Table I. Reaction of *N*-Nitrosodibenzylamine (1) with Azides[a]

Registry no.	Azide	Recovered 1, %	$PhCH_2$-CH_2Ph, %	Other products
622-37-7	(1) Phenyl azide	81.5	Traces	*b*
1516-60-5	(2) 4-Nitrophenyl azide	48.5	46	
18523-41-6	(3) 4-Cyanophenyl azide	93.7	48	
31656-77-6	(4) 2-Cyanophenyl azide	66.5	47	
3296-05-7	(5) 4-Chlorophenyl azide	79.5	0	*c*
62416-01-7	(6) 2-Chloro-4-nitrophenyl azide	63	14	*d*
62416-02-8	(7) 2,3-Dichlorophenyl azide	0	1	*c, d*
16714-27-5	(8) 2-Benzoylphenyl azide	90	Traces	*e*
1516-58-1	(9) 2-Nitrophenyl azide	80	0	*f*
62460-41-7	(10) 2-Cyano-4-nitrophenyl azide	9.7	69.5	*d*
941-55-9	(11) Tosyl azide	>99	Traces	
1070-19-5	(12) *tert*-Butyl azidoformate	89	0	

[a] All reactions were carried out in enough chlorobenzene to give a homogeneous solution using 5 mmol of *N*-nitrosodibenzylamine and 10 mmol of the azides. The yields of bibenzyl are based on the amount of unrecovered nitrosamine. Other products such as the azo compounds and biphenyls were formed. [b] In this reaction, azoxybenzene was sought (see ref 8) and detected by thin-layer chromatography. [c] Trace amounts of benzylidenedibenzylhydrazine (5) were detected. [d] Benzaldehyde was identified as a by-product. [e] 3-Phenylanthranil was isolated in nearly quantitative yield. [f] Benzofuroxan was isolated in 32% yield.

the deoxygenation of 1. That this expectation was fully warranted was shown by the fact that a 70% yield of bibenzyl was obtained with less than 10% of recovered *N*-nitrosamine. Our results indicated that aryl azides may be useful for the removal of semiionic oxygen in compounds such as *N*-oxides, azoxy compounds, and nitrones.

Experimental Section

Materials. *N*-Nitrosodibenzylamine was prepared according to the literature procedure.[12] The azides were obtained from the corresponding amines.[13] Three new azides were prepared by the same method.

2-Chloro-4-nitrophenyl azide (70% yield): mp 65–66 °C, pale yellow needles from a mixture of acetone–95% ethanol. Anal. Calcd for $C_6H_3ClN_4O_2$: C, 36.29; H, 1.52; N, 28.22. Found: C, 36.23; H, 1.71; N, 28.29.

2,3-Dichlorophenyl azide (76% yield): mp 61–62 °C, pale yellow needles from 95% ethanol. Anal. Calcd for $C_6H_3Cl_2N_3$: C, 38.33; H, 1.61; N, 22.35. Found: C, 38.54; H, 1.90; N, 22.50.

2-Cyano-4-nitrophenyl azide (17% yield): mp 107–108 °C, pale yellow needles from 95% ethanol. Since this azide deteriorates on standing, an elemental analysis was performed on its triphenylphosphine imine adduct, mp 247–248 °C (from benzene). Anal. Calcd for $C_{25}H_{18}N_3O_2P$: C, 70.92; H, 4.25; N, 9.93. Found: C, 71.09; H, 4.35; N, 9.87.

Typical Procedure. A solution of 1.14 g (5 mmol) of *N*-nitrosodibenzylamine and of 2-cyano-4-nitrophenyl azide (1.89 g, 10 mmol) in 40 mL of chlorobenzene was purged with nitrogen for 30 min. The solution was then heated to reflux for 48 h with stirring in a nitrogen atmosphere. After careful evaporation of the solvent, the residue was chromatographed on silica gel (60–200 mesh, 50 g) using hexane, varying mixtures of hexane–benzene, and finally benzene.

All products reported were characterized by direct comparison with an authentic sample by at least one of the following methods: IR, NMR, mixture melting point, and TLC retention time.[14]

Acknowledgment. The support of this work by the National Institutes of Health (GM 13689-10) is acknowledged with gratitude.

