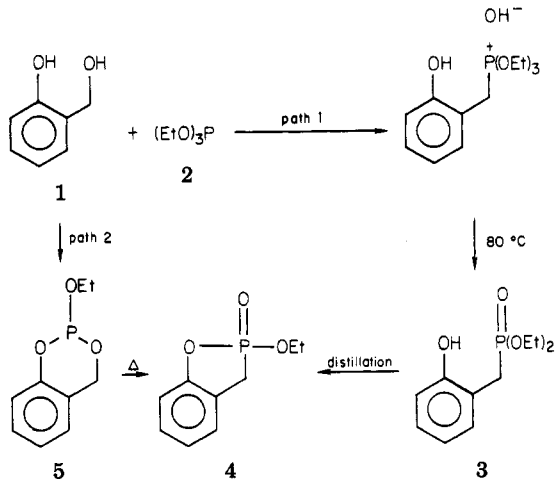
Scheme I



with examples of uncatalyzed rearrangements of 2-alkynyl, 2,3-butadienyl, and 2-alkenyl phosphite esters to phosphonates,[3] there is little precedent for the similar rearrangement of benzyl-type phosphites. However, Kamai and Kharrasova[4] did report that benzyl diphenyl phosphite in CCl_4 for 32 h at 160–165 °C afforded benzyl diphenyl phosphonate in unspecified yield. Thus, it remains unclear whether **3** or **5** is an intermediate in the reaction of **1** with **2**. Since we had an interest in some similar chemistry,[5] we decided to reexamine the reaction in Scheme I.

When equal molar quantities of **1** and **2** were mixed in DMF at room temperature, a reaction that could be followed by GC began immediately. This reaction proceeded to near completion after 1.5 h at 90–100 °C. Half of this solution was removed and the solvent evaporated under vacuum and at or below 36 °C. The ^1H, ^{13}C, and ^{31}P NMR spectra (see Experimental Section) and the field-desorption mass spectrum (FD/MS) were consistent with this material being **3**. In particular, both the smaller (29.18 ppm) ^{31}P chemical shift and the large 140-Hz ^{13}C–^{31}P coupling constant are consistent with the phosphonate **3** rather than phosphite **5** structure. Phosphonates typically give rise to ^{31}P absorptions in the 10–40-ppm range[6] and $^1J_{C-P}$ coupling constants in the 130–160-Hz range[7] while phosphites exhibit ^{31}P absorptions in the 120–140-ppm range[6] and $^2J_{COP}$ coupling constants in the 2–18-Hz range.[7] The remaining contents of the reaction flask were heated to 145–150 °C and held there for 4 h, during which time ethanol was distilled off. Distillation afforded a clear liquid whose ^1H, ^{13}C, and ^{31}P NMR spectra and FD/MS spectrum were consistent with this material being **4**. Thus, it is clear that Ivanov and co-workers were correct in their conjecture that **3**, and not **5**, is an intermediate to **4** in Scheme I. However, in a future communication, we will discuss our observations on the rearrangement of benzylic cyclic phosphites to phosphonates.

Experimental Section

The ^1H (200.13 MHz) and ^{13}C (50.28 MHz) NMR spectra were obtained on a Bruker WH-200 instrument, and the chemical shifts are reported in ppm downfield from internal tetramethylsilane. The ^{31}P NMR spectra were obtained on a Bruker Model HX-90E instrument at 36.44 MHz, and the chemical shifts are in ppm downfield from external phosphoric acid. The field-desorption mass spectra (FD/MS) were obtained on a Finnigan MAT 311A spectrometer. Gas chromatograms were obtained on an HP 5840A instrument equipped with a 46 × 0.3 cm stainless steel column packed with 3% OV-17 on 80/100 Chromasorb WHP.

