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Reaction Between Isocyanides and Dialkyl Acetylenedicarboxylates in the Presence of Hydantoins – A One-pot Synthesis of Stable Ketenimines

Mehdi Adib[1,*], **Mohammad Hosein Sayahi**[1], **Behrooz Behnam**[1], and **Esmaeil Sheibani**[2]

[1] Department of Chemistry, Faculty of Science, University of Tehran, P.O. Box 14155-6455, Tehran, Iran
[2] Department of Chemistry, Tarbiat Modarres University, Tehran, Iran

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Summary. The reactive 1:1 intermediate produced in the reaction between isocyanides and dialkyl acetylenedicarboxylates was trapped by hydantoins to yield highly functionalized stable ketenimines in fairly good yields.

Keywords. Acetylenic esters; Isocyanides; Ketenimines; Hydantoins; Three-component reactions.

Introduction

5,5-Diphenylhydantoin (phenytoin) and its derivatives are of interest because they exhibit useful pharmacological properties in wound healing [1], in the treatment of various types of convulsions and seizures [2], in actions on the brain and nervous system in the treatment of epilepsy [3], to control irregular heartbeat [4], and also to treat migraine headaches and facial nerve pain [5], among other applications. A considerable number of papers are published concerning new pharmacological effects of phenytoin.

Multi-component reactions (MCRs), by virtue of their convergence, productivity, facile execution, and generally high yields of products, have attracted much attention from the point of combinatorial chemistry. Of pivotal importance in this area are the isocyanide-based MCRs such as the versatile *Ugi* and *Passerini* reactions [6–8]. In recent years, applications of multifunctional heteroallenes have also been widely investigated [9, 10]. Compounds containing a heterocumulene entity

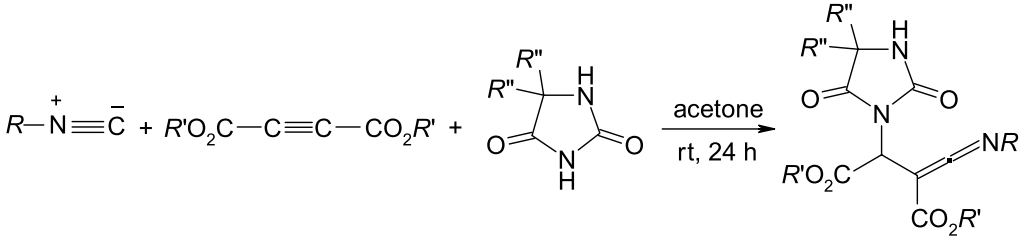
* Corresponding author. E-mail: madib@khayam.ut.ac.ir

are expected to have synthesis potential as a result of their ability to take part in dimerization, cycloaddition, and polymerization [11]. Ketenimines play a role as discrete but transient intermediates in many interconversions, especially in elimination-addition processes and in the formation of heterocyclic ring systems [12–14]. These compounds have attracted interest as dehydrating agents for peptide synthesis, as complexing agents for transition-metal ions, and co-reagents for *DMSO* oxidations [15]. In spite of extensive developments in the chemistry of modified ketenes and isocyanates [16], little attention has been paid to the synthesis of ketenimines [17]. In general, unsubstituted ketenimines and those with small unbranched alkyl substituents are elusive, but their spectroscopic properties have been intensively investigated [18]. The reactions of isocyanides and acetylenic esters in the presence of OH- and NH-acids have been reported [19]. In this paper, we wish to report a simple one-pot synthesis of highly functionalized stable ketenimines derived from 5,5-disubstituted hydantoins.

Results and Discussion

The reaction of isocyanides **1** with electron-deficient acetylenic esters **2** in the presence of hydantoins **3** proceeded at room temperature in acetone, and was completed within 24 h (Scheme 1). The structures of compounds **4a**–**4i** were deduced from their elemental analyses and their IR, ^{1}H, and ^{13}C NMR spectra as given in the experimental part.

