Synthesis of α-Diaminomethylenebutyrolactams by the Reaction of α-Cyanobutyrolactams with *N*-Trimethylsilylamines

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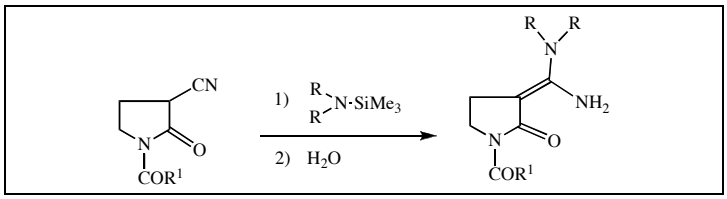

Successive treatment of α-cyanobutyrolactams **1** with *N*-trimethylsilylamines and water gave the corresponding α-diaminomethylenebutyrolacatms **2** in good yields. In the NOESY spectra of **2**, the *E* isomer, the NOE observed between β-CH_2 (butyrolactam) and *N*-CH_2 (morpholine, pyrrolidine) or *N*-CH_3 (dimethylamine) indicated a *cis* configuration of these groups.

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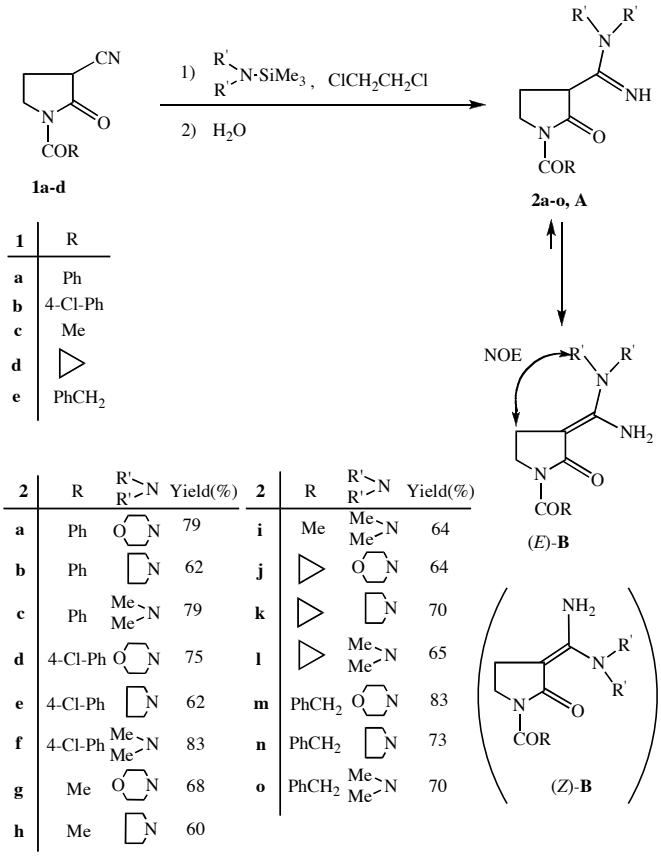
α-Methylene- [1-3] and α-aminomethylenebutyrolactams [4-8] are well known with a number of publications in the literature dealing with synthetic aspects of these compounds. α-Methylenebutyrolactams are also known as potential antitumor agents [2]. However, α-diaminomethylenebutyrolactams have been the subject of very few studies. This has been due to the relative difficulty of the synthesis of α-diaminomethylenebutyrolactams in earlier years. On a search in the literature only a single paper was found reporting the synthesis of α-diaminomethylenebutyrolactam. Kantlehner *et al.* reported the synthesis of 3-[bis(dimethylamino)-methylene]-1-methyl-2-pyrrolidinone from the reaction of 1-methyl-2-pyrrolidinone with ethoxy-*N*,*N*,*N*',*N*',*N*'',*N*''-hexamethylmethanetriamine [9]. One marked feature of α-diaminomethylenebutyrolactams is the enhanced enaminic reactivity. The nucleophilicity of the β-carbon atom (C-3) is higher than that of the nitrogen atoms of primary and tertiary amino moieties. In addition, the carbon-carbon double bond of these species is highly polarized. Furthermore, the amino substituents can also participate in the reaction of the system by acting as either the nucleophilic site or as a leaving group. For these reason, we are interested in the development of new method for the synthesis of α-diaminomethylenebutyrolactams.

