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A Versatile Synthesis of 1-Aryl-2-nitro-3-(3,4,5-trimethoxyphenyl)propenes as Precursors of Novel Mescaline Derivatives

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A convenient one-step procedure to synthesize the title compounds (**3a—v**) by the reaction between 1,2,3-trimethoxy-5-(2-nitroethyl)benzene (**1**) and aromatic or heterocyclic aldehydes is reported. The obtained 1-aryl-2-nitro-3-(3,4,5-trimethoxyphenyl)propenes can easily be reduced to provide new mescaline derivatives. As an example, 1-(3-indolyl)-2-amino-3-(3,4,5-trimethoxyphenyl)propane (**4**), which contains in the same molecule the structural features of both mescaline and tryptamine, has been prepared.

Keywords—1-aryl-2-nitro-3-(3,4,5-trimethoxyphenyl)propene; 1,2,3-trimethoxy-5-(2-nitroethyl)benzene; aromatic aldehyde; heterocyclic aldehyde; tryptamine; mescaline

We have recently reported a novel method for synthesizing *o*-hydroxy- β -nitrostyrenes using the system of dimethylammonium chloride–potassium fluoride as the condensing agent.¹⁾ In an extension of our investigations in this field, we have found that these reagents are also efficient for the preparation of the title compounds, which are obviously attractive intermediates for the synthesis of new mescaline derivatives of biological and pharmaceutical interest.

These trimethoxy derivatives belong to an almost unexplored chemical class. Thus, to our knowledge, only one report has appeared in the literature regarding the synthesis of 1,3-diaryl-2-nitropropenes and it only deals with five examples (limited to methoxy and benzyloxy derivatives), the preparation of which is not detailed.²⁾

The procedure we report here proved to be a general and convenient method to prepare 1-aryl-2-nitro-3-(3,4,5-trimethoxyphenyl)propenes (**3a—v**) by the condensation of 1,2,3-trimethoxy-5-(2-nitroethyl)benzene (**1**) and aldehydes **2a—v** in the presence of dialkylammonium chloride and a small amount of potassium fluoride (Chart 1).

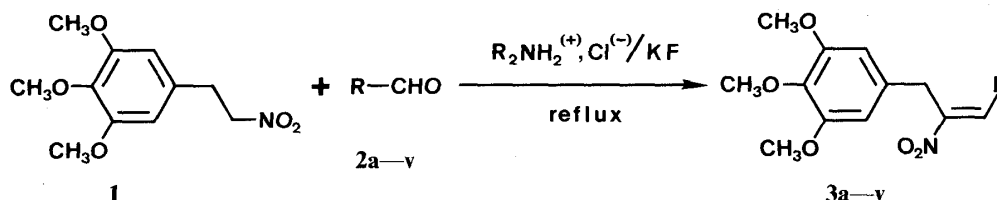


Chart 1

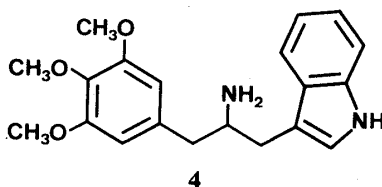
This procedure offers two major advantages: It is applicable to a large range of aldehydes, including compounds bearing sensitive functional groups (Table I); It provides multigram quantities of **3** and allows subsequent transformations on a scale sufficient to provide reasonable amounts of the products for pharmacological tests.

The preparative conditions have been experimentally optimized. We have found that the

best results are obtained by using dimethylammonium chloride in refluxing xylene in most cases (**3a—u**) and di-*n*-butylammonium chloride in refluxing isoamyl acetate in the case of **3v**. In this context, it is worth pointing out that the use of primary amine hydrochlorides lowers the yields because it induces the formation in variable amounts of the corresponding aldimines as by-products.

The products **3a—v** are new compounds. Their structures are well supported by microanalyses and proton nuclear magnetic resonance ($^1\text{H-NMR}$) data. These compounds are clearly the pure *E* isomers (the two aromatic rings are located on the same side of the double bond) on the basis that the ethylenic protons appear at low field. In this respect, comparison with the results of a theoretical study devoted to the $^1\text{H-NMR}$ of the closely related *Z* and *E* α -nitrostilbenes is unambiguous.³⁾

As a preliminary trial we have checked in the particular case of **3v** that these nitropropenes can be easily reduced with lithium aluminum hydride in tetrahydrofuran (THF). The resulting amine **4**, which is a combination of the structures of both mescaline and tryptamine, is however surprisingly devoid of the expected psychotomimetic properties. The lack of activity in this instance does not imply that other mescaline derivatives of the same type must be biologically inactive. Pharmacological tests relating to several amines derived from the nitro compounds **3** are in progress, and the results will be reported in the near future.



