

round-bottomed flask was charged with 10 mL of dioxane, 5 mmol of the carbonyl compound, 6 mmol of α -bromoethyl acetate, 9 mmol of zinc dust, and 1 mmol of iodine. The flask was then partially submerged in the sonicator in a place that produced the greatest agitation of the reaction mixture. The reaction conditions for potassium iodide aided reactions were similar except that relatively larger quantities of zinc (15 mmol) and promoter (4.2 mmol of KI) were used. The progress of the reaction, i.e., disappearance of carbonyl substrate, was monitored by ^1H NMR.

Product isolation was straightforward. The crude product was poured into an ice-water mixture (100 mL) which was extracted with methylene chloride (2×200 mL) followed by separation and drying of the organic fraction. Removal of the volatiles by flash evaporation and high vacuum (~ 0.1 torr) gave the desired β -hydroxy ester. Each product was characterized by IR, NMR, and mass spectroscopy. These spectra were compared to those of authentic samples. Yields in the table are based on quantities of isolated product with $>95\%$ purity by NMR.

Larger Scale Reformatsky Reaction Using *n*-Butanal and Ethyl Bromoacetate. An oven-dried, nitrogen-filled, 250-mL, single-necked, round-bottomed flask was charged with 25 mL of dioxane, 5.4 g (75 mmol) of butanal, 15 g (90 mmol) of ethyl bromoacetate, and 8.5 g (130 mmol) of zinc dust. The flask was immersed to the solvent level in the sonicator, and iodine was added to the slurry until it became exothermic. This required about 0.5 g (~ 2 mmol) of I_2 . The progress of the reaction was monitored by following the disappearance of the aldehyde proton (triplet, δ 9.8) by ^1H NMR. The absorption was gone in <5 min.

The product mixture was poured slowly into an ether-ice slurry with stirring, and 1 g of KI was added to remove I_2 from the organic layer. This was extracted with ether (2×200 mL), and the combined extracts were dried over CaCl_2 . Removal of the volatiles by flash evaporation followed by vacuum distillation gave 10.9 g (91%) of ethyl 3-hydroxyhexanoate [bp 75°C (0.1 torr)] which was identified by ^1H NMR and mass spectroscopy.

Acknowledgment. We thank the Air Force Office of Scientific Research for financial support through Grant No. 80-0239.

Registry No. 1, 123-72-8; 2, 124-13-0; 3, 100-52-7; 4, 66-77-3; 5, 66-99-9; 6, 120-92-3; 7, 123-19-3; 8, 98-86-2; 9, 119-61-9; $\text{C}_3\text{H}_7\text{CH}(\text{OH})\text{CH}_2\text{CO}_2\text{Et}$, 2305-25-1; $\text{C}_7\text{H}_{15}\text{CH}(\text{OH})\text{CH}_2\text{CO}_2\text{Et}$, 6071-25-6; $\text{C}_6\text{H}_5\text{CH}(\text{OH})\text{CH}_2\text{CO}_2\text{Et}$, 5764-85-2; $1\text{-C}_{10}\text{H}_7\text{CH}(\text{OH})\text{CH}_2\text{CO}_2\text{Et}$, 83463-90-5; $2\text{-C}_{10}\text{H}_7\text{CH}(\text{OH})\text{CH}_2\text{CO}_2\text{Et}$, 83463-91-6; $\text{C}_3\text{H}_7(\text{C}_3\text{H}_7)\text{C}(\text{OH})\text{CH}_2\text{CO}_2\text{Et}$, 10297-62-8; $\text{C}_6\text{H}_5(\text{CH}_3)\text{C}(\text{OH})\text{CH}_2\text{CO}_2\text{Et}$, 2293-60-9; $\text{C}_6\text{H}_5(\text{C}_6\text{H}_5)\text{C}(\text{OH})\text{CH}_2\text{CO}_2\text{Et}$, 894-18-8; $\text{BrCH}_2\text{CO}_2\text{Et}$, 105-36-2; I_2 , 7553-56-2; 1-[(ethoxycarbonyl)methyl]-1-cyclopentanol, 3197-76-0.

