Synthesis and Reactions of an Open-Chain bis-Reissert Salt Analog

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An open-chain bis-Reissert salt analog **5** has been synthesized and fully characterized. The reaction of the salt analog with methyl acrylate yields the pyrrole **7** which exists in solution in the azafulvene form **7a**. The reaction of the salt analog with dimethylacetylene dicarboxylate (DMAD) affords the bispyrrole **9**.

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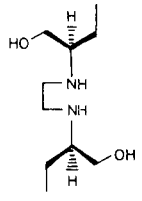
The chemistry of Reissert compounds has been extensively investigated during the last few decades [1a-e]. In comparison however, the chemistry of open-chain Reissert analogs and their salts have received much less attention except for a recent comprehensive study by McEwen *et al.* [2]. That there exists a difference between the chemistry of a Reissert compound and an open-chain analog was first recognized by Elliot [3] who reported that while acid-catalyzed hydrolysis of a Reissert compound gave aldehydes (the so-called Reissert aldehyde synthesis), an open-chain Reissert analog gave a carboxylic acid (the so-called anomalous behaviour of Reissert analog) [4a-b]. In a preliminary communication [5], we had reported the preparation, characterization and synthetic utility of an open-chain bis-Reissert salt analog. We wish to present details of our results in this paper.



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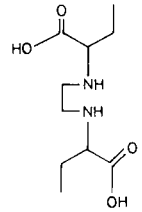

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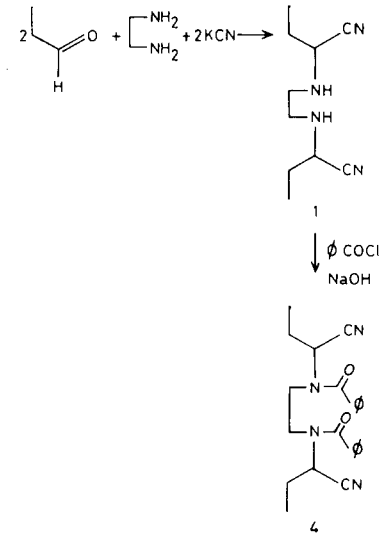
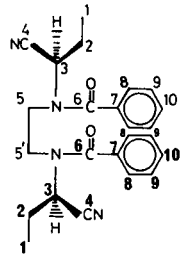
We had recognized [6] that the hitherto unreported open-chain bis-Reissert analog **4** is a precursor to a well-known antitubercular drug, ethambutol **2** [7] and its metabolite, the bis-α-aminoacid **3** [8-10]. The analog **4** was therefore synthesized as shown in Scheme 1. Thus a double Strecker reaction of propionaldehyde with ethylene diamine and potassium cyanide gave the bis-α-aminonitrile **1** in 65-75% yield as a *meso*-dl mixture. Attempts to separate the *meso*-dl mixture of **1** were unsuccessful, because it was unstable and therefore had to be used as such for further work.

The Schotten-Baumann benzoylation of the *meso*-dl mixture of **1** gave the bis-Reissert analog **4** in 70-75% yield. High pressure liquid chromatography (hplc) of **4** indicated it to be a diastereomeric mixture (80:20). The individual diastereomers (major, mp, 172-173°; minor, mp, 109-111°) could be separated by fractional crystallization and the purity ascertained by hplc.

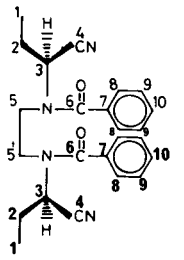
SCHEME 1



Although the knowledge of the relative configuration of the major and minor diastereomers of **4** was not of a major consequence in our work, the configurational assignment appeared to be a challenge. Firstly, there was no "handle" to attempt and determine which among the two diastereomers could be resolved. Secondly, conversion of the individual diastereomers into compounds of known configuration could not possibly be done without the attended risk of racemization because of the well-known facility with which Reissert compounds and their open-chain analogs are converted to 5-aminooxazolium cations [1a-d]. Therefore, both these methods could not be used for configurational assignment. However the ^{13}C-nmr spectrum of each of the diastereomers (Table 1) showed significant differences which was helpful in configurational assignment. Thus a broad-band decoupled ^{13}C-nmr spectrum of the major diastereomer (mp, 172-173°) showed eleven distinct signals whereas that of the minor



