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ORIGINAL PAPER

Synthesis of stable ketenimines via a four-component reaction

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Abstract An efficient one-pot synthesis of ketenimine derivatives is described. It involves a four-component reaction between benzylamines, 2,2-dichloroethanoyl chloride, alkyl isocyanide, and dialkyl acetylenedicarboxylates under mild conditions at ambient temperature.

Keywords Amine · 2,2-Dichloroethanoyl chloride · Dialkyl acetylenedicarboxylate · Alkyl isocyanide · Multicomponent reaction

Introduction

Multicomponent reactions (MCRs) are at a premium for achievement of high diversity and brevity, because they enable three or more simple and flexible building blocks to be combined in practical, one-pot operations. MCRs have attracted much interest owing to their exceptional synthetic efficiency [1]. A multitude of MCRs exist today, of which isocyanide-based MCRs are the most documented. Isocyanide-based MCRs are most frequently exploited because the isocyanide is an extraordinary functional group.

Ketenimines are extensively involved in cascade reactions and have attracted much attention in recent years because of their easy formation, the relative reactivity, tolerance of different procedures, and the diversity of the products [2]. Ketenimines are important reactive intermediates that occur as transient compounds in many thermal and photochemical reactions [3–6]. They have also found widespread use as reactive starting materials for the formation of four, five, and six-membered heterocyclic ring systems [7–11].

The trapping of the 1:1 intermediate formed between dialkyl acetylenedicarboxylates and isocyanides with OH, NH, and CH acids has been widely studied [12–16]. Previously, we reported a similar reaction between alkyl isocyanides, acetylenic esters, and oximes to produce thermally stable ketenimines [15].

Here we report a simple one-pot preparation of stable ketenimines using alkyl isocyanides, dialkyl acetylenedicarboxylates, and, as N–H acids, *N*-benzyl-2,2-dichloroacetamides, which were obtained by reaction of benzylamine derivatives with 2,2-dichloroethanoyl chloride.

Results and discussion

Alkyl isocyanides **1**, dialkyl acetylenedicarboxylates **2**, and benzylamine derivatives **3** in the presence of 2,2-dichloroethanoyl chloride undergo a addition reaction in CH_2Cl_2 at ambient temperature to produce dialkyl 2-[benzyl (2,2-dichloroacetyl)amino]-3-(alkylcarbonimidoyl)succinate derivatives **4** in excellent yields (Scheme 1). The structures of compounds **4a**–**4h** were deduced from their mass, IR, and high-field ^{1}H and ^{13}C NMR spectra. The mass spectrum of **4a** contained the molecular ion peak at $m/z = 443$, which is in agreement with the proposed structure. The IR spectrum of this compound contained absorption bands at 2,055, 1,744, and 1,684 cm^{-1} arising from the stretching frequencies of ketenimine, CO_2Me, and NCO groups, respectively. The ^{1}H NMR spectrum of **4a** contained five singlets for *t*-Bu, two methoxy, $CHCl_2$, and CH groups at $\delta = 1.45$, 3.66, 3.72, 5.09, and 6.14 ppm, respectively, and an AB system for the diastereotopic benzylic hydrogen atoms at $\delta = 4.84$ ppm ($^2J_{HH} = 16.9$ Hz). Aromatic

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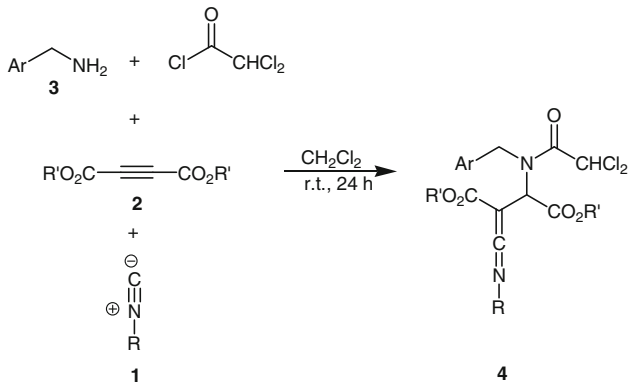