N-Trimethylsilylamines are well known as reagents for silylation of alcohols [10] or amines [11], dealkylation of esters [12], ring opening of oxiranes [13], synthesis of silylenol ethers [14] or enamines [15] and carbon-carbon bond forming reactions [16]. We have recently reported on the reaction of 3-cyanobutyrolactones with *N*-trimethylsilyl-morpholine to give 3-(amino(morpholino)-methylene)-butyrolactones [17]. In an extension of this



1	R
a	Ph
b	4-Cl-Ph
c	Me
d	▷
e	$PhCH_2$

2	R	R', R'N	Yield(%)
a	Ph	O⌒N	79
b	Ph	⌒N	62
c	Ph	Me, MeN	79
d	4-Cl-Ph	O⌒N	75
e	4-Cl-Ph	⌒N	62
f	4-Cl-Ph	Me, MeN	83
g	Me	O⌒N	68
h	Me	⌒N	60
i	Me	Me, MeN	64
j	▷	O⌒N	64
k	▷	⌒N	70
l	▷	Me, MeN	65
m	$PhCH_2$	O⌒N	83
n	$PhCH_2$	⌒N	73
o	$PhCH_2$	Me, MeN	70

work, this note describes a synthesis of α-diaminomethylenebutyrolactams by the reaction of α-cyanobutyrolactams **1** with *N*-trimethylsilylamines. *N*-Trimethylsilylamines used in this study include *N*-trimethylmorpholine, *N*-trimethylsilylpyrrolidine and *N*-trimethylsilyldimethylamine. The starting compounds **1** were prepared by a procedure described in the literature [18].

When a mixture of 1-benzoyl-2-oxo-3-pyrrolidinecarbonitrile (**1a**) and *N*-trimethylsilylmorpholine in 1,2-dichloroethane was stirred at room temperature, 3-(amino(morpholino)methylene)-1-benzoyl-2-pyrrolidinone (**2a**) was obtained in 79% yield. The ir spectrum of **2a** reveals two bands at 3385 and 3200 cm^{-1} due to a primary amino group. The absorption band at near 2240 cm^{-1} due to a non-conjugated cyano group is not observed. The ^{13}C nmr spectrum exhibits the signals at δ = 76.6 and 169.8 ppm due to olefinic carbon atoms of the enamine moiety. These observations indicate that **2a** exists in the enamine structure **B** rather than the imine structure **A**. Furthermore, in the NOESY spectrum of **2a** ((*E*)-**B**), the presence of a NOE effect between the 4-H and N-CH_2 (morpholine) indicated a *cis* configuration of these groups, and the (*Z*)-isomer ((*Z*)-**B**) could be excluded. Elemental analysis, mass and ^1H nmr spectra of **2a** are consistent with the assigned structure. Compounds **1b-e** react with *N*-trimethylsilylmorpholine under the same conditions to afford the corresponding α-diaminomethylenebutyrolactams **2d**, **g**, **j**, **m** in good yields. Subsequently, the reaction of **1a-e** with *N*-trimethylsilylpyrrolidine or *N*-trimethylsilyldimethylamine resulted in the formation of the corresponding 3-(amino(pyrrolidino)methylene)-2-pyrrolidinones **2b**, **e**, **h**, **k**, **n** or 3-(amino(dimethylamino)methylene)-2-pyrrolidinones **2c**, **f**, **i**, **l**, **o** in 60-83% yields. The stereochemistry of **2b-o** was assigned by means of NOE between the 4-H and NCH_2 (morpholine or pyrrolidine) or N-CH_3 (dimethylamine). In the NOESY spectra of **2b-o**, the NOE showed that these groups are located on the same side of the carbon-carbon double bond system of the enamine moiety. Although the nucleophilic attack of *N*-trimethylsilylamines to **1** might be expected to occur at three possible positions of the lactam carbonyl, acyl carbonyl, or cyano group, we have shown that for **1** studied in this work nucleophilic attack takes place exclusively at cyano group. In conclusion, the reaction of α-cyanobutyrolactams with *N*-trimethylsilylamines is a versatile new access to α-diaminomethylenebutyrolactam compounds.