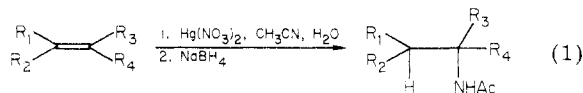
Mechanistic Diversity in the Acetamidomercuration of Substituted Cyclohexenes¹

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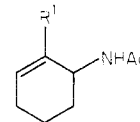
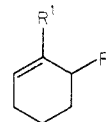
Reaction between an alkene and a mercury(II) salt in the presence of acetonitrile, followed by reductive cleavage of the intermediate organomercurial, constitutes a synthetic procedure for overall addition of the elements of acetamide to the carbon-carbon double bond (eq 1).²



(1) Financial support was supplied by the National Science Foundation (Grant No. CHE 79-04907).

(2) Brown, H. C.; Kurek, J. T. *J. Am. Chem. Soc.* **1969**, *91*, 5647.

However, unlike its better-known counterpart, the oxymercuration-demercuration of alkenes,³ the sequence represented by eq 1 has not been applied to a very wide range of structural types. In fact, studies to date have been limited mainly to monosubstituted ethylenes, cyclohexene, and cyclopentene, although Brown and Kurek² reported failure to obtain the expected product from 2-methyl-1-pentene (the product was not identified), and Delpech and Khuong-Huu⁴ reported formation of a series of allylic amides from a steroidal ethylenecyclopentane. We recently had occasion to attempt the acetamidomercuration-demercuration of several substituted cyclohexenes (1a-d) and observed a variety of reactions in ad-



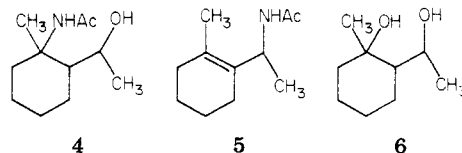
- 1a, $\text{R}^1 = \text{C}_6\text{H}_5$; $\text{R} = \text{CH}(\text{OH})\text{CH}_3$
 b, $\text{R}^1 = \text{C}_6\text{H}_5$; $\text{R} = \text{H}$
 c, $\text{R}^1 = \text{CH}_3$; $\text{R} = \text{CH}(\text{OH})\text{CH}_3$
 d, $\text{R}^1 = \text{CH}_3$; $\text{R} = \text{H}$

- 2, $\text{R} = \text{C}_6\text{H}_5$
 3, $\text{R} = \text{CH}_3$

dition to that embodied above as eq 1. Although we plan no further work in this area, we wish to call our results to the attention of others who may have interest in this reaction.

Lithium aluminum hydride reduction of 6-acetyl-1-phenylcyclohexene⁵ afforded 6-(1-hydroxyethyl)-1-phenylcyclohexene (1a), the respective diastereomers of which could be separated cleanly by medium-pressure liquid chromatography (MPLC)⁶ over silica gel. Reduction of 6-acetyl-1-methylcyclohexene⁵ afforded the corresponding alcohol (1c), the diastereomers of which were not separable in this case by either TLC or MPLC. Reaction of either diastereomer of 1a under standard acetamidomercuration conditions, that is, mercuric nitrate in wet acetonitrile at room temperature for 0.5-24 h,² afforded the allylic amide 2 in 80% isolated yield. 1-Phenylcyclohexene (1b) also afforded 2, in 70% yield, under the same conditions. In addition to the carbon-carbon bond cleavage observed with 1a, these reactions are notable for the ease with which reaction takes place, given the inertness of 1a (our observation) and 1b³ toward oxymercuration, and the surprising mildness with which allylic oxidation of 1b takes place, in view of the considerably more vigorous conditions required to effect allylic oxidation of alkenes by using mercuric acetate in acetic acid.⁷

The similarity in reaction products from 1a and 1b does not carry over to their methyl analogues 1c and 1d. 1-Methylcyclohexene (1d) does undergo allylic oxidation to amide 3 in 80% yield, but 1c is converted to a pair of products consisting of the expected product (4, 52%) and



the allylic amide 5 (37%). 1c is also converted to diol 6 ($>80\%$) under the usual³ oxymercuration conditions. Both