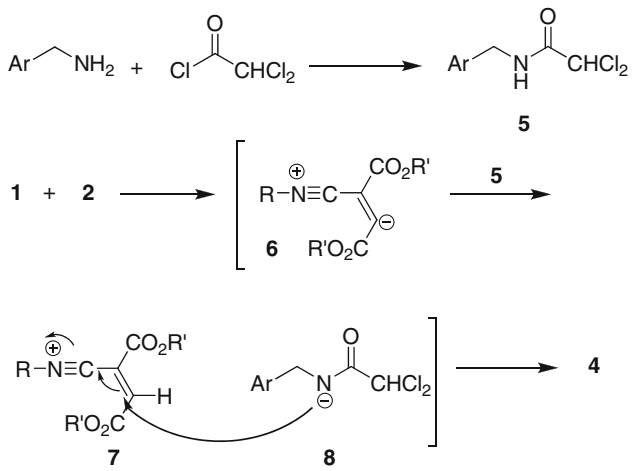
4	R	R'	Ar
a	*t*-Bu	Me	Ph
b	*t*-Bu	Me	4-MeC_6H_4
c	*t*-Bu	Me	4-$MeOC_6H_4$
d	*t*-Bu	Me	4-ClC_6H_4
e	*t*-Bu	Et	Ph
f	*t*-Bu	Et	4-MeC_6H_4
g	Cyhex	Me	Ph
h	Cyhex	Et	4-MeC_6H_4

Scheme 1

hydrogens gave rise to characteristic signals in the aromatic region of the spectrum. The ^{1}H-decoupled ^{13}C NMR spectrum of **4a** contained 16 distinct resonances, in agreement with the suggested structure. In the aliphatic region there were six signals related to two methoxy, $CHCl_2$, *t*-Bu, CH, and CH_2 groups. The characteristic carbon (RN=C=C) of the compound **4a** resonates at 164.07 ppm. The other important region of the spectrum is related to carbonyl groups which produce two C=O signals at 168.26 and 170.63 ppm. The ^{1}H and ^{13}C NMR spectra of compounds **4b**–**4h** are similar to those of **4a** except for the aromatic and OR which give characteristic signals with appropriate chemical shifts.

The ketenimine structure was established by IR (2,055 cm^{-1} absorbance) and NMR spectroscopy. In the off-resonance ^{13}C NMR spectrum of **4a**, the CH signal ($\delta = 64.40$ ppm) is a doublet, and no exchange is observed in the ^{1}H NMR spectrum of the same compound in deuterium oxide solution.

Although we have not established the mechanism of the reaction between an isocyanide and an acetylenic ester in the presence of an *N*-benzyl-2,2-dichloroacetamide **5** experimentally, a possible explanation is proposed in Scheme 2. On the basis of the well-established chemistry of isocyanides [17–21], it is reasonable to assume that the functionalized ketenimine **4** results from initial addition of the isocyanide to the acetylenic ester and subsequent protonation of the 1:1 adduct **6** by *N*-benzyl-2,2-dichloroacetamide **5** as N–H acid; this is followed by attack of the anion **8** on the positively charged ion **7** to give the



Scheme 2

corresponding dialkyl 2-[benzyl(2,2-dichloroacetyl)amino]-3-(alkylcarbonimidoyl)succinate derivatives **4** (Scheme 2).

In summary, this simple one-pot reaction between isocyanides, dialkyl acetylenedicarboxylates, and benzylamine derivatives in the presence of 2,2-dichloroethanoyl chloride provides access to stable ketenimine derivatives of potential synthetic interest. The method presented has the advantage of being performed under neutral conditions and requires no activation or modification of the reagents.

Experimental

Dimethyl, diethyl, and the isocyanides were obtained from Merck (Germany) and Fluka (Switzerland) and were used without further purification. Mass spectra were recorded on a Finnigan MAT 8430 mass spectrometer operating at an electron energy of 70 eV. ^{1}H NMR and ^{13}C NMR spectra were measured in $CDCl_3$ solution, at 500.1 and 125.7 MHz, respectively, using a Bruker DRX-500 Avance spectrometer. IR spectra were recorded on a Shimadzu IR-460 spectrometer. Chromatography columns were prepared from Merck silica gel (230–240 mesh).