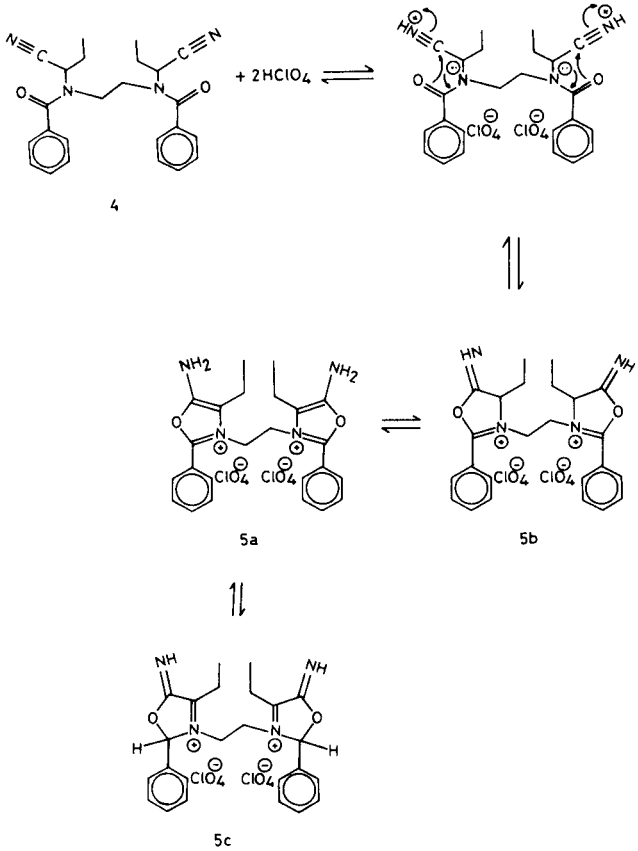
4a



4b

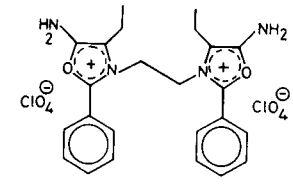
diastereomer (mp, 109-111°) showed only ten signals. Since that diastereomer of **4** which possesses a plane of symmetry is expected to give only ten signals, the minor diastereomer (mp, 109-111°) is the *meso*-compound **4b**. The major diastereomer (mp, 172-173°) is the dl-compound **4a**.

SCHEME 2



4 5a 5b 5c

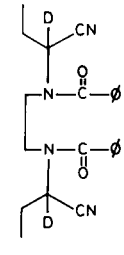
Treatment of either **4a** or **4b** with 60% perchloric acid (25°, 0.5 hour) gave the salt analog **5** quantitatively (Scheme-2). An ^{1}H-nmr examination of the salt in d_6-DMSO showed a six-proton triplet at δ 1.03, a four-proton singlet at δ 4.64, a four-proton broad signal at δ 6.83 (exchangeable with deuterium oxide) and a ten-proton singlet at δ 7.77. The ir spectrum displayed bands at 3400, 3300 and 3200 cm^{-1} characteristic of a ν-NH_2. The uv spectrum of the salt in methanol showed λ max at 214 nm (ϵ, log 4.11) and 336 nm (ϵ, log 3.81). These data indicate that the predominant tautomeric form of the salt in solution is the amino-form **5a** [11-12]. The ^{13}C-nmr data is



5

also in agreement with the structure **5a** for the salt [13]. Additional evidence for **5a** being the predominant form is obtained by examination of the Reissert analog **4** formed on treatment of the salt **5** with aqueous sodium bicarbonate. The hplc of **4** thus obtained indicated it to be a *meso*-dl mixture (1:1). A ^{13}C-nmr of **4** indicated separate signals for the carbons C-2, C-4 and C-5 (the C-3 signals were quite broadened) with almost equal signal areas thus representing a 1:1 *meso*-dl mixture [14]. Treatment of **5** with sodium carbonate/deuterium oxide gave the deutero-analog **4c** as a *meso*-dl mixture (1:1) as ascertained by the signal areas of C-2, C-4 and C-5 carbons in the ^{13}C-nmr [14]. All of these data are in agreement with the structure **5a** as representing the major tautomeric form of **5**.

That cations of the type **5b** and **5c** could be transient intermediates [11] is evidenced by the fact that upon heating **5** with hydrochloric acid (36%), a faint odor of benzaldehyde could be detected in the residue after the removal of **3** and benzoic acid [2,6].



