cdot mol^{-1}$ of probase was used in the electrolyses. Moderate increases (5−10%) of the above value did not significantly improve the yield of **5a.** The yield of **5a** is affected by the pyrrolidone/dichloro ketone/oxazolidinone molar ratio, it becomes greater with an increase in probase and ketone with respect to oxazolidinone (Table 1, Entries 1−3, 5, 6). However, a strong excess of probase produces a drastic decrease in the yield of **5a** (Table 1, Entry 4). In all cases, **6** is isolated in low yields.

To simplify the synthesis of **5a** (i.e., without EGB and the formation of the by-product **6**), we have performed the deprotonation of **1a** by direct electrochemical reduction (Scheme 2, reaction 1′) instead of acid-base reaction with the EGB (reaction 2). A comparison of the molecular structure of 2-pyrrolidone with oxazolidin-2-one suggests that

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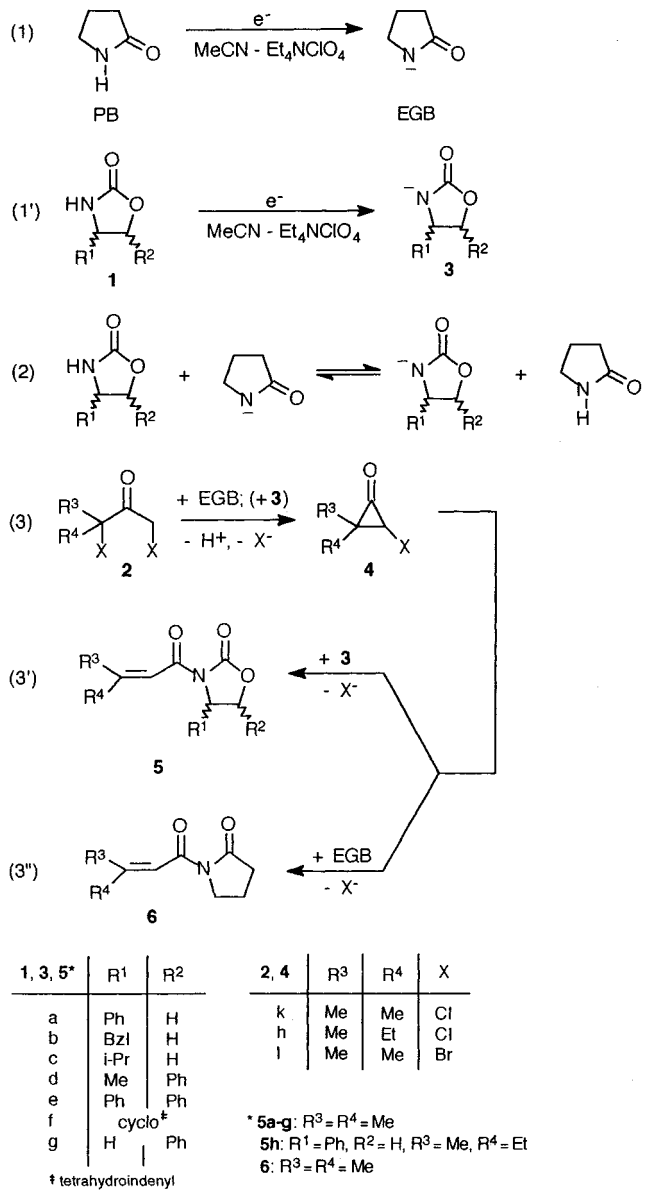


Scheme 2

Table 1. Reaction of 2-pyrrolidone EGB with oxazolidin-2-one **1a** and dichloro ketone **2k** under different conditions (MeCN/0.1 mol·dm^{-3} TEAP as solvent, n = 1.0[a])

Entry	ρ[b]	Yield of **5a** (%)[c]	Yield of **6** (%)[d]
1	1.0:1.0:1.0	33	5
2	1.5:1.0:1.0	42	4
3	2.0:1.0:1.0	64	4
4	3.0:1.0:1.0	17	14
5	2.0:1.5:1.0	75	3
6	2.0:2.0:1.0	81	traces

[a] Number of Faradays per mol of 2-pyrrolidone supplied to the electrodes. – [b] Mol ratio 2-pyrrolidone/α,α′-dichloro ketone/oxazolidinone. – [c] Yields of isolated **5a** based on starting oxazolidin-2-one **1a**. – [d] Yields of isolated **6** based on starting dichloro ketone **2k**.

