Amino Acids (2006) 31: 397–401
DOI 10.1007/s00726-006-0340-z

Amino Acids
Printed in Austria

Preparation and characterization of novel pyrrol-3-ones attached to α/β-amino acids, esters and amides

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Received October 29, 2005
Accepted March 24, 2006
Published online May 26, 2006;

Summary. Various α/β amino acid derivatives **5** were attached to compounds **3** to yield 2,3-dihydro-1*H*-pyrrol-3-ones amino acids derivatives **6**. This rare heterocyclic amino acid skeleton including the pyrrolo[1,2-b][1,3]oxazol moiety was also successfully prepared in the esteric form. The structure of the new compounds was characterized by spectroscopic methods.

Keywords: Pyrrol-3-ones – Amino acids – Amino acid esters – Amino acid amides – Pyrrolo[2,1-b][1,3]oxazole – Furan-3(2*H*)-ones

Introduction

The development and application of new practical methods for the preparation of structurally diverse amino acid derivatives is of fundamental importance due to their widespread use in all areas of physical and life sciences. Non-naturally occurring amino acids and derivatives constitute an important resource for new chemotherapeutic agents, antibacterial compounds and enzyme inhibitors (Hoz et al., 2001). In this respect, heterocyclic amino acids are of much interest for biological and medicinal reasons. Such amino acids, containing isoxazole (Betler and Mulle, 1995), tetrazole (Schoepp et al., 1991), triazole (Ikegami and Murakoshi, 1994), and pyrazole (Dunnil and Fowden, 1965), are bioactive materials. Among these, the pyrrole amino acid derivatives, holds a special important status. Some of them, e.g. domoic acids, kainic acids (Clayden et al., 2005), lamellarins (Bailly, 2004), lukianol A (Yoshida et al., 1992), halitulin (Kashman et al., 1999), were isolated from natural sources. Some act as DNA targeting compounds with considerable cytotoxicity while others function as multi-drug resistant reversal agents (Bailly, 2004; Rudi et al., 1994; Kashman et al., 1999; Boger et al., 1999).

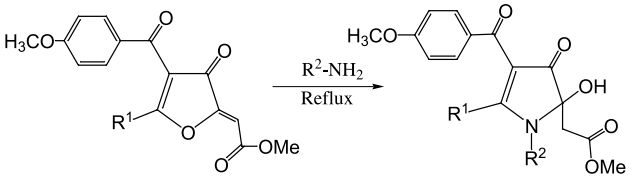
A variety of methods are known for the synthesis of pyrrolo-amino acids and esters. Recently, we have reported (Üngören et al., 2004; Saçmacı et al., 2005) about the reaction of furan-3(2*H*)-ones with primary amines and hydrazines to yield pyrroles containing N-alkyl/aryl/hydroxy substituted β-amino acid esters. We were able (Saçmacı et al., 2005) to prepare the interesting N-acetic acid substituted pyrrole derivative (Scheme 1). This last synthesis paved the way to a general procedure for a new type of pyrrolo-amino acid derivatives. In the present study, we extend the scope to various pyrrole containing α-amino acids, amides and esters. We also describe the successful preparation of the rare heterocyclic amino acid ester skeleton including the pyrrolo[1,2-b][1,3]oxazol moiety.

Materials and methods

4-(4-Methoxybenzoyl)-5-(4-methoxyphenyl)-2,3-dihydro-2,3-furandione **1** was prepared by cyclization of 1,3-bis(4-methoxybenzoyl)-propane-1,3-dione with oxalyl chloride (Hökelek et al., 2002). Melting points were determined on an electrothermal 9200 apparatus and are uncorrected. Elemental analyses (C, H, N) were carried out using LECO-932 CHNSO analyzer. IR spectra were recorded on a Jasco Plus Model 460 FT-IR Spectrometer as KBr pellets. ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra were obtained on a Bruker Avance DPX-400 spectrometer in DMSO-d_6 and $CDCl_3$ with TMS as an internal standart. Mass spectra, were measured with an Agilent 1100 MSD instrument. All experiments were followed by tlc using DC Alufolien Kieselgel 60 F 254 Merck and Camag TLC lamp (254/366 nm).