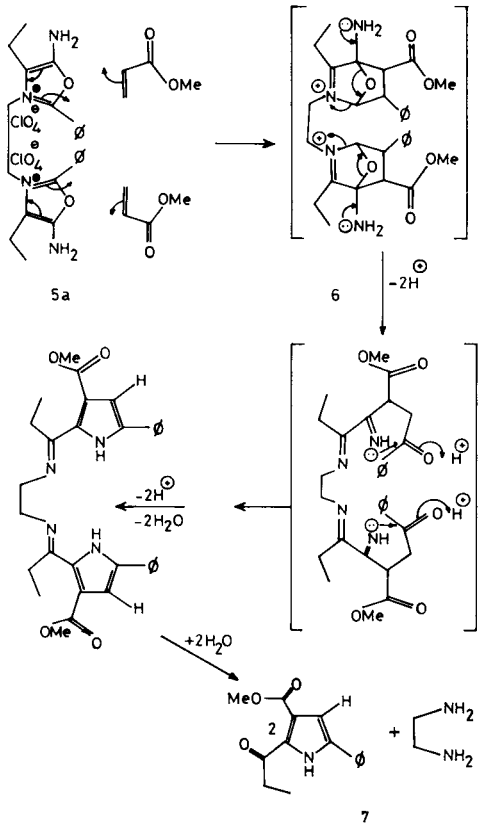
4c

Reaction of **5** with Methyl Acrylate.

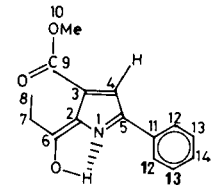
McEwen *et al.* [2] have reported that open-chain Reissert-salt analogs undergo Diels-Alder type reaction with alkenes to give highly functionalized pyrroles. When our salt analog **5** was reacted with methyl acrylate in DMF (25-30°, 8 hours), a product with a molecular formula $C_{15}H_{15}NO_3$ was obtained. In analogy with McEwen's studies [2], a tentative structure **7** could indeed be proposed for the product. However the ir spectrum did not show a distinct ν NH absorption band characteristic of pyrroles [15a-b], but a funnel-shaped band between 2700-3200 cm^{-1}

characteristic of a chelated enolic hydroxyl group [16]. The ^{1}H-nmr showed the presence of a highly deshielded proton *ca* δ 13 as a broad signal suggestive of a chelated enolic hydroxyl group [16]. The position of this signal was invarient with concentration and the signal disappeared on addition of deuterium oxide.

SCHEME 3



Consistent with the above facts, **7** gave a positive ferric chloride test characteristic of enols [17]. Also **7** failed to give an oxime or hydrazone derivative. These data seem to suggest that the pyrrole **7** exists in solution in the pyrrolenine (azafulvene) form **7a**. Further, the ^{13}C-nmr spectrum (Figure 1) is in better agreement with **7a** rather than **7**. Of the thirteen signals in the proton-decoupled ^{13}C-nmr, the ones at δ 52.19, 20.88 and 13.11 could be assigned to C-10, C-7 and C-8 respectively using correlation tables [18a] and the proton-coupled spectrum. The region between δ 150-200 was most informative in favouring **7a** over **7**. Thus for a structure **7** one would expect a signal in the δ 180-200 region characteristic of a carbonyl group in acyl pyrroles [18b]. Instead, two signals at δ 167 and 165 were observed. The signal at δ 167 could be assigned to the ester carbonyl C-9 where as that at δ 165 could be assigned to the enolic carbon **6**. Due to paucity of ^{13}C-nmr data on substituted aza-fulvenes definitive assignments for all the Sp^2 carbons, especially the quaternary carbons in **7a** could



7a

not be made. However, based on the assigned δ values for the α and β pyrrolenine carbons in chlorophyll [18c] and porphyrins [18d] the following tentative assignments for the Sp^2 carbons of **7a** can be made. Thus the signals at δ 143.58, 139.52 and 134.42 are due to quaternary carbons. The signal at δ 143.58 is due to C-5, consistent with α-pyrrolenic carbon resonances [18c]. In the proton coupled spectrum, this signal appeared as doublet (J_{CCH} = 3 Hz) due to long-range coupling with the C-4 proton. The signal at δ 139.52 could be assigned to C-2 and that at δ 134.42 to

Table 1

^{13}C-nmr Assignments [a] for Carbons in **4a**, **4b**. (The spectra were determined in deuteriochloroform and reported in ppm downfield from TMS.)