Registry No.—1, 5336-53-8; $PhCH_2CH_2Ph$, 103-29-7; 2-cyano-4-nitrophenyl azide triphenylphosphine imine adduct, 55210-55-4.

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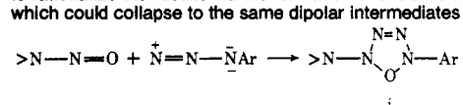
(9) R. A. Abramovitch and E. P. Kyba, "Chemistry of the Azido Group", S. Patai, Ed., Interscience Publishers, New York, N.Y., 1971, p 270. It is also possible to rationalize the results via the formation of unstable oxatetrazolines (i) which could collapse to the same dipolar intermediates (ii). On the basis



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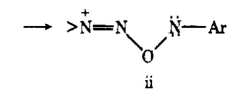
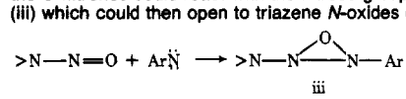


ii

of the present data, we favor the nitrene mechanism. In the same context, the *C*-nitrenes could react with the nitroso group to give oxadiaziridines (iii) which could then open to triazene *N*-oxides (iv) which are known to



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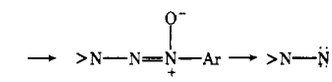
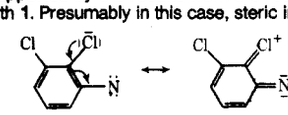


iv

fragment to the *N*-nitrenes (M. Koga and J.-P. Anselme, unpublished results).
(10) While the inductive effect of a chloro substituted at any position would be expected to make the nitrene *more* electron deficient, in the ortho and para positions the chlorine may act as an electron-donating group to render the nitrene *less* electron deficient. Thus, if this putative resonance stabilization and concomitant deactivation of the nitrene by a chloro substituent could be inhibited, then deoxygenation of 1 should be possible. This hypothesis seems to be supported by the results from the reaction of 2,3-dichlorophenyl azide with 1. Presumably in this case, steric inhibition of resonance,



i ii

such as shown, allows the chlorine in the ortho position to exert only its inductive effect. See Y. T. Struckhov and S. L. Solenova-Sidorova, *Bull. Acad. Sci. USSR, Div. Chem. Sci.*, 93 (1960); S. L. Chien and R. Adams, *J. Am. Chem. Soc.*, **56**, 1787 (1934); see also J. March, "Advanced Organic Chemistry", McGraw-Hill, New York, N.Y., 1968, p 123.
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(14) See footnote *a* of Table I.

2-Methyl-3-butyn-2-ol as an Acetylene Precursor in the Mannich Reaction. A New Synthesis of Suicide Inactivators of Monoamine Oxidase[1]

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The propargylamines, *N*-[3-(2,4-dichlorophenoxy)propyl]-*N*-methyl-2-propynylamine (clorgyline, **1a**) and L-

N,α-dimethyl-*N*-2-propynylphenethylamine (L-deprenyl, **1b**) have been shown to be selective irreversible inhibitors of the isoenzymes of monoamine oxidase (MAO) type A and B, respectively.[2,3] Another propargylamine of this type, *N*-methyl-*N*-2-propynylbenzylamine (pargyline, **1c**), a more general MAO inhibitor, has been used therapeutically as an antihypertensive agent.[4]

In our studies of the metabolic fate of suicide enzyme inactivators of MAO A and B, we required a synthetic sequence which could be used to label the methylene carbon of the propargyl group of clorgyline (**1a**) and deprenyl (**1b**) with either radioactive (^{14}C or ^{11}C) or stable (^{13}C) isotopes of carbon. The introduction of isotopic carbon at this position using labeled formaldehyde in the Mannich reaction with acetylene and the appropriate secondary amine formally represented a sequence which could be used to yield the appropriately labeled propargylamines. However, the inconvenience and potential hazards associated with the use of acetylene as well as the formation of disubstituted by-products prompted us to investigate an alternative synthesis of **1a** and **1b**. 2-Methyl-3-butyn-2-ol (bp 104 °C) appeared to offer an attractive alternative to the use of acetylene in the Mannich reaction since it has been reported to act as an active hydrogen compound in the presence of cuprous catalysts,[5] and it is well known that acetylenic carbinols undergo KOH-catalyzed decomposition to the acetylene and carbonyl compound.[5,6]

