yields (68, 62, and 72%) of monoadduct, which consisted solely of α-isomer **3d** (^{1}H and ^{13}C NMR). Because of its high regioselectivity, EDAA should be very useful for α-substitution of pyrroles.

Experimental Section

General Procedures. Infrared (IR) spectra were recorded on Perkin-Elmer 727B or 283 spectrophotometers. Proton NMR spectra were determined on a Varian EM-360 (60 MHz) or Perkin-Elmer R32 (90 MHz) instrument using $CDCl_3$ as solvent and Me_4Si as an internal reference (20 mg of compound in 0.3 mL of solvent).[1b] Carbon-13 NMR spectra were obtained on a JEOL FX60Q spectrometer (15.00 MHz) in $CDCl_3$ with Me_4Si as an internal reference (150 mg of compound in 2 mL of solvent). Both proton noise-decoupled and off-resonance decoupled ^{13}C NMR spectra were recorded; only noise-decoupled data are presented. GLC analyses were performed on a Perkin-Elmer 3920B instrument with a flame-ionization detector, equipped with a Hewlett-Packard 3352B data system and 18652A A/D converter; a 6 ft × $^1/_8$ in. glass 1.35% OV-17 on Chromosorb W AW/DMS (100–120 mesh) column was employed. GLC–mass spectra (electron impact) were obtained on a Hitachi Perkin-Elmer RMU-6E instrument at an ionizing voltage of 70 eV. GLC/MS exact mass measurements were performed on a VG Micromass 7035 spectrometer at 70 eV. All reactions with diazo compounds were conducted under a dry nitrogen atmosphere.

General Results of *N*-Methylpyrrole and DMDM. *N*-Methylpyrrole (1.21 g, 15 mmol; commercial material from Aldrich Chemical Co. was distilled from CaH_2 and stored at −20 °C) and metal-promoting agents (Table I) were placed in a flask and heated at 70 °C in an oil bath with stirring. Two drops of dimethyl diazomalonate[11] solution (0.93 g in 200 μL of CH_2Cl_2,[12] 5 mmol) were added. If nitrogen evolution began within 0–2 min, the remainder of the DMDM solution was added drop by drop over about 5 min at 70 °C. Otherwise, the temperature was slowly increased (110 °C maximum) until nitrogen evolution was initiated and then the remainder of the DMDM was added drop by drop at the initiation temperature. After 50–60 min of additional heating, the reaction was concentrated, and the residue was distilled on a Kugelrohr apparatus (ca. 90 °C at 0.1 torr) to give a pale yellow liquid. Specific experimental results are collected in Table I. In some of the reactions (entries 1–5) small amounts (1–3%) of **3c** were detected by GLC (confirmed by coinjection of authentic **3c**[7] and GLC/MS), in addition to the malonate adducts. Malonates **3b** and **4b** were first identified by GLC/MS. The isomers showed virtually identical mass spectra with major peaks at m/z 211 ($M^{+\cdot}$), 179 (M − CH_3O, H), 152 (M − $COOCH_3$, base peak), 125, 121 (M − $COOCH_3$, OCH_3), 108, 94, 93 (M − $2COOCH_3$), 92, 65, 44, 42, 39; exact mass, m/z 211.0835 (found for $C_{10}H_{13}NO_4$, 211.0840), 121.0478 (found for C_7H_9NO, 121.0503). Spectral data for a mixture of **3b** and **4b** in an 8:1 GLC ratio: IR (neat) ν_{max} 2960, 1743 (C=O), 1496, 1438, 1300, 1244, 1152, 1027, 718 cm^{-1}; ^{1}H NMR δ 3.55 (s, 3, NCH_3), 3.74 (s, 6, OCH_3), 4.73 (s, 1, CH), 6.0–6.25 (m, 1.90, β pyrrole H), 6.5–6.7 (m, 1.12, α pyrrole H); the ^{1}H NMR β-proton/α-proton integral ratio of 1.70 (= 1.90/1.12) corresponds to the GLC α/β isomer ratio of 8.0:1 [β/α proton ratio = (mole fraction of α isomer + 1)/(mole fraction of β isomer + 1) = 1.889/1.111 = 1.70]; ^{13}C NMR for **3b**, δ 34.2 (NCH_3), 50.2 (CH), 52.9 (OCH_3), 107.3 (C_3), 109.9 (C_4), 123.0 (C_2), 123.8 (C_5), 167.8 (CO); for **4b**, δ 36.2 (NCH_3), 50.7 (CH), 52.6 (OCH_3), 108.8 (C_4), 114.4 (C_3), 121.1 (C_2), 122.0 (C_5), 169.3 (CO). Anal. Calcd for $C_{10}H_{13}NO_4$: C, 56.87; H, 6.20; N, 6.63. Found (mixture of **3b** and **4b**): C, 56.82; H, 6.25; N, 6.60.