Carbon Atom	**4a**	**4b**
1	9.97	9.80
2	25.18	24.74
3	50.78 [b]	49.86 [b]
4	117.15	117.38
5	43.30	43.62
5'	43.17	--
6	171.56	171.44
7	134.14	134.06
8	128.82	128.72
9	126.13	126.13
10	130.27	130.18

[a] The spectra were calibrated using the central line of the deuteriochloroform signal as 76.9 ppm. The carbon numbering system is found in the structure **4a** and **4b**. [b] Signals were broad.

C-3 as the former is an α-pyrrolenic carbon [18c,d]. The signals at δ 132.78, 129.64, 128.7 and 126.4 are due to C-11, C-14, C-12 and C-13 respectively, based on signal intensity in the decoupled spectrum and signal multiplicity in the coupled spectrum. Lastly, the signal at 103.54 J_{CH} = 170 Hz) can be assigned to C-4, consistent with a δ value for a β-pyrrolenic carbon bearing a proton [18b-d].

In view of our findings, we have examined the ^{13}C-nmr spectrum of McEwen's pyrrole **8** which was synthesized by modifications of the reported procedure [2] and it also is suggestive of an azafulvene structure **8a** in solution.

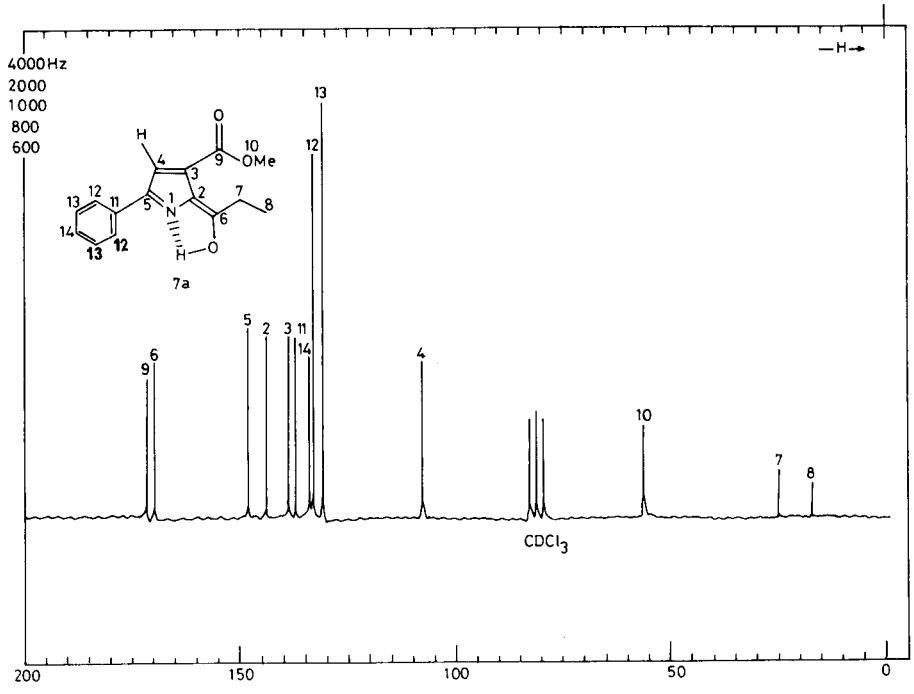
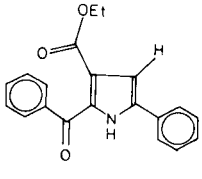
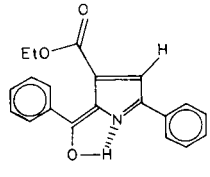