Preparation of 3. 2-Hydroxybenzyl alcohol (15 g, 0.12 mol) and triethyl phosphite (20 g, 0.12 mol) were dissolved in dimethylformamide (50 mL) and heated at 100 °C for 1.5 h. Twenty milliliters of solution were removed, and the solvent was evaporated under vacuum at 36 °C to afford a clear liquid: ^1H NMR ($CDCl_3$) δ 1.26 (t, J = 7, 6 H, CH_3), 3.95–4.10 (m, 4 H, OCH_2), 3.20 (d, J_{P-H} = 21, 2 H, PCH_2Ar), 4.8 (s, 1 H, OH), 6.82–7.12 (m, 4 H, Ar H); ^{13}C NMR ($CDCl_3$) (^1H decoupled) δ 16.11 (d, J_{POCCH_3} = 5.9), 28.22 (d, J_{P-CH_2} = 140), 62.43 (d, J_{POCH_2} = 7.3), 117.21, 118.51 (d, J = 9.1), 120.08, 128.37 (d, J = 3.1), 131.27 (d, J = 6.6), 155.63 (d, J = 6.0); ^{31}P NMR ($CDCl_3$) (^1H decoupled) δ 29.18; FD/MS, m/e 244 (M^+).

Preparation of 4. The remaining reaction solution from the preparation of **3** was heated to 145–150 °C for 4 h while the ethanol was distilled off. Distillation in vacuuo then afforded a clear liquid: bp 116–118 °C (0.2–0.4 mm) [lit.[1c] bp 122 °C (0.03 mm)]; ^1H NMR ($CDCl_3$) δ 1.37 (t, J = 7.3, 3 H, CH_3), 4.20–4.30 (m, 2 H, OCH_2), 3.13 (dd, J = 4.2, J = 15.2, 2 H, PCH_2Ar), 6.97–7.25 (m, 4 H, Ar H); ^{13}C NMR ($CDCl_3$) (^1H decoupled) δ 16.38 (d, J_{POCCH_3} = 4.9), 24.58 (d, J_{P-C} = 123), 63.41 (d, J_{POCH_2} = 6.3), 113.39 (d, J = 16), 122.79 (d, J = 3.4), 123.47, 127.41 (d, J = 19), 129.17, 153.45 (d, J = 12); ^{31}P NMR ($CDCl_3$) (^1H decoupled) δ 45.62; FD/MS, m/e 198 (M^+).

Acknowledgment. I thank Mr. Perry Matheny for his experimental assistance.

Registry No. **1**, 90-01-7; **2**, 122-52-1; **3**, 50375-72-9; **4**, 14707-41-6; **5**, 87451-61-4.

(3) V. Mark in "Mechanisms of Molecular Migrations", Vol. 2, B. S. Thyagaragan, Ed., Interscience, New York, 1969, p 319.
(4) G. Kamai and F. M. Kharrasova, *Tr. Kazan. Khim.-Tekhnol. Inst.*, **23**, 122 (1957); *Chem. Abstr.*, **52**, 9980i (1958).
(5) D. W. Chasar, U.S. Patent applied for.
(6) M. M. Crutchfield, C. H. Dungan, J. H. Letcher, V. Mark, and J. R. VanWazer, "^{31}P Nuclear Magnetic Resonance", Interscience, New York, 1967, Chapter 4.
(7) F. W. Wehrli and T. Wirthlin, "Interpretation of Carbon-13 NMR Spectra", Heyden and Son, Philadelphia, 1980, p 60.

Use of a New Protecting Group in an Attempted Synthesis of Cyclopropyldihydroxyphenylalanine

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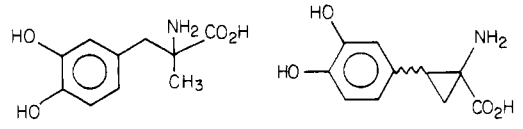
Received May 3, 1983

Since α-methyl-3,4-dihydroxyphenylalanine (**1**) is a clinically effective drug useful against hypertension, the corresponding cyclopropyl compound (**2**), cyclopropyl-



