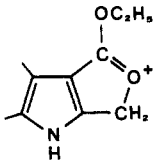
polarization of the C–Cl bond which makes hydrolysis of the chloromethyl group easier.

Pyrroles **1**, **2**, and **4** undergo hydrolysis as easily as pyrroles **3** and **5**; the hydroxymethyl derivatives of the former are, however, unable to alkylate *p*-xylene in the presence of acids, giving rise instead to self-condensation products (Table I). An obvious conclusion is that the enhanced alkylating ability of the azafulvalenium ions deriving from pyrroles **3** and **5** depends on the presence of an ethoxycarbonyl function adjacent (β) to the methylene group. This specific neighboring-group effect would stabilize the positive charge through the contribution of acyloxonium-like structures[22] such as **13**, which strongly



13

reduces the extent of its delocalization on the pyrrole nucleus, thus causing an overall increase in reactivity.

Registry No. **1**, 73018-14-1; **2**, 73018-12-9; **3**, 57745-26-3; **4**, 51740-95-5; **5**, 5408-12-8; **6**, 5422-89-9; **6** (cation), 83633-46-9; **7**, 83633-45-8; **8**, 83633-44-7; **9**, 7164-22-9; **10**, 83633-42-5; **11**, 83633-43-6; benzene, 71-43-2; *p*-xylene, 106-42-3; durene, 95-93-2; diethyl 3,5-dimethylpyrrole-2,4-dicarboxylate, 2436-79-5; [3,3′,5,5′-tetrakis(ethoxycarbonyl)-4,4′-dimethyl-2,2′-dipyrryl]-methane, 5431-96-