Ethyl (Z)-[2,3-Dihydro-4-(4-methoxybenzoyl)-5-(4-methoxyphenyl)-3-oxofuran-2-ylidene]acetate (**3b**)

To a boiling solution of 2,3-dihydro-furan-2,3-dione **1** (0.338 g, 1 mmole) in 25 ml benzene, a solution of **2** (0.348 g, 1 mmole) in dry benzene (15 ml)



R^1: Anisyl, Styryl R^2: -CH_2COOH, NR_2, Alkyl, Aryl

Scheme 1

was added and the mixture was refluxed for 20 min. After removal of the solvent, the oily residue was triturated with a mixture of petrolum ether (40–60) and diethyl ether (3:1) for 24 h. The bright yellow crystals were filtered off and washed with cyclohexane (0.32 g; 78%). m.p. 99–100 °C; ^{1}H NMR δ (ppm): 7.95–6.91 (m, 8H, Ar–H), 6.13 (s, 1H, –C=CH), 4.34 (q, $J = 7.1$ Hz, 2H, OCH_2), 3.85, 3.81 (s, 6H, OCH_3), 1.39 (t, $J = 7.1$ Hz, 3H, CH_3); ^{13}C NMR δ (ppm): 188.5 (Ar–CO), 184.3 (ring's CO), 177.4 (C=O, ester), 164.7, 164.6, 163.6, 151.2, 132.3, 131.7, 129.7, 118.8, 114.8, 114.7, 114.0, 100.7 (C=C, arom. and aliph.), 61.35 (OCH_2), 55.68, 55.59 (OCH_3), 14.24 (CH_3); IR ν (cm^{-1}): 1719, 1705, 1645 (C=O).

Anal. calcd. for $C_{23}H_{20}O_7$: C, 67.64; H, 4.94. Found C, 67.60; H, 4.95.

General procedure for preparation of 2,3-dihydro-1H-pyrrol-3-ones **6**

1 mmole amino acid derivatives (**5a**–**e**) and 1 mmole **3a**,**b** were refluxed in a mixture of 50 ml methanol and 2 ml pyridin for 5 h. After the solvents were removed by evaporation, the oil residue was triturated with mixtures of dry diethyl ether and n-hexane (3:1) to get the corresponding crude pyrrolones (**6a**–**i**), which were purified by recrystallization.

Methyl [2-hydroxy-4-(4-methoxybenzoyl)-2-(2-methoxy-2-oxoethyl)-5-(4-methoxyphenyl)-3-oxo-2,3-dihydro-1H-pyrrol-1-yl]acetate (**6a**)

White crystal from water; m.p. 156–157 °C; yield 0.270 g, 56%; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.80–6.83 (m, 8H, Ar–H), no detection (OH), 4.23 (q, $J = 18.2$ Hz, 2H, NCH_2COOMe), 3.84, 3.82, 3.75, 3.73 (s, 12H, –OCH_3), 3.0 (q, $J = 16.2$ Hz, 2H, –CH_2COOMe); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 193.6 (Ar–CO), 188.5 (pyrrol's CO), 181.2 ($CH_2\underline{C}OOMe$), 170.9 ($NCH_2\underline{C}OOMe$), 163.2–111.7 (C=C, arom. and aliph.), 88.1 (C–OH), 55.4, 55.4, 52.7, 52.3 (OCH_3), 44.6 (N–CH_2), 39.7 (C–$\underline{C}H_2$). IR ν (cm^{-1}): 3139 (OH), 1750, 1690 (C=O).

Anal. calcd. for $C_{25}H_{25}NO_9$: C, 62.11; H, 5.21; N, 2.90. Found C, 62.27; H, 5.22; N, 2.93.