Conversion of 3b and 4b to 3c and 4c.[4a] A mixture of **3b** and **4b** from the $Cu(acac)_2$ reaction (148 mg, 0.7 mmol, **3b**/**4b** = 7.3:1) was combined with LiCl (60 mg, 0.4 mmol), 13 μL of water, and 1 mL of dimethylformamide. The mixture was heated at reflux for 1 h, cooled, diluted with brine, and extracted with CCl_4. The organic extract was dried (Na_2SO_4) and concentrated. Distillation by Kugelrohr gave 50 mg (47%) of colorless oil, almost entirely composed of **3c** and **4c** in a 6.5:1 ratio (GLC/MS).[11]

General Reaction of *N*-Methylpyrrole and EDAA. *N*-Methylpyrrole (1.21 g, 15 mmol) was reacted with ethyl 2-diazoacetoacetate[13] (0.905 g, 5 mmol) according to the DMDM procedure, using $Cu(hfacac)_2$ (28 mg, 0.05 mmol), $Cu(IPSAL)_2$ (30 mg, 0.05 mmol), or $Rh_2(OAc)_4$ (11 mg, 0.025 mmol) (at 75, 90, and 70 °C, respectively). GLC analyses of the yellow, oily distillates were erratic, probably due to thermal decomposition; GLC/MS was thus precluded. Spectral data were collected on the $Cu(hfacac)_2$ reaction product: direct-inlet MS showed major peaks at m/z 209 ($M^{+\cdot}$), 167, 166 (M − $COCH_3$), 163 (M − OC_2H_5), 138, 121 (M − CH_3, $COOC_2H_5$; base peak), 94, 93; IR (neat) ν_{max} 2988, 1721, 1646, 1616, 1334, 1253, 1228, 1090, 709 cm^{-1}; ^{1}H NMR δ 1.21 (t, 3, OCH_2CH_3), 1.86 (s, 2.53, enol CH_3C=), 2.18 (s, 0.50, keto CH_3CO), 3.38 (s, 2.40, enol NCH_3), 3.52 (s, 0.65, keto NCH_3), 4.24 (q, 2, OCH_2), 4.68 (s, 0.26, keto CH), 5.85–6.15 (m, 1.87, β pyrrole H), 6.6–6.7 (m, 0.94, α pyrrole H), 13.23 (s, 0.78, enol OH); the ^{1}H NMR β-proton/α-proton integral ratio of 1.99 indicates that virtually no β isomer, **4d**, is present (limit of detection is ca. 5%),[14] and ^{1}H NMR indicates an ca. 4:1 enol/keto ratio; ^{13}C NMR δ 14.1 (keto CH_2CH_3), 14.4 (enol CH_2CH_3), 19.9 (=CCH_3), 28.5 ($COCH_3$), 33.7 (NCH_3), 58.6 (keto CH), 60.7 (enol OCH_2), 61.8 (keto OCH_2), 95.0 (=COH), 107.0 (enol C_3), 107.7 (keto C_3), 109.9 (keto C_4), 110.1 (enol C_4), 121.7 (enol C_5), 123.9 (keto C_5), 126.3 (enol C_2), keto C_2 not observed, 172.9/177.7 ($CO_2C_2H_5$, 2:1 intensity ratio, *E* and *Z* enol double bond isomers), $C(O)CH_3$ was not observed. Anal. Calcd for $C_{11}H_{15}NO_3$: C, 63.14; H, 7.23; N, 6.69. Found: C, 63.09; H, 7.27; N, 6.70. ^{1}H NMR spectra for the products from the $Cu(IPSAL)_2$ and $Rh_2(OAc)_4$ reactions were virtually identical with spectra for the $Cu(hfacac)_2$ reaction product.