direct electrochemical reduction of **1a** could produce the nitrogen anion **3a** (Scheme 2, reaction 1′). When **2k** or **2l** were treated with electrogenerated **3a**, compound **5a** was isolated from the reaction mixture, and the starting halo ketones were completely absent. However, with the direct electrochemical reduction the yields of **5a** (from **1a**) were reduced (Table 2, Entries 1 and 2). Under all these conditions, the oxazolidin-2-one anion **3a** reacts both as a nucleophile (yielding **5a**, reaction 3′) and as a base [yielding **4** and thus regenerating **1a**; reaction 3 (+**3**), theoretical maximum yield in **5a**: 50%]. The trouble of recovery of **1a** from the reaction mixture should also be taken into account.

Table 2. Reaction of the oxazolidin-2-one anion (**3a**) and of the pyrrolidone anion (EGB) with α,α′-dihalo ketones **2k** and **2l** (MeCN/0.1 mol·dm^{-3} TEAP as solvent, n[a] = 1.0, ρ[b] = 1.0)

Entry	Anion	Halo ketone	Products and yields[c] (%)
1	**3a**	**2k**	**5a** (38)[d]
2	**3a**	**2l**	**5a** (43)[e]
3	EGB	**2k**	**6** (24)
4	EGB	**2l**	**6** (32)

[a] Number of Faradays per mol of oxazolidin-2-one **1a** or of 2-pyrrolidone supplied to the electrodes. – [b] Molar ratio oxazolidinone/α,α′-dichloro ketone or 2-pyrrolidone/α,α′-dichloro ketone. – [c] Yields of isolated **5a** based on starting oxazolidin-2-ones **1a** (Entries 1, 2) or yields of isolated **6** based on starting 2-pyrrolidone (Entries 3, 4). – [d] Oxazolidin-2-one **1a** (61%) was also recovered. – [e] Oxazolidin-2-one **1a** (39%) was also recovered.

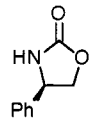
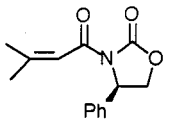
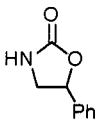
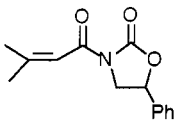
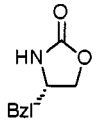
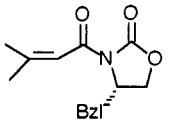
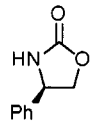
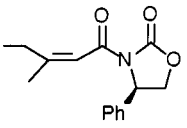
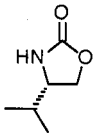
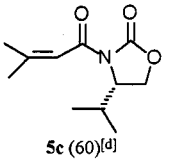
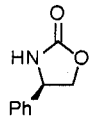
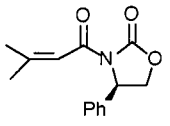
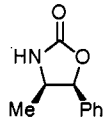
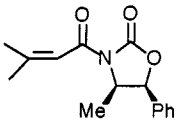
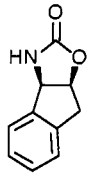
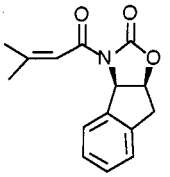
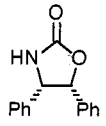
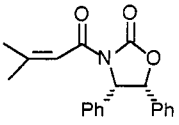
A comparison between the reactivity of the EGB and of **3a** with the α,α′-dihalo ketones could be helpful for a better insight into the overall synthetic process. Therefore, we have performed the reaction between the EGB and **2k** or **2l** (in the absence of oxazolidinone). From the reaction mixture, **6** has been isolated in moderate yields (yields of **6**: 24 and 32% respectively, based on the starting PB; Table 2, Entries 3 and 4).

According to the results reported in Table 2, both EGB and **3a** are strong enough bases to deprotonate α,α′-dihalo ketones **2k** and **2l**. Nevertheless, the higher yields of **5a** than **6**, show that **3a** is a stronger nucleophile than the EGB (Table 2, Entries 1 vs. 3 and 2 vs. 4). In addition, the results in Table 2 suggest a higher reactivity of bromocyclopropanone, than chlorocyclopropanone, with the nucleophilic agent (i.e., **3a** or EGB), according to the assumption that bromide is a better leaving group than chloride. In fact, both **5a** and **6** were obtained with higher yields from α,α′-dibromo ketone **2l** than from α,α′-dichloro ketone **2k**.