Experimental

All melting points are uncorrected. The $^1\text{H-NMR}$ spectra were recorded at 90 MHz using a Varian EM 390 spectrometer. Chemical shifts are given on the δ (ppm) scale with tetramethylsilane (TMS) as an internal standard. The following abbreviations are used: s = singlet, d = doublet, dd = doublet of doublet, m = multiplet, and br = broad.

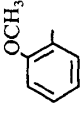
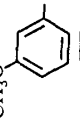
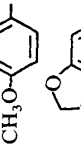
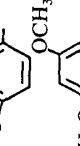
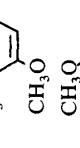
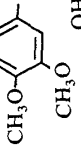
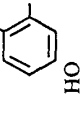
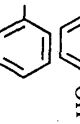
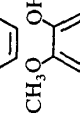
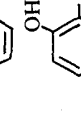
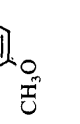
The starting aldehydes **2a—r** and **2v** are commercially available. Compounds **2s** and **2t** were prepared according to a previously reported procedure,⁴⁾ and **2u** was synthesized as described.⁵⁾

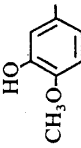
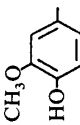
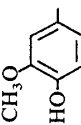
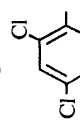
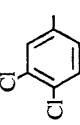
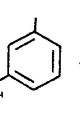
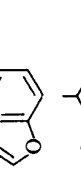
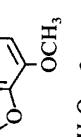
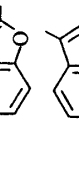
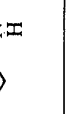
1,2,3-Trimethoxy-5-(2-nitroethyl)benzene (1)—This compound has been synthesized on a preparative scale by adapting a method recently described for work on a reduced scale.⁶⁾ The required large amounts of the starting nitrostyrene were obtained as follows.

1,2,3-Trimethoxy-5-(2-nitroethyl)benzene: The following components were placed in a one-necked 1 l conical flask equipped with a Dean-Stark water trap (capacity about 30 ml): commercial 3,4,5-trimethoxybenzaldehyde (98.1 g, 0.5 ml), dimethylammonium chloride (81.5 g, 1 mol), nitromethane (300 ml), toluene (300 ml), and anhydrous potassium fluoride (4.36 g, 75 mmol). This mixture was vigorously refluxed with stirring for 5 h. The reaction flask was cooled down, then fitted to a rotary evaporator in order to remove the volatiles by gradual heating under reduced pressure. To the tepid residue (*ca.* 55 °C), chloroform (125 ml) and 0.2 N HCl (400 ml) were added. The mixture was heated in the water bath until complete dissolution, then the flask was stored overnight in a refrigerator (–5 °C). A crystalline solid was filtered out by suction, carefully rinsed with water and thoroughly dried in a vacuum oven. The filtrate was poured into a separatory funnel, the layers were separated and the aqueous phase was extracted with chloroform (3 $\times$ 100 ml). The organic extracts were combined then evaporated to give a crude oily material, which was chromatographed over silica gel (400 g, eluent dichloromethane–ethyl acetate, 95 : 5). After removal of the solvents, the resulting solid and the previously separated product were recrystallized together from isopropanol; yield 99.2 g (83%); mp 125.5–126.5 °C (allotropic change at 121–121.5 °C); (lit.,⁷⁾ mp 119–120 °C).

Conversion of 1,2,3-Trimethoxy-5-(2-nitroethyl)benzene to 1: The following components were placed in a 6 l wide-mouthed reaction flask provided with an efficient mechanical stirrer: isopropanol (750 ml), chloroform, (2.5 l) and the above nitrostyrene (47.8 g, 0.2 mol). When the crystals had dissolved completely, silica gel 200–400 mesh ASTM (400 g) was poured into the flask whilst the mixture was continuously stirred vigorously. Sodium borohydride (33.25 g, 0.88 mol) was then added portionwise over a period of 15 min. The slurry was stirred for an additional 2 h, and acetic acid (*ca.* 50 ml) was carefully added. The insoluble material was separated by suction and the filtrate was