EXPERIMENTAL

All melting points are uncorrected. Ir spectra were recorded on a JASCO FT/IR-230 spectrometer. The ^1H nmr and ^{13}C nmr spectra were measured with a JEOL JNM-A500 instrument (500.00 MHz for ^1H, 125.65 MHz for ^{13}C) with TMS as internal standard. ^{13}C signal assignments were confirmed by the DEPT techniques. FAB mass spectra were taken with a JEOL JMS-HX100 instrument at 70 eV. Elemental analyses were performed using a YANACO MT-6 elemental analyzer.

General Procedure for the Preparation of 2. A mixture of **1** (10 mmoles) and *N*-trimethylsilylamine (10 mmoles) in 1,2-dichloroethane (20 ml) was stirred at room tempurature for 2 days. After removal of the solvent *in vacuo*, cold water (5 ml) was added to the residue. The precipitate was collected by filtration, dried and recrystallized from an appropriate solvent to yield **2**.

(*E*)-3-(Amino(morpholino)methylene)-1-benzoyl-2-pyrrolidinone (2a). This compound was obtained as pale yellow prisms (2.38 g, 79%), mp 204-205° (acetone); ir (potassium bromide): ν 3385, 3200 (NH), 1665 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 2.62-2.66 (m, 2H, 4-H), 3.22-3.26 (m, 4H, $2CH_2$(morpholine)), 3.58-3.63 (m, 4H, $2CH_2$(morpholine)), 3.64-3.68 (m, 2H, 5-H), 7.30-7.41 ppm (m, 7H, NH_2, aryl H); ^{13}C nmr (DMSO-d_6): δ 23.2 (4-C), 43.3 (5-C), 46.7 (NCH_2), 66.0 (OCH_2), 76.6 (3-C), 127.0, 128.2, 129.9, 136.7 (aryl C), 161.4, 169.4 (C=O), 169.8 ppm (=C(NH_2)N); ms: m/z 302 $[M+H]^+$. *Anal.* Calcd. for $C_{16}H_{19}N_3O_3$ (MW 301.3): C, 63.77; H, 6.36; N, 13.94. Found: C, 63.70; H, 6.32; N, 13.82.

(*E*)-3-(Amino(pyrrolidino)methylene)-1-benzoyl-2-pyrrolidinone (2b). This compound was obtained as yellow columns (1.76 g, 62%), mp 203-204° (acetone); ir (potassium bromide): ν 3402, 3177 (NH), 1646 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 1.79-1.86 (m, 4H, $2CH_2$(pyrrolidine)), 2.85-2.90 (m, 2H, 4-H), 3.39-3.48 (m, 4H, $2CH_2$(pyrrolidine)), 3.60-3.66 (m, 2H, 5-H), 7.29-7.58 ppm (m, 7H, NH_2, aryl H); ^{13}C nmr (DMSO-d_6): δ 23.1 (4-C), 24.7 (NCH_2CH_2), 42.9 (5-C), 47.9 (NCH_2), 73.3 (3-C), 126.9, 128.1, 129.5, 137.1 (aryl C), 158.7 (C=O), 168.0 (=C(NH_2)N), 169.1 ppm (C=O); ms: m/z 286 $[M+H]^+$. *Anal.* Calcd. for $C_{16}H_{19}N_3O_2$ (MW 285.3): C, 67.35; H, 6.71; N, 14.73. Found: C, 67.36; H, 6.69; N, 14.73.