To ascertain whether this method for *N*-acylation of 2-oxazolidinones could be generalized, the reactivity of different dihalo ketones **2** as well as of different oxazolidinones **1**, under optimized reaction conditions (i.e. ρ = 2:2:1, MeCN, 0.1 mol dm^{-3} TEAP as solvent) has been studied (Table 3).

The reactivity is affected by the nature of the substituents on the carbon atoms in positions 4 and 5 on the oxazolidi-

Table 3. Reaction of 2-pyrrolidone EGB with oxazolidin-2-ones **1a**−**g** and dihaloketones **2k** (Entries 1−6, 9) or **2h** (Entry 7) or **2l** (Entry 8); MeCN/0.1 mol·dm^{-3} TEAP as solvent, n = 1.0[a], ρ =2:2:1;[b] yields[c] and stereochemistry of reaction products

Entry	Oxazolidinone	Product (yield, %)	Entry	Oxazolidinone	Product (yield, %)
1	 **1a**	 **5a** (81)[d]	6	 **1g**	 **5g** (81)
2	 **1b**	 **5b** (98)[d]	7	 **1a**	 **5h** (60)[e]
3	 **1c**	 **5c** (60)[d]	8	 **1a**	 **5a** (63)[d]
4	 **1d**	 **5d** (70)[d]	9	 **1f**	 **5f** (53)
5	 **1e**	 **5e** (53)[d]			

[a] Number of Faradays per mol of 2-pyrrolidone supplied to the electrodes. − [b] Molar ratio 2-pyrrolidone/α,α′-dichloro ketone/oxazolidinone. − [c] Yields of isolated **5a**−**f** based on starting oxazolidin-2-ones **1a**−**f**. − [d] **6** was also recovered at the end of the reaction (Entries 1, 4, 5: traces; Entry 2: 5%; Entry 3: 9%; Entry 8: 15%; yields based on starting dihalo ketones). − [e] A mixture of (*Z*) and (*E*) isomers in a 1.25 ratio was obtained.

none and in the α-position to the carbonyl group for the dihalo ketone as well as by the nature of the halogen atom. For the oxazolidin-2-one, the steric hindrance and electronic structure of the substituent groups at both 4 (Table 3, Entries 1−3 and 4−6) and 5 position (Table 3, Entries 1 and 5) seems to play a significant role. As for the α,α′-dihalo ketones, the replacement of chlorine with bromine increases the reactivity of the intermediate halocyclopropanone **4** (see above) and consequently decreases the selectivity [chloro ketone: **5a** (81%), **6** (traces); bromo ketone: **5a** (63%), **6** (15%); Table 3, Entries 1, 8, footnote[d]]. Thus, *N*-enoyloxazolidin-2-ones have been obtained in good to high yields. In addition, we have ascertained that the absolute configurations of all the chiral atoms of **5a**−**f** remained unchanged with respect to **1a**−**f** (Table 3, Entries 1−5 and 7−9).

Conclusion

In conclusion, a new method for the *N*-enoylation of Evans' chiral oxazolidin-2-ones auxiliaries has been established, namely the reaction of α,α′-dihalo ketones with oxazolidin-2-ones in aprotic solvent with the EGB 2-pyrrolidone anion. *N*-Enoyloxazolidin-2-ones have been obtained under mild reaction conditions in good to high yields.

Experimental Section

General Remarks: The electrochemical apparatus, the cells, and the reference electrode[17] as well as the method of purification of acetonitrile (MeCN) and tetraethylammonium perchlorate (TEAP) have already been described.[18] − 2-Pyrrolidone and all oxazolidin-2-ones were commercially available and used as received. Dihalo ketones were prepared as described in the literature.[19] − ^{1}H and ^{13}C NMR spectra were recorded using a Bruker AC 200 spectrometer with $CDCl_3$ as internal standard. − GC-MS measurements were carried out with an SE 54 capillary column using a Fisons 8000 gas chromatograph coupled with a Fisons MD 800 quadrupole mass-selective detector. − Optical rotations were measured with a Perkin−Elmer 241 polarimeter.