Typical procedure: dimethyl 2-[benzyl(2,2-dichloroacetyl)amino]-3-(tert-butylcarbonimidoyl)-succinate (**4a**, $C_{20}H_{24}Cl_2N_2O_5$)

To a magnetically stirred solution of 0.22 g benzylamine (2 mmol) and 0.15 g 2,2-dichloroethanoyl chloride (1 mmol) dissolved in 2 cm^3 dry CH_2Cl_2, a solution of 0.14 g dimethyl acetylenedicarboxylate (1 mmol) in 2 cm^3 dry CH_2Cl_2 and a solution of 0.08 g *t*-BuNC (1 mmol) in 2 cm^3 dry CH_2Cl_2 were added simultaneously dropwise at room temperature over 10 min. The mixture was then stirred for 24 h, and the progress of the reaction was followed by thin-layer chromatography. After completion,

the reaction mixture was filtered and the precipitate of benzylammonium chloride was separated. Solvent was removed from the filtrate under reduced pressure, and the residue was separated by column chromatography (silica gel, hexane–EtOAc, 5:1) to give the product **4a** as yellow oil, yield 0.38 g (86%). IR (KBr): $\bar{\nu}$ = 2,055 (N=C=C), 1,744 (CO_2Me), 1,684 (NCO), 1,531, 1,493 (Ph), 1,256, 1,236 (C–O of esters), 1,175 (C–N) cm^{-1}; ^1H NMR (500.1 MHz, $CDCl_3$): δ = 1.45 (9H, s, $C(CH_3)_3$), 3.66 (3H, s, OCH_3), 3.72 (3H, s, OCH_3), 4.84 (2H, AB system, $^2J_{HH}$ = 16.9 Hz, CH_2), 5.09 (1H, s, $CHCl_2$), 6.14 (1H, s, CH), 7.32–7.41 (5H, m, 5CH of Ph) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 30.07 ($C(CH_3)_3$), 51.72 (CH_2), 52.08 (OCH_3), 52.69 (OCH_3), 58.75 ($CHCl_2$), 60.02 ($C(CH_3)_3$), 62.43 (C=C=N), 64.40 (CH), 127.18 ($2CH_{meta}$ of Ph), 128.10 (CH_{para} of Ph), 128.86 ($2CH_{ortho}$ of Ph), 135.32 (C_{ipso}), 162.51 (C=O), 164.07 (C=C=N), 168.26 (CO_2Me), 170.63 (CO_2Me) ppm; MS (EI, 70 eV): *m/z* (%) = 443 (M^+, 26), 387 (16), 327 (10), 260 (10), 226 (100), 170 (6), 91 (94), 57 (65), 41 (28).

Dimethyl 2-(tert-butylcarbonimidoyl)-3-[(2,2-dichloroacetyl)(4-methylbenzyl)amino]succinate (**4b**, $C_{21}H_{26}Cl_2N_2O_5$)

Yellow wax, yield 0.39 g (85%); IR (KBr): $\bar{\nu}$ = 2,055 (N=C=C), 1,746 (CO_2Me), 1,671 (NCO), 1,553, 1,513 (Ph), 1,256, 1,236 (C–O of esters), 1,173 (C–N) cm^{-1}; ^1H NMR (500.1 MHz, $CDCl_3$): δ = 1.45 (9H, s, $C(CH_3)_3$), 2.36 (3H, s, Me), 3.66 (3H, s, OCH_3), 3.71 (3H, s, OCH_3), 4.79 (2H, AB system, $^2J_{HH}$ = 16.9 Hz, CH_2), 5.07 (1H, s, $CHCl_2$), 6.14 (1H, s, CH), 7.18 (2H, d, $^3J_{HH}$ = 6.5 Hz, 2CH of Ar), 7.28 (2H, d, $^3J_{HH}$ = 7.95 Hz, 2CH of Ar) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 21.11 (Me), 30.06 ($C(CH_3)_3$), 44.11 (CH_2), 51.92 (OCH_3), 52.66 (OCH_3), 58.68 (CH), 60.11 ($C(CH_3)_3$), 62.33 (C=C=N), 64.77 ($CHCl_2$), 127.18 (2CH of Ar), 127.77 (2CH of Ar), 132.21 (C_{ipso}–Me), 133.70 (C_{ipso}–CH_2), 162.65 (C=O), 164.02 (C=C=N), 168.28 (CO_2Me), 170.63 (CO_2Me) ppm; MS (EI, 70 eV): *m/z* (%) = 457 (M^+, 11), 401 (3), 232 (17), 198 (35), 196 (99), 160 (67), 117 (22), 105 (100), 91 (16), 77 (16), 65 (8), 51 (6).

