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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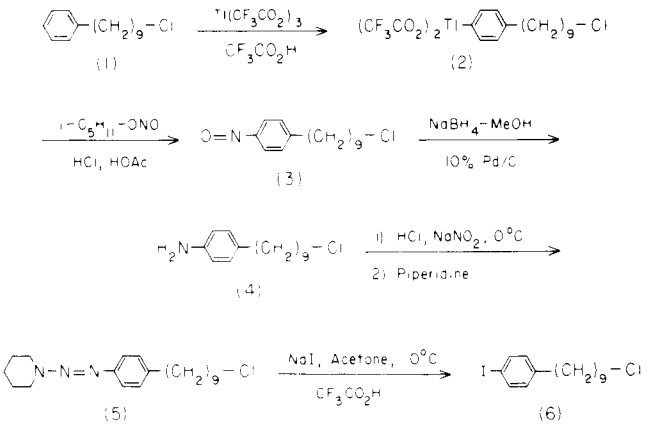
Radiolabeled long-chain fatty acids have important applications in nuclear medicine for the diagnosis of heart disease and can be used to delineate regions of abnormal fatty acid metabolism within the myocardium.[1] The most extensively investigated agents of this type are the terminal ^{123}I-labeled 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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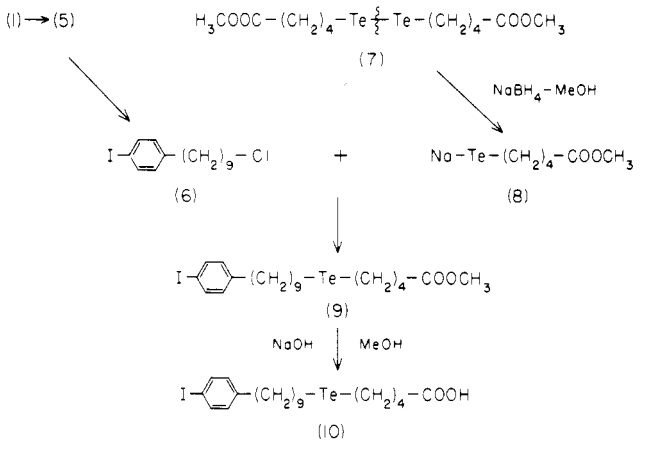
Scheme II



sponding to the C-9 and C-1 methylene protons, respectively, and the AA'BB' pattern centered at δ 6.8 (J = 7 Hz) for the aromatic protons.

Although the Sandmeyer reaction[12] is a commonly used multistep procedure for the introduction of radioiodide into aromatic molecules, it is not conducive for the facile preparation of high specific activity (Curies/millimole) iodinated aromatics. Recently, a simple and rapid method has been reported[13-16] for the introduction of halogens into aromatic substrates by the decomposition of aryltriazene intermediates. This method was first reported for aryl halogenation in 1886 by Wallach.[17] On application of this approach, treatment of 1-chloro-9-(*p*-aminophenyl)nonane (**4**) at 0 °C with nitrous acid followed by aqueous piperidine and subsequent purification by adsorption chromatography gave 1-[4-(9-chlorononyl)phenyl]-3,3-(1,5-pentanediyl)triazene (**5**) in 40% yield. The NMR spectrum showed characteristic multiplets at δ 1.63 and 3.70 for the piperidinyl protons and an AA'BB' pattern centered at δ 7.2 (J = 8 Hz) for the aromatic protons which is consistent with the assigned structure. On employment of a modification of a recently published procedure,[15] compound **5** was treated with sodium iodide and trifluoroacetic acid. After purification by column chromatography, 1-chloro-9-(*p*-iodophenyl)nonane (**6**) was obtained in 60% yield. The *p*-iodophenyl chloride **6** was characterized by IR, low-resolution mass spectrometry, ^{1}H NMR, and elemental analyses. The structure assignment was further confirmed by the preparation of compound **6** by a direct route involving the displacement of the thallium ditrifluoroacetate moiety of intermediate **2** with aqueous potassium iodide.[18] The products from both synthetic routes possessed identical physical and spectral properties.[19]

