Studies on Quinones. Part 23. Synthesis of Azepinones Fused to Quinone Systems [1]

J. A. Valderrama*, H. Pessoa-Mahana and R. Tapia

Facultad de Química, Pontificia Universidad
Católica de Chile, Casilla 306,
Santiago-22, Chile

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The Diels-Alder reactions of 3,4-dihydro-5*H*-1-benzazepine-2,6,9-trione **1** with butadiene, cyclopentadiene, 1-(*E*)-trimethylsiloxybutadiene **4**, and methacrolein dimethylhydrazone are described. Cycloaddition with the unsymmetrical dienes **4** and **18** occurs regiospecifically affording **5** and **19** respectively. The structure of **5** was established through naphthazepine **13** by comparison of its ^{1}H nmr with model compounds. The probable course of cycloaddition with 1-azadiene **18** is analyzed considering the polarization of azadiene **18** and the behavior of quinone **1** in the reaction with **4**. Michael addition of quinone **1** with isobutenylmorpholine **23** which afforded exclusively the cyclic O,N-acetal **24** or **25** and its convertion to benzepinquinone **26** or **27** is also reported.

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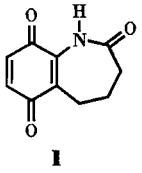
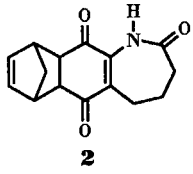
Introduction.

In a previous work [1] we have discovered a short and efficient preparation of 3,4-dihydro-5*H*-1-benzazepine-2,6,9-trione **1**. Our interest in this substance is derived from its potential as a precursor of new azepines condensed with carbocyclic and heterocyclic quinones taking advantage of the synthetic flexibility of the quinone moiety. Benzazepines are substances of current interest due to the variety of their pharmacological activities [2-5], and the synthetic methods have been normally carried out by heterocyclic ring formation of aromatic precursors [6].



1



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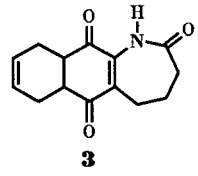

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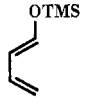

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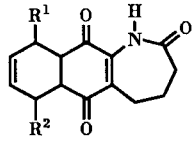
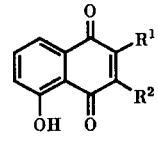


5, R^1 = OTMS, R^2 = H
6, R^1 = H, R^2 = OTMS



7, R^1 = NH_2, R^2 = H
8, R^1 = H, R^2 = NH_2

In this communication we wish to report the reactivity of the quinone **1** with dienes and with a Michael donor, that provides routes to the synthesis of new azepinones condensed with the benzo-, naphtho-, and quinolinequinone nucleus.

Results and Discussion.

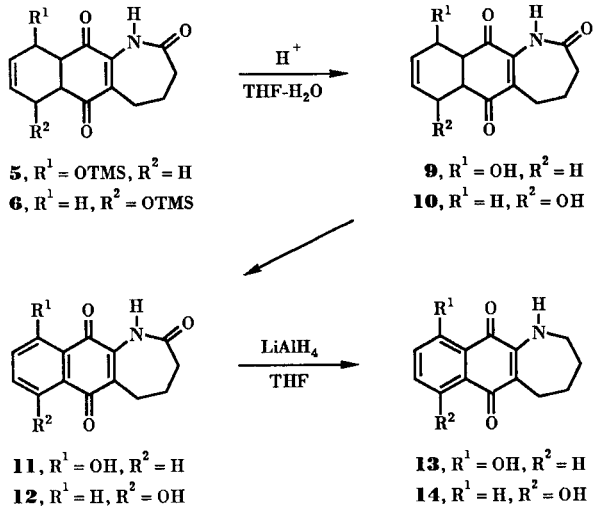
Quinone **1** reacts with cyclopentadiene in benzene solution at room temperature to afford the corresponding cycloadduct **2** in 96% yield. Under similar conditions the treatment of quinone **1** with butadiene furnished adduct **3** in nearly quantitative yield.

We also have investigated the cycloaddition of **1** with (*E*)-1-trimethylsiloxybutadiene **4**. The reaction was conducted in dichloromethane solution at room temperature to give a sole regioisomer **5** or **6** as was observed by tlc and ^{1}H nmr spectra.