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Registry No. 3a, 49669-45-6; **3b**, 81643-08-5; **3c**, 51856-79-2; **3d**, 81643-09-6; **4a**, 62822-16-6; **4b**, 81643-10-9; **4c**, 81643-11-0; *N*-methylpyrrole, 96-54-8; DMDM, 6773-29-1; EDAA, 2009-97-4.

(11) Peace, B. W.; Carman, F. C.; Wulfman, D. S. *Synthesis* **1971**, 658.
(12) The small amount of CH_2Cl_2 was used to facilitate slow addition of the DMDM. The DMDM may be added undiluted or dissolved in a small amount (200 μL) of *N*-methylpyrrole, which is already present in excess. (The excess *N*-methylpyrrole serves as a solvent and minimizes formation of bisadducts.)
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(14) ^{1}H NMR spectra of the products from each of the catalysts tested were identical, indicating a regioselectivity for **3d** of ≥95%.

Mono- and Bisdiazotization of Proflavine

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In the course of developing photoaffinity probes of biologically active compounds, we undertook the synthesis of the monoazido analogue, **4**, of proflavine (**1**) by selective diazotization and appropriate substitution of the diazonium salt. An early claim[1] to the preparation of the monodiazonium salt **2** and its subsequent reduction in ethanol to yield 3-aminoacridine (**6**) has been disputed.[2] Other workers have prepared the monodiazonium salt by first monoacetylation and then diazotization.[3] We report that the major product from the diazotization of proflavine

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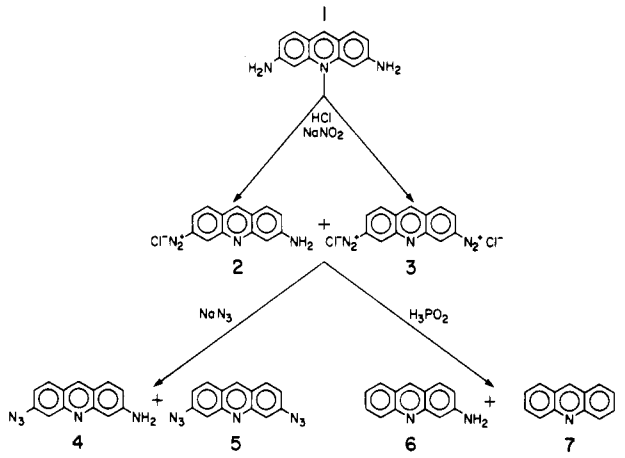
0022-3263/82/1947-3002$01.25/0

Scheme I



is indeed the monodiazonium salt **2**, with the bisdiazonium derivative **3** making up a small fraction of the product (Scheme I). Samples of 3-aminoacridine (**6**) and 3-amino-6-azidoacridine (**4**) have been prepared from proflavine, and their structures have been confirmed by mass spectrometry and NMR and IR spectroscopy and by comparison with authentic samples. In each case, the monosubstituted derivative was the major product with less than 15% of the disubstituted analogues, i.e., acridine (**7**) and 3,6-diazidoacridine (**5**), being formed.

When proflavine was diazotized with 4 equiv of nitrous acid, followed by the addition of 6 equiv of sodium azide, two major products, **4** (the monoazide) and **5** (the diazide), and some unreacted proflavine could be detected by chromatography on silica gel. The IR spectra of compounds **4** and **5** showed the characteristic azido group absorption at 2100 cm^{-1}. Product **4** could be diazotized again and coupled to β-naphthol to give a cerise-colored solution, indicating the presence of a primary amino group.

When the diazonium and bisdiazonium salts of proflavine were subsequently reacted with 6 equiv of hypophosphorous acid, again two major products, **6** and **7**, were found. The color, fluorescence, and R_f values on TLC of compounds **6** and **7** were compared with those of authentic samples of acridine (**7**) obtained commercially and 3-aminoacridine (**6**) synthesized by established procedures,[4] thus confirming the identities of **6** and **7**. Also IR and NMR spectroscopy and mass spectrometry conclusively established the chemical structures of **6** as 3-aminoacridine and of **7** as acridine. Monoacetylation of **1**, followed by diazotization, reduction, and hydrolysis did not improve the yield of **6**. Clearly, the acetylation step reported by other workers[3] is unnecessary.