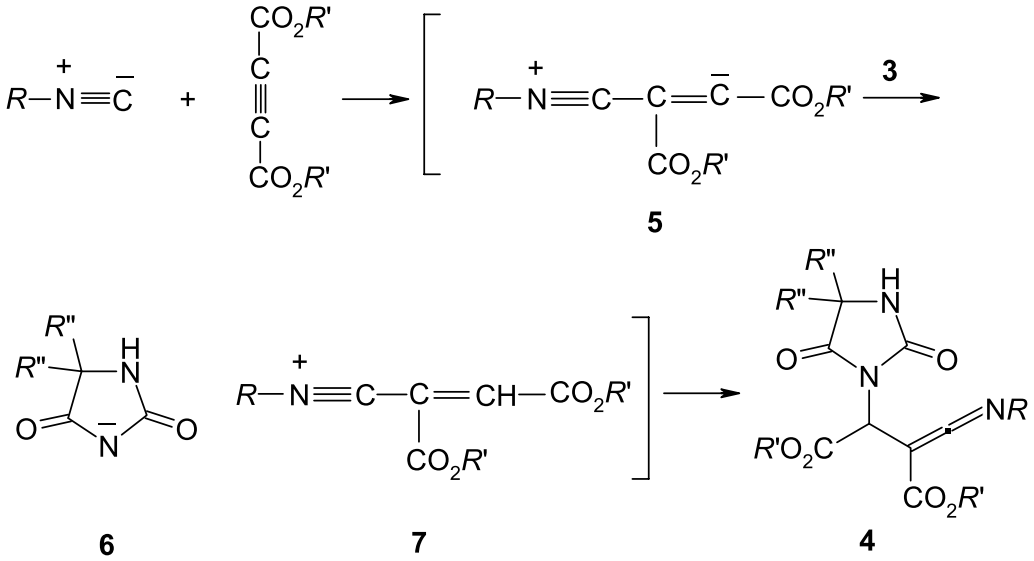
Although we have not established the mechanism of the reaction between the isocyanides and the acetylenic esters in the presence of the hydantoins in an experi-



4

	R		R′		R″		R	R′	R″
1a	*tBu*	**2a**	*Me*	**3a**	*Ph*	**4a**	*tBu*	*Me*	*Ph*
1b	Cyclohexyl	**2b**	*Et*	**3b**	*Me*	**4b**	*tBu*	*Et*	*Ph*
		2c	*tBu*			**4c**	Cyclohexyl	*Me*	*Ph*
						4d	Cyclohexyl	*Et*	*Ph*
						4e	Cyclohexyl	*tBu*	*Ph*
						4f	*tBu*	*Me*	*Me*
						4g	*tBu*	*Et*	*Me*
						4h	Cyclohexyl	*Me*	*Me*
						4i	Cyclohexyl	*Et*	*Me*

Scheme 1



Scheme 2

mental manner, a possible explanation is proposed in Scheme 2. On the basis of the well-established chemistry of isocyanides [20] it is reasonable to assume that **4** apparantly results from an initial addition of the isocyanide to the acetylenic ester and the subsequent protonation of the 1:1 adduct **5** by the hydantoin. This is followed by attack of the anion of the NH-acid **6** on the positively charged ion **7** to form **4**.

In conclusion, the three-component reaction between isocyanides and electron deficient acetylenic esters in the presence of 5,5-disubstituted hydantoins provides a simple one-pot entry into the synthesis of polyfunctionalized ketenimines, which are of potential synthesis and pharmaceutical interest. The present method carries the advantage that not only the reaction is performed under neutral conditions, but also the educts can just be mixed without any activation or modification.

Experimental

Dialkyl acetylenedicarboxylates, alkyl isocyanides, and 5,5-disubstituted hydantoins were obtained from Merck (Germany) and Fluka (Switzerland) and were used without further purification. Melting points were measured on an Electrothermal 9100 apparatus. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer. Mass spectra were recorded on a FINNIGAN-MATT 8430 mass spectrometer operating at an ionization potential of 20 eV. ^{1}H and ^{13}C NMR spectra were measured ($CDCl_3$ solution) with a Bruker DRX-500 AVANCE spectrometer at 500.1 and 125.8 MHz. Chemical shifts are given in ppm (δ) relative to internal *TMS*. IR spectra were recorded on a Shimadzu IR-460 spectrometer. Chromatography columns were prepared from Merck silica gel 60 mesh.