Ethyl [2-hydroxy-4-(4-methoxybenzoyl)-2-(2-methoxy-2-oxoethyl)-5-(4-methoxyphenyl)-3-oxo-2,3-dihydro-1H-pyrrol-1-yl]acetate (**6b**)

White crystal from water; m.p. 156–157 °C; yield 0.427 g, 86%; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.75–6.76 (m, 8H, Ar–H), 6.22 (s, 1H, OH), 4.17 (q, $J = 18.2$ Hz, 2H, –NCH_2), 4.15 (q, $J = 7.1$ Hz, 2H, OCH_2), 3.79, 3.77, 3.70 (s, 9H, –OCH_3), 3.02 (q, $J = 16.2$ Hz, 2H, $C\underline{H}_2COOMe$), 1.21 (t, $J = 7.1$ Hz, 3H, $CH_2\underline{C}H_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 194.2, 188.8, 180.9, 170.0 (C=O), 169.9–111.7 (C=C, arom. and aliph.), 88.1 (C–OH), 61.8 (OCH_2), 55.3, 55.1 (OCH_3), 44.6 (N–CH_2), 40.1 ($\underline{C}H_2$ COOMe), 14.05 ($OCH_2\underline{C}H_3$); IR ν (cm^{-1}): 3136 (OH), 1749, 1688 (C=O).

Anal. calcd. for $C_{26}H_{27}NO_9$; C, 62.77; H, 5.47; N, 2.82. Found C, 62.50; H, 5.36; N, 2.95.

Methyl [2-hydroxy-4-(4-methoxybenzoyl)-2-(2-ethoxy-2-oxoethyl)-5-(4-methoxyphenyl)-3-oxo-2,3-dihydro-1H-pyrrol-1-yl]acetate (**6c**)

White crystal from toluene; m.p. 151–153 °C; yield 0.417 g, 84%; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.80–6.83 (m, 8H, Ar–H), no detection (OH), 4.23 (q, $J = 18.8$ Hz, 2H, –NCH_2), 4.20 (q, $J = 7.1$ Hz, 2H, OCH_2), 3.84, 3.82, 3.72 (s, 9H, –OCH_3), 2.99 (q, $J = 16.1$ Hz, 2H, C–CH_2–COOMe), 1.29 (t, $J = 7.0$ Hz, 3H, –CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 194.0, 188.6, 181.1, 170.25 (C=O), 169.9–111.8 (C=C, arom. and aliph.), 88.2 (C–OH), 61.3 (OCH_2), 55.6, 55.3, 52.6 (OCH_3), 44.6 (N–CH_2), 40.1 ($\underline{C}H_2$ COOEt), 14.07 ($OCH_2\underline{C}H_3$); IR ν (cm^{-1}): 3127 (OH), 1753, 1747, 1687 (C=O).

Anal. calcd. for $C_{26}H_{27}NO_9$; C, 62.77; H, 5.47; N, 2.82. Found C, 62.60; H, 5.55; N, 2.61.

Ethyl [2-hydroxy-4-(4-methoxybenzoyl)-2-(2-ethoxy-2-oxoethyl)-5-(4-methoxyphenyl)-3-oxo-2,3-dihydro-1H-pyrrol-1-yl]acetate (**6d**)

White crystal from benzene; m.p. 178–180 °C; yield 0.429 g, 84%; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.79–6.81 (m, 8H, Ar–H), 6.20 (broad OH), 4.21 (q, $J = 18.2$ Hz, 2H, –NCH_2), 4.19 (q, $J = 7.4$ Hz, 2H, OCH_2) and 4.18 (q, $J = 7.4$ Hz, 2H, other OCH_2), 3.83, 3.81 (s, 6H, OCH_3), 2.99 (q, $J = 16.3$ Hz, 2H, –C–$\underline{C}H_2COOR$), 1.28 (t, $J = 7.2$ Hz, 3H, $OCH_2\underline{C}H_3$) and 1.25 (t, $J = 7.1$ Hz, 3H, other $OCH_2\underline{C}H_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 193.9, 188.7, 181.2, 170.3 (C=O), 163.1–111.7 (C=C, arom. and aliph.), 88.2 (C–OH), 61.9, 61.4 (–OCH_2), 55.4, 55.4 (OCH_3), 44.80 (N–CH_2), 40.8 (CH_2COOR), 14.1, 14.0 (–CH_3); IR ν (cm^{-1}): 3129 (OH), 1745, 1685 (C=O).