We report here the synthesis of **1a–c** via the Mannich reaction with 2-methyl-3-butyn-2-ol followed by KOH-catalyzed elimination of acetone from the acetylenic carbinols **3a–c** (Scheme I). This reaction sequence has been used to synthesize ^{14}C-labeled **1a** and **1b**.[7]

In summary, this two-step reaction results in the formation of propargylamines in moderate yields (33–48%). The overall transformation is that of the Mannich reaction of acetylene without the hazards and inconvenience of using acetylene or the formation of complex mixtures of products by the substitution of both active hydrogen atoms on acetylene.

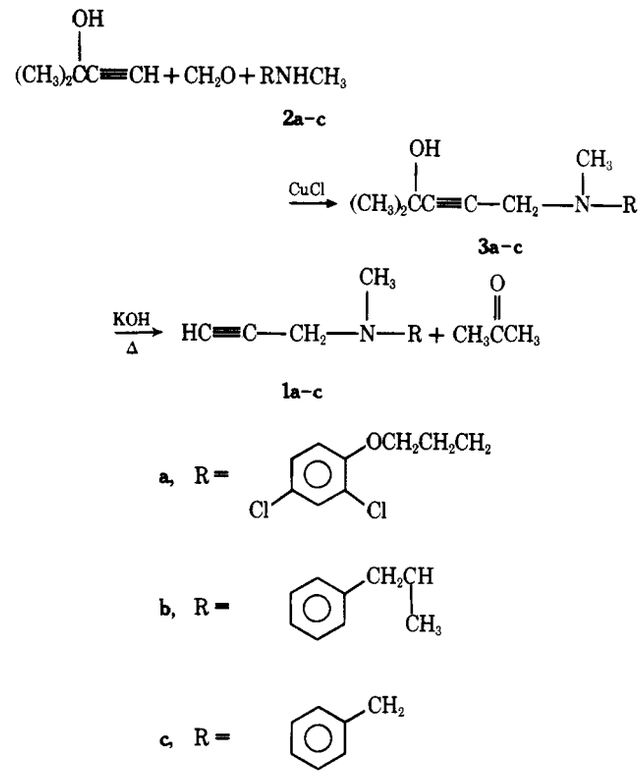
Experimental Section

Melting points are uncorrected. NMR spectra were run on a JEOL JNM-MH-100 using Me_4Si as an internal standard. Optical rotations were obtained using a Rudolf Research automatic polarimeter (Model 26202).

***N*-[3-(2,4-Dichlorophenoxy)propyl]-*N*-methyl-2-propynylamine (Clorgyline, 1a).** To 0.048 g (0.177 mmol) of 3-(2,4-dichlorophenoxy)-*N*-methylpropylamine hydrochloride[8] (**2a** HCl) in 0.2 mL of H_2O was added 0.0144 mL (0.177 mmol) of 37% CH_2O in 0.2 mL of H_2O. This was warmed slightly and 9 mg of CuCl added. The mixture was adjusted to pH 8 with 0.2 mL of 7.5% $NaHCO_3$ and 0.0172 mL of 2-methyl-3-butyn-2-ol in 0.6 mL of dioxane. The mixture was heated (110 °C) and stirred for 3 h and allowed to cool to room temperature, and 0.6 mL of ammonium hydroxide (concentrated) was added. The mixture was extracted with ether, the extracts were dried (K_2CO_3), and the solvent was removed to yield 0.050 g (86%) of **3a** a pale yellow oil having NMR ($CDCl_3$) δ 7.46–6.76 (m, 3 H, aromatic H), 4.06 (t, 2 H, J = 6 Hz, $-OCH_2-$), 3.34 (s, 2 H, $-C{\equiv}CCH_2N<$), 2.60 (t, 2 H, J = 7 Hz, $>NCH_2CH_2$), 2.30 (s, 3 H, $>NCH_3$), 1.96 (p, 2 H, J = 7 Hz, $-CH_2CH_2CH_2-$), 1.50 [s, 6 H, $(CH_3)_2C-$]. The compound was used in the next step without further purification.