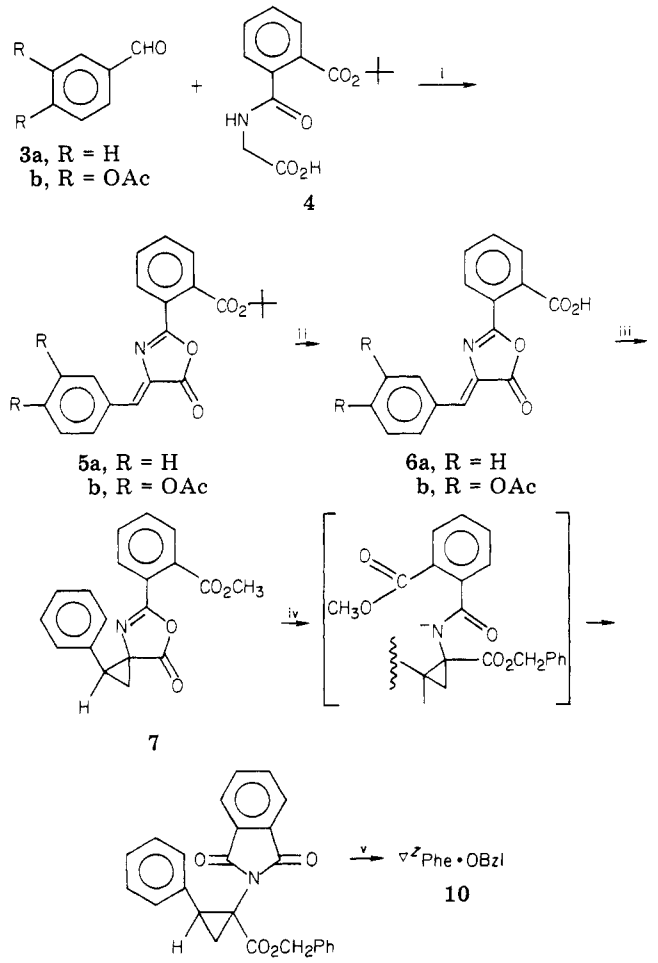
1 2

3,4-dihydroxyphenylalanine (▽Dopa),[1] of either the E or Z configuration, is of considerable interest as a possible antihypertensive drug. Two earlier attepts at the synthesis of **2** have been published.[2] The key to the synthesis of aromatic cyclopropyl amino acids, when prepared using the oxazolone process, resides in the development of mild

(1) We use the ▽ symbol to mean "cyclopropyl" in which the cyclopropane ring requires the C_α–C_β carbon atoms of the amino acid residue as one of its sides. In an earlier publication (ref 3) this symbol was inverted inadvertantly. The superscript E or Z, i.e., ∇^E, ∇^Z, refers to the configuration about the cyclopropane ring.
(2) (a) Bernabe, M.; Cuevas O.; Fernandez-Alvarez, E. *Eur. J. Med. Chem.* **1979**, *14*, 33. (b) Hines, J. W., Jr.; Breitholle, E. G.; Sato, M.; Stammer, C. H. *J. Org. Chem.* **1976**, *41*, 1466.

0022-3263/83/1948-4769$01.50/0

Scheme I[a]

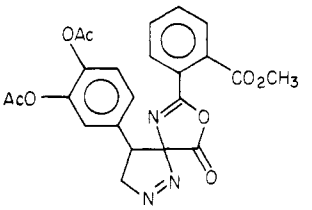


[a] Reagents: i, Ac_2O/NaOAc; ii, CF_3CO_2H; iii, CH_2N_2; iv, $PhCH_2OH$/DMAP; v, NH_2NH_2/MeOH.