(*E*)-3-(Amino(dimethylamino)methylene)-1-benzoyl-2-pyrrolidinone (2c). This compound was obtained as pale brown columns (2.04 g, 79%), mp 193-194° (acetone); ir (potassium bromide): ν 3373, 3208 (NH), 1655, 1635 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 2.72-2.78 (m, 2H, 4-H), 2.90 (s, 6H, $2CH_3$), 3.61-3.66 (m, 2H, 5-H), 7.28-7.42 ppm (m, 7H, NH_2, aryl H); ^{13}C nmr (DMSO-d_6): δ 23.6 (4-C), 39.0 (CH_3), 43.1 (5-C), 74.5 (3-C), 126.9, 128.1, 129.7, 136.9 (aryl C), 162.1 (C=O), 168.6 (=C(NH_2)N), 169.2 ppm (C=O); ms: m/z 260 $[M+H]^+$. *Anal.* Calcd. for $C_{14}H_{17}N_3O_2$ (MW 259.3): C, 64.85; H, 6.61; N, 16.20. Found: C, 64.83; H, 6.63; N, 16.08.

(*E*)-3-(Amino(morpholino)methylene)-1-(4-chlorobenzoyl)-2-pyrrolidinone (2d). This compound was obtained as colorless needles (2.52 g, 75%), mp 221-222° (acetone); ir (potassium bromide): ν 3395, 3208 (NH), 1657, 1630 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 2.62-2.66 (m, 2H, 4-H), 3.23-3.27 (m, 4H, $2CH_2$(morpholine)), 3.58-3.63 (m, 4H, $2CH_2$(morpholine)), 3.63-3.68 (m, 2H, 5-H), 7.34 (br. s, 2H, NH_2), 7.36-7.45 ppm (m, 4H, aryl H); ^{13}C nmr (DMSO-d_6): δ 23.2 (4-C), 43.2 (5-C), 46.7 (NCH_2), 66.0 (OCH_2), 76.5 (3-C), 127.1, 130.0, 134.6, 135.5 (aryl C), 161.5, 168.2 (C=O), 168.8 ppm (=C(NH_2)N); ms: m/z 336 $[M+H]^+$. *Anal.* Calcd. for $C_{16}H_{18}N_3O_3Cl$ (MW 335.8): C, 57.23; H, 5.40; N, 12.51. Found: C, 57.15; H, 5.41; N, 12.56.

(*E*)-3-(Amino(pyrrolidino)methylene)-1-(4-chlorobenzoyl)-2-pyrrolidinone (2e). This compound was obtained as yellow prisms (1.97 g, 62%), mp 205-206° (acetone); ir (potassium bromide): ν 3393, 3200 (NH), 1640 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 1.80-1.87 (m, 4H, $2CH_2$(pyrrolidine)), 2.84-2.90 (m, 2H, 4-H), 3.40-3.48 (m, 4H, $2CH_2$(pyrrolidine)), 3.60-3.65 (m, 2H, 5-H), 7.39-7.60 ppm (m, 6H, NH_2, aryl H); ^{13}C nmr (DMSO-d_6): δ 23.1 (4-C), 24.7 (NCH_2CH_2), 42.8 (5-C), 47.8 (NCH_2), 73.3 (3-C), 127.0, 129.9, 134.2, 135.9 (aryl C), 158.8 (C=O), 167.8 (=C(NH_2)N), 167.9 ppm (C=O); ms: m/z 320 $[M+H]^+$. *Anal.* Calcd. for $C_{16}H_{18}N_3O_2Cl$ (MW 319.8): C, 60.06; H, 5.67; N, 13.14. Found: C, 60.11; H, 5.67; N, 13.09.