Fig. 1

Decoupled ^{13}C-NMR of 7a



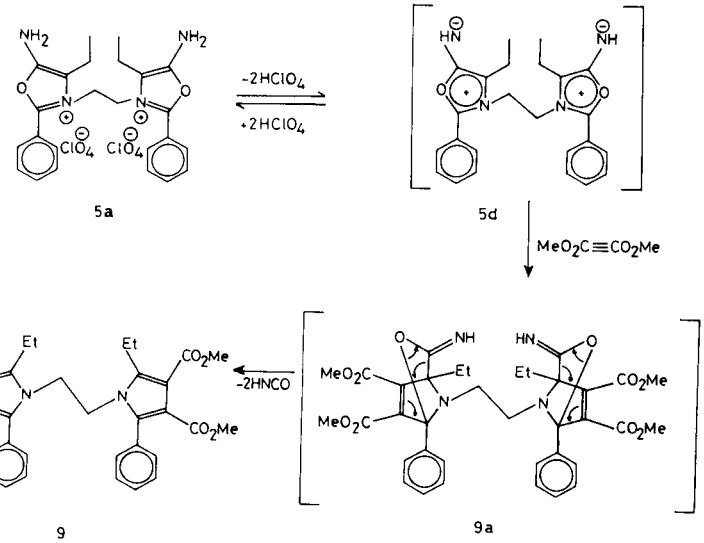
8



8a

The formation of **7a** can be explained in terms of the participation of **5a** in a Diels-Alder type reaction with methyl acrylate followed by fragmentation of the bis-bicyclic intermediate **6** and ring closure [2]. (Scheme 3). Interestingly, ethylene diamine could be isolated from the reaction mixture and identified as its dibenzoyl derivative. This key evidence provides additional support for the

SCHEME 4



mechanism suggested by McEwen *et al.* for the formation of pyrroles (azafulvenes) by the reaction of Reissert compounds and Reissert analogs with alkenes [2,19-22].

Reaction of **5** with Dimethylacetylene Dicarboxylate (DMAD).

Reissert salt analogs have been reported to undergo cycloaddition reactions with alkynes to give highly functionalized pyrroles [2]. When the salt analog **5** was reacted with DMAD (10 equivalents, 70-80°, 12 hours), the bispyrrole **9** was obtained in 40-45% yield. The formation of **9** can be rationalized if one were to assume the dissociation of the salt **5** into the meso-ionic form **5d** and its participation in a 1,3-dipolar cycloaddition reaction with DMAD to give the adduct **9a** which could then undergo cycloreversion with loss of HNCO *via* a *retro*-Diels-Alder process (Scheme 4) [2,23]. It is pertinent to mention that attempts to isolate the meso-ionic **5d** were unsuccessful. Therefore a direct reaction of the salt **5** as a 4 π system with DMAD leading to the formation of **9** *via* **9a** cannot be ruled out.

The bispyrrole **9** and their analogs are precursors to the various vinylogous carbinolamine-type derivatives described as potential anticancer agents [24a-b]. Efforts are under way to carry out such an evaluation on **9** and their analogs.

EXPERIMENTAL

General.

The proton nmr spectra were obtained on a Varian 360L NMR Spectrometer. The ^{13}C-nmr spectra were obtained on a Varian FT-80A NMR spectrometer, in either deuteriochloroform (99.8% d_1) or d_6-dimethylsulfoxide (d_6-DMSO) (99.5% d_6), both obtained from Aldrich Chemical Co., Milwaukee, Wisconsin. All ir spectra were obtained on a Perkin-Elmer model 397 double beam instrument and all uv were run on a Beckmann 25 spectrophotometer. Melting points were determined on a Tempo melting point unit and are uncorrected. Thin layer chromatography (tlc) was carried out using silica gel (Acme) on analytical plates prepared from chloroform-methanol slurry. Column chromatography was carried out with silica gel, 60-120 mesh (Acme). High pressure liquid chromatography (hplc) was performed on a Waters instrument. *Note*: Adequate safety precautions must be taken during the preparation and handling of perchlorate salts, even though no difficulties were encountered during the course of this work.

2,2'-(1,2-ethanediyldimino)bis-butanenitrile **1**.

To a stirred solution of potassium cyanide (16.25 g, 0.25 mole) in water (135 ml) at 5° was added ethylene diamine dihydrochloride (17.1 g) followed by freshly prepared propanol [25] (14.5 g, 0.25 mole) in one portion. Precipitation of the snowy white bis-α-aminonitrile **1** started within five minutes. The reaction mixture was kept cooled and stirred for three more hours. It was then filtered under vacuum, the precipitate washed with cold water (6 x 25 ml) and dried *in vacuo* to give the bis-α-aminonitrile **1** (19 g, 75%), mp 44-48°; ir (nujol): 3250, 2238, 1670, 1120, 1102 cm^{-1}; ^{1}H nmr (deuteriochloroform): δ ppm 1.1 (t, 6H, J = 7 Hz), 1.4-2.1 (m, 6H, 2H exchangeable with deuterium oxide), 2.4-3.2 (m, 4H), 3.45 (t, 2H, J = 7 Hz).