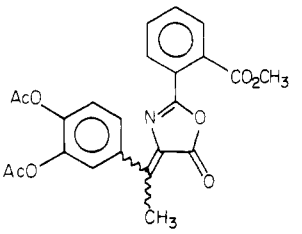
methods for the removal of the *N*-acyl group. We found,[2b] as did Bernabe,[2a] that conditions sufficient for the hydrolysis of aromatic *N*-benzoyl cyclopropyl amino acids destroyed the ring completely. We have successfully treated these acyl intermediates with Meerwein's reagent, followed by mild hydrolysis of the resulting imino ether in our recent work,[3] but we were unsuccessful in applying this method to Dopa derivatives. In this paper we report a new deblocking method, developed during our work on the synthesis of (*Z*)-cyclopropylphenylalanine (∇^ZPhe).[1]

In view of the ease of removal of the *N*-phthaloyl group,[4] we introduced a masked phthaloyl function into the starting hippuric acid used in the synthesis of the required oxazolone. As shown in Scheme I, *N*-(*o*-*tert*-butoxycarbonylbenzoyl)glycine (**4**) could be condensed with benzaldehyde (**3a**), in the usual manner, to yield the oxazolone **5a**. Removal of the *tert*-butyl blocking group followed by treatment of the acid **6a** with excess diazomethane afforded the spirooxazolone methyl ester **7** in quite acceptable yield. Methanolysis of an oxazolone ring normally gives an *N*-benzoyl methyl ester,[3] but solvolysis of **7** afforded an intermediate anion **8**, having an *o*-carbomethoxy group that underwent the desired spontaneous rearrangement to the phthaloyl derivative **9** in high yield. Conversion of **9** into ∇^ZPhe by hydrazinolysis followed by ester removal was then routine.

When this reaction sequence was applied to the synthesis of ∇Dopa using 3,4-diacetoxybenzaldehyde (**3b**), the oxazolone **5b** was obtained in good yield and its conversion to the acid **6b** was uneventful. Surprisingly,[5] when **6b** was treated with diazomethane, the spiropyrazoline **11** was obtained in excellent yield. The ^{1}H NMR spectrum of



11



12

11 showed a characteristic[6] methylene multiplet at δ 5.3 ppm and no peaks for cyclopropyl protons at higher field. Pyrolysis of **11** gave an excellent yield of the β-methyl compound **12**, which showed a methyl singlet at δ 2.70 rather than the expected cyclopropyl proton signals at higher field. No further work on ∇Dopa is planned.

Experimental Section

Instrumentation. All melting points were taken on a Thomas-Hoover capillary melting point apparatus and are uncorrected. Infrared spectra were measured with a Perkin-Elmer Model 297 recording spectrophotometer with polystyrene as a standard. Elemental analyses were provided by Atlantic Microlab, Atlanta, GA. ^{1}H NMR spectra were recorded on a Varian EM-390 spectrometer at 90 MHz.

***N*-[*p*-(*tert*-Butoxycarbonyl)benzoyl]glycine (4).** A solution of 22.2 g (0.1 mol) of *tert*-butyl hydrogen phthalate[7] and 10 g (0.1 mol) of *N*-methylmorpholine in 200 mL of anhydrous THF was cooled to −15 °C and 13.6 g (0.1 mol) of isobutyl chloroformate was added. After the mixture was stirred at 0 °C for 25 min, the white solid was filtered and the filtrate was cooled to 0 °C. To it was added a solution of glycine (9 g, 0.12 mol) and sodium hydroxide (4.8 g, 0.12 mol) in a minumum quantity of water. The reaction mixture was stirred vigorously for 30 min at 0 °C and removed and the residue was dissolved in water (50 mL), acidified with 1 N HCl, and extracted with ethyl acetate (3 × 50 mL), and the extracts were dried over anhydrous Na_2SO_4. The solution was concentrated and allowed to stand in a refrigerator overnight. The white crystals of **4** were collected by filtration: 16.76 g (60%); mp 146–147 °C; IR (KBr) 3280, 1765, 1685, 1640 cm^{-1}; NMR ($CDCl_3$) δ 1.4 (9 H, s, $COO(CH_3)_3$), 3.9 (2 H, d, $NHCH_2$), 7.6 (4 H, m, Ar H).

Anal. Calcd for $C_{14}H_{17}NO_5$: C, 60.21; H, 6.13; N, 5.01. Found: C, 60.17; H, 6.15; N, 5.01.