To a film of KOH (0.2 mL of 1 M KOH, lyophilized on the bottom and sides of a test tube with a vacuum adapter and heated under vacuum for 2 min at 150 °C) was added an ethereal solution of **3a** (0.050 g). This was evaporated to dryness using a stream of N_2, a cold finger attached, and the residue heated under vacuum (1 mm) at 150 °C for 1 min. The material adhering to the cold finger was dissolved in ether–hexane (1:1) and passed over a 4 × 0.5 cm silica gel column and eluted with ~3 mL of the same solvent. The solvent was removed leaving 0.023 g (48% based on **2a**) of **1a**, a pale yellow oil, having NMR ($CDCl_3$) δ 7.46–6.76 (m, 3 H, aromatic H), 4.04 (t, 2 H, $-OCH_2-$), 3.32 (d, 2 H, J = 2 Hz, $HC{\equiv}CCH_2-$), 2.60 (t, 2 H, J = 7 Hz, $>NCH_2CH_2-$), 2.30 (s, 3 H, $>NCH_3$), 2.18 (t, 1 H, J = 2 Hz, $HC{\equiv}C-$), 1.96 (p, 2 H, J = 7 Hz, $-CH_2CH_2CH_2-$). The NMR spectrum was identical with that of an authentic sample of clorgyline.[9] TLC on silica gel in ether–hexane (1:1) showed one spot with R_f 0.36. Treatment of 0.023 g of **1a** in dry ether with HCl produced 0.025 g of **1a** HCl, mp 97–99 °C (lit.[8] mp 98.5–100 °C).

Scheme I



L-*N*,α-Dimethylphenethylamine (2b). To 1.6 g (0.0098 mol) of L-*N*-formyl-1-phenyl-2-aminopropane (prepared from L-amphetamine according to the procedure of Cavallito and Gray[10]) in 15 mL of ether at 0 °C was added dropwise 8 mL of $LiAlH_4$ solution (1 M in ether). The mixture was stirred at 25 °C for 8 h and decomposed with 1.25 mL of 3% NaOH. The mixture was filtered and the filtrate extracted with 1 N HCl. Addition of NaOH to the HCl solution followed by extraction with ether gave 0.63 g (43%) of a colorless oil. The NMR ($CDCl_3$) showed δ 7.50–7.14 (m, 5 H, aromatics), 3.02–2.54 (m, 3 H, $-CH_2CH<$), 2.42 (s, 3 H, $>NCH_3$), 1.34 (s, 1 H, $-NH-$), 1.06 (d, 3 H, J = 6 Hz, $CH_3CH<$). Treatment of an ethereal solution of the amine with HCl produced **2b** HCl which had mp 168–171 °C (lit.[11] 170–171 °C) and α^{25}_D −12.6° (*c* 16.1, H_2O) (lit.[11] α^{22}_D −14.8°).

L-*N*,α-Dimethyl-*N*-2-propynylphenethylamine (L-Deprenyl, 1b). Using the same general procedure described above, 0.033 g (0.178 mmol) of **2b** HCl was allowed to react with formaldehyde and 2-methyl-3-butyn-2-ol at 110 °C for 4 h to yield 0.037 g of a pale yellow oil which NMR showed to contain 71% of **3b** (60% yield). The NMR ($CDCl_3$) showed δ 7.42–7.06 (m, 5 H, aromatics), 3.42 (s, 2 H, $-C{\equiv}CCH_2N<$), 3.14–2.90 (m, 2 H, $-CH_2C_6H_5$), 2.52–2.22 (m, 1 H, $>NCH<$), 2.38 (s, 3 H, $>NCH_3$), 1.52 [s, 6 H, $(CH_3)_2C<$], 0.96 (d, 3 H, J = 6 Hz, $CH_3CH<$). This compound was used in the next step without further purification.