It is not easy to establish the structure of the adduct generated in this reaction, but it might by anticipated that product **5** is the most probable reaction product. This assumption is supported by the electron-releasing properties of the acylamino group in **1**, that decrease the electrophilic character of the carbonyl group in C-6, favoring the attack of the nucleophilic terminus of the diene to the 7-position.

Goldstein [7] and Parker [8] have reported useful evidence to correlate the substitution pattern with the chemical shifts of aromatic protons in the nmr spectra of some 1,4-naphthoquinones. It is noteworthy that quinones **7** and **8**, which appeared among the reported examples [8], exhibited a noticeable difference in the chemical shift of the

Scheme 1



5, R^1 = OTMS, R^2 = H
6, R^1 = H, R^2 = OTMS

9, R^1 = OH, R^2 = H
10, R^1 = H, R^2 = OH

11, R^1 = OH, R^2 = H
12, R^1 = H, R^2 = OH

13, R^1 = OH, R^2 = H
14, R^1 = H, R^2 = OH

chelated protons which probably is due to the influence of the nitrogen atom over the donor properties of the carbonyl group at the 4-position.

We have considered that naphthazepine **13** or **14**, which could arise from **5** or **6** through the sequence outlined in Scheme 1, is structurally related with quinone **7** or **8** and therefore the ^{1}H nmr properties of quinones **7** and **8** can be used to establish the structures of **13** or **14** and indirectly the structure of their precursors.

The conversion of adducts **5** or **6** to the corresponding naphthazepinequinones was performed through the synthetic sequence outlined in Scheme 1. Adducts **5** or **6** were reacted with 1.3 *N* hydrochloric acid in aqueous THF to give alcohols **9** or **10** in 99% yield. Then, compounds **9** or **10** were oxidized with pyridinium chlorochromate (PCC) in dichloromethane solution to afford hydroxyquinones **11** or **12** in 96% yield which, by subsequent treatment with lithium aluminium hydride in tetrahydrofuran solution, gives naphthazepinequinones **13** or **14** in 50% yield.

The heterocyclic quinone obtained through the sequence showed the ^{1}H nmr signal of the upfield aromatic proton at δ 7.08 ppm, in good accord with the chemical shift of quinone **8**, in which the aromatic proton at the 6-position appeared at δ 7.16 ppm (**7**, δ 7.23 ppm) [8]. Furthermore, the ^{1}H nmr spectrum of the new quinone displays the chelated proton at δ 11.53 ppm in good agreement with the chemical shift of the chelated proton of **8** that appeared at δ 11.56 ppm (**7**, δ 12.83 ppm) [8]. These facts support structure **13** for the quinone resulting from the synthetic sequence of Scheme 1, and those of its corresponding precursors **5**, **9**, and **11**.

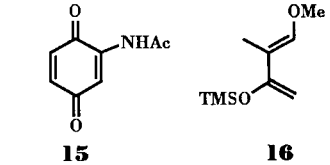
It is noteworthy that quinone **15**, which is structurally related to quinone **1**, reacts with the polarized diene **16** to give, after some transformations, a single naphthoquinone **17** [9]. This behavior explained on the basis of the electron-donating properties of the acylamino group that favored the cycloaddition, is in agreement with our results on the cycloaddition between quinone **1** and diene **4** in which, the control of the cycloaddition is exerted by an acylamino group.

The high regioselectivity observed in the cycloaddition between heterocyclic quinone **1** and diene **4** led us to study the cycloaddition of **1** with methacrolein dimethylhydrazone **18**.

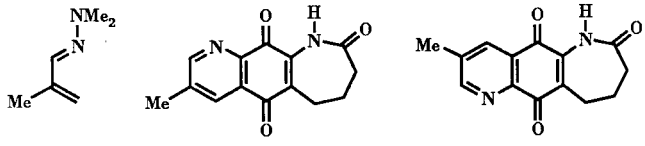
The use of α,β-unsaturated *N,N*-dimethylhydrazones in synthesis of aza-heterocyclic quinones have increased in the last few years [10-15] due to the high reactivity and regioselectivity observed with these 1-azadienes in the Diels-Alder reaction. The polarisation of the 1-azadiene system in these dimethylhydrazones has been firmly established and is atributed to the electron-releasing properties of the dimethylhydrazonyl group. There are no theorical calculations to evaluate the magnitude of the donor effect of this group in the 1-azadiene system however, Potts *et al.* [16] have reported calculations which show that by introducing the dimethylhydrazonyl group into the furan nucleus an increase in its HOMO energy, relative to that of furan and 2-vinylfuran, is observed.