Dimethyl 2-(tert-butylcarbonimidoyl)-3-[(2,2-dichloroacetyl)(4-methoxybenzyl)amino]succinate (**4c**, $C_{21}H_{26}Cl_2N_2O_6$)

White wax, yield 0.41 g (87%); IR (KBr): $\bar{\nu}$ = 2,068 (N=C=C), 1,746 (CO_2Me), 1,669 (NCO), 1,514, 1,460 (Ph), 1,253, 1,228 (C–O of esters), 1,177 (C–N) cm^{-1}; ^1H NMR (500.1 MHz, $CDCl_3$) : δ = 1.45 (9H, s, $C(CH_3)_3$), 3.67 (3H, s, OCH_3), 3.70 (3H, s, OCH_3), 3.81 (3H, s, OCH_3), 4.77 (2H, AB system, $^2J_{HH}$ = 16.3 Hz, CH_2), 5.02 (1H, s, $CHCl_2$), 6.17 (1H, s, CH), 6.91 (2H, d, $^3J_{HH}$ = 8.55 Hz, 2CH of Ar), 7.34 (2H, d, $^3J_{HH}$ = 8.6 Hz, 2CH of Ar) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 30.05 ($C(CH_3)_3$), 43.85 (CH_2), 51.71 (OCH_3), 52.66 (OCH_3), 55.31 (4-$CH_3OC_6H_4$), 58.45 (CH), 60.1 ($C(CH_3)_3$), 62.31 (C=C=N), 64.82 ($CHCl_2$), 114.94 (2CH of Ar), 128.78 (2CH of Ar), 129.19 (C_{ipso}–CH_2), 159.52 (C_{ipso}–OCH_3), 162.61 (C=O), 163.85 (C=C=N), 168.25 (CO_2Me), 170.74 (CO_2Me) ppm; MS (EI, 70 eV): *m/z* (%) = 473 (M^+, 1), 351 (12), 295 (23), 246 (9), 226 (8), 170 (7), 122 (46), 121 (100), 91 (8), 77 (9), 57 (75), 41 (23).

Dimethyl 2-(tert-butylcarbonimidoyl)-3-[(4-chlorobenzyl)-(2,2-dichloroacetyl)amino]succinate (**4d**, $C_{20}H_{23}Cl_3N_2O_5$)

White wax, yield 0.43 g (90%); IR (KBr): $\bar{\nu}$ = 2,071 (N=C=C), 1,745 (CO_2Me), 1,684 (NCO), 1,540, 1,493 (Ph), 1,266 (C–O of esters), 1,178 (C–N) cm^{-1}; ^1H NMR (500.1 MHz, $CDCl_3$): δ = 1.45 (9H, s, $C(CH_3)_3$), 3.67 (3H, s, OCH_3), 3.72 (3H, s, OCH_3), 4.83 (2H, AB system, $^2J_{HH}$ = 16.9 Hz, CH_2), 4.99 (1H, s, $CHCl_2$), 6.12 (1H, s, CH), 7.24 (2H, d, $^3J_{HH}$ = 8.4 Hz, 2CH of Ar), 7.34 (2H, d, $^3J_{HH}$ = 8.4 Hz, 2CH of Ar) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 30.06 ($C(CH_3)_3$), 43.56 (CH_2), 51.50 (OCH_3), 52.76 (OCH_3), 58.58 (CH), 59.80 ($C(CH_3)_3$), 62.39 (C=C=N), 64.77 ($CHCl_2$), 129.00 (2CH of Ar), 129.07 (2CH of Ar), 133.95 (C_{ipso}–Cl), 134.07 (C_{ipso}–CH_2), 162.00 (C=O), 163.88 (C=C=N), 168.13 (CO_2Me), 170.34 (CO_2Me) ppm; MS (EI, 70 eV): *m/z* (%) = 478 (M^+, 1), 420 (5), 361 (6), 295 (20), 263 (12), 125 (100), 112 (13), 91 (43), 70 (20), 57 (75), 41 (29).