Pyrolysis of **3b** as described above yielded 11 mg of **1b** (33% based on **2b**), a colorless oil having NMR ($CDCl_3$) δ 7.4–7.04 (m, 5 H, aromatics), 3.42 (d, 2 H, J = 2 Hz, $HC{\equiv}CCH_2-$), 3.16–2.84 (m, 2 H, $-CH_2C_6H_5$), 2.56–2.32 (m, 1 H, $>NCH<$), 2.40 (s, 1 H, $>NCH_3$), 2.22 (t, 1 H, J = 2 Hz, $HC{\equiv}C-$), 0.96 (d, 3 H, J = 6 Hz, $CH_3CH<$). The NMR spectrum was identical with that of an authentic sample of deprenyl[12] and showed no other signals. Treatment of **1b** (0.011 g) in dry ether with HCl followed by crystallization from methanol–ether gave 0.0095 g of **1b** HCl, mp 141–142 °C (lit.[13] mp 141 °C), α^{25}_D −10.8° (*c* 6.48, H_2O) (lit.[13] α^{20}_D −11.2°).

***N*-Methyl-*N*-2-propynylbenzylamine (Pargyline, 1c).** The reaction of **2c** HCl (0.028 g, 0.178 mmol) with CH_2O and 2-methyl-3-butyn-2-ol at 110 °C for 8 h as described yielded 0.032 g of **3c** (83%), a pale yellow oil having NMR ($CDCl_3$) δ 7.45–7.21 (m, 5 H, aromatics), 3.57 (s, 2 H, $C_6H_5CH_2N<$), 3.31 (s, 2 H, $>NCH_2C{\equiv}C-$), 2.32 (s, 3 H, $>NCH_3$), 2.28 (s, broad, 1 H, $-OH$), 1.55 [s, 6 H, $(CH_3)_2C<$].

To 0.2 mL of 1 M KOH was added **3c** (0.032 g) in 0.2 mL of MeOH. The mixture was evaporated (1 mm), leaving a residue which was pyrolyzed and purified as described previously to yield 8 mg (41% based on **2c**) of **1c**, a colorless oil having NMR ($CDCl_3$) δ 7.44–7.16 (m,

5 H, aromatics), 3.58 (s, 2 H, $C_6H_5CH_2N<$), 3.32 (d, 2 H, J = 2 Hz, $>NCH_2C{\equiv}C-$), 2.33 (s, 3 H, $>NCH_3$), 2.25 (t, 1 H, J = 2 Hz, $HC{\equiv}C-$). The NMR spectrum was identical with that of authentic pargyline.[14] Treatment of **1c** in dry ether with HCl followed by crystallization from MeOH–ether gave 0.007 g of **1c** HCl, mp 158.5–159 °C (lit.[15] mp 154–155 °C).

Acknowledgment. The author is grateful to Richard Ehrenkaufer, Brian Gallagher, David Lloyd, Robert MacGregor, and Alfred Wolf for helpful discussions and suggestions.

Registry No.—**1a,** 17780-72-2; **1a** HCl, 17780-75-5; **1b,** 14611-51-9; **1b** HCl, 14611-52-0; **1c,** 555-57-7; **1c** HCl, 306-07-0; **2a** HCl, 62505-88-8; **2b,** 33817-09-3; **2b** HCl, 826-10-8; **2c** HCl, 13426-94-3; **3a,** 62505-89-9; **3b,** 62505-90-2; **3c,** 62505-91-3; 2-methyl-3-butyn-2-ol, 115-19-5; L-*N*-formyl-1-phenyl-2-aminopropane, 62532-67-6.