(*E*)-3-(Amino(dimethylamino)methylene)-1-(4-chlorobenzoyl)-2-pyrrolidinone (2f). This compound was obtained as yellow prisms (2.43 g, 83%), mp 182-183° (acetone); ir (potassium bromide): ν 3411, 3165 (NH), 1650 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 2.73-2.77 (m, 2H, 4-H), 2.91 (s, 6H, $2CH_3$), 3.61-3.66 (m, 2H, 5-H), 7.38-7.50 ppm (m, 6H, NH_2, aryl H); ^{13}C nmr (DMSO-d_6): δ 23.6 (4-C), 39.0 (CH_3), 43.0 (5-C), 74.4 (3-C), 127.1, 130.0, 134.4, 135.8 (aryl C), 162.1 168.1 (C=O), 168.3 ppm (=C(NH_2)N); ms: m/z 294 $[M+H]^+$. *Anal.* Calcd. for $C_{14}H_{16}N_3O_2Cl$ (MW 293.8): C, 57.24; H, 5.49; N, 14.30. Found: C, 57.10; H, 5.49; N, 14.21.

(*E*)-1-Acetyl-3-(Amino(morpholino)methylene)-2-pyrrolidinone (2g). This compound was obtained as colorless columns (1.63g, 68%), mp 198-199° (acetone); ir (potassium bromide): ν 3392, 3190 (NH), 1648 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 2.33 (s, 3H, CH_3), 2.51-2.56 (m, 2H, 4-H), 3.22-3.25 (m, 4H, $2CH_2$(morpholine)), 3.48-3.54 (m, 2H, 5-H), 3.59-3.64 (m, 4H, $2CH_2$(morpholine)), 7.38 ppm (br. s, 2H, NH_2); ^{13}C nmr (DMSO-d_6): δ 22.3 (4-C), 23.7 (CH_3), 41.7 (5-C), 46.8 (NCH_2), 66.1 (OCH_2), 77.1 (3-C), 161.4, 169.3 (C=O), 169.7 ppm (=C(NH_2)N); ms: m/z 240 $[M+H]^+$. *Anal.* Calcd. for $C_{11}H_{17}N_3O_3$ (MW 239.3): C, 55.22; H, 7.16; N, 17.56. Found: C, 55.09; H, 7.13; N, 17.50.

(*E*)-1-Acetyl-3-(Amino(pyrrolidino)methylene)-2-pyrrolidinone (2h). This compound was obtained as colorless prisms (1.33 g, 60%), mp 201-202° (acetone); ir (potassium bromide): ν 3404, 3135 (NH), 1655 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 1.81-1.85 (m, 4H, $2CH_2$(pyrrolidine)), 2.33 (s, 3H, CH_3), 2.75-2.79 (m, 2H, 4-H), 3.40-3.46 (m, 4H, $2CH_2$(pyrrolidine)), 3.46-3.51 (m, 2H, 5-H), 7.54 ppm (br. s, 2H, NH_2); ^{13}C nmr (DMSO-d_6): δ 22.3 (4-C), 23.6 (CH_3), 24.7 (NCH_2CH_2), 41.4 (5-C), 47.8 (NCH_2), 73.5 (3-C), 158.7 (C=O), 168.9 (=C(NH_2)N), 169.1 ppm (C=O); ms: m/z 224 $[M+H]^+$. *Anal.* Calcd. for $C_{11}H_{17}N_3O_2$ (MW 223.3): C, 59.17; H, 7.67; N, 18.82. Found: C, 59.09; H, 7.65; N, 18.78.

(*E*)-1-Acetyl-3-(Amino(dimethylamino)methylene)-2-pyrrolidinone (2i). This compound was obtained as colorless prisms (1.26 g, 64%), mp 168-169° (acetone); ir (potassium bromide): ν 3378, 3135 (NH), 1645 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 2.33 (s, 3H, CH_3), 2.63-2.68 (m, 2H, 4-H), 2.90 (s, 6H, $2CH_3$), 3.47-3.52 (m, 2H, 5-H), 7.43 ppm (br. s, 2H, NH_2); ^{13}C nmr (DMSO-d_6): δ 22.8 (4-C), 23.7 ($COCH_3$), 39.1 (NCH_3), 41.5 (5-C), 74.8 (3-C), 162.1 169.2 (C=O), 169.3 ppm (=C(NH_2)N); ms: m/z 198 $[M+H]^+$. *Anal.* Calcd. for $C_9H_{15}N_3O_2$ (MW 197.2): C, 54.81; H, 7.67; N, 21.30. Found: C, 54.83; H, 7.71; N, 21.27