The synthesis of 15-(*p*-iodophenyl)-6-tellurapentadecanoic acid (**10**) was achieved by employing a method recently developed[8] for the synthesis of terminal halogenated tellurium fatty acids. In this approach (Scheme II) the (*p*-iodophenyl)alkyl chloride **6** was coupled with sodium (methylvaleryl)telluride (**8**) to afford methyl-15-(*p*-iodophenyl)-6-tellurapentadecanoate (**9**) in 90% yield. Hydrolysis of compound **9** gave the desired 15-(*p*-iodophenyl)-6-tellurapentadecanoic acid (**10**) in 65% yield. The spectral properties, elemental analyses, and chromatographic properties substantiated the structures of compounds **9** and **10**.

The method described in this paper for the synthesis of 15-(*p*-iodophenyl)-6-tellurapentadecanoic acid (**10**) can be readily applied to the preparation of other tellurium fatty acid analogues. It has recently been used for the preparation of compound **10** with ^{125}I and ^{131}I labels, and these new agents showed the expected pronounced and prolonged myocardial retention in rats with little in vivo deiodination.[20]

Experimental Section

General Methods. The melting points were determined in capillary tubes by using a Büchi SP apparatus and are uncorrected. The petroleum ether was analytical grade and had a boiling range of 30–60 °C. The thin-layer chromatographic analysis (TLC) were performed by using 250-μm thick layers of silica gel G PF-254 coated on glass plates (Analtech, Inc.). Column chromatography was performed by using 60–200-mesh acidic or basic grade silicic acid (Sigma Chemical Co.). The infrared spectra (IR) were recorded on a Beckman 18-A spectrophotometer with NaCl plates or KBr pellets. The low-resolution mass spectra (MS) were recorded by using a Kratos MS-25 low-resolution instrument under the following conditions: ionizing energy, 70 eV; accelerating potential, 8000 V; trap current, 100 μA; probe temperature, 200–300 °C. The proton nuclear magnetic resonance spectra (^{1}H NMR) were obtained at 60 MHz with a Varian 360-A instrument or at 200 MHz with a Nicolet high-resolution instrument. Samples (30–40 mg) were dissolved in deuteriochloroform ($CDCl_3$), and resonances are reported downfield (δ) from the internal tetramethylsilane standard.

1-Chloro-9-(*p*-nitrosophenyl)nonane (3). A mixture of 1-chloro-9-phenylnonane (1.19 g, 5 mmol), thallium(III) trifluoroacetate (2.72 g, 5 mmol), and trifluoroacetic acid (5 mL) were protected from light and stirred at room temperature for 16 h. The resulting dark green solution was vacuum distilled (0.6 mm, bath temperature at 40 °C) followed by two vacuum codistillations with 1,2-dichloroethane to remove the trifluoroacetic acid. The amber oil remaining in the distillation flask consisted of 3.12 g (90%) of the crude 1-chloro-9-[*p*-[bis(trifluoroacetyl)thallium]-phenyl]nonane (**2**) which was used without further purification. The thallium intermediate **2** (3.12 g, 4.5 mmol) was stirred in CH_2Cl_2 (50 mL) at room temperature under red lights with isoamyl nitrite (0.7 g, 6 mmol). A mixture of 12 N HCl (1.6 mL) and HOAc (2.4 mL) was then added and the solution stirred 10 min. After the addition of 1.2 N HCl (20 mL), the solution was stirred an additional 10 min. The green mixture was washed several times with 0.1 N HCl and water and dried over anhydrous Na_2SO_4, and the CH_2Cl_2 was removed in vacuo. The resulting green oil was chromatographed on a silicic acid (25 g). Fractions were eluted with petroleum ether (1–5) and 2% ether–petroleum ether (6–16 and 17–32). Fractions 17–32 were combined to give 0.83 g (70%) of 1-chloro-9-(*p*-nitrosophenyl)nonane (**3**) as a green oil. Analysis by TLC (2% ether–petroleum ether) indicated the presence of a single component: R_f 0.30; IR (neat, NaCl) 2930 (CH_2), 1510 (PhN=O), 840 (1,4-substituted Ph) cm^{-1}; MS, m/z (relative intensity) 267 (M^+[^{35}Cl], 27), 238 (M^+[^{35}Cl] – NO + H, 28), 91 (tropylium, 100); ^{1}H NMR ($CDCl_3$) 1.38 (s, 10 H, $(CH_2)_5$), 1.63 (m, 4 H, $PhCH_2CH_2$ and $ClCH_2CH_2$), 2.70 (t, 2 H, J = 6.6 Hz, $PhCH_2$), 3.50 (t, 2 H, J = 6.6 Hz, $ClCH_2$), 7.60 (AA'BB', 4 H, J = 6.6 Hz, aromatic). Anal. Calcd for $C_{15}H_{22}NOCl$: C, 67.29; H, 8.22; N, 5.23. Found: C, 67.26; H, 8.40; N, 5.17.