Reduction of 2-Pyrrolidone (PB): MeCN (30 mL, 0.1 mol·L^{-1}) in TEAP with 2-pyrrolidone (1.0 mmol) was electrolyzed under N_2 at

room temperature in a divided cell (Pt anode and cathode) under galvanostatic control (I = 25 mA·cm^{-2}). After the flow of 1.0 F·mol^{-1} of PB, the cathodic solution was added to a mixture of the dihalo ketone (0.5 or 1.0 mmol) and/or oxazolidin-2-one (0.5 mmol) and the resulting solution was stirred overnight at room temperature. After the usual workup, the electrolysis products were purified by flash chromatography using a mixture of *n*-hexane and ethyl acetate as eluent in a 9:1 to 7:3 ratio. The products and yields are reported in Tables 1, 2, and 3.

Reduction of Oxazolidin-2-one 1a: MeCN (30 mL, 0.1·mol L^{-1}) in TEAP with oxazolidin-2-one **1a** (1.0 mmol) was electrolyzed under N_2 at room temperature in a divided cell (Pt anode and cathode) under galvanostatic control (I = 25 mA·cm^{-2}). After the flow of 1.0 F mol^{-1} of substrate, the cathodic solution was added to the dihalo ketone (0.5 mmol) and the resulting solution was stirred overnight at room temperature. After the usual workup, the electrolysis products were purified by flash chromatography using a mixture of *n*-hexane and ethyl acetate as eluent in an 8:2 ratio. The products and yields are reported in Table 2.

(*S*)-(−)-4-Benzyl-*N*-(3-methylbut-2-enoyl)oxazolidin-2-one (5b),[20] **(*S*)-(−)-4-Isopropyl-*N*-(3-methylbut-2-enoyl)oxazolidin-2-one (5c),**[21] **(4*R*,5*S*)-(+)-4-Methyl-*N*-(3-methylbut-2-enoyl)-5-phenyloxazolidin-2-one (5d):**[22] These electrolysis products gave spectroscopic data according to the literature.

(*R*)-(−)-*N*-(3-Methylbut-2-enoyl)-4-phenyloxazolidin-2-one (5a): ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.36−7.27 (m, 5 H, ar), 6.94−6.93 (m, 1 H, *H*C=C), 5.46 (dd, J = 8.6, J = 4.3 Hz, 1 H,CH_2O), 4.65 (t, J = 8.6 Hz, 1 H, PhC*H*N), 4.20 (dd, J = 8.6, J = 4.3 Hz, 1 H, CH_2O), 2.07 (d, J = 1.0 Hz, 3 H, *Z* H_3C), 1.95 (d, J = 1.0 Hz, 3 H, *E* H_3C). − ^{13}C NMR ($CDCl_3$, 50.3 MHz): δ = 164.37, 159.63, 153.67, 139.43, 129.07, 128.43, 125.76, 115.70, 69.65, 57.58, 27.98, 21.28. − GC-MS *m*/*z*: 245 (8) [M$^+$], 162 (24), 83 (100). − $[\alpha]_D^{20}$= −123.1 (*c* = 0.52, $CHCl_3$).

(4*S*,5*R*)-(−)-*N*-(3-Methylbut-2-enoyl)-*cis*-4,5-diphenyloxazolidin-2-one (5e): ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.12−6.84 (m, 11 H, ar and *H*C=C), 5.88 (d, J = 7.5 Hz, 1 H, PhC*H*O), 5.69 (d, J = 7.5 Hz, 1 H, PhC*H*N), 2.11 (s, 3 H, *Z* H_3C), 2.01 (s, 3 H, *E* H_3C). − ^{13}C NMR ($CDCl_3$, 50.3 MHz): δ = 164.18, 160.05, 153.63, 134.84, 133.08, 128.26, 128.18, 127.93, 126.54, 126.13, 115.54, 80.01, 62.82, 28.06, 22.58. − GC-MS *m*/*z*: 321 (3) [M$^+$], 83 (100). − $[\alpha]_D^{20}$= −43.8 (*c* = 0.53, $CHCl_3$).

(3a*R*)-(−)-*N*-(3-Methylbut-2-enoyl)-3,3a,8,8a-tetrahydro-*cis*-2*H*-indeno[1,2-*d*]oxazol-2-one (5f): ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.67−7.64 (m, 1 H, ar), 7.31−7.23 (m, 3 H, ar) 6.91 (s, 1 H, *H*C=C), 5.97 (d, J = 6.9 Hz, 1 H, PhC*H*N), 5.25 (dt, J = 6.9, J = 3.6 Hz, 1 H, CH_2C*H*O), 3.36 (d, J = 3.6 Hz, 2 H, CH_2CHO), 2.22 (s, 3 H, *Z* H_3C), 1.96 (s, 3 H, *E* H_3C). − ^{13}C NMR ($CDCl_3$, 50.3 MHz): δ = 165.34, 159.11, 153.41, 139.48, 129.69, 128.06, 127.38, 125.12, 115.76, 77.73, 63.05, 38.02, 28.03, 21.41. − GC-MS *m*/*z*: 257 (1) [M$^+$], 83 (100). − $[\alpha]_D^{20}$= −189.3 (*c* = 0.38, $CHCl_3$).