Preparation of 2,2'-(1,2-Ethanediyl(N,N'-dibenzoyl)diimino)bis-butanenitrile **4**.

To a stirred solution of the bis-α-aminonitrile **1** (9.7 g, 0.05 mole) in benzene (10 ml) at 10° was added sodium hydroxide solution (30%, 20 ml), followed immediately by freshly distilled benzoyl chloride (15.5 g, 0.11 mole). The contents were vigorously stirred for five minutes. The sticky mass so obtained was transferred to a mortar and pestle and triturated so as to obtain a suspension of the benzoyl derivative **4**. This was filtered, washed thoroughly with saturated sodium bicarbonate solution, water and ether to give 16 g (80%) of crude **4**. The major diastereomer **4a** was obtained by repeated fractional crystallization of the crude **4** from hot methanol. The mother liquor after removal of most of the major diastereomer was concentrated and chromatographed on a silica gel column. Elution with benzene containing 1% methanol gave the minor diastereomer **4b**.

Compound **4a**.

This compound had mp 172-173° (aqueous methanol); ir (potassium bromide); 3064, 3046, 2978, 2956, 2886, 2246, 1646, 1601, 1493, 1467, 1446, 1412, 1338, 1321, 1301, 1264, 1198, 1133, 1113, 1078, 1028, 935, 793, 775, 748, 728, 709, 644 cm^{-1}; ^{1}H-nmr (deuteriochloroform): δ ppm 0.95 (t, J = 7 Hz, 6H), 1.48-2.10 (m, 4H), 3.75 (s, 4H), 4.8 (broad t, 2H), 7.5 (s, 10H); hplc (porasil, methylene chloride:cyclohexane 80:20, 1 ml/minute) retention time 2.82 minutes.

Anal. Calcd. for $C_{24}H_{26}N_4O_2$ (402.5): C, 71.6; H, 6.5; N, 13.9. Found: C, 71.8; H, 6.6; N, 13.4.

Compound **4b**.

This compound had mp 109-111° (aqueous methanol); ir (potassium bromide): 3056, 2976, 2936, 2881, 2246, 1646, 1601, 1495, 1469, 1446, 1426, 1406, 1350, 1316, 1301, 1261, 1196, 1136, 1111, 1080, 1031, 936, 778, 751, 732, 711, 645 cm^{-1}; ^{1}H-nmr (deuteriochloroform): δ ppm 0.93 (t, J = 7 Hz, 6H), 1.48-2.1 (m, 4H), 3.75 (s, 4H), 4.8 (bt, 2H), 7.45 (s, 10H); hplc (porasil, methylene chloride:cyclohexane 80:20, 1 ml/minute) retention time 4.82 minutes. For ^{13}C-nmr data of **4a** and **4b** see Table 1.

Anal. Calcd. for $C_{24}H_{26}N_4O_2$ (402.5): C, 71.6; H, 6.5; N, 13.9. Found: C, 71.9; H, 6.4; N, 13.5.

Preparation of 3,3'-Ethylene-bis(2-phenyl-4-ethyl-5-aminooxazolium Perchlorate) **5**.

To the bis-Reissert analog **4a** or **4b** (804 mg, 2 mmoles) was added perchloric acid (60%, 4 ml) slowly and with cooling (10°). The reaction mixture was stirred vigorously for 15 minutes and the resulting yellow precipitate was filtered and washed with dry ether (3 x 15 ml). The crude salt **5** (870 mg) thus obtained was crystallized from dry acetonitrile-dry ether to give 740 mg (60% yield). Recrystallization thrice from the same solvent system afforded an analytically pure sample, mp 196-198° dec; uv (methanol): λ max 214 nm, ε 12,852; 336 nm, 6426; ir (nujol): 3400, 3300, 3200, 1680, 1620, 1570, 1420, 1070, 765 cm^{-1}; ^{1}H-nmr (d_6-DMSO): δ ppm 1.03 (t, J = 7 Hz, 6H), 2.1-2.7 (m, 4H), 4.64 (s, 4H), 6.83 (broad s, 4H, exchangeable with deuterium oxide), 7.77 (s, 10H); ^{13}C-nmr (d_6-DMSO): δ ppm 152.69, 150.24, 133.59, 129.85, 128.88, 119.82, 107.08, 43.73, 43.67, 13.98, 13.92, 12.22.