1-Chloro-9-(*p*-aminophenyl)nonane (4). A mixture of 1-chloro-9-(*p*-nitrosophenyl)nonane (**3**; 1.0 g, 3.75 mmol) and 10%

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(19) For the synthesis of the unlabeled iodinated fatty acid **9** the direct conversion of the thallium intermediate **2** to **6** by treatment with potassium iodide is the more direct and higher yield route. For radioiodination of **9**, however, the conversion of the triazene **5** to the radioiodinated intermediate **6** is the preferred route since the iodide is the limiting reactant, and thus all of the radioiodide is consumed.
(20) M. M. Goodman and F. F. Knapp, Jr., unpublished results.

palladium on charcoal (50 mg) was stirred in MeOH (5 mL) at room temperature under argon. Following the addition of $NaBH_4$ (400 mg, 10 mmol) in MeOH (5 mL), the mixture was stirred 30 min. The yellow solution was then filtered into H_2O (50 mL) and extracted with Et_2O. The combined Et_2O extracts were washed thoroughly with H_2O and dried over anhydrous Na_2SO_4, and the solvent removed in vacuo to yield 910 mg (97%) of compound **4** as an orange oil: TLC (C_6H_6) R_f 0.20; IR (neat, NaCl) 3450 and 3380 (NH_2), 2930 and 2860 (CH_2), 820 (1,4-substituted Ph) cm^{-1}; MS, m/z (relative intensity) 253 ($M^+[^{35}Cl]$, 6), 106 (100); ^1H NMR ($CDCl_3$) 1.38 (s, 10 H, $(CH_2)_5$), 1,63 (m, 4 H, $PhCH_2CH_2$ and $ClCH_2CH_2$), 2.45 (t, 2 H, J = 6 Hz, $PhCH_2$), 3.50 (t, 2 H, J = 6.6 Hz, $ClCH_2$), 6.8 (AA'BB', 4 H, J = 7 Hz, aromatic). Anal. Calcd for $C_{15}H_{24}NCl$: C, 71.01; H, 9.47; N, 5.5. Found: C, 70.94; H, 9.56; N, 5.70.

1-[4-(9-Chlorononyl)phenyl]-3,3-(1,5-pentanediyl)triazene (5). The amine intermediate **4** (253 mg, 1 mmol) was stirred at 0–5 °C with 0.5 N HCl (4 mL). Sodium nitrite (69 mg, 1 mmol) in H_2O (1 mL) was added dropwise, and the mixture was stirred at 0–5 °C for 5 min. Piperidine (403 mg, 4.5 mmol) in H_2O (3 mL) was then added dropwise while the temperature of the reaction mixture was maintained at 0–5 °C. The solution was stirred at 0–5 °C for 30 min, poured into H_2O (50 mL), and extracted several times with CH_2Cl_2. The combined organic extracts were washed thoroughly with H_2O and dried over anhydrous Na_2SO_4, and the solvent was removed in vacuo. The crude product was dissolved in C_6H_6 (2 mL) and chromatographed on silicic acid (basic, 25 g) slurried in petroleum ether. Elution with 2% ether–petroleum ether gave **5** in fractions 8–13 (143 mg, 41%) as an orange oil. A single component was detected by TLC (2% ether–petroleum ether, R_f 0.20): IR (neat, NaCl) 2910 and 2840 (CH_2), 835 (1,4-substituted Ph) cm^{-1}; MS, m/z (relative intensity) 351 ($M^+[^{35}Cl]$, 100); ^1H NMR (CDCl3) 1.38 (s, 10 H, $(CH_2)_5$), 1.63 (m, 10 H, $PhCH_2CH_2$, $ClCH_2CH_2$ and piperidinyl $CH_2CH_2CH_2$), 2.60 (t, 2 H, J = 6.6 Hz, $PhCH_2$), 3.50 (t, 2 H, J = 6.6 Hz, $ClCH_2$), 3.70 (m, 4 H, piperidinyl CH_2NCH_2), 7.20 (AA'BB', 4 H, J = 8 Hz, aromatic). Anal. Calcd for $C_{20}H_{32}N_3Cl$: C, 68.67; H, 9.16. Found: C, 69.18; H, 9.00.