Diethyl 2-[benzyl(2,2-dichloroacetyl)amino]-3-(tert-butylcarbonimidoyl)succinate (**4e**, $C_{22}H_{28}Cl_2N_2O_5$)

Yellow oil, yield 0.40 g (85%); IR (KBr): $\bar{\nu}$ = 2,055 (N=C=C), 1,744 (CO_2Et), 1,684 (NCO), 1,532, 1,493 (Ph), 1,248, 1,227 (C–O of esters), 1,175 (C–N) cm^{-1}; ^1H NMR (500.1 MHz, $CDCl_3$): δ = 1.20 (3H, t, $^3J_{HH}$ = 7.1 Hz, OCH_2CH_3), 1.25 (3H, t, $^3J_{HH}$ = 7.1 Hz, OCH_2CH_3), 1.43 (9H, s, $C(CH_3)_3$), 4.10 (2H, q, $^3J_{HH}$ = 7.1 Hz, OCH_2CH_3), 4.15 (2H, q, $^3J_{HH}$ = 7.1 Hz, OCH_2CH_3), 4.84 (2H, AB system, $^2J_{HH}$ = 16.9 Hz, CH_2), 5.03 (1H, s, $CHCl_2$), 6.14 (1H, s, CH), 7.30–7.41 (5H, m, 5CH of Ph) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 14.04 (OCH_2CH_3), 14.12 (OCH_2CH_3), 30.06 ($C(CH_3)_3$), 52.16 (CH_2), 58.85 ($CHCl_2$), 60.35 (OCH_2CH_3), 60.54 ($C(CH_3)_3$), 61.90 (OCH_2CH_3), 62.23 (C = C=N), 64.67 (CH), 127.27 (2 CH_{meta} of Ph), 128.04 (CH_{para} of Ph), 128.79 ($2CH_{ortho}$ of Ph), 135.41 (C_{ipso}), 163.41 (C=O), 163.88 (C=C=N), 167.63 (CO_2Et), 170.29 (CO_2Et) ppm; MS (EI, 70 eV): *m/z* (%) = 471 (M^+, 12), 415 (13), 341 (12), 288 (8), 254 (67), 198 (15), 91 (100), 57 (48), 41 (16).

Diethyl 2-(tert-butylcarbonimidoyl)-3-[(2,2-dichloroacetyl)-(4-methylbenzyl)amino]succinate (**4f**, $C_{23}H_{30}Cl_2N_2O_5$)

Yellow wax, yield 0.39 g (80%); IR (KBr): $\bar{\nu}$ = 2,058 (N=C=C), 1,743 (CO_2Et), 1,670 (NCO), 1,555, 1,512 (Ph),

1,249, 1,206 (C–O of esters), 1,176 (C–N) cm^{-1}; ^1H NMR (500.1 MHz, $CDCl_3$): $\delta = 1.22$ (3H, t, $^3J_{HH} = 7.1$ Hz, OCH_2CH_3), 1.26 (3H, t, $^3J_{HH} = 7.1$ Hz, OCH_2CH_3), 1.45 (9H, s, $C(CH_3)_3$), 2.36 (3H, s, Me), 4.12 (2H, q, $^3J_{HH} = 7.1$ Hz, OCH_2CH_3), 4.17 (2H, q, $^3J_{HH} = 7.2$ Hz, OCH_2CH_3), 4.80 (2H, AB system, $^2J_{HH} = 16.9$ Hz, CH_2), 5.02 (1H, s, $CHCl_2$), 6.15 (1H, s, CH), 7.18 (2H, d, $^3J_{HH} = 7.9$ Hz, 2CH of Ar), 7.30 (2H, d, $^3J_{HH} = 7.9$ Hz, 2CH of Ar) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): $\delta = 14.04$ (OCH_2CH_3), 14.34 (OCH_2CH_3), 21.11 (4-MeC_6H_4), 30.08 ($C(CH_3)_3$), 44.12 (CH_2), 52.04 ($C(CH_3)_3$), 58.83 (C=C=N), 60.35 (OCH_2CH_3), 61.81 (CH), 64.73 (OCH_2CH_3), 66.45 ($CHCl_2$), 127.77 (2CH of Ar), 129.61 (2CH of Ar), 133.68 (C_{ipso}–CH_3), 137.85 (C_{ipso}–CH_2), 163.85 (C=O), 163.96 (C=C=N), 167.67 (CO_2Et), 170.35 (CO_2Et) ppm; MS (EI, 70 eV): *m/z* (%) = 485 (M^+, 8), 429 (15), 355 (17), 323 (22), 277 (10), 254 (55), 198 (15), 170 (6), 106 (36), 105 (100), 79 (9), 57 (72), 41 (25).