TABLE I. 1-Aryl-2-nitro-3-(3,4,5-trimethoxyphenyl)propenes 3 from Aldehydes 2

Product No.	R	Reaction time (h)	Yield (%) ^{a)}	mp (°C) (Solvent)	Formula and Analysis (%)			¹ H-NMR (CDCl ₃ -TMS) δ (ppm)
					Calcd	Found	C H N	
3a		48	88	119—120 (benzene-cyclohexane)	C ₁₉ H ₂₁ NO ₆ 63.50 5.89 (63.32 5.99)	3.90 3.80)		3.77 (s, 6H), 3.80 (s, 3H), 3.87 (s, 3H), 4.11 (s, 2H), 6.37 (s, 2H), 6.77—7.06 (m, 2H), 7.20—7.57 (m, 2H), 8.44 (s, 1H)
3b		48	82	67—68 (ether-pentane)	C ₁₉ H ₂₁ NO ₆ 63.50 5.89 (63.63 5.89)	3.90 3.81)		3.72 (s, 3H), 3.78 (s, 6H), 3.80 (s, 3H), 4.18 (s, 2H), 6.38 (s, 2H), 6.86—7.13 (m, 3H), 7.24—7.48 (m, 1H), 8.22 (s, 1H)
3c		48	75	82—83 (ether-pentane)	C ₁₉ H ₂₁ NO ₆ 63.50 5.89 (63.53 5.79)	3.90 3.87)		3.80 (s, 6H), 3.83 (s, 3H), 3.85 (s, 3H), 4.22 (s, 2H), 6.40 (s, 2H), 6.92 (br d, 2H, J=9 Hz), 7.43 (br d, 2H, J=9 Hz), 8.22 (s, 1H)
3d		48	81	119—120 (isopropanol-isopropyl ether)	C ₁₉ H ₁₉ NO ₇ 61.12 5.13 (61.30 5.17)	3.75 3.66)		3.80 (s, 6H), 3.82 (s, 3H), 4.20 (s, 2H), 5.99 (s, 2H), 6.39 (s, 2H), 6.75—7.12 (m, 3H), 8.22 (s, 1H)
3e		48	87	162.5—163.5 (benzene-isopropanol)	C ₂₁ H ₂₅ NO ₈ 60.14 6.01 (60.01 6.11)	3.34 3.32)		3.45 (s, 3H), 3.78 (br s, 9H), 3.87 (s, 3H), 3.91 (s, 3H), 4.22 (s, 2H), 6.40 (s, 2H), 6.52 (s, 1H), 6.79 (s, 1H), 8.61 (s, 1H)
3f		48	86	148—149 (benzene-isopropanol)	C ₂₁ H ₂₅ NO ₈ 60.14 6.01 (60.26 6.00)	3.34 3.41)		3.70 (s, 6H), 3.80 (br s, 9H), 3.86 (s, 3H), 4.24 (s, 2H), 6.39 (s, 2H), 6.64 (s, 2H), 8.25 (s, 1H)
3g		24	55	140—141 (benzene-cyclohexane)	C ₁₈ H ₁₉ NO ₆ 62.61 5.55 (62.85 5.59)	4.06 3.90)		3.76 (s, 6H), 3.80 (s, 3H), 4.13 (s, 2H), 6.24 (br s, 1H), exchangeable with D ₂ O, 6.38 (s, 2H), 6.78—7.03 (m, 2H), 7.15—7.38 (m, 2H), 8.45 (s, 1H)
3h		24	46	154—155 (trichloroethylene)	C ₁₈ H ₁₉ NO ₆ 62.61 5.55 (62.36 5.58)	4.06 4.04)		3.76 (s, 6H), 3.80 (s, 3H), 4.17 (s, 2H), 5.90—6.12 (1H, exchangeable with D ₂ O), 6.37 (s, 2H), 6.81—7.11 (m, 3H), 7.18—7.45 (m, 1H), 8.18 (s, 1H)
3i		24	63	136.5—137.5 (benzene-cyclohexane)	C ₁₈ H ₁₉ NO ₆ 62.61 5.55 (62.40 5.54)	4.06 4.01)		3.77 (s, 6H), 3.82 (s, 3H), 4.22 (s, 2H), 6.40 (s, 2H), 6.48 (s, 1H, exchangeable with D ₂ O), 6.85 (br d, 2H, J=8.9 Hz), 7.37 (br d, 2H, J=8.9 Hz), 8.29 (s, 1H)
3i		24	87	140—143 (benzene-cyclohexane)	C ₁₉ H ₂₁ NO ₇ 60.79 5.64 (60.99 5.55)	3.73 3.67)		3.77 (s, 6H), 3.79 (s, 3H), 3.89 (s, 3H), 4.14 (s, 2H), 6.11 (s, 1H, exchangeable with D ₂ O), 6.37 (s, 2H), 6.79—7.03 (m, 3H), 8.45 (s, 1H)
3k		24	50	149—150 (trichloroethylene)	C ₁₉ H ₂₁ NO ₇ 60.79 5.64 (60.61 5.76)	3.73 3.56)		3.57 (s, 3H), 3.77 (s, 6H), 3.80 (s, 3H), 5.63—5.90 (1H, exchangeable with D ₂ O), 6.38 (s, 2H), 6.70—6.90 (m, 3H), 8.42 (s, 1H)