Experimental Section

General Methods. IR spectra were performed on a Perkin-Elmer Model 221 grating infrared spectrometer; mass spectra were determined on a Hewlett-Packard Model 5985 GC/MS spectrometer by direct insertion; ^1H NMR spectra were run in $CDCl_3$ on a Varian 220-MHz instrument with Me_4Si as the internal standard; melting points were determined on an Electrothermal melting point apparatus. Thin-layer chromatography was carried out on 20 × 20 cm plates by using silica gel "G" (Merck), and column chromatography was performed with 40 × 2.5 cm glass columns packed with cation-exchange (carboxymethyl)cellulose (Cellex CM, Bio-Rad). Visualization of compounds during and after chromatography was effected by UV and visible light.

Preparation of 3-Amino-6-azidoacridine (4) and 3,6-Diazidoacridine (5). All steps involved in the synthesis of the azido compounds were carried out under photographic safelights since the azides were photodegradable. Proflavine hydrochloride (0.5 g, 2 mmol) was added to 50 mL of 1 N HCl and cooled to 0 °C in an ice bath. Sodium nitrite (0.6 g, 8 mmol) dissolved in 5 mL of 1 N HCl (0 °C) was added dropwise to the proflavine mixture and allowed to react 1 min. Sodium azide (0.78 g, 12 mmol) dissolved in 5 mL of 1 N HCl (0 °C) was then added to the diazotized solution. The reaction was allowed to continue in the cold for 30 min. The acridine-free bases were precipitated at pH 10 with NaOH, and the filtered precipitate was redissolved in dilute acid (HCl). The compound mixture was then chromatographed on (carboxymethyl)cellulose columns (Bio-Rad Cellex CM) with an H_2O–HCl gradient (pH 3.5–2.0). Three major bands were detected with UV and visible light. A pale yellow band, the diazide **5**, was the first to elute and displayed little fluorescence. The monoazide **4**, a yellow band with bright yellow fluorescence, was next. An orange band, unreacted proflavine (**1**), fluoresced brilliant yellow-green and was last to elute. Each band was taken individually, with the diazide **5** eluting at pH 3.2, the monoazide **4** at pH 2.6, and the proflavine (**1**) at pH 2.0. The eluants were lyophilized and were determined to be pure by thin-layer chromatography with a benzene–methanol mixture (5:2): R_f for proflavine (**1**) was 0.2; R_f for the monoazide **4** was 0.5; R_f for the diazide **5** was 0.9. Samples of each band were collected and irradiated with UV light to determine the photostability of the compounds. An aqueous solution of **1** remained unchanged while solutions of **4** and **5** turned brown upon irradiation and fluoresced violet and pale green, respectively. Because of the photosensitivity and heat lability of the azido derivatives **4** and **5**, they could not be accurately characterized by elemental analysis. Furthermore, all storage and experimental handling of these compounds by necessity was performed in the dark. The products were intially isolated as the hydrochloride salts, but the free bases were obtained by titration with NaOH, lyophilization, and selective extraction with anhydrous ethanol. For **1**: 50 mg (10%); mp 285–287 °C. For **4**: 350 mg (62.3%); mp 157–159 °C dec; IR (KBr) 3500 (m), 3450 (m), 2400 (s), 1610 (s), 1475 (s), 1300 (s) cm^{-1}; mass spectrum, m/e 235 (M^+), 207, 179, 152; NMR ($CDCl_3$) δ 4.29 (br s, 2, NH_2), 6.98, 7.02 (2 overlapping d, 2, C_2H and C_7H, J = 9.0, 9.0 Hz), 7.18 (s, 1 , C_4H), 7.70 (3, 1, C_5H), 7.76 (d, 1, C_1H, J = 9.0 Hz), 7.84 (d, 1, C_8H, J = 9.0 Hz), 8.48 (s, 1, C_9H). For **5**: 80 mg (12.8%); mp 168–169 °C dec; IR (KBr) 2100 (s), 1610 (m), 1450 (s) cm^{-1}; mass spectrum, m/e 261 (M^+), 233, 205, 179, 152; NMR ($CDCl_3$) δ 7.15 (dd, 2, C_2H and C_7H, J = 9.0, 2.1 Hz), 7.78 (d, 2, C_4H and C_5H, J = 2.1 Hz), 7.93 (d, 2, C_1H and C_8H, J = 9.0 Hz), 8.66 (s, 1, C_9H).