***N*-(3-Methylbut-2-enoyl)-5-phenyloxazolidin-2-one (5g) (Racemic Mmixture):** ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.37−7.31 (m, 5 H, ar), 6.95 (s, 1 H, *H*C=C), 5.52 (t, J = 8.2 Hz, 1 H, PhC*H*O), 4.39 (dd, J = 11.2, J = 8.2 Hz, 1 H, CH_2N), 3.90 (dd, J = 11.2, J = 8.2 Hz, 1 H, CH_2N), 2.16 (s, 3 H, *Z* H_3C), 1.98 (s, 3 H, *E* H_3C). − ^{13}C NMR ($CDCl_3$, 50.3 MHz): δ = 164.99, 159.47, 152.86, 137.30, 129.19, 129.00, 125.69, 115.39, 74.55, 49.85, 28.03, 21.31. − GC-MS *m*/*z*: 245 (16) [M$^+$], 230 (16) [M$^+$ − CH_3], 83 (100).

(*R*)-(−)-*N*-(3-Methylpent-2-enoyl)-4-phenyloxazolidin-2-one (5h). − (*E*) Isomer: ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.40−7.24 (m, 5 H, ar), 6.95 (s, 1 H, COC*H*=C), 5.46 (dd, J = 8.7, J = 4.0 Hz, 1 H, PhC*H*), 4.64 (t, J = 8.7 Hz, 1 H, OCH_2), 4.24−4.18 (m, 1 H, OCH_2), 2.22 (q, J = 7.3 Hz, 2 H, CH_3CH_2), 2.07 (s, 3 H, CH_3C=), 1.08 (t, J = 7.3 Hz, 3 H, CH_3CH$_2$). − ^{13}C NMR ($CDCl_3$, 50.3 MHz): δ = 164.65, 163.95, 153.66, 139.44, 129.06, 128.41, 125.67, 114.14, 69.64, 57.55, 34.29, 19.88, 12.29. − GC-MS *m*/*z*: 260 [M$^+$ + 1] (3), 259 (27) [M$^+$], 164 (15), 162 (9), 104 (27) [PhCHCH$_2^+$], 97 (87), 96 (100). − **(*Z*) Isomer:** ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.40−7.24 (m, 5 H, ar), 6.89 (s, 1 H, COC*H*=C), 5.46 (dd, J = 8.7 Hz, J = 4.02 Hz, 1 H, PhC*H*), 4.64 (t, J = 8.7 Hz, 1 H, OCH_2), 4.24−4.18 (m, 1 H, OCH_2), 2.53−2.45 (m, 2 H, CH_3CH_2), 1.94 (s, 3 H, CH_3C=), 0.99 (t, J = 7.5 Hz, 3 H, CH_3CH$_2$). − ^{13}C NMR ($CDCl_3$, 50.3 MHz): δ = 165.02, 163.95, 153.66, 139.41, 129.06, 125.77, 125.67, 115.04, 69.64, 57.57, 27.46, 25.18, 11.86. − GC-MS *m*/*z*: 260 (3) [M$^+$ + 1], 259 (27) [M$^+$], 164 (15), 162 (9), 104 (27) [PhCHCH$_2^+$], 97 (87), 96 (100).

***N*-(3-Methylbut-2-enoyl)-2-pyrrolidone (6):** ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 6.91 (s, 1 H, *H*C=C), 3.81 (t, J = 7.1 Hz, 2 H, CH_2N), 2.58 (t, J = 8.1 Hz, 2 H, CH_2C=O), 2.14 (s, 3 H, *Z* H_3C), 2.03−1.95 (m, 2 H, CH_2CH_2CH$_2$), 1.95 (s, 3 H, *E* H_3C). − ^{13}C NMR ($CDCl_3$, 50.3 MHz): δ = 175.08, 166.31, 157.63, 117.61, 45.58, 34.03, 27.94, 21.34, 17.12. − GC-MS *m*/*z*: 167 (9) [M$^+$], 84 (14), 83 (100).

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