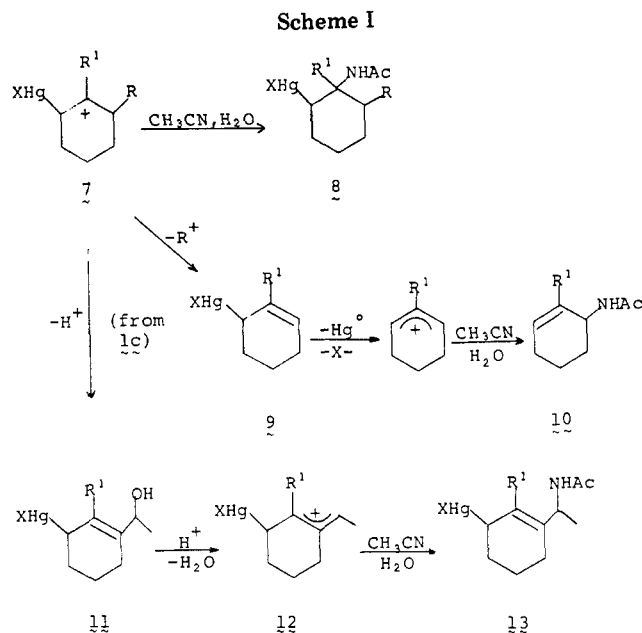
(3) Brown, H. C.; Geoghegan, P. J., Jr. *J. Org. Chem.* **1972**, *37*, 1937.

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4 and 6 appear to be largely one diastereomer, judging from the relative simplicity of their 200-MHz NMR spectra (see Experimental Section).

Four distinct modes of reaction⁸ are represented among these results (1a → 2, 1b → 2, 1c → 4, 1c → 5); 1c is the only substance for which the expected acetamidomercuration sequence occurred even in part. All of the reactions may be rationalized as proceeding via initial attack by electrophilic mercury on the alkene (Scheme I). [The possibility that 2 and 3 arise by electron transfer⁹ from the alkene to mercury(II) could be excluded by our observation that, when a limited amount of mercuric nitrate was allowed to react with an equimolar mixture of 1b and 1d, 1d reacted much faster than 1b.]¹⁰ The initial carbonium ion 7 might undergo nucleophilic attack by acetonitrile, affording the acetamidomercurated product 8. Alternatively, loss of the R group as a proton or protonated acetaldehyde would afford an allylic mercurial (9), solvolysis of which would then afford allylic amide 10. Finally, loss of a proton from 7 will afford an allylic alcohol (11), which should be converted to an allylic cation (12) under the acidic reaction conditions (HNO₃ is formed in the first step). Attack by acetonitrile and water on 12 would then afford 13. Demercuration of 8 and 13 would take place upon borohydride treatment.

The reasons different reaction paths are followed by the different cyclohexenes 1a–d are not completely evident. Presumably the relative rates of the various competing reactions are a function of steric, electronic, and (perhaps) conformational factors, about which it would be premature to speculate. It should be apparent, however, that the scope of the amidomercuration–demercuration process (eq 1) must be considered undefined at this time.

Experimental Section

¹H NMR spectra were obtained on a Varian XL-200 spectrometer in CDCl₃. Elemental analyses were performed by Robertson Laboratories, Florham Park, NJ. 1-Phenylcyclohexene

(8) To these we might add a fifth, in view of our observation that 1-methylcyclopentene is rapidly converted to a dark intractable water-soluble mixture under the usual² acetamidomercuration conditions.

(9) Wolfe, S.; Campbell, P. G. C.; Palmer, G. E. *Tetrahedron Lett.* 1966, 4203.

(10) We have measured the oxidation potentials of 1b and 1d as 1.25 and 1.5 V, respectively (vs. Ag/0.1 M AgNO₃) at a Pt disk in acetonitrile containing 0.1 M tetramethylammonium tetrafluoroborate.

was prepared by iodine-catalyzed dehydration of 1-phenylcyclohexanol (Aldrich) in benzene. 1-Methylcyclohexene was obtained from the Aldrich Chemical Co.

6-(1-Hydroxyethyl)-1-phenylcyclohexene (1a). Reduction of 6-acetyl-1-phenylcyclohexene⁵ by lithium aluminum hydride afforded 1a as a mixture of diastereomers, which was separated by medium-pressure liquid chromatography over silica gel with 40:60 ethyl acetate/hexane as the eluent. The slow-moving diastereomer (B) crystallized upon standing and was recrystallized from heptane: mp 71.4–72.0 °C; NMR (CDCl₃) δ 7.4 (s, 5 H), 6.07 (t, 1 H), 4.04 (quintet, 1 H), 2.3 (m, 1 H), 1.4–2.0 (m, 6 H), 1.10 (d, 3 H). Diastereomer A, a yellow oil, failed to crystallize. Its NMR spectrum was essentially identical with that of B, except that the methyl doublet appeared at δ 1.12.