Anal. calcd. for $C_{27}H_{29}NO_9$; C, 63.40; H, 5.71; N, 2.74. Found C, 63.20; H, 5.85; N, 2.50.

1-[2-Hydroxy-4-(4-methoxybenzoyl)-2-(2-ethoxy-2-oxoethyl)-5-(4-methoxyphenyl)-3-oxo-2,3-dihydro-1H-pyrrol-1-yl]acetamid (**6e**)

White crystal from n-buthanol; m.p. 187–188 °C; yield 0.347 g, 72%; ^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.70–6.90 (m, 8H, Ar–H), 7.16 (s, 2H, NH_2), no detection (OH), 4.03 (q, $J = 6.9$ Hz, OCH_2), 3.92 (q, $J = 17.1$ Hz, 2H, NCH_2) 3.88, 3.84 (s, 6H, –OCH_3), 2.90 (s, 2H, C–$\underline{C}H_2COOEt$), 1.16 (t, $J = 7.1$ Hz, 3H, $CH_2\underline{C}H_3$); ^{13}C NMR (100 MHz, DMSO) δ (ppm): 193.8, 187.7, 179.9, 170.3 (C=O), 162.7–110.7 (C=C, arom. and aliph.), 88.3 (C–OH), 60.6 (OCH_2), 55.84, 55.74 (OCH_3), 45.28 (NCH_2), 39.3 $\underline{C}H_2CH_3$), 14.42 ($CH_2\underline{C}H_3$); IR ν (cm^{-1}): 3430, 3190 (NH_2), 3280–3050 (OH), 1740, 1696 (C=O).

Anal. calcd. for $C_{25}H_{26}N_2O_8$; C, 62.23; H, 5.43; N, 5.81. Found C, 62.15; H, 5.48; N, 5.82.

[2-Hydroxy-4-(4-methoxybenzoyl)-2-(2-ethoxy-2-oxoethyl)-5-(4-methoxyphenyl)-3-oxo-2,3-dihydro-1H-pyrrol-1-yl]acetic acid (**6f**)

White crystal from water; m.p. 191–193 °C; yield 0.319 g, 66%; ^{1}H NMR (400 MHz, DMSO) δ (ppm): 7.71–6.89 (m, 8H, Ar–H), 4.05 (q, $J = 18.2$ Hz, 2H, CH_2COOEt), 4.08–4.00 (m, 2H, –OCH_2), 3.80, 3.79 (s, 6H, –OCH_3) 3.40 (br.,1H, COOH), 2.92 (s, 2H, NCH_2), (OH alcohol) no detection, 1.16 (t, $J = 7.1$ Hz, 3H, $CH_2\underline{C}H_3$); ^{13}C NMR (100 MHz, DMSO) δ (ppm): 193.8, 187.8, 179.7, 170.8 (C=O), 162.8–111.3 (C=C, arom. and aliph.), 88.1 (C–OH), 60.6 (–OCH_2), 55.9, 55.7 (–OCH_3), 44.2 (NCH_2), due to DMSO signals there is no detection of $\underline{C}H_2COOEt$ signal, 14.4 ($CH_2\underline{C}H_3$); IR ν (cm^{-1}): 3428 (OH, acid), 3136 (OH alcohol), 1743, 1724, 1690 (C=O).

Anal. calcd. for $C_{25}H_{25}NO_9$: C, 62.11; H, 5.21; N, 2.90. Found C, 61.95, H, 5.35, N, 2.70.

Ethyl 3-[2-hydroxy-4-(4-methoxybenzoyl)-2-(2-methoxy-2-oxoethyl)-5-(4-methoxyphenyl)-3-oxo-2,3-dihydro-1H-pyrrol-1-yl]propanoate (**6h**)

White crystal from CCl_4–CH_2Cl_2 (1:1); m.p. 113–115 °C; yield 0.245 g, 48%; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.68–6.75 (m, 8H, Ar–H),