*General Procedure (examplified by **4a**)*

To a magnetically stirred solution of 0.252 g 5,5-diphenylhydantoin (1 mmol) and 0.142 g dimethyl acetylenedicarboxylate (1 mmol) in 6 cm^3 acetone was added dropwise a solution of 0.083 g *tert*-butyl isocyanide (1 mmol) in 2 cm^3 acetone at −5°C for 10 min. The reaction mixture was then allowed to warm up to room temperature and stirred for 24 h. The solvent was removed and 3 cm^3 CH_2Cl_2 were added. The unreacted 5,5-diphenylhydantoin precipitated and was filtered off. The filtrate was evapo-

rated and the residue was crystallized from 3:1 *n*-hexane:ethyl acetate. Compounds **4e**–**4i** were purified by column chromatography using *n*-hexane:ethyl acetate (1:2) as eluent.

Dimethyl 2-[(tert-butylimino)methylene]-3-(4,4-diphenyl-2,5-dioxoimidazolidin-1-yl) butanedioate (**4a**, $C_{26}H_{27}N_3O_6$)
Colorless crystals, yield 0.42 g (89%), mp 165–167°C; IR (KBr): $\bar{\nu} = 3170$ (NH), 3100, 2985, and 2856 (CH), 2073 (C=C=N), 1780 (shoulder, C=O), 1751, 1721 (broad, C=O), 1665 (shoulder, C=O), 1436, 1265, 1010, 754, 700 cm^{-1}; ^{1}H NMR: $\delta = 1.28$ (s, $C(CH_3)_3$), 3.62 and 3.68 (2s, 2 OCH_3), 5.80 (s, $NCHCO_2CH_3$), 7.16 (fairly br s, NH), 7.20–7.50 (m, 2 C_6H_5) ppm; ^{13}C NMR: $\delta = 29.56$ ($C(CH_3)_3$), 49.71 ($NCHCO_2CH_3$), 51.44 and 52.74 (2 OCH_3), 59.86 (C=C=N), 62.13 ($C(CH_3)_3$), 70.03 (Ph–C–Ph), 127.28 and 127.33 (2 CH_{meta}), 128.55 and 128.56 (2 CH_{para}), 128.74 and 128.80 (2 CH_{ortho}), 138.94 and 139.37 (2 C_{ipso}), 155.93 (C=O, urea), 163.51 (C=C=N), 168.00 (C=O, amide), 169.87 and 172.67 (2 C=O, ester) ppm; MS: m/z (%) = 477 (M^+, 14), 223 (34), 180 (100), 138 (43), 104 (33), 86 (18).

Diethyl 2-[(tert-butylimino)methylene]-3-(4,4-diphenyl-2,5-dioxoimidazolidin-1-yl) butanedioate (**4b**, $C_{28}H_{31}N_3O_6$)
Colorless crystals, yield 0.43 g (85%), mp 144–147°C; IR (KBr): $\bar{\nu} = 3207$ (NH), 3099, and 2979 (CH), 2069 (C=C=N), 1775 (shoulder, C=O), 1747, 1722 (broad, C=O), 1685 (shoulder, C=O), 1421, 1259, 1026, 757, 698 cm^{-1}; ^{1}H NMR: $\delta = 1.14$ and 1.16 (2t, $J = 7.2$ Hz, 2 OCH_2CH_3), 1.32 (s, $C(CH_3)_3$), 4.10 and 4.15 (2q, $J = 7.2$ Hz, 2 OCH_2CH_3), 5.80 (s, $NCHCO_2C_2H_5$), 7.10–7.50 (m, NH and 2 C_6H_5) ppm; ^{13}C NMR: $\delta = 14.64$ and 14.95 (2 OCH_2CH_3), 30.67 ($C(CH_3)_3$), 50.74 ($NCHCO_2C_2H_5$), 61.05 (OCH_2CH_3), 61.23 (C=C=N), 62.96 (OCH_2CH_3), 62.98 ($C(CH_3)_3$), 70.58 (Ph–C–Ph), 127.55 and 127.91 (2 CH_{meta}), 128.92 and 129.11 (2 CH_{para}), 129.30 and 129.38 (2 CH_{ortho}), 139.69 and 139.83 (2 C_{ipso}), 156.28 (C=O, urea), 163.05 (C=C=N), 167.78 (C=O, amide), 169.81 and 172.92 (2 C=O, ester) ppm; MS: m/z (%) = 506 (M^+, 4), 451 (56), 420 (26), 376 (11), 330 (10), 302 (13), 223 (26), 180 (100), 104 (37).