Dimethyl 2-[benzyl(2,2-dichloroacetyl)amino]-3-(cyclohexylcarbonimidoyl)succinate
(**4g**, $C_{22}H_{26}Cl_2N_2O_5$)
Yellow oil, yield 0.38 g (81%); IR (KBr): $\overline{\nu} = 2{,}060$ (N=C=C), 1,741 (CO_2Me), 1,708 (CO_2Me), 1,674 (NCO), 1,525, 1,492 (Ph), 1,246, 1,206 (C–O of esters), 1,163 (C–N) cm^{-1}; ^1H NMR (500.1 MHz, $CDCl_3$): $\delta = 1.26$–1.60 (10H, m, $5CH_2$ of Cy), 3.66 (3H, s, OCH_3), 3.72 (3H, s, OCH_3), 3.60–3.64 (1H, m, CHN), 4.84 (2H, AB system, $^2J_{HH} = 18.0$ Hz, CH_2), 5.09 (1H, s, $CHCl_2$), 6.14 (1H, s, CH), 7.30–7.40 (5H, m, 5CH of Ph) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$) : $\delta = 23.95$, 25.22, 32.99 (Cy), 44.31 (CH_2), 51.68 (C=C=N), 52.13 (OCH_3), 52.76 (OCH_3), 60.05 (CH), 64.71 (CHN), 66.42 ($CHCl_2$), 127.2 ($2CH_{meta}$ of Ph), 128.05 (CH_{para} of Ph), 128.97 ($2CH_{ortho}$ of Ph), 136.76 (C_{ipso}), 162.30 (C=O), 164.15 (C=C=N), 168.30 (CO_2Me), 170.62 (CO_2Me) ppm; MS (EI, 70 eV): *m/z* (%) = 469 (M^+, 2), 427 (16), 403 (27), 375 (23), 343 (19), 295 (17), 271 (54), 239 (27), 212 (88), 171 (20), 113 (21), 92 (43), 91 (100), 65 (28), 55 (77), 41 (55).

Diethyl 2-(cyclohexylcarbonimidoyl)-3-[(2,2-dichloroacetyl)-(4-methylbenzyl)amino]succinate (**4h**, $C_{25}H_{32}Cl_2N_2O_5$)
Yellow oil, yield 0.43 g (84%); IR (KBr): $\overline{\nu} = 2{,}075$ (N=C=C), 1,739 (CO_2Et), 1,680 (NCO), 1,520, 1,449 (Ph), 1,264 (C–O of esters), 1,105 (C–N) cm^{-1}; ^1H NMR (500.1 MHz, $CDCl_3$): $\delta = 1.36$–1.96 (10H, m, $5CH_2$ of Cy), 1.22 (3H, t, $^3J_{HH} = 7.1$ Hz, OCH_2CH_3), 1.25 (3H, t, $^3J_{HH} = 7.1$ Hz, OCH_2CH_3), 2.35 (3H, s, Me), 3.84–3.88 (1H, m, CHN), 4.13 (2H, q, $^3J_{HH} = 7.1$ Hz, OCH_2CH_3), 4.16 (2H, q, $^3J_{HH} = 7.2$ Hz, OCH_2CH_3), 4.80 (2H, AB system, $^2J_{HH} = 16.9$ Hz, CH_2), 5.04 (1H, s, $CHCl_2$), 6.15 (1H, s, CH), 7.18 (2H, d, $^3J_{HH} = 7.9$ Hz, 2CH of Ar), 7.30 (2H, d, $^3J_{HH} = 7.9$ Hz, 2CH of Ar) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): $\delta = 13.85$ (OCH_2CH_3), 14.4 (OCH_2CH_3), 21.10 (Me), 23.79, 25.30, 32.9, 32.96 (Cy), 52.08 (CH_2), 59.07 (CH), 59.25 (C=C=N), 60.35 (OCH_2CH_3), 61.90 (CHN), 61.84 (OCH_2CH_3), 64.70 ($CHCl_2$), 127.96 (2CH of Ar), 129.48 (2CH of Ar), 132.33 (C_{ipso}–Me), 137.8 (C_{ipso}–CH_2), 163.09 (C=O), 163.91 (C=C=N), 167.68 (CO_2Et), 170.32 (CO_2Et) ppm; MS (EI, 70 eV): *m/z* (%) = 511 (M^+, 12), 427 (12), 405 (29), 355 (17), 323 (30), 299 (16), 280 (84), 252 (10), 226 (32), 198 (29), 170 (12), 105 (100), 83 (26), 55 (58), 41 (26).

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