Preparation of 3-Aminoacridine (6) and Acridine (7). Diazotization of proflavine was performed as above. To the diazotization mixture was added 1 mL of 48% hypophosphorous acid (12 mmol), and the reaction mixture was allowed to stand for 48 h at 0 °C. The acridine-free bases were precipitated with NaOH. The precipitate was redissolved in pH 4.0 H_2O (HCl), and chromatographed on (carboxymethyl)cellulose cation-exchange columns eluted over a pH range of 3.2–2.0. Three bands were observed. A pale yellow band, **7**, with a violet fluorescence was first to elute from the column. 3-Aminoacridine (**6**) was next and appeared as a yellow band with a bright yellow fluorescence. Proflavine (**1**), and orange band with brilliant yellow-green fluorescence, again eluted last. The three bands were collected separately and were lyophilized. Their purity was checked by TLC chromatography on silica gel plates with 20% methanol–benzene: R_f of proflavine (**1**) was 0.1, R_f of 3-aminoacridine (**6**) was 0.3, and R_f of acridine (**7**) was 0.7. The hydrochlorides were obtained from the lyophilized fractions, but the free bases could be formed by back-titration with NaOH, lyophilization, and selective extraction with anhydrous ethanol to give **1** [42 mg (8%); mp 285–287 °C], **6** [266 mg (57.4%); mp 218–219 °C; IR (KBr) 3350 (m), 1650 (s), 1500 (s), 1390 (m), 745 cm^{-1}; mass spectrum, m/e 194 (M^+), 167, 140], and **7** [46 mg (10.7%); mp 110–111 °C; IR (KBr) 2950 (m), 1640 (m), 1610 (s), 750 (m), 730 (m) cm^{-1}; mass spectrum, m/e 179 (M^+), 152, 80]. Alternatively, **6** was prepared by condensation of 2-chloro-4-nitrobenzoic acid (50 g, 0.25 mol) with aniline (35 g, 0.37 mol) in n-amyl alcohol. The yellow product was recrystallized from acetic acid to give (2-carboxy-5-nitrophenyl)phenylamine (27 g, 0.11 mol). Ring closure with phosphorus oxychloride gave 3-nitroacridone which was reduced to

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6 with sodium amalgam.[4] Compound 6 prepared from the diazotization of proflavine and 3-aminoacridine (6) synthesized by the above published synthetic route[4] were found to be identical by IR and NMR spectroscopy, mass spectrometry, and TLC criteria.

In addition, compound 7 was compared to an authentic sample of acridine (Aldrich), and they were found to be indistinguishable from one another by IR and NMR spectroscopy, mass spectrometry, and TLC chromatography.

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Registry No. 1, 92-62-6; 1·HCl, 7459-75-8; 2, 81771-14-4; 3, 81771-15-5; 4, 78276-16-1; 4·HCl, 81771-16-6; 5, 57459-61-7; 5·HCl, 81771-17-7; 6, 581-29-3; 6·HCl, 81771-18-8; 7, 260-94-6; 7·HCl, 17784-47-3; 2-chloro-4-nitrobenzoic acid, 99-60-5; aniline, 62-53-3; *N*-(2-carboxy-5-nitrophenyl)phenylamine, 49551-01-1.