(*E*)-3-(Amino(morpholino)methylene)-1-cyclpropancarbonyl-2-pyrrolidinone (2j). This compound was obtained as colorless prisms (1.70g, 64%), mp 170-171° (acetone); ir (potassium bromide): ν 3392, 3206 (NH), 1656 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 0.74-0.85 (m, 4H, $2CH_2$(cyclopropane)), 2.52-2.56 (m, 2H, 4-H), 3.23-3.28 (m, 4H, $2CH_2$(morpholine)), 3.30-3.36 (m, 1H, CH), 3.50-3.54 (m, 2H, 5-H), 3.60-3.65 (m, 4H, $2CH_2$(morpholine)), 7.42 ppm (br. s, 2H, NH_2); ^{13}C nmr (DMSO-d_6): δ 8.5 (CH_2), 12.2 (CH), 22.4 (4-C), 42.3 (5-C), 46.8 (NCH_2), 66.1 (OCH_2), 77.4 (3-C), 161.5(C=O), 170.0 (=C(NH_2)N), 173.3 ppm (C=O),; ms: m/z 266 $[M+H]^+$. *Anal.* Calcd. for $C_{13}H_{19}N_3O_3$ (MW 265.3): C, 58.85; H, 7.22; N, 15.58. Found: C, 58.91; H, 7.14; N, 15.84.

(*E*)-3-(Amino(pyrrolidino)methylene)-1-cyclpropanecarbonyl-2-pyrrolidinone (2k). This compound was obtained as colorless prisms (1.73 g, 70%), mp 214-215° (acetone); ir (potassium bromide): ν 3406, 3154 (NH), 1642 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 0.70-0.83 (m, 4H, $2CH_2$(cyclopropane)), 1.78-1.85 (m, 4H, $2CH_2$(pyrrolidine)), 2.75-2.80 (m, 2H, 4-H), 3.36-3.50 (m, 7H, 5-H, CH, $2CH_2$(pyrrolidine)), 7.55 ppm (br. s, 2H, NH_2); ^{13}C nmr (DMSO-d_6): δ 8.3 (CH_2), 12.0 (CH), 22.4 (4-C), 24.6 (NCH_2CH_2), 42.1 (5-C), 47.8 (NCH_2), 73.8 (3-C), 158.8 (C=O), 169.2 (=C(NH_2)N), 173.1 ppm (C=O); ms: m/z 250 $[M+H]^+$. *Anal.* Calcd. for $C_{13}H_{19}N_3O_2$ (MW 249.3): C, 62.63; H, 7.68; N, 16.85. Found: C, 62.66; H, 7.71; N, 16.82.

(*E*)-3-(Amino(dimethylamino)methylene)-1-cyclopropanecarbonyl-2-pyrrolidinone (2l). This compound was obtained as colorless needles (1.45 g, 65%), mp 188-190° (acetone/petroleum ether); ir (potassium bromide): ν 3408, 3153 (NH), 1645, 1630 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 0.75-0.85 (m, 4H, CH_2 (cyclopropane)), 2.65-2.75 (m, 2H, 4-H), 2.91 (s, 6H, $2CH_3$), 3.35-3.40 (m, 1H, CH), 3.47-3.53 (m, 2H, 5-H), 7.45 ppm (br. s, 2H, NH_2); ^{13}C nmr (DMSO-d_6): δ 8.3 (CH_2), 12.1 (CH), 22.9 (4-C), 39.1 (CH_3), 42.2 (5-C), 75.1 (3-C), 162.2 (C=O), 169.6 (=C(NH_2)N), 173.2 ppm (C=O); ms: m/z 224 $[M+H]^+$. *Anal.* Calcd. for $C_{11}H_{17}N_3O_2$ (MW 223.3): C, 59.17; H, 7.67; N, 18.82. Found: C, 59.07; H, 7.63; N, 18.80