5.50 (br., 1H, OH), 4.12–4.04 (m, 2H, OCH_2), 3.91–3.66 (m, 2H, NCH_2), 3.85, 3.83, 3.80 (s, 9H, OCH_3), 3.10 (q, $J = 15.2$ Hz, 2H, CH_2COOMe), 2.81–2.44 (m, 2H, $CH_2\underline{CH_2}COOEt$), 1.22 (t, $J = 7.1$ Hz, Hz, 3H, $OCH_2\underline{CH_3}$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 194.3, 188.4, 180.5, 171.1 (C=O), 169.0–110.8 (C=C, arom. and aliph.), 89.2 (C–OH), 60.8 (–OCH_2), 55.3, 52.1 (OCH_3), 40.2 (N–CH_2), 38.9 ($\underline{CH_2}COOMe$), 34.3 ($\underline{CH_2}COOEt$), 14.10 (CH_3); IR ν (cm^{-1}): 3130 (OH), 1735, 1685 (C=O).

Anal. calcd. for $C_{27}H_{29}NO_9$; C, 63.40; H, 5.71; N, 2.74. Found C, 63.23; H, 5.78; N, 2.66.

Ethyl 3-[2-(2-ethoxy-2-oxoethyl)-2-hydroxy-4-(4-methoxybenzoyl)-5-(4-methoxyphenyl) -3-oxo-2,3-dihydro-1H-pyrrol-1yl]propanoate (**6i**)

White crystal from Water; m.p. 111–112 °C; yield 0.215 g, 41%; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.79–6.85 (m, 8H, Ar–H), no detection (OH), 4.20 (q, $J = 7.1$ Hz, 2H, $NCH_2CH_2COOC\underline{H_2}$), 4.14–4.09 (m, 2H, $CCH_2COOC\underline{H_2}CH_3$), 3.93–3.71 (m, 2H, NCH_2), 3.85 (s, 6H, OCH_3), 3.02 (q, $J = 15.2$ Hz, 2H, $CC\underline{H_2}COOEt$), 2.81–2.44 (m, 2H, $NCH_2C\underline{H_2}COOEt$), 1.27 (t, $J = 7.1$ Hz, 3H, $NCH_2CH_2COOCH_2C\underline{H_3}$), 1.25 (t, $J = 7.1$ Hz, 2H, $CCH_2COOCH_2C\underline{H_3}$); IR ν (cm^{-1}): 3153 (OH), 1732, 1689, 1625 (C=O).

Anal. calcd. for $C_{28}H_{31}NO_9$; C, 63.99; H, 5.95; N, 2.67. Found C, 63.90; H, 5.85; N, 2.75.

Methyl [6-(4-methoxybenzoyl)-2,7-dioxo-5-(4-methoxyphenyl)-2,3-dihydropyrrolo[2,1-b][1,3]oxazol-7a(7H)-yl]acetate (**8**)

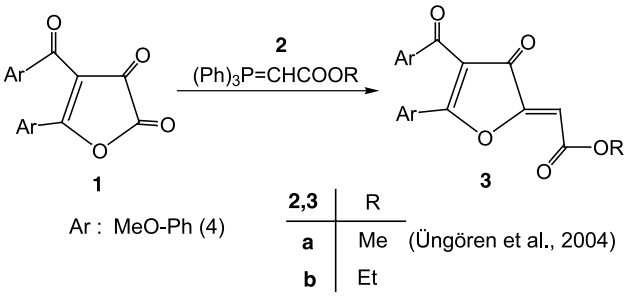
Compound **6g** (1 mmol, 0451 g) and thionyl chloride (1 ml, 13.8 mmol) were stirred on an ice-bath at 0–5 °C for 3 h and to solution was added diethyl ether. The crude precipitate was filtered off and recrystallized from CCl_4–CH_2Cl_2 (1:2). Yield 0.226 g, 50%; yellow crystal; m.p. 170–171 °C; ^1H NMR (400 MHz, DMSO) δ (ppm): 7.96–6.75 (m, 8H, Ar–H), 4.17 (q, $J = 18.1$ Hz, 2H, $NCH_2C{=}O$), 3.02 (q, $J = 16.2$, 2H, CH_2COOMe), 3.78, 3.77, 3.70 (s, 9H, –OCH_3); ^{13}C NMR (100 MHz, DMSO) δ (ppm): 194.0, 189.1, 175.3, 171.5 (C=O), 163.8–114.2 (C=C, arom. and aliph.), 78.4 (C–O–C=O), 56.1, 55.9, 53.0 (OCH_3), 47.6 (N–CH_2), 39.6 ($\underline{CH_2}COOMe$); IR ν (cm^{-1}): 1779, 1747, 1697, 1632 (C=O), 1255 (O–CO).