We investigated the Diels-Alder reaction of the quinone **1** with the 1-azadiene **18** which was prepared by condensation of the methacrolein and 1,1-dimethylhydrazine as reported [17]. The reaction conducted in dichloromethane solution at room temperature afforded the azepinoquinolinequinones **19** or **20**, along with a secondary product. The ^{1}H nmr spectrum of the mixture indicated that the minor product is 7- or 8-dimethylaminobenzazepinoquinone **21** or **22** generated, probably, by Michael addition of dimethylamine to quinone **1** under aerobic conditions. The formation of dimethylaminoquinones in the reaction of the 1-azadiene **18** with quinones has also been observed by other authors [14].

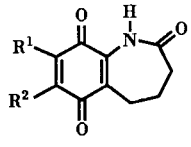
Taking into account the regiochemistry of the cycloaddition of quinone **1** with the polarized diene **4** and the well known directional effect exerted by the dimethylhydrazonyl group in **18**, it is probable that the structure of the



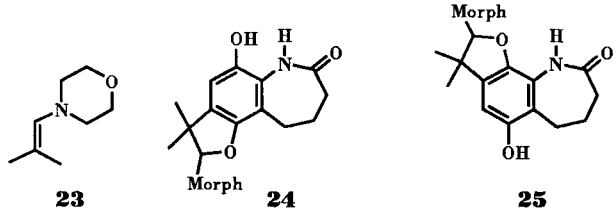
15 **16** **17**



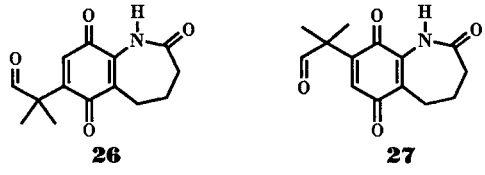
18 **19** **20**



21, $R^1 = H$, $R^2 = NMe_2$
22, $R^1 = NMe_2$, $R^2 = H$



23 **24** **25**



26 **27**

isolated product is **19**. This quinone probably was generated by attack of the nucleophilic terminus of the 1-azadiene to the more electrophilic carbon atom on the 8-position in quinone **1**, followed by elimination and aromatization.

We also investigate the Michael addition of enamine **23** to quinone **1** in order to prepare a functionalized benzazepinetrione. The reaction afforded a sole naphthofuran **24** or **25** as was observed by tlc and ^{1}H nmr spectroscopy in quantitative yield. The heterocycle **24** or **25** was treated with ferric chloride in aqueous solution to give the quinones **26** or **27** in 98% yield. The high regioselectivity observed in the reaction of **1** with the Michael donor **23**, as in the Diels-Alder reaction, probably is due to the control exerted by the acylamino group in **1**. Therefore, in the synthetic sequence described here compounds **24** and **26** are proposed as the most likely.

In conclusion, we have developed routes to the synthesis of azepinones fused to a quinone system by functionalization of the heterocyclic quinone **1**, through cycloaddition and Michael reactions. This study indicates a remarkable regioselectivity of this reactions in which the control of the process is atributed, in part, to the acylamino group of the quinone.

EXPERIMENTAL

Melting points were determined on a Kofler hot-stage apparatus and are uncorrected. The ir spectra were recorded on a Perkin-Elmer 1310 spectrophotometer in potassium bromide discs and the wave numbers are given in cm^{-1}. The ^{1}H nmr spectra were determined on either a Varian XL-100 or a Varian XL-300 spectrometer. The ^{13}C nmr spectra were determined on a Bruker AM-200 spectrometer in deuteriochloroform, unless otherwise stated. Chemical shifts are reported in δ ppm downfield from TMS, and J-values are given in Hertz. Mass spectra were recorded on VB-12-250 spectrometer in the Instituto de Química General (C.S.I.C.), Madrid, Spain. Silica gel Merck 60 (70-230 mesh) and DC-Alufolien $60F_{254}$ were normally used for preparative column and analytical tlc, respectively.

Cycloaddition of Quinone **1** with Cyclopentadiene.