(*E*)-3-(Amino(morpholino)methylene)-1-phenylacetyl-2-pyrrolidinone (2m). This compound was obtained as colorless needles (2.63 g, 83%), mp 154-155° (acetone); ir (potassium bromide): ν 3366, 3208 (NH), 1655, 1630 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 2.52-2.57 (m, 2H, 4-H), 3.23-3.27 (m, 4H, $2CH_2$(morpholine)), 3.52-3.56 (m, 2H, 5-H), 3.60-3.63 (m, 4H, $2CH_2$(morpholine)), 4.24 (s, 2H, $COCH_2$), 7.15-7.30 (m, 5H, aryl H), 7.45 ppm (br. s, 2H, NH_2); ^{13}C nmr (DMSO-d_6): δ 22.2 (4-C), 41.1 ($COCH_2$), 42.1 (5-C), 46.8 (NCH_2), 66.1(5-C), 77.1 (3-C), 126.1, 128.0, 129.3, 136.3 (aryl C), 161.6 (C=O), 169.4 (=C(NH_2)N), 170.2 (C=O) ppm; ms: m/z 316 $[M+H]^+$. *Anal.* Calcd. for $C_{17}H_{21}N_3O_3$ (MW 315.4): C, 64.74; H, 6.71; N, 13.32. Found: C, 64.63; H, 6.78; N, 13.19.

(*E*)-3-(Amino(pyrrolidino)methylene)-1-phenylacetyl-2-pyrrolidinone (2n). This compound was obtained as colorless needles (2.20 g, 73%), mp 170-171° (acetone); ir (potassium bromide): ν 3381, 3181 (NH), 1655 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 1.80-1.86 (m, 4H, $2CH_2$(pyrrolidine)), 2.76-2.80 (m, 2H, 4-H), 3.41-3.46 (m, 4H, $2CH_2$(pyrrolidine)), 3.48-3.54 (m, 2H, 5-H), 4.26 (s, 2H, $COCH_2$), 7.17-7.30 (m, 5H, aryl H), 7.50-7.70 ppm (br. s, 2H, NH_2); ^{13}C nmr (DMSO-d_6): δ 22.3 (4-C), 24.7 (NCH_2CH_2), 40.9 ($COCH_2$), 41.8 (5-C), 47.8 (NCH_2), 73.3 (3-C), 126.0, 127.9, 129.3, 136.6 (aryl C), 158.9 (C=O), 168.6 (=C(NH_2)N), 170.0 ppm (C=O); ms: m/z 300 $[M+H]^+$. *Anal.* Calcd. for $C_{17}H_{21}N_3O_2$ (MW 299.4): C, 68.20; H, 7.07; N, 14.04. Found: C, 68.29; H, 7.15; N, 14.02.

(*E*)-3-(Amino(dimethylamino)methylene)-1-phenylacetyl-2-pyrrolidinone (2o). This compound was obtained as pale brown prisms (1.90g, 70%), mp 130-131° (acetone); ir (potassium bromide): ν 3425, 3153 (NH), 1650 (C=O) cm^{-1}; ^1H nmr (DMSO-d_6): δ 2.64-2.67 (m, 2H, 4-H), 2.91 (s, 6H, $2CH_3$), 3.50-3.53 (m, 2H, 5-H), 4.25 (s, 2H, $COCH_2$), 7.16-7.30 (m, 5H, aryl H), 7.40-7.60 ppm (br. s, 2H, NH_2); ^{13}C nmr (DMSO-d_6): δ 22.7 (4-C), 39.1 (CH_3), 41.0 ($COCH_2$), 41.9 (5-C), 74.9 (3-C), 126.0, 127.9, 129.3, 136.5 (aryl C), 162.2 (C=O), 169.0 (=C(NH_2)N), 170.1 ppm (C=O); ms: m/z 274 $[M+H]^+$. *Anal.* Calcd. for $C_{15}H_{19}N_3O_2$ (MW 273.3): C, 65.91; H, 7.01; N, 15.37. Found: C, 65.98; H, 6.98; N, 15.34.

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