Anal. Calcd. for $C_{24}H_{28}Cl_2N_4O_{10}$ (603.4): C, 47.8; H, 4.7; N, 9.3. Found: C, 48.2; H, 4.6; N, 9.4.

Reaction of **5** with Saturated Sodium Bicarbonate Solution.

To the salt analog **5** (121 mg, 0.2 mmole) obtained from **4a** or **4b** was added saturated sodium bicarbonate solution and the contents stirred at 25° for one hour. The white precipitate of **4** thus obtained (76 mg, 95%) was filtered, washed with water and dried. The hplc (μ porasil, methylene chloride: cyclohexane 80:20, 1 ml/minute, 254 nm), indicated 4 to be *ca.* 1:1 diastereomeric mixture, mp 132-134°. The ^{13}C-nmr spectrum of **4** indicated separate signals for the carbons C-2, C-4, and C-5 corresponding to a 1:1 diastereomeric mixture.

Reaction of **5** with Sodium Carbonate/Deuterium Oxide.

In a 5-ml reacti-vial with a triangular spin-vane was placed **5** (100 mg) and 310 mg of dry sodium carbonate. The vial was capped with a rubber septum and 4 ml of deuterium oxide was injected into it with a syringe. The reaction mixture was stirred for one hour at 25°. The precipitate of

4c which had formed was filtered in a nitrogen atmosphere and taken up in deuteriochloroform for ^{13}C-nmr. Separate signals for the carbons C-2 and C-4 and C-5 corresponding to a 1:1 diastereomeric mixture was found. Further, the ^{1}H-nmr of **4c** was identical to that of **4** except for the disappearance of the two-proton triplet at δ 4.8 and collapse of the broad multiplet at δ 1.48-2.10 to a broad quartet.

Reaction of **5** with Methyl Acrylate.

Dry DMF (4 ml), the salt analog **5** (603 mg, 1 mmole) and methyl acrylate (0.4 ml) were stirred together at 25-30° for 8 hours. The reaction mixture became a yellow semi-solid mass. The excess methyl acrylate and most of the DMF were removed on a rotary evaporator. The residue so obtained was triturated with aqueous methanol and filtered. The filtrate was processed further for the recovery of ethylene diamine (see below). The residue washed with sodium bicarbonate solution (2 x 5 ml) followed by water (3 x 5 ml). Crystallization from aqueous methanol gave fine yellow needles of **7a** (154 mg, yield 60%). An analytically pure sample was obtained by column chromatography on silica gel. Elution with chloroform:methanol (98:2) and evaporation of the solvent *in vacuo* left behind yellow needles of **7a**. Crystallization from aqueous methanol gave **7a**, mp 185-187°; ir (potassium bromide): 2700, 3200, 1734, 1646, 1624, 1581, 1554, 1506, 1454, 1441, 1366, 1286, 1261, 1238, 1186, 1081, 1065, 1001, 975, 911, 869, 841, 801, 766, 755, 691, 631 cm^{-1}; uv (methanol): λ max 248 nm, ε, 7506; 340 nm, ε, 11,394; ^{1}H-nmr (deuteriochloroform): δ ppm 1.21 (t, J = 8 Hz, 3H), 2.85 (q, J = 8 Hz, 2H), 3.92 (s, 3H), 6.72 (s, 1H), 7.16-8.03 (m, 5H), 12.75 (broad s, 1H, invarient with concentration, exchangeable with deuterium oxide); ^{13}C-nmr (deuteriochloroform): δ ppm 166.97, 165.2, 143.58, 139.51, 134.42, 132.78, 129.64, 128.7, 126.47, 103.54, 52.19, 20.88, 13.11.

Anal. Calcd. For $C_{15}H_{15}NO_3$ (257.2): C, 70.0; H, 5.9; N, 5.44. Found: C, 69.6; H, 6.0; N, 5.2.

Isolation of Ethylenediamine from the Reaction of **5** with Methyl Acrylate.