3l		24	68	150—150.5 (benzene-cyclohexane)	$C_{19}H_{21}NO_7$ 60.79 5.64 (60.99 5.54 3.65)	3.73 3.80 (s, 6H), 3.83 (s, 3H), 3.92 (s, 3H), 4.22 (s, 2H), 5.73 (s, 1H, exchangeable with D_2O), 6.42 (s, 2H), 6.76—7.16 (m, 3H), 8.21 (s, 1H)
3m		24	73	168—169 (benzene-cyclohexane)	$C_{19}H_{21}NO_7$ 60.79 5.64 (60.91 5.65 3.80)	3.73 3.72 (s, 3H), 3.79 (s, 6H), 3.81 (s, 3H), 4.25 (s, 2H), 5.98 (s, 1H, exchangeable with D_2O), 6.42 (s, 2H), 6.85—7.17 (m, 3H), 8.28 (s, 1H)
3n		24	72	132—133 (benzene-cyclohexane)	$C_{20}H_{23}NO_8$ 59.25 5.72 (59.50 5.73 3.49)	3.46 3.72 (s, 6H), 3.78 (s, 6H), 3.80 (s, 3H), 4.26 (s, 2H), 5.84 (s, 1H, exchangeable with D_2O), 6.42 (s, 2H), 6.68 (s, 2H), 8.28 (s, 1H)
3o		24	42	165—166 (benzene-cyclohexane)	$C_{18}H_{18}BrNO_6$ 50.96 4.28 (51.17 4.16 3.18)	3.30 3.78 (s, 6H), 3.81 (s, 3H), 4.09 (s, 2H), 6.37 (s, 2H), 6.55 (br s, 1H, exchangeable with D_2O), 6.77 (d, 1H, $J=9.4$ Hz), 7.23—7.47 (m, 2H), 8.28 (s, 1H)
3p		48	40	112—112.5 (isopropyl ether-hexane)	$C_{18}H_{17}Cl_2NO_5$ 54.29 4.30 3.52 (54.26 4.27 3.57)	3.52 3.79 (br s, 9H), 4.02 (s, 2H), 6.28 (s, 2H), 7.22—7.38 (m, 2H), 7.47—7.59 (m, 1H), 8.23 (s, 1H)
3q		48	50	122—122.5 (isopropanol- isopropyl ether)	$C_{18}H_{17}Cl_2NO_5$ 54.29 4.30 3.52 (54.45 4.18 3.47)	3.52 3.81 (s, 6H), 3.83 (s, 3H), 4.14 (s, 2H), 6.35 (s, 2H), 7.25 (dd, 1H, $J=2.1, 8.4$ Hz), 7.51 (d, 1H, $J=8.4$ Hz), 7.55 (d, 1H, $J=2.1$ Hz), 8.12 (s, 1H)
3r		48	59	140—141 (benzene-isopropanol)	$C_{18}H_{18}N_2O_7$ 57.75 4.85 7.48 (57.94 4.79 7.42)	7.48 3.81 (s, 9H), 4.19 (s, 2H), 6.40 (s, 2H), 7.51—7.87 (m, 2H), 8.19—8.43 (m, 2H), 8.25 (s, 1H)
3s		48	81	138.5—140 (isopropanol)	$C_{21}H_{21}NO_7$ 63.15 5.30 3.51 (63.39 5.30 3.49)	3.51 3.70 (s, 6H), 3.75 (s, 3H), 3.85 (s, 3H), 3.93 (s, 2H), 6.24 (s, 2H), 6.58—6.70 (m, 1H), 6.95 (d, 1H, $J=9$ Hz), 7.52 (br d, 1H, $J=9$ Hz), 7.68 (d, 1H, $J=2.2$ Hz), 8.30 (s, 1H)
3t		48	75	115.5—116.5 (isopropanol- isopropyl ether)	$C_{21}H_{21}NO_7$ 63.15 5.30 3.51 (63.28 5.36 3.44)	3.51 3.79 (s, 6H), 3.83 (s, 3H), 4.04 (s, 3H), 4.27 (s, 2H), 6.41 (s, 2H), 6.81 (d, 1H, $J=8.4$ Hz), 6.95 (d, 1H, $J=2.4$ Hz), 7.32 (d, 1H, $J=8.4$ Hz), 7.75 (d, 1H, $J=2.4$ Hz), 8.54 (s, 1H)
3u		24	57	137—138.5 (isopropanol- cyclohexane)	$C_{21}H_{21}NO_7$ 63.15 5.30 3.51 (63.20 5.41 3.47)	3.51 3.76 (s, 6H), 3.80 (s, 3H), 3.85 (s, 3H), 4.51 (s, 2H), 6.57 (s, 2H), 6.93—7.18 (m, 3H), 7.40 (br d, 1H, $J=9.9$ Hz), 7.99 (s, 1H)
3v		8	52	191.5—193 (toluene- nitromethane)	$C_{20}H_{20}N_2O_5$ 65.21 5.47 7.60 (65.38 5.45 7.48)	7.60 3.75 (s, 6H), 3.82 (s, 3H), 4.31 (s, 2H), 6.45 (s, 2H), 7.14—7.58 (m, 4H), 7.74—8.00 (m, 1H), 8.81 (s, 1H), 9.05—9.25 (1H, exchangeable with D_2O)