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α-Alkylation and Michael Addition of Amino Acids—a Practical Method

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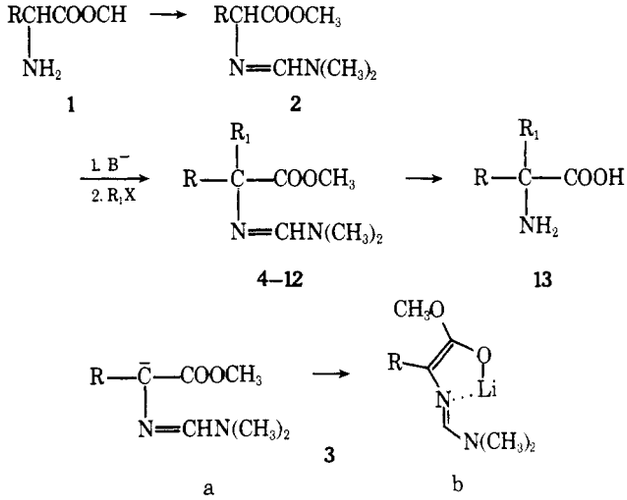
In recent years various methods have been developed to effect alkylation of amino acid derivatives at their α carbon.[1] One of the more versatile variants is the alkylation of deprotonated α-isocyano esters developed by Schöllkopf.[1a] Yet most of these procedures—the latter one included—suffer from the drawback of a multistep procedure necessary to sequentially protect carboxylic acid and amine functions in order to prepare a derivative suitable for α-deprotonation. We here wish to report a new and practical approach which is general for *all* α-amino acids and obviates the need for a laborious sequential protection.

The treatment of carboxylic acids in general and N-protected amino acids in particular with the acetals of dimethylformamide is known as an efficient and high-yielding method to prepare the corresponding esters.[2–6] Aminolysis of these reagents, on the other hand, particularly with primary aromatic amines, leads to substituted formamidines.[7–9] The simultaneous application of these two reactions for the conversion of a free amino acid into its α-formamidine methyl ester has not been used previously for preparative synthetic purposes.[10] Our results indicate that such ester formamidines are ideally suited as intermediates for α-alkylations.

By refluxing any α-amino acid **1** in 2–2.5 equiv of dimethylformamide dimethyl acetal (cf. ref 10) for 1–6 h, an essentially quantitative conversion to the distillable and reasonably stable[11] amidino esters **2** is achieved.[12] As we have discovered



independently, the conditions for deprotonation to **3** as well as its reactivity are very similar to those recently reported by Stork[1b] for the benzylidene derivative of glycine ethyl ester. Deprotonation can be achieved either with lithium diisopropylamide in THF at temperatures ranging from 0 to −70 °C or in certain instances with potassium *tert*-butoxide in CH_3OH. The anion **3** is sufficiently reactive toward alkylating agents such as alkyl iodides, allylic halides and even epoxides to give the products **4–12** in good to excellent yields[13] (see Table I). We have reason to believe that deprotonation of **2** initially leads to **3a** which readily tautomerizes to the lithium enolate **3b,** highly favored by the chelating effect of the unshared pair of electrons of the amidine nitrogen. In the case of the phenylglycine derivative **2c** (R = C_6H_5), low-temperature (−78 °C) deprotonation by LDA produces an intense red-orange color, characteristic of stabilized benzylic anions, gradually fading to a light orange-yellow. The infrared spectrum of the anionic species **3** (in CH_3CN) indicates no ester absorption but instead a strong band at 1630 cm^{-1} (C=N and C=C).[14]

The high degree of chelation in **3** appears to be the reason for its unusually soft character (cf. ref 1b): in sharp contrast to the reactivity of the α-isocyano esters,[15,16] **3** does not react with ketones (benzophenone), and only sluggishly with benzaldehyde. This reactivity pattern is ideal for 1,4-additions which indeed occur readily either in aprotic or protic solvents (cf. ref 1b) (see Table I). Hydrolysis of the products **4–12** can be achieved in refluxing concentrated hydrochloric acid to produce the amino acids **13–15.** Unlike the imine functionality (cf. ref 1b), the dimethylformamidine moiety appears to be remarkably stable toward dilute mineral acids at room temperature.

Thus, we have outlined a practical method which permits the effective α-alkylation of any α-amino acid in a total of three steps: (1) simultaneous protection of both functional groups with dimethylformamide dimethyl acetal, (2) α-alkylation (or Michael addition), (3) acidic hydrolysis.

Experimental Section

The physical data were obtained as follows: melting points in a Thomas-Hoover melting point apparatus (uncorrected); IR spectra on a Perkin-Elmer 521; mass spectra on a AEI MS 902 by direct in-