2-[*o*-(*tert*-Butoxycarbonyl)phenyl]-4-benzylidene-5-oxazolone (5a). A mixture of **4** (15 g, 0.054 mol), benzaldehyde (8.6 g, 0.081 mol), sodium acetate (13.3 g, 0.162 mol), and acetic anhydride (60 mL) was stirred at room temperature for 24 h. The excess of acetic anhydride was removed under reduced pressure and the oily residue was dissolved in ethyl acetate (100 mL) and the solution was washed with dilute sodium carbonate (2 × 30 mL) and water (2 × 40 mL) and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was dissolved in hot isoproyl alcohol. The yellow solid that crystallized at 5 °C weighed 9.3 g (49%): mp 103–104 °C; IR (KBr) 1800, 1715, 1650 cm^{-1}; NMR ($CDCl_3$) δ 1.6 (9 H, s, $COO(CH_3)_3$),

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(4) The careful work of Wolfe and Hasan (Wolfe, S.; Hasan, S. K. *Can. J. Chem.* **1970**, *48*, 3572) Losse and Raue (Losse, G.; Raue, H. *Chem. Ber.* **1965**, *98*, 1522), and Aberhart and Lin (Aberhart, D. J.; Lin, H.-J. *J. Org. Chem.* **1981**, *46*, 3749) inspired this idea.

(5) This is the first time in our experience that a pyrazoline has been obtained from an arylidene oxazolone upon reaction with diazomethane.

(6) We have observed this signal at δ 4.9–5.3 in several pyrazolines similarly substituted with *alkyl* groups.

(7) Davies, A. G.; Kenyon, J.; Salami, L. W. F. *J. Chem. Soc.* **1957**, 3148.

7.3–8.3 (10 H, m, Ar H and vinylic H).

Anal. Calcd for $C_{21}H_{19}NO_4$: C, 72.19; H, 5.48; N, 4.01. Found: C, 72.22; H, 5.48; N, 3.99.

(*Z*)-2-(*o*-Carboxyphenyl)-4-benzylidene-5-oxazolone (6a). A solution of **5a** (5.34 g, 0.015 mol) in 100 mL of CF_3CO_2H/CH_2Cl_2 solution (1:3) was stirred for 2 h at room temperature. The solvent was removed in vacuo and the residue was triturated with ethyl acetate (100 mL) to give 4.2 g (94%) of **6a**: mp 194–195 °C; IR (KBr) 1795, 1775, 1695, 1650 cm^{-1}; NMR (Me_2SO-d_6) δ 7.4 (5 H, m, Ar H), 7.9 (4 H, s, Ar H), 8.1 (1 H, s, vinylic H).

Anal. Calcd for $C_{17}H_{11}NO_4$: C, 69.62; H, 3.78; N, 4.77. Found: C, 69.50; H, 3.84; N, 4.73.

(*Z*)-1-Phenyl-5-[*o*-(methoxycarbonyl)phenyl]-6-oxo-4-azaspiro[2.4]hept-4-en-7-one (7). To a stirred suspension of **6a** (5 g, 0.0172 mol) in methylene chloride (40 mL) was added dropwise a solution of diazomethane in 250 mL of ether, prepared from 32.3 g (0.15 mol) of Diazald. After the reaction mixture was stirred at room temperature for 24 h, the excess of diazomethane was removed under a stream of dry nitrogen and the solvent was evaporated under reduced pressure to give a yellow oil. The residue was dissolved in 20 mL of ether and 2 g (33%) of spiro compound **7** deposited on cooling: mp 93–96 °C; IR (KBr) 1810, 1725, 1630 cm^{-1}; NMR ($CDCl_3$) δ 2.2–2.6 (2 H, m, CH_2), 3.2–3.4 (1 H, m, CH), 3.6 (3 H, s, $COOCH_3$), 7.2–7.9 (9 H, m, Ar H).