Dimethyl 2-[(cyclohexylimino)methylene]-3-(4,4-diphenyl-2,5-dioxoimidazolidin-1-yl) butanedioate (**4c**, $C_{28}H_{29}N_3O_6$)
Colorless crystals, yield 0.42 g (84%), mp 169–171°C; IR (KBr): $\bar{\nu} = 3178$ (NH), 3097, 2980, and 2856 (CH), 2073 (C=C=N), 1775 (shoulder, C=O), 1751, 1722 (broad, C=O), 1685 (shoulder, C=O), 1436, 1265, 1010, 754, 700 cm^{-1}; ^{1}H NMR: $\delta = 1.20$–1.85 (m, $CH(CH_2)_5$), 3.65 and 3.71 (2s, 2 OCH_3), 3.73 (m, $NCH(CH_2)_5$), 5.84 (s, $NCHCO_2CH_3$), 7.26 (br s, NH), 7.30–7.50 (m, 2 C_6H_5) ppm; ^{13}C NMR: $\delta = 23.78$, 25.05 and 32.80 (3 CH_2), 50.05 ($NCHCO_2CH_3$), 51.61 and 53.03 (2 OCH_3), 58.58 (C=C=N), 60.36 ($NCH(CH_2)_5$), 70.05 (Ph–C–Ph), 127.02 and 127.09 (2 CH_{meta}), 128.38 and 128.41 (2 CH_{para}), 128.53 and 128.68 (2 CH_{ortho}), 138.66 and 139.15 (2 C_{ipso}), 155.32 (C=O, urea), 162.68 (C=C=N), 167.68 (C=O, amide), 169.42 and 172.37 (2 C=O, ester) ppm; MS: m/z (%) = 503 (M^+, 18), 484 (54), 442 (100), 311 (22), 272 (33), 211 (27).

Diethyl 2-[(cyclohexylimino)methylene]-3-(4,4-diphenyl-2,5-dioxoimidazolidin-1-yl) butanedioate (**4d**, $C_{30}H_{33}N_3O_6$)
Colorless crystals, yield 0.43 g (80%), mp 154–157°C; IR (KBr): $\bar{\nu} = 3211$ (NH), 3095, 2975, 2858 (CH), 2073 (C=C=N), 1776 (shoulder, C=O), 1747, 1724 (broad, C=O), 1685 (shoulder, C=O), 1423, 1255, 1028, 756, 700 cm^{-1}; ^{1}H NMR: $\delta = 1.13$ and 1.16 (2t, $J = 7.2$ Hz, 2 OCH_2CH_3), 1.20–1.85 (m, $CH(CH_2)_5$), 3.75 (m, $NCH(CH_2)_5$), 4.10 and 4.15 (2q, $J = 7.2$ Hz, 2 OCH_2CH_3), 5.81 (s, $NCHCO_2C_2H_5$), 7.20–7.50 (m, NH and 2 C_6H_5) ppm; ^{13}C NMR: $\delta = 13.51$ and 13.86 (2 OCH_2CH_3), 23.28, 24.80 and 32.50 (3 CH_2), 49.90 ($NCHCO_2C_2H_5$), 58.88 (C=C=N), 60.13 ($NCH(CH_2)_5$), 60.15 and 62.13 (2 OCH_2CH_3), 69.92 (Ph–C–Ph), 127.07 and 127.28 (2 CH_{meta}), 128.49 and 128.51 (2 CH_{para}), 128.69 and 128.79 (2 CH_{ortho}), 139.14 and 139.29 (2 C_{ipso}), 155.88 (C=O, urea), 163.91 (C=C=N), 167.50 (C=O, amide), 169.42 and 172.67 (2 C=O, ester) ppm; MS: m/z (%) = 531 (M^+, 15), 458 (34), 450 (36), 376 (23), 330 (15), 298 (56), 252 (47), 180 (100), 143 (55), 104 (60).