A solution of **1** (65 mg, 0.34 mmole), freshly distilled cyclopentadiene (23 mg, 0.35 mmole) in benzene solution (7 ml) was allowed to stand for 3 hours at room temperature. The solvent was evaporated to afford adduct **2** (87 mg, 99%), mp 128-130° (benzene-ligh petroleum, 1:2); ir: 3210 (N-H), 1650 (C=O), 1600 (C=C); ^{1}H nmr: 300 MHz δ 1.47 (d, 1H, J 8.8), 1.57 (dt, 1H, J 8.8 and 1.8), 1.98 (m, 2H), 2.60 (m, 2H), 2.67 (m, 2H), 3.29 (m, 2H), 3.55 (signal with fine coupling, 2H), 6.04 (m, 2H), 8.10 (s, 1H); ^{13}C nmr: δ 21.47, 26.36, 36.81, 47.11, 48.01, 48.99, 49.49, 132.72, 134.66, 135.69, 140.98, 173.83, 193.57, 197.20.

Anal. Calcd. for $C_{15}H_{15}NO_3$: C, 70.03; H, 5.83; N, 5.45. Found: C, 69.76; H, 5.94; N, 5.82.

Cycloaddition of Quinone **1** with Butadiene.

Through a solution of **1** (60 mg, 0.31 mmole) in benzene (7 ml) in a round bottom flask was bubbled butadiene at room temperature for 5 minutes. The flask was stopped tightly and the mixture was allowed to stand at room temperature for 4 days. Removal of the solvent gave adduct **3** (153 mg, 96%), mp 181-182.5° (benzene-ligh petroleum, 2:1); ir: 3220 (N-H), 1670 (C=O), 1650 (C=O), 1600 (C=C); ^{1}H nmr: 300 MHz δ 2.02 (m, 2H), 2.17-2.30 (m, 2H), 2.42-2.57 (m, 2H), 2.69 (m, 2H), 2.74 (m, 2H), 3.20-3.32 (m, 2H), 5.71 (m, 2H), 8.05 (br s, 1H); ^{13}C nmr: 50 MHz δ 20.38, 24.58, 27.85, 37.20, 44.12, 45.05, 123.98, 124.57, 127.81, 137.22, 174.05, 194.86, 198.41.

Anal. Calcd. for $C_{14}H_{15}NO_3$: C, 68.57; H, 6.12; N, 5.71. Found: C, 68.36; H, 6.10; N, 6.09.

Reaction of Quinone **1** with Diene **4**.

A mixture of quinone **1** (155 mg, 0.81 mmole), diene **4** (115 mg, 0.81 mmole) in benzene (12 ml) was kept at ambient temperature for 2 days. The solvent was evaporated off to yield adduct **5** (267 mg, 99%) as a yellow liquid which solidified on standing; mp 114-115° (benzene-cyclohexane, 1:1); ir: 3240 (N-H), 1680-1640 (C=O), 1600 (C=C), 1240 ($SiMe_3$), 1060 (Si-O-C), 840 (Si-O-C); ^{1}H nmr: 300 MHz δ 1.98-2.24 (m, 3H), 2.61-2.70 (m, 2H), 2.74-2.84 (m, 2H), 3.11 (dd, 1H, J 21 and 4), 3.35 (dd, 1H, J 7 and 4), 3.48 (t, 1H, J 7), 4.46 (m, 1H), 5.80 (m, 2H), 8.12 (br s, 1H); ^{13}C nmr (acetone-d_6): 50 MHz δ 0.10, 22.27, 22.92, 27.47, 37.01, 41.39, 51.18, 65.93, 127.65, 129.77, 133.67, 140.88, 174.33, 195.61, 196.29; ms: m/z (%) 333 (M^+, 7), 318 (17), 243 (52), 142 (100).

Anal. Calcd. for $C_{17}H_{23}NO_4Si$: C, 61.26; H, 6.90; N, 4.20. Found: C, 61.47; H, 6.78; N, 4.45.

Hydrolysis of Adduct **5**.