Anal. calcd. for $C_{24}H_{21}NO_8$; C, 63.85; H, 4.69; N, 3.10. Found C, 63.60, H, 4.50, N, 2.95.

Results and discussion

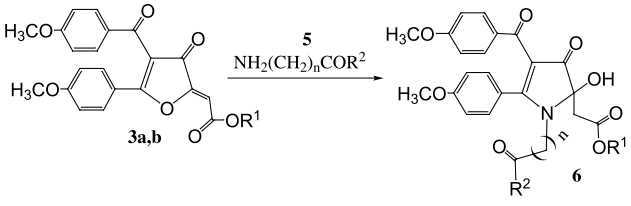
As described in Scheme 2, the starting material **3a** was prepared *via* Wittig reaction of 4-(4-methoxybenzoyl)-5-(4-methoxyphenyl)-2,3-dihydro-2,3-furandione (**1**) with methyl (Z)-[2,3-dihydro-4-(4-methoxybenzoyl)-5-(4-methoxyphenyl)-3-oxofuran-2-ylidene]acetate (**2a**). Various α- and β-amino acid derivatives **5** reacted with compounds **3** in methanol in the presence of pyridine to give the corresponding 2,3-dihydro-1*H*-pyrrol-3-ones amino acid compounds (Scheme 3). Ring closure of **6g** was achieved using $SOCl_2$ without solvent to generate the intermediate **7** which was spontaneously cyclized to the ester pyrrolo[1,2-b][1,3] oxazol **8** (Scheme 4).

The structures of all the new compounds were characterized by elemental analysis, NMR, IR and MS. Major fragmentation of compounds **6b**, **c**, **e**, **h**, in the MS are shown



2,3	R
a	Me
b	Et

Scheme 2

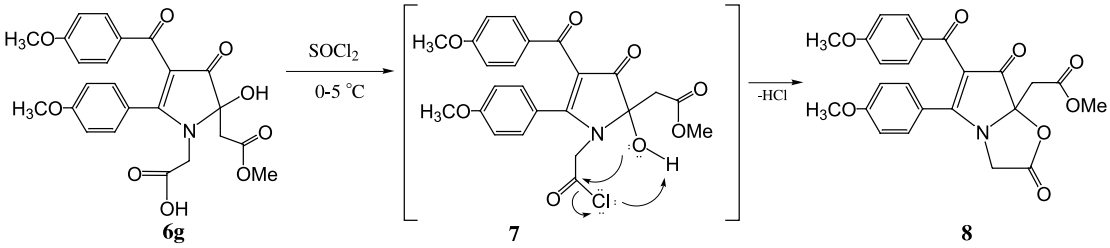


5	n	R^2
a	1	OMe
b	1	OEt
c	1	OH
d	1	NH_2
e	2	OEt

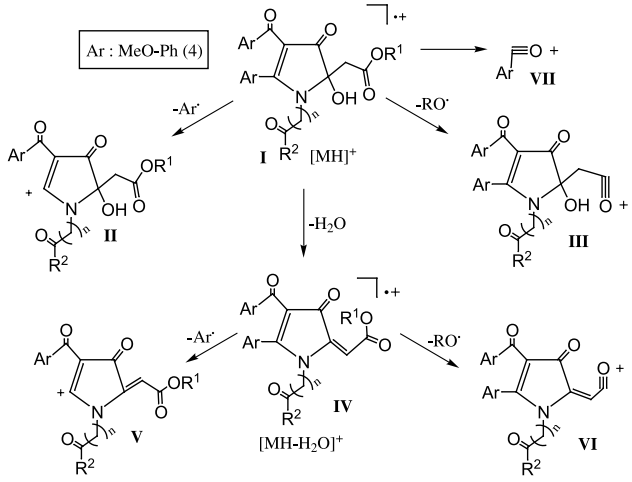
6	n	R^1	R^2
a	1	Me	OMe
b	1	Me	OEt
c	1	Et	OMe
d	1	Et	OEt
e	1	Et	NH_2
f	1	Et	OH
g	1	Me	OH