6-(1-Hydroxyethyl)-1-methylcyclohexene (1c) was prepared by lithium aluminum hydride reduction of the corresponding ketone.⁵ The alcohol, not separable into its respective diastereomers by TLC (silica gel, 40:60 hexane/ethylacetate), has the following: bp 100–102 °C (30–35 mm); NMR (CDCl₃) δ 5.60 (br, 1 H), 4.08 (br, 1 H), 1.84 (m, 2 H), 1.62 (d, 3 H), 1.10 (d, 3 H), 0.9–2.0 (m, 6 H). Its α-naphthylurethane (mp 113.8–115.0 °) was recrystallized from heptane for microanalysis. Anal. Calcd for C₂₀H₂₃NO₂: C, 77.64; H, 7.49. Found: C, 77.80; H, 7.60.

Reaction of 6-(1-Hydroxyethyl)-1-phenylcyclohexene (1a) with Mercuric Nitrate. To a suspension of 1.61 g (4.95 mmol) of mercuric nitrate in 50 mL of acetonitrile was added, dropwise with stirring, a solution of 1.00 g (4.95 mmol) of 1b in 15 mL of acetonitrile. After the mixture was stirred 8 h at room temperature, 10 mL of 3 M sodium hydroxide was added dropwise, followed by 10 mL of a 0.5 M solution of sodium borohydride in 3 M sodium hydroxide. After separation of the two phases, the aqueous layer was washed with ether (3 × 25 mL). The combined organic fractions were washed with saturated sodium chloride, dried over magnesium sulfate, and concentrated under reduced pressure to afford 1.02 g of crude product. Preparative TLC was carried out on silica gel (3:1 hexane/ethyl acetate) on a 100-mg sample of the crude product to afford 86.5 mg (80%) of 6-acetamido-1-phenylcyclohexene (2) after recrystallization from heptane: mp 140.3–142.0 °C; IR (CDCl₃) 3400 (NH), 1670 (C=O), 1500 cm⁻¹ (C–N); mass spectrum, *m/e* (relative intensity) 215 (2), 172 (6), 156 (100), 141 (3), 128 (14); NMR (CDCl₃) δ 7.25–7.45 (m, 5 H), 6.32 (t, 1 H), 5.50 (br, 1 H), 5.10 (br, 1 H), 2.20 (br, 2 H), 1.80 (s, 3 H), 1.5–2.3 (m, 4 H). Anal. Calcd for C₁₄H₁₇NO: C, 78.10; H, 7.96. Found C, 77.97; H, 8.19.

The results were identical for both diastereomers of 1a.

Reaction of 1-phenylcyclohexene (1b) with mercuric nitrate was carried out upon 1.0 g of 1b as described for 1a. The crude product, (1.2 g, 70%) crystallized upon evaporation of the ether extract. It was shown to be 2 by TLC *R_f* and its NMR spectrum.

Reaction of 6-(1-hydroxyethyl)-1-methylcyclohexene (1c) with mercuric nitrate was carried out as described for 1a. From 1.0 g of 1c there was obtained 0.9 g of a yellow oil. Analysis of this material by VPC (3 ft × 0.25 in. column packed with 20% SE-30 on 60/80-mesh Fluoropak and operated at 150 °C) indicated it to consist of two major and three minor constituents. Areas under the respective peaks were used to calculate the relative yields of the two major components, which were then isolated by preparative VPC. The faster moving component, 2-(1-acetamidoethyl)-1-acetamidoethyl-1-methylcyclohexene (5, 37%), was identified by spectroscopic and microanalytical data: IR (CDCl₃) 3630, 1645 cm⁻¹; mass spectrum, *m/e* (relative intensity) 181 (7), 135 (100), 79 (94); NMR δ 5.58 (br, 1 H), 4.42 (quartet, 1 H), 2.53 (br, 1 H), 1.86 (s, 3 H), 1.14 (d, 3 H), 1.0–2.0 (m, 10 H). Anal. Calcd for C₁₁H₁₉NO: C, 72.89; H, 10.56. Found: C, 72.63; H, 10.62.