Benzyl (*Z*)-1-Phthalimido-2-phenylcyclopropanecarboxylate (9). A mixture of spirooxazolone **7** (1 g, 0.003 mol), DMAP (380 mg, 0.003 mol), and 5 mL of benzyl alcohol was stirred at room temperature of $2^1/_2$ h. The reaction mixture was dissolved in ethyl acetate (25 mL) and the solution was washed with 10% citric acid, water, and saturated sodium chloride and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure. The residue was crystallized from ether/petroleum ether to yield 838 mg of **9** (68%): mp 143–144 °C; IR (KBr) 1710 cm^{-1}; NMR ($CDCl_3$) δ 2.2–2.6 (2 H, m, CH_2), 3.4–3.6 (1 H, m, CH), 5.3 (2 H, s, $OCH_2C_6H_5$), 7.2 (5 H, s, Ar H), 7.4 (5 H, s, Ar H), 7.8 (4 H, s, Ar H).

Anal. Calcd for $C_{25}H_{19}NO_4$: C, 75.55; H, 4.82; N, 3.52. Found: C, 75.41; H, 4.89; N, 3.48.

Benzyl (*Z*)-1-Amino-2-phenylcyclopropanecarboxylate Hydrochloride (10). A mixture of **9** (795 mg, 2 mmol), hydrazine hydrate (0.2 g, 4 mmol), and methanol (5 mL) was stirred at room temperature for 30 min. The solvent was removed under reduced pressure and the residue was dissolved in 10 mL of 1 N HCl, heated 15 min on a steam bath, and filtered and the filtrate was evaporated to dryness. The residue was crystallized from isopropyl alcohol/ether to yield 379 mg (63%) of **10**, mp 161–63 °C dec, identical with that of the known compound.[3]

(*Z*)-2-(*o*-Carboxyphenyl)-4-(3,4-diacetoxybenzylidene)-5-oxazolone (6b). A mixture of 3,4-diacetoxybenzaldehyde (7.55 g, 0.034 mol), *o*-(*tert*-butoxycarbonyl)hippuric acid (4; 6.3 g, 0.03 mol), NaOAc (5.1 g, 0.068 mol), and Ac_2O (50 mL) was stirred for 2 days at room temperature. Excess acetic anhydride was removed in vacuo and the resulting residual syrup was extracted with AcOEt (3 × 50 mL). The extract was washed with water, 10% Na_2CO_3 solution, and saturated NaCl solution and then dried over anhydrous Na_2SO_4. The solvent was removed in vacuo to give a yellow oil, which was dissolved in CH_2Cl_2 (110 mL), and CF_3COOH (36 mL) was added to the solution. After the solution was stirred overnight at room temperature, the solvents were evaporated in vacuo, and the residue was chromatographed (50 g silica gel, 60–200 mesh, J. T. Baker Chemical Co.) with $CHCl_3$/AcOEt (5:1) to give a pale yellow solid, **6b** (2.0 g). Recrystallization from MeOH/AcOEt (1:1) gave 1.6 g (17.2%) of **6b** as light yellow prisms: mp 214–215 °C; IR (KBr) 1750, 1680, 1630, 1600 cm^{-1}; NMR (Me_2SO-d_6) δ 7.2–8.5 (m, 8 H, Ar H, CH=), 2.22 (s, 3 H, CH_3), 2.20 (s, 3 H, CH_3).

Anal. Calcd for $C_{21}H_{15}NO_8$: C, 61.61; H, 3.70; N, 3.42. Found: C, 61.56; H, 3.99; N, 3.28.

1-(3,4-Diacetoxyphenyl)-7-[*o*-(methoxycarbonyl)-phenyl]-8-oxa-3,4,6-triazaspiro[4.4]nona-3,6-dien-9-one (11). To a suspension of **6b** (1.3 g, 3.18 mmol) in CH_2Cl_2 (10 mL) was added dropwise an ethereal diazomethane solution prepared from Diazald (5.25 g, 245 mol) with ice cooling over a period of 45 min. After stirring was continued for 24 h at room temperature, the solvent was evaporated in vacuo and the resulting syrup was column chromatographed (silica gel 20 g, 60–200 mesh Baker Analyzed Reagent), using $CHCl_3$. The syrup obtained was triturated with Et_2O/*n*-hexane and the resulting solid was collected by suction to give **11** (1.4 g, 94.6%). Recrystallization from AcOEt/*n*-hexane gave colorless prisms: 1.2 g (81.1%); mp 82–83 °C; NMR ($CDCl_3$) δ 7.75 (s, 4 H, Ar H), 6.8–7.0 (m, 3 H, Ar H), 5.2–5.45 (m, 2 H, CH_2N), 4.4–4.6 (m, 1 H, CH), 3.95 (s, 3 H, OCH_3), 2.2 (s, 6 H, $2CH_3CO_2$).