6	n	R^1	R^2
h	2	Me	OEt
i	2	Et	OEt

Scheme 3

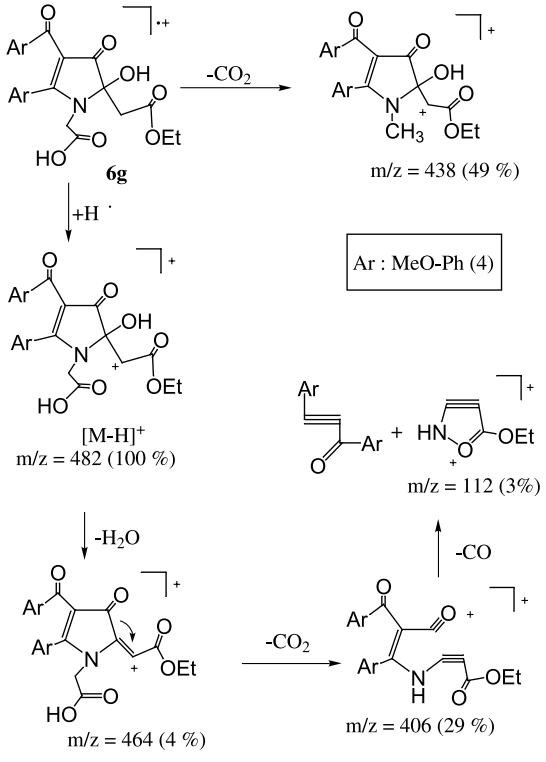


Scheme 4

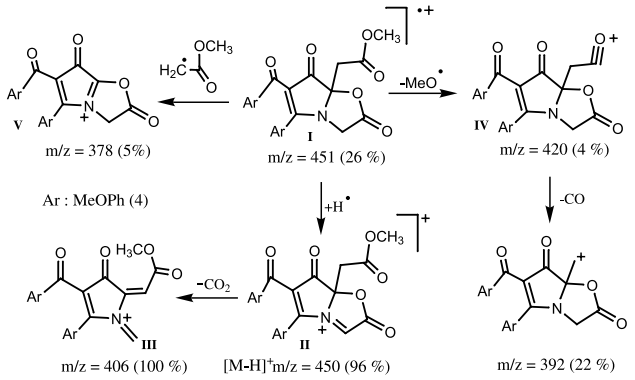


Frag.	b	c	e	h
I	498, 65 %	498, 100 %	483, 25 %	512, 18 %
II	390, 10 %	390, 17 %	375, 2 %	404, 1 %
III	-	-	-	480, 12 %
IV	480, 100 %	480, 86 %	465, 100 %	494, 100 %
V	372, 3 %	372, 2 %	357, 7 %	-
VI	448, 12 %	434, 22 %	419, 15 %	426, 9 %
VII	135, 2 %	135, 4 %	135, 3 %	135, 2 %

Scheme 5. Structure of fragments I–VII derived from compounds **6b**, **c**, **e**, **h**, their m/z and relative abundance values



Scheme 6



Scheme 7

in Scheme 5. The different mode of fragmentation of **6f** is shown in Scheme 6 and the mass fragmentations of compound **8** is detailed in Scheme 7.

In the IR spectra of compounds **6**, the broad OH absorption band is displayed around 3130 cm^{-1}. The carboxylic OH broad absorption of **6f** appears at 3428 cm^{-1}. Strong asymmetric and symmetric stretching vibrations of the NH_2 group of compound **6e** are observed at 3430, and 3190 cm^{-1}. Compound **8** exhibits in the IR the lactonic absorption at 1779 and other absorptions at 1747, 1697, 1632 cm^{-1}.

Acknowledgement

This work was financially supported by the Research Center of Erciyes University (Project No: FBA-04-27).

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