Di-tert-butyl 2-[(cyclohexylimino)methylene]-3-(4,4-diphenyl-2,5-dioxoimidazolidin-1-yl) butanedioate (**4e**, $C_{34}H_{41}N_3O_6$)
Colorless crystals, yield 0.52 g (88%), mp 120–122°C; IR (KBr): $\bar{\nu} = 3190$ (NH), 3090, 2981, 2856 (CH), 2070 (C=C=N), 1770 (shoulder, C=O), 1750, 1729 (broad, C=O), 1683 (shoulder, C=O), 1420, 1258, 1024, 758, 700 cm^{-1}; ^1H NMR: $\delta = 1.25$–1.84 (m, $CH(CH_2)_5$), 1.38 and 1.52 (2s, 2 $C(CH_3)_3$), 3.75 (m, $NCH(CH_2)_5$), 5.75 (s, $NCHCO_2C_4H_9$), 7.20–7.50 (m, NH and 2 C_6H_5) ppm; ^{13}C NMR: $\delta = 23.38$ and 24.91 (2 CH_2), 27.32 and 27.94 (2 $C(CH_3)_3$), 32.53 (CH_2), 50.56 ($NCHCO_2CCH_3$), 59.85 (C=C=N), 61.12 ($NCH(CH_2)_5$), 70.60 (Ph–C–Ph), 79.83 and 81.86 (2 $C(CH_3)_3$), 127.15 and 127.88 (2 CH_{meta}), 128.66 and 129.15 (2 CH_{para}), 129.41 and 129.47 (2 CH_{ortho}), 136.66 and 137.66 (2 C_{ipso}), 154.28 (C=O, urea), 163.32 (C=C=N), 167.00 (C=O, amide), 168.78 and 172.50 (2 C=O, ester) ppm; MS: m/z (%) = 587 (M^+, 8), 530 (22), 485 (37), 450 (21), 376 (45), 330 (46), 223 (33), 180 (100), 104 (57), 85 (19), 57 (72).

Dimethyl 2-[(tert-butylimino)methylene]-3-(4,4-dimethyl-2,5-dioxoimidazolidin-1-yl) butanedioate (**4f**, $C_{16}H_{23}N_3O_6$)
Colorless crystals, yield 0.30 g (87%), mp 96–99°C; IR (KBr): $\bar{\nu} = 3215$ (NH), 2986 (CH), 2091 (C=C=N), 1781 (shoulder, C=O), 1745, 1720 (broad, C=O), 1686 (shoulder, C=O), 1429, 1365, 1313, 1263, 1184, 1114, 1039, 997, 773, 607 cm^{-1}; ^1H NMR: $\delta = 1.42$ and 1.43 (2s, 2 CH_3), 1.46 (s, $C(CH_3)_3$), 3.72 and 3.76 (2s, 2 OCH_3), 5.72 (s, $NCHCO_2CH_3$), 6.90 (br s, NH) ppm; ^{13}C NMR: $\delta = 24.61$ and 24.67 (2 CH_3), 30.00 ($C(CH_3)_3$), 49.47 ($NCHCO_2CH_3$), 51.67 and 52.97 (2 OCH_3), 58.67 (CH_3–C–CH_3), 60.12 (C=C=N), 62.29 ($C(CH_3)_3$), 155.32 (C=O, urea), 163.79 (C=C=N), 167.81 (C=O, amide), 169.58 and 176.32 (2 C=O, ester) ppm; MS: m/z (%) = 353 (M^+, 14), 297 (65), 282 (70), 165 (42), 238 (100), 206 (72), 178 (90), 153 (37), 121 (30), 84 (70), 57 (95).

Diethyl 2-[(tert-butylimino)methylene]-3-(4,4-dimethyl-2,5-dioxoimidazolidin-1-yl) butanedioate (**4g**, $C_{18}H_{27}N_3O_6$)
Colorless crystals, yield 0.33 g (86%), mp 128–130°C; IR (KBr): $\bar{\nu} = 3325$ (NH), 2980 (CH), 2068 (C=C=N), 1780 (C=O), 1745, 1724 (broad, C=O), 1685 (shoulder, C=O), 1420, 1369, 1259, 1180, 1105, 1028, 775 cm^{-1}; ^1H NMR: $\delta = 1.25$ and 1.26 (2t, $J = 7.1$ Hz, 2 OCH_2CH_3), 1.42 and 1.43 (2s, 2 CH_3), 1.46 (s, $C(CH_3)_3$), 4.10–4.30 (m, 2 OCH_2CH_3), 5.69 (s, $NCHCO_2C_2H_5$), 7.16 (br s, NH) ppm; ^{13}C NMR: $\delta = 14.00$ and 14.22 (2 OCH_2CH_3), 24.57 and 24.61 (2 CH_3), 30.02 ($C(CH_3)_3$), 49.42 ($NCHCO_2C_2H_5$), 58.59 (CH_3–C–CH_3), 60.30 (C=C=N), 60.62 (OCH_2CH_3), 62.10 ($C(CH_3)_3$), 62.20 (OCH_2CH_3), 155.44 (C=O, urea), 164.67 (C=C=N), 167.26 (C=O, amide), 169.21 and 176.41 (2 C=O, ester) ppm; MS: m/z (%) = 381 (M^+, 5), 362 (4), 344 (10), 334 (45), 296 (70), 252 (100), 206 (85), 178 (20), 84 (74), 55 (60).