2-[*o*-(Methoxycarbonyl)phenyl]-4-[1-(3,4-diacetoxyphenyl)ethylidene]-5-oxazolone (12). A mixture of **11** (1.2 g, 2.6 mmol) and toluene (20 mL) was stirred at 95–100 °C (bath temperature) for 1.5 h. The solvent was evaporated in vacuo and the residual syrup was triturated with *n*-hexane (20 mL). The crystals were filtered by suction to give **12** (1.1 g, 97.3%), which was recrystallized from AcOEt/*n*-hexane to give colorless prisms: mp 136–138 °C; NMR ($CDCl_3$) δ 7.65–7.9 (m, 4 H, Ar H), 7.00–7.25 (m, 3 H, Ar H), 3.77 (s, 3 H, OMe), 2.70 (s, 3 H, CH_3), 2.20 (s, 6 H, $2CH_3CO_2$).

Anal. Calcd for $C_{23}H_{19}NO_8$: C, 63.16; H, 4.38; N, 3.20. Found: C, 62.98; H, 4.40; N, 3.16.

Acknowledgment. We gratefully acknowledge the support of NSF Grant CHE-8122011 and NIH NIDA Grant 02938.

Registry No. *cis*-**2**, 87483-06-5; *trans*-**2**, 87483-05-4; **4**, 87483-07-6; **5a**, 87483-08-7; **6a**, 87483-09-8; **6b**, 87483-10-1; **7**, 87483-11-2; **9**, 87483-12-3; **10**, 87483-13-4; **11**, 87483-14-5; **12**, 87483-15-6; *tert*-butyl hydrogen phthalate, 33693-84-4; glycine, 56-40-6; benzaldehyde, 100-52-7; 3,4-diacetoxybenzaldehyde, 67727-64-4; diazomethane, 334-88-3.

Nitration of Estrone into 2-Nitroestrone by Clay-Supported Ferric Nitrate[1]

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Received June 1, 1983

The large spectrum of potential biological activities[2-6] of estrone derivatives functionalized in ring A explains the major preparative interest in 2- and 4-nitroestrone as starting materials. The classic procedure, via concentrated nitric acid dissolved in glacial acetic acid,[7,8] suffers from a lack of discrimination, hence the importance of developing a regiospecific mononitration. The recent report by Santaniello et al.[9] of their new procedure, using either silver nitrate or *N*-nitropyrazole as nitrating agents in association with boron trifluoride etherate, prompts us to disclose our results with inexpensive reagents (clay-supported ferric nitrate, "clayfen",[10] a reagent which we have introduced for oxidation of alcohols[10] and used also for oxidative coupling of thiols[1]) under very mild and straightforward conditions (room temperature, toluene suspension, ease of setup and of workup). We obtained the best isolated yields (>55%) reported so far in the nitration of estrone (**1**) in the 2-position. The remainder of the reaction mixture is adsorbed into the clay, and we are hoping to

(1) Clay-supported Reagents. 5. Previous publication in this series: Cornélis, A.; Depaye, N.; Gerstmans, A.; Laszlo, P. *Tetrahedron Lett.* **1983**, *24*, 3103.
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(4) Emons, G.; Ball, P. *Acta Endocrinol. (Copenhagen)* **1982**, *100*, 105.
(5) Rao, N. K.; Sweet, F.; Warren, J. C. *Steroids* **1974**, *24*, 63.
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(10) Cornélis, A.; Herzé, P. Y.; Laszlo, P.; *Tetrahedron Lett.* **1982**, *23*, 5035.

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