Compound **5** (268 mg, 0.80 mmole) was dissolved in water-THF (9:1, 25 ml) containing 1.3 *N* hydrochloric acid (1 ml) and the mixture was allowed to stand at room temperature for 1 hour. The mixture was diluted with water (25 ml) and extracted with chloroform (25 ml). The extract was washed with water, dried (magnesium sulfate) and evaporated to afford alcohol **9** (207 mg, 99%), mp 139.5-141° (benzene-ethanol-ligh petroleum, 2:1:1); ir: 3480, 3440 (O-H), 3220 (N-H), 1660 br (C=O); ^{1}H nmr: 300 MHz δ 1.80-2.02 (m, 3H), 2.45 (m, 2H), 2.56 (m, 2H), 2.86 (d, with fine coupling, 1H, J 18), 3.15 (dd, 1H, J 9.5 and 6), 3.25 (t, with fine coupling, 1H, J 8), 4.00 (d, 1H, J 5), 4.20 (m, 1H), 5.70 (m, 2H), 7.82 (s, 1H); ^{13}C nmr: 50 MHz δ 20.98, 23.17, 27.33, 36.99, 42.48, 49.80, 65.51, 127.65, 128.79, 131.02, 139.26, 173.99, 194.86, 197.21; ms: m/z (%) 261 (M^+, 22), 243 (100), 215 (72), 187 (48).

Anal. Calcd. for $C_{14}H_{15}NO_4$: C, 64.36; H, 5.74; N, 5.36. Found: C, 64.15; H, 5.70; N, 5.60.

Hydroxynaphthazepinetrione **11**.

A solution of PCC (250 mg, 1.16 mmoles), anhydrous sodium acetate (125 mg) in dichloromethane (7 ml) was dropwise added to a solution of alcohol **9** (130 mg, 0.5 mmole) in dichloromethane (7 ml) and the mixture was magnetically stirred for 2 hours at room temperature. The mixture was chromatographed on silica gel (dichloromethane) to yield heterocyclic quinone **11** (126 mg, 98%), mp 204-205° (ethanol-benzene, 3:1); ir: 3440 br (O-H), 3220 (N-H), 1690, 1630 (C=O), 1600 (C=C); ^{1}H nmr: 300 MHz δ 2.12 (q, 2H, J 6.8), 2.69 (t, 2H, J 6.8), 2.87 (t, 2H, J 6.8), 7.28 (dd, 1H, J 8 and 1), 7.61 (dd, 1H, J 8), 7.77 (t, 1H, J 8), 8.36 (br s, 1H), 11.43 (br s, 1H); ^{13}C nmr: 50 MHz δ 20.48, 27.15, 37.18, 113.30, 119.47, 123.72, 128.38, 131.99, 137.49, 161.59, 173.92, 183.35, 184.22; ms: m/z (%) 257 (M^+, 100), 228 (30%), 202 (25).

Anal. Calcd. for $C_{14}H_{11}NO_4$: C, 65.36; H, 4.28; N, 5.44. Found: C, 65.47; H, 4.30; N, 5.65.

Hydroxynaphthazepinedione **13**.

To a suspension of lithium aluminum hydride (95 mg, 2.52 mmoles) in anhydrous THF (30 ml) at 0° was dropwise added to a solution of compound **11** (130 mg, 0.51 mmole) in THF (15 ml) under nitrogen atmosphere and the mixture was heated to reflux for 3 hours. The mixture was diluted with water, acidified with acetic acid, and extracted with ethyl ether (2 x 50 ml). The organic layer was washed with saturated sodium bicarbonate solution, water and dried over magnesium sulfate. Removal of the solvent afforded **13** (80 mg, 50%). An analytical sample of **13** was obtained by preparative tlc on silica gel (chloroform), mp 126-127.5°; ir: 3600-3200 (O-H), 3320 (N-H), 1660, 1620 (C=O); ^{1}H nmr: 100 MHz δ 1.86 (m, 4H), 2.87 (m, 2H), 3.46 (m, 2H), 6.00 (br s, 1H), 7.08 (dd, 1H, J 7 and 3), 7.58 (m, 1H), 8.04 (t, 1H, J 7), 11.53 (s, 1H); ms: m/z (%) 243 (M^+, 62), 227 (100), 212 (35).

Anal. Calcd. for $C_{14}H_{13}NO_3$: C, 69.13; H, 5.35; N, 5.76. Found: C, 69.30; H, 5.35; N, 5.48.

Cycloaddition of **1** with Azadiene **18**.