The slower-moving component, 2-(1-hydroxyethyl)-1-methyl-1-acetamidocyclohexane (4, 52%), was also identified by spectral and microanalytical data: NMR δ 4.4 (m, 1 H), 2.53 (br, 1 H), 1.86 (s, 3 H), 1.12 (s, 3 H), 1.1–2.0 (m, 13 H). Anal. Calcd for C₁₁H₂₁NO₂: C, 66.33; H, 10.56. Found: C, 68.17; H, 11.22.

Reaction of 1-methylcyclohexene (1d) with mercuric nitrate was carried out as described for 1a. From 1.0 g of 1d there was isolated 0.85 g (80%) of crude 6-acetamido-1-methylcyclohexene (3), which was purified by preparative TLC and recrystallization from heptane: mp 99.5–101.0 °C; IR (CDCl₃) 3450, 1660, 1530 cm⁻¹; mass spectrum, *m/e* (relative intensity) 153 (12),

96 (47), 94 (100), 83 (54), 79 (82); NMR δ 5.98 (br, 1 H), 5.40 (vbr, 1 H), 4.36 (br, 1 H), 1.97 (s, 3 H), 0.7-2.0 (m, 12 H). Anal. Calcd for $C_9H_{15}NO$: C, 70.56; H, 9.86. Found: C, 70.32; H, 9.64.

2-(1-Hydroxyethyl)-1-methylcyclohexanol (6). Hydroboration of 1.0 g of **1c** was carried out by using a commercial solution of borane in tetrahydrofuran (Alfa), followed by alkaline hydrogen peroxide workup to afford 1.10 g (97%) of diol **6**: mp 81.0-84.0 °C; NMR δ 4.26 (br, 1 H) 1.25-1.95 (m, 11 H), 1.22 (s, 3 H), 1.18 (d, 3 H).

Registry No. **1a** (isomer 1), 83528-99-8; **1a** (isomer 2), 83529-00-4; **1b**, 771-98-2; **1c**, 79828-21-0; **1c** α -naphthylurethane, 83529-06-0; **1d**, 591-49-1; **2**, 83529-01-5; **3**, 83529-02-6; **4**, 83529-03-7; **5**, 83529-04-8; **6**, 83529-05-9; mercuric nitrate, 10045-94-0.

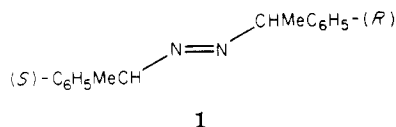
Convenient Synthesis of *meso*-Azobis(α -phenylethane)

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We had the need to develop a method of synthesis of *meso*-azobis(α -phenylethane) [*meso*-bis(1-phenylethyl)-diazene, **1**] which could be used to prepare large quantities (100 g-1 kg) of pure material.



The synthesis of **1** has been reported,¹⁻⁷ and in all cases the starting material of choice has been acetophenone azine (**2**). Catalytic reduction of **2** over Pt or Pd/C in glacial acetic acid^{2,3} (ethanol⁴ or dioxane⁵) afforded the substituted hydrazine **3** as a mixture of racemic and *meso* forms. Oxidation of the isolated hydrazines to **1** was accomplished by treatment of **3** with hydrogen peroxide in the presence of aqueous $NaHCO_3$ ^{2a} (34% yield), oxygen gas over a heptane solution of **3**⁴ (72% yield), air over **3** as a neat oil³ (73% yield), or mercuric oxide in an ether⁶ or benzene^{2b} solution of **3** (47% yield). The method of Scheppele⁷ involved the addition of 1 mol of chlorine to **2** followed by reduction with lithium aluminum hydride (or deuteride) to afford **1** (or *1-d*₂) in 52% yield. In some of these reported syntheses, the description of the procedures was vague¹⁻⁵ and lacked the necessary detail to allow one to synthesize **1** in reasonable quantity and quality without considerable experimental study. In others, the use of expensive reagents (mercuric chloride,^{2b,6} lithium aluminum hydride⁷) discourage their use in preparing larger quantities of **1**.

In our hands, the reduction of the azine **2** (20 g) in glacial acetic acid (200 mL) with hydrogen (~40-50 psi) and 5% or 10% Pd/C proceeded readily to give **3** contaminated

with an appreciable amount of side product,⁸ *dl*- α -phenylethylamine, which remained unchanged in the oxidation step (using either mercuric oxide, oxygen, or air).