a) Yield of recrystallized product.

evaporated *in vacuo* (the recovered solvents were used to rinse thoroughly the silica gel). The resulting crude material was taken up with dichloromethane (500 ml) and water (300 ml). The organic layer was separated, and the aqueous phase was extracted with dichloromethane (3 × 100 ml). The combined extracts were dried with magnesium sulfate, filtered, then evaporated to dryness. The residue was chromatographed on a silica gel column (400 g, eluent dichloromethane-ethyl acetate, 95:5) to give **1** (46 g), which was further purified by recrystallization (benzene-cyclohexane) to yield 44.6 g (92.5%) as colorless crystals, mp 82–83 °C.

General Procedure for Preparation of 1-Aryl-2-nitro-3-(3,4,5-trimethoxyphenyl)propenes (3a–u)—Dimethylammonium chloride (8.15 g, 0.1 mol), 1,2,3-trimethoxy-5-(2-nitroethyl)benzene (**1**; 12.05 g, 50 mmol), an aldehyde (**2a–u**, 55 mmol), xylene (170 ml) and potassium fluoride (581 mg, 10 mmol) were placed in a one-necked 500 ml conical flask fitted with a Dean-Stark apparatus (capacity about 20 ml). The mixture was vigorously refluxed for the indicated times (Table I) then allowed to cool to room temperature. Direct rotary evaporation of the xylene from the reaction vessel under reduced pressure yielded a crude residue which was taken up with chloroform (250 ml) and 0.5 N HCl (125 ml). This mixture was filtered through a sintered Büchner filter, and insoluble material, if present (e.g. with **3g–o**), was thoroughly rinsed with hot chloroform. The organic layer was separated, and the aqueous phase was extracted with chloroform (3 × 75 ml). The organic extracts were combined, dried with magnesium sulfate, filtered, then evaporated. The resulting material was column-chromatographed on silica gel (400 g, eluent dichloromethane-ethyl acetate, 95:5 for **3a–d** and **3p–u**, 90:10 for **3e–o**). Removal of the solvents followed by recrystallization gave analytically pure **3a–u** in the yields shown in Table I.