A solution of quinone **1** (127 mg, 0.67 mmole), diene **18** (148 mg, 1.33 mmoles) in dichloromethane (15 ml) was kept at room temperature for 1 hour. Removal of the solvent and excess diene **18** under reduced pressure afforded a mixture of compounds **19** and **21** (^{1}H nmr). Product **19** was isolated from the mixture by column chromatography on silica gel (chloroform-ethyl acetate, 1:1) to give pure **19** (94 mg, 56%), mp >410°; ir: 3600-3300 (N-H), 1610 (C=C), 1690, 1660, 1630 (C=O), 1610 (C=C); ^{1}H nmr: 300 MHz δ 2.11 (m, 2H), 2.54 (s, 3H), 2.77 (t, 2H, J 6.5), 2.95 (t, 2H, J 6.5), 8.21 (d, 1H, J 1.8), 8.52 (br s, 1H, exchangeable with deuterium oxide), 8.82 (d, 1H, J 1.8); ^{13}C nmr: 50 MHz δ 18.99, 20.20, 27.07, 37.26, 126.44, 128.93, 134.21, 137.66, 139.66, 143.89, 155.09, 173.72, 178.56, 183.66; ms: m/z (%) 256 (M^+, 100), 227 (55), 202 (52).

Anal. Calcd. for $C_{14}H_{12}N_2O_3$: C, 65.62; H, 4.69; N, 10.93. Found: C, 66.48; H, 5.32; N, 10.63.

Reaction of Quinone **1** with Isobutenylmorpholine **23**.

A solution of quinone **1** (86 mg, 0.45 mmole) in dichloromethane (10 ml) was dropwise added to a solution of enamine **23** (69 mg, 0.49 mmole) in dichloromethane (5 ml) and the resulting mixture was allowed to stand at room temperature for 1 hour. A white precipitate began to separate after 30 minutes. The mixture was cooled and the precipitate product was collected by filtration to give crude **24** or **25** (149 mg, 100%). An analytical sample of heterocycle **24** or **25** was obtained by column chromatography on silica gel (chloroform-ethyl acetate, 2:1), mp 264-266°; ir: 3600-3000 (O-H and N-H), 1620 (C=O), ^{1}H nmr: 300 MHz δ 1.15 (s, 3H), 1.28 (s, 3H), 2.00-2.20 (m, 4H), 2.40-2.58 (m, 4H), 2.63 (t, 2H, J 7.6), 3.48 (t, 2H, J 4.3), 4.82 (s, 1H), 6.48 (s, 1H), 8.45 (s, 1H), 8.85 (s, 1H); ^{13}C nmr: 50 MHz δ 19.79, 22.44, 26.74, 31.84, 32.91, 43.51, 48.91, 65.96, 106.44, 106.95, 114.94, 125.02, 132.27, 142.56, 148.01, 172.69; ms: m/z (%) 332 (M^+, 100), 317 (12), 162 (81).

Anal. Calcd. for $C_{18}H_{24}N_2O_4$: C, 65.06; H, 7.22; N, 8.43. Found: C, 64.85; H, 7.41; N, 8.25.

Oxidation of Heterocycles **24** or **25** with Ferric Chloride.

To a stirred suspension of compounds **24** or **25** (60 mg, 0.18 mmole) in water (25 ml) was added ferric chloride (130 mg, 0.80 mmole) and the mixture was heated to 40° for 1.5 hours. The resulting mixture was extracted with chloroform and the extract was washed with water and dried over magnesium sulfate. Removal of the solvent gave crude quinone **26** or **27** (48 mg, 98%), mp 132-134° (ethanol); ir: 3200 (N-H), 1720 (C=O), 1680 (C=O), 1650 (C=O), 1630 (C=O), 1600 (C=C); ^{1}H nmr: 300 MHz δ 1.35 (s, 6H), 2.00 (m, 2H), 2.73 (m, 4H), 6.70 (s, 1H), 8.13 (s, 1H), 9.62 (s, 1H); ^{13}C nmr: 50 MHz δ 19.04, 22.33, 27.35, 37.34, 48.36, 123.87, 130.03, 134.68, 154.15, 173.62, 181.76, 186.06, 200.57; ms: m/z (%) 263 (M^++2, 8), 261 (M^+, 5), 233 (100), 205 (21).

Anal. Calcd. for $C_{14}H_{15}NO_4$: C, 64.36; H, 5.74; N, 5.36. Found: C, 64.57; H, 6.00; N, 5.15.

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