Dimethyl 2-[(cyclohexylimino)methylene]-3-(4,4-dimethyl-2,5-dioxoimidazolidin-1-yl) butanedioate (**4h**, $C_{18}H_{25}N_3O_6$)
Colorless crystals, yield 0.33 g (89%), mp 129–131°C; IR (KBr): $\bar{\nu} = 3321$ (NH), 2987 (CH), 2079 (C=C=N), 1775 (shoulder, C=O), 1746, 1722 (broad, C=O), 1687 (shoulder, C=O), 1431, 1355, 1258, 1109, 1014, 915, 775, 732 cm^{-1}; ^1H NMR: $\delta = 1.20$–1.95 (m, $CH(CH_2)_5$), 1.35 and 1.36 (2s, 2 CH_3), 3.64 and 3.67 (2s, 2 OCH_3), 3.83 (m, $NCH(CH_2)_5$), 5.64 (s, $NCHCO_2CH_3$), 7.05 (br s, NH) ppm; ^{13}C NMR: $\delta = 23.67$ (CH_2), 24.38 and 24.44 (2 CH_3), 25.03 and 32.85 (2 CH_2), 49.45 ($NCHCO_2CH_3$), 51.50 and 52.89 (2 OCH_3), 58.53 (CH_3–C–CH_3), 60.18 (C=C=N), 60.31 ($NCH(CH_2)_5$), 155.11 (C=O, urea), 163.22 (C=C=N), 167.85 (C=O, amide), 169.47 and 176.48 (2 C=O, ester) ppm; MS: m/z (%) = 379 (M^+, 33), 364 (25), 320 (45), 282 (70), 238 (100), 206 (75), 178 (45), 149 (30), 84 (80).

Diethyl 2-[(cyclohexylimino)methylene]-3-(4,4-dimethyl-2,5-dioxoimidazolidin-1-yl) butanedioate (**4i**, $C_{20}H_{29}N_3O_6$)
Colorless crystals, yield 0.34 g (85%), mp 105–108°C; IR (KBr): $\bar{\nu} = 3321$ (NH), 2980 (CH), 2077 (C=C=N), 1780 (C=O), 1745, 1720 (broad, C=O), 1684 (shoulder, C=O), 1421, 1369, 1254, 1191,

1105, 1028, 997, 775, 732 cm^{-1}; ^1H NMR: $\delta = 1.25$ and 1.26 (2t, $J = 7.1$ Hz, 2 OCH_2CH_3), 1.43 and 1.44 (2s, 2 CH_3), 1.20–2.02 (m, $CH(CH_2)_5$), 3.93 (m, $NCH(CH_2)_5$), 4.10–4.29 (m, 2 OCH_2CH_3), 5.71 (s, $NCHCO_2C_2H_5$), 7.00 (br s, NH) ppm; ^{13}C NMR: $\delta = 13.96$ and 14.23 (2 OCH_2CH_3), 23.61 (CH_2), 24.52 and 24.57 (2 CH_3), 25.14 and 32.88 (2 CH_2), 49.60 ($NCHCO_2C_2H_5$), 58.55 (CH_3–C–CH_3), 58.95 (C=C=N), 60.27 ($NCH(CH_2)_5$), 60.28 and 62.10 (2 OCH_2CH_3), 155.35 (C=O, urea), 164.08 (C=C=N), 167.33 (C=O, amide), 169.15 and 176.44 (2 C=O, ester) ppm; MS: m/z (%) = 408 ($M^+ + 1$, 4), 381 (6), 325 (40), 296 (69), 280 (15), 252 (65), 206 (100), 178 (30), 84 (95), 57 (35).

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