The filtrate obtained as above was concentrated *in vacuo* to remove most of the methanol and an excess of benzoyl chloride was added to the residue. After stirring for four hours, water was added to the reaction mixture and contents extracted with chloroform. The chloroform layer was washed with dilute sodium hydroxide solution, followed by water. The organic layer was then dried over anhydrous sodium sulfate, and the solvent removed *in vacuo* to give a red oil. On trituration with chloroform, a white solid separated out. Crystallization from chloroform gave the N,N'-dibenzoyl ethylenediamine mp 242°. No depression in mp on admixture of the compound with an authentic sample prepared by benzoylation of ethylenediamine; ^{1}H-nmr (d_6-DMSO): δ ppm 3.40-3.73 (m, 4H), 7.33-8.1 (m, 10H), 8.65 (broad s, 2H, exchangeable with deuterium oxide); ^{13}C-nmr (d_6-DMSO): δ ppm 166.62, 134.64, 131.11, 128.25, 127.21, 39.25.

Preparation of McEwen's Pyrrole.

For the preparation of McEwen's pyrrole, the procedure reported by McEwen *et al.* was followed as reported [2] except that the perchlorate salt was used rather than the fluroborate salt for the reaction with ethyl acrylate. The perchlorate salt was prepared by treatment of the Reissert analog (600 mg, 1.45 mmoles) with perchloric acid (60%, 3 ml) at 26° for 10 minutes. The yellow solid thus obtained was filtered, washed with anhydrous ether (3 x 10 ml) and dry dichloromethane, to give 630 mg (79%) of the salt.

McEwen's pyrrole prepared as above had a mp 212° (lit [2] 214-215°); ir (potassium bromide): 2600-3200, 1941, 1726, 1634, 1591, 1571, 1541, 1501, 1468, 1449, 1381, 1361, 1347, 1336, 1281, 1256, 1231, 1161, 1156, 1091, 1036, 1026, 1001, 979, 921, 906, 861, 836, 797, 771, 743, 711,701 cm^{-1}; ^{1}H-nmr (deuteriochloroform): δ ppm 0.93 (t, J = 7 Hz, 3H), 4.02 (q, J = 7 Hz, 2H), 6.72 (s, 1H), 7.10-8.00 (m, 10H), 12.85 (broad s, 1H, exchangeable with deuterium oxide); ^{13}C-nmr (deuteriochloroform): δ ppm 167.52, 164.27, 145.93, 142.72, 134.76, 132.41, 130.04, 129.53, 129.04, 128.89, 127.67, 127.59, 126.59,103.62, 61.46, 13.37.

Reaction of **5** with Dimethyl Acetylenedicarboxylate (DMAD).

The bis-Reissert salt analog (603 mg, 1 mmole) and DMAD (1.2 ml, 10 mmoles) were heated together at 100-110° for 11 hours. To the cooled reaction mixture, dichloromethane (30 ml) was added. A precipitate, presumably a polymer of isocyanic acid [2] separated out immediately. The reaction mixture was filtered and the polymeric residue washed with dichloromethane (2 x 10 ml). The filtrate and the washings were combined, washed with sodium bicarbonate solution and dried over anhydrous sodium sulfate. On removal of the solvent *in vacuo* a yellow solid was obtained. Column chromatography over silica gel and elution with dichloromethane afforded 185 mg (yield 40-45%) of the bispyrrole **9**. Crystallization from dichloromethane-methanol gave analytically pure sample, mp 267-268°; ir (potassium bromide): 2970, 2950, 1725, 1710, 1610, 1540, 1490, 1445, 1425, 1400, 1300, 1270, 1245, 1215, 1200, 1180, 1100, 1050, 1000, 950, 760, 705 cm^{-1}; uv (acetonitrile): λ max 264 nm, ε, 14,828; ms: 600, 568, 539, 536, 507, 282, 268, 224, 196, 181, 167, 77, 59; ^{1}H-nmr (deuteriochloroform): δ ppm 0.85 (t, J = 7 Hz, 6H), 1.95 (q, J = 7 Hz, 4H), 3.55 (s, 6H), 3.65 (s, 4H), 3.76 (s, 6H), 7.10-7.60 (m, 10H); ^{13}C-nmr (deuteriochloroform): δ ppm 165.35, 164.81, 139.87, 134.07, 130.46, 130.17, 129.07, 128.67, 115.81, 111.57, 51.40, 51.22, 43.02, 17.28, 14.34.

Anal. Calcd. for $C_{34}H_{36}N_2O_8$ (600.6): C, 67.98; H, 6.04; N, 4.66. Found: C, 68.13; H, 5.93; N, 4.98.

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