In the cases of **3i** and **3n**, the chromatographed product was contaminated with unreacted starting aldehyde as judged by NMR spectroscopy, and the following purification process was applied before recrystallization. The impure material was dissolved in dichloromethane (250 ml), then an aqueous 50% solution of sodium hydrogen sulfite (100 ml) was added. The mixture was efficiently stirred for 2 h and then allowed to stand overnight at room temperature. The organic layer was separated, and the aqueous phase was extracted with dichloromethane (3 × 40 ml). The combined organic extracts were washed with water (3 × 50 ml) then dried with magnesium sulfate. Filtration followed by removal of the solvent provided aldehyde-free products.

In some other cases (**3o–r**), the recrystallization was hindered by the presence of a sticky impurity. In these instances, it proved efficient to pool the chromatographed fractions containing the desired product and to stir them with activated carbon for 2 h. After filtration through a short pad of Celite, the solvents were distilled off to yield a material that could be recrystallized under proper conditions.

1-(3-Indolyl)-2-amino-3-(3,4,5-trimethoxyphenyl)propene (3v)—Di-*n*-butylammonium chloride (16.55 g, 0.1 mol), 1,2,3-trimethoxy-5-(2-nitroethyl)benzene (**1**; 12.05 g, 50 mmol), commercial indole-3-carboxaldehyde (**2v**; 8.0 g, 55 mmol), isoamyl acetate (170 ml) and potassium fluoride (581 mg, 10 mmol) were placed in a one-necked 500 ml conical flask equipped with a Dean-Stark water trap (capacity about 20 ml). This mixture was refluxed for 8 h, then allowed to cool to about 60 °C, and 0.5 N HCl (125 ml) was added. The flask was afterwards allowed to stand overnight in a refrigerator (–5 °C). A solid was filtered out and the filtrate was decanted off. The aqueous layer was extracted with ethyl acetate (3 × 100 ml). The combined organic extracts were washed with water (100 ml), dried over magnesium sulfate and then concentrated *in vacuo*. The residual material and the previously filtered solid were combined and taken up with a mixture of dichloromethane-acetone, 97:3 (55 ml). This suspension was refluxed with stirring for 10 min, then allowed to cool in a refrigerator overnight. A powder was filtered off and rinsed with small portions of benzene. The filtrate was evaporated to leave a material which was chromatographed on silica gel (350 g, eluent dichloromethane-acetone, 94:6). After removal of the solvents, the resulting solid and the previously separated product were recrystallized together from toluene-nitromethane to yield 9.6 g (52%) of **3v** as orange crystals, mp 191.5–193 °C.

1-(3-Indolyl)-2-amino-3-(3,4,5-trimethoxyphenyl)propane (4)—A solution of **3v** (9.2 g, 25 mmol) in dry THF (100 ml) was added dropwise to a refluxing suspension of lithium aluminium hydride (4.95 g, 0.13 mol) in THF (100 ml) under an inert atmosphere. The mixture was refluxed for 3 h after the addition was completed, then cooled to –12 °C using an ice-salt bath. Water (30 ml) then 12.5 N NaOH (10 ml) were successively added with caution. The mixture was allowed to warm to room temperature. A precipitate was filtered off by suction, then thoroughly rinsed with several portions of THF. After removal of the solvent *in vacuo*, the residue was taken up with dichloromethane (250 ml) and water (75 ml). The organic layer was separated and the aqueous phase was extracted with dichloromethane (3 × 40 ml). The combined extracts were dried with potassium carbonate, then evaporated to dryness. The crude product was flash-chromatographed on silica gel (100 g, eluent ethyl acetate then methanol) and the fractions containing the desired product were pooled then stirred with activated charcoal for 2 h. After filtration through a short pad of Celite, removal of the solvents gave an oil, which was triturated with hot ether (130 ml). The mixture was allowed to stand overnight in a refrigerator, then filtered to yield **4** (5.45 g, 64%) as a pale yellow powder. This product was satisfactorily pure as judged by NMR spectroscopy and microanalytical results, mp 118–122 °C. ¹H-NMR (CDCl₃) δ: 1.64 (br s, 2H, exchangeable with D₂O), 2.36–3.19 (m, 4H), 3.23–3.56 (m, 1H), 3.82 (s, 9H), 6.43 (s, 2H), 6.97–7.43 (m, 4H), 7.50–7.72 (m, 1H), 8.49 (br s, 1H, exchangeable with D₂O). *Anal.* Calcd for C₂₀H₂₄N₂O₃: C, 70.57; H, 7.11; N, 8.23. Found: C, 70.31; H, 7.13; N, 8.05. Hydrochloride: mp 230–232 °C (from acetonitrile-ethanol).

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