1-Chloro-9-(*p*-iodophenyl)nonane (6). Method A. Sodium iodide (15 mg, 0.1 mmol) and 2 mL of acetone were stirred at 0–5 °C. Trifluoroacetic acid (114 mg, 1 mmol) was added in 3 mL of acetone. The triazene **5** (35 mg, 0.1 mmol) in 3 mL of acetone was added, and the resulting mixture was stirred at 0–5 °C for 5 min, poured into 50 mL of H_2O, and extracted several times with ether. The combined ether extracts were washed twice with 25 mL of 10% sodium metabisulfite and several times with H_2O and dried over anhydrous Na_2SO_4, and the solvent was removed in vacuo. The crude product was chromatographed on silicic acid (acidic, 25 g). Elution with petroleum ether gave **6** in fractions 7–10 (21 mg, 60%) as a colorless oil.

Method B. To a solution of 1-chloro-9-[*p*-[bis(trifluoroacetyl)thallium]phenyl]nonane (**2**; 2.04 g, 3 mmol) in water (30 mL) was added potassium iodide (3.00 g, 17 mmol), and the resulting cloudy solution was stirred at room temperature for 15 min. Sodium metabisulfite (1 g) was then added, and the mixture was stirred until the solution turned yellow. After being stirred 30 min, the solution was made basic with 1 N NaOH, filtered, and extracted thoroughly with ether. The combined ether extracts were washed with H_2O, dried over anhydrous Na_2SO_4, and evaporated in vacuo to afford a yellow residue. The residue was taken up in benzene (2 mL) and chromatographed as described in method A to yield 0.64 g (64%) of 1-chloro-9-(*p*-iodophenyl)nonane (**6**).

The 1-chloro-9-(*p*-iodophenyl)nonane (**6**) exhibited one component (R_f 0.65) upon TLC analysis (petroleum ether): IR (neat, NaCl) 2930 and 2860 (CH_2) cm^{-1}; MS, m/z (relative intensity) 364 ($M^+[^{35}Cl]$, 40), 217 (100), 91 (tropylium, 85); ^1H NMR ($CDCl_3$) 1.38 (s, 10 H, $(CH_2)_5$), 1.63 (m, 4 H, $PhCH_2CH_2$ and $ClCH_2CH_2$), 2.52 (t, 2 H, J = 5 Hz, $PhCH_2$), 3.50 (t, 2 H, J = 6.6 Hz, $ClCH_2$), 7.20 (AA'BB', 4 H, J = 7 Hz, aromatic protons).