Synthesis of 15-(*p*-Iodophenyl)-6-tellurapentadecanoic Acid: A New Myocardial Imaging Agent

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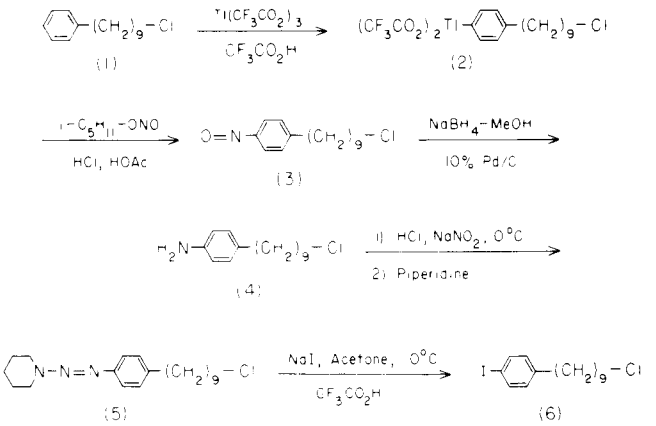
Radiolabeled long-chain fatty acids have important applications in nuclear medicine for the diagnosis of heart disease and can be used to delineate regions of abnormal fatty acid metabolism within the myocardium.[1] The most extensively investigated agents of this type are the terminal ^{123}I-labeled long-chain fatty acids.[2-4] The widespread clinical use of these agents, however, appears to be limited because of the significant in vivo deiodination and the short myocardial residence time. Recently, a new class of agents has been developed by Knapp et al. in which the tellurium-123m isotope has been incorporated into long-chain fatty acids.[5] Animal studies with tellurium-123m-labeled 9-telluraheptadecanoic acid (9-THDA) have demonstrated rapid myocardial concentration and the retention of significant levels of radioactivity in the heart.[6,7] The problems associated with rapid myocardial "washout" have thus been overcome with this unique agent, and introduction of the tellurium heteroatom within the fatty acid chain appears to be an effective means of trapping the fatty acid in the myocardium.

Because of the more attractive physical properties of iodine-123 ($T_{1/2}$ = 13.2 h), a method has recently been developed for the synthesis of 17-iodo-9-telluraheptadecanoic acid (17-I-9-THDA).[8] This agent shows rapid in vivo deiodination, and a variety of methods have thus been considered for chemically stabilizing the iodine on the tellurium fatty acid. One approach is the attachment of the *p*-iodophenyl moiety to the tellurium fatty acid, since studies by other workers have demonstrated the pronounced heart uptake and in vivo stability of radioiodinated 15-(*p*-iodophenyl)pentadecanoic acid.[9] These results suggest the radioiodinated *p*-iodophenyl fatty acids containing stable tellurium may exhibit the pronounced uptake and the unique prolonged myocardial retention

Scheme I



demonstrated with 9-THDA. Because of the potential clinical importance of using the trapping phenomenon of radioiodinated tellurium fatty acids to diagnose heart disease, we have developed a general synthetic method for the preparation of *p*-iodophenyl tellurium fatty acids. In this paper we describe the synthesis of 15-(*p*-iodophenyl)-6-tellurapentadecanoic acid as a model agent.

Discussion

We chose 1-chloro-9-phenylnonane (1) as the starting material on the bases of its commercial availability and the length of the primary alkyl halide chain. Aromatic thallation[10] of compound 1 with thallium(III) trifluoroacetate gave the organothallium intermediate 1-chloro-9-[*p*-[bis(trifluoroacetyl)thallium]phenyl]nonane (2, Scheme I). Following removal of the trifluoroacetic acid by vacuum distillation, the arylthallium compound 2 was treated with nitrosyl chloride[11] which was generated by in situ treatment of isoamyl nitrite in $CHCl_3$ with HCl–HOAc. The 1-chloro-9-(*p*-nitrosophenyl)nonane (3) was obtained in 70% yield based on compound 1. The NMR spectrum of compound 3 exhibited a two-doublet pattern (AA'BB') centered at δ 7.60 (J = 6.6 Hz) for the aromatic protons. The infrared spectrum of compound 3 showed intense bands at 1510 and 840 cm^{-1} which are characteristic frequencies for ArN=O and para-disubstituted benzenes, respectively. Reduction of nitroso compound 3 with $NaBH_4$ and 10% Pd/C in MeOH gave 1-chloro-9-(*p*-aminophenyl)nonane (4) in excellent yield. The infrared spectrum of compound 4 exhibited the characteristic asymmetric N–H stretching frequency at 3450 cm^{-1} and the symmetric stretching frequency at 3380 cm^{-1} observed for primary aromatic amines. The NMR spectrum of compound 4 displayed triplets at δ 2.45 and 3.50 corre-

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