The hydrogenation of the azine **2** in ethyl acetate over 10% Pd/C proceeded at a reasonable rate at 30-50 psi and ambient temperature to afford a colorless solution of **3** (single spot by TLC) after removal of the catalyst. This solution can be directly treated (15 min) with dry freshly prepared mercuric oxide to afford **1** (~50% yield after workup); however, it was more convenient to oxidize **3** to **1** by stirring the ethyl acetate solution of **3** under an atmosphere of oxygen at ambient temperature (1.5-3 h) to give **1** in ~50% isolated yield. The procedure described here can be successfully used to prepare **1** on a scale of 10-100 g of isolated crystalline product (mp 72-73 °C) in less than 2 days.

Experimental Section

Melting points were obtained on a Thomas-Hoover capillary melting point apparatus and are uncorrected. The ¹H NMR spectrum of **1** was measured on a Varian Model FT-80 spectrometer. Reactions were monitored and product purity was checked by thin-layer chromatography on precoated silica gel plates (EM Laboratories, 60F-254) by using 3:1 toluene/EtOAc as the developing solvent. The compounds and their approximate *R*_f values were as follows: **1**, 0.80; **2**, 0.76; **3**, 0.26.

Acetophenone Azine (2). The azine **2** was prepared essentially according to the procedure described by Cohen^{2a} by using acetophenone (350 mL, 3.0 mol), 95+% hydrazine⁹ (48 mL, 1.5 mol), 95% EtOH (1500 mL), and glacial HOAc (6 mL) in a 3-L Erlenmeyer flask equipped with a reflux condenser and magnetic stirrer. After the mixture was refluxed for 3.5 h, the reaction was complete (TLC), and after the mixture cooled, 336 g of acetophenone azine (95% yield) was collected as bright yellow prisms, mp 122-123 °C. An additional 11 g of **2** of equal quality could be obtained by concentration of the mother liquor (to 400 mL) for a total yield of 98%.

***meso*-Azobis(α -phenylethane) (1).**¹⁰ A 2-L Parr hydrogenation bottle was charged with acetophenone azine (mp 122-123 °C; 200.0 g, 846 mmol), EtOAc (1000 mL),¹¹ and 10% Pd/C (5.0 g) and shaken with H₂ on a Parr Model 3921 low-pressure hydrogenation apparatus at 30-50 psi for 10 h, at the end of which time the absorption of hydrogen ceased, and 2 equiv of H₂ had been absorbed. The reaction mixture was filtered through a pad of Celite (suction) to remove the catalyst. The colorless filtrate (TLC, single spot, *R*_f 0.26, 3:1 toluene/EtOAc) was placed in a 2-L round-bottomed flask equipped with a magnetic stirrer, and the system was flushed with oxygen and connected to a reservoir of oxygen (balloon). After 0.5 h, the solution had turned pale yellow and warmed slightly. After 3 h, the oxidation was complete (TLC, single spot, *R*_f 0.80, 3:1 toluene/EtOAc). Most of the EtOAc was removed on a rotary evaporator at 30 °C (25 mmHg) to give 240 g of a pale yellow oil (product plus EtOAc) to which was added 100 mL of MeOH. The resulting solution was cooled to 0 °C and stored for several hours in an ice bath, after which time **1** was collected as colorless crystals which were washed with two 20-mL portions of cold (ice bath) MeOH.¹² The air-dried product (105 g, 52% yield; mp 68-70 °C) was recrystallized from 230 mL of

(8) The side product (7.6 g) was isolated after oxidation of the crude **3** in ether solution with oxygen (1.5 days) by extraction with 5% aqueous HCl from which it was liberated with aqueous KOH. It was shown to be identical with *dl*- α -phenylethylamine (formed by hydrogenolysis of the NH-NH bond in **3**) by comparison with an authentic sample (¹H NMR, IR, and benzamide melting point, 120-121 °C).

(9) The use of 99% hydrazine hydrate (73 mL, 1.5 mol) could be employed with equal success.

(10) This procedure has also been carried out on smaller scales by using the same proportions of **2** (160, 40, or 20 g) to EtOAc (800, 200, or 100 mL) and 10% Pd/C (4, 1, or 0.5 g) with the same overall percent yields of pure **1**.

(11) The amount of azine used is only partly soluble in this amount of EtOAc but dissolves as the hydrogenation proceeds.

(12) The mother liquor from this crystallization contained only azo compound (TLC, *R*_f 0.80) which was shown by ¹H NMR analysis to have the composition of approximately 7 parts of racemic **1** to 1 part of *meso*-**1**.

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