Dimethyl 6,7-Ditelluradodecanedioate (7). Tellurium metal (1.27 g, 10 mmol), sodium hydride (0.44 g, 11 mmol), and dry dimethylformamide (DMF, 50 mL) were stirred at 70 °C under an argon atmosphere for 3 h. The purple Na_2Te_2 solution was cooled to room temperature, and a mixture of methyl 5-bromovalerate (2.15 g, 11 mmol) in 10 mL of argon-purged dry DMF was added. The resulting mixture was stirred at room temperature for 60 min, cooled, poured into water (100 mL), and extracted several times with Et_2O. The combined orange extracts were washed thoroughly with H_2O and dried over anhydrous Na_2SO_4, and the Et_2O was removed in vacuo to give a dark orange oil. The crude ditelluride was chromatographed on silicic acid (basic, 125 g). Elution with $CHCl_3$ gave 1.38 g of **7** as a dark orange oil (53%) which exhibited a single component (R_f 0.56) on TLC (solvent, $CHCl_3$): IR (neat, NaCl) 2920 (CH_2), 1740 (C=O) cm^{-1}; MS, m/z (relative intensity) 490 ($M^+[^{130}Te]$, 12), 360 ($M^+[^{130}Te]$ – Te, 50), 260 (Te_2^+, 10); ^1H NMR ($CDCl_3$) 1.63 (m, 4 H, $(CH_2)_2$), 2.32 (t, 2 H, J = 7 Hz, CH_2COO), 3.06 (t, 4 H, J = 7 Hz, CH_2TeCH_2), 3.65 (s, 3 H, OCH_3).

Methyl 15-(*p*-Iodophenyl)-6-tellurapentadecanoate (9). The ditelluride **7** (800 mg, 1.65 mmol) was stirred at room temperature in MeOH under argon and reduced to a colorless solution of sodium (methylvaleryl)telluride (**8**) by cautious stepwise addition of $NaBH_4$. The substrate **6** (1.0 g, 3 mmol) was added in MeOH (2 mL). The mixture was refluxed 1 h, cooled, poured into H_2O, and extracted twice with Et_2O, and the combined organic extracts were washed thoroughly with H_2O and dried over anhydrous Na_2SO_4. Following evaporation of the solvent in vacuo, the oily product was chromatographed on silicic acid (basic, 25 g). Elution with C_6H_6 gave **9** in fractions 5–7 (1.60 g, 90%) as a light yellow oil: TLC (C_6H_6) R_f 0.47; IR (neat, NaCl) 1060 (*p*-iodophenyl), 1740 (C=O) cm^{-1}; MS, m/z (relative intensity) 574 (M^+, 14), 543 ($M^+ - OCH_3$, 4), 447 (M^+ – I, 20), 330 (100), 279 (40); ^1H NMR ($CDCl_3$, 200 MHz) 2.27 (t, J = 7 Hz, 2 H, CH_2COO), 2.56 (t, 6 H, J = 7 Hz, CH_2TeCH_2 and CH_2Ph), 3.61 (s, 3 H, $COOCH_3$), 7.19 (AA'BB', 4 H, J = 7 Hz, aromatic).

15-(*p*-Iodophenyl)-6-tellurapentadecanoic Acid (10). The methyl ester **9** (97 mg, 0.168 mmol) was dissolved in EtOH (20 mL) and refluxed with 1 N NaOH (1 mL) under argon for 30 min. The mixture was cooled, poured into H_2O, acidified to pH 2–3 with 10% H_2SO_4, and extracted twice with Et_2O. Following thorough washing with H_2O, the organic layer was dried over anhydrous Na_2SO_4 and the solvent evaporated to give 66 mg (70%) of 15-(*p*-iodophenyl)-6-tellurapentadecanoic acid (**10**). Crystallization from petroleum ether gave plates: mp 72–73 °C; TLC (8% MeOH–$CHCl_3$) R_f 0.55; ^1H NMR ($CDCl_3$) 2.39 (t, $J \simeq 7$ Hz, 2 H, CH_2COO), 2.62 (t, $J \simeq 7$ Hz, 6 H, CH_2TeCH_2 and CH_2Ph) and 7.23 (AA'BB', 4 H, J = 7 Hz, aromatic); MS, m/z (relative intensity) 560 (M^+, 11), 433 (M^+ – I, 25), 330 (31), 230 (22). Anal. Calcd for $C_{20}H_{31}O_2TeI$: C, 43.03; H, 5.60. Found: C, 43.19; H, 5.69.

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Oxidative Decarboxylation of α-Hydroxy Carboxylic Acids with *N*-Iodosuccinimide

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Recently,[1] we found that 1,2-diols were easily cleaved with *N*-iodosuccinimide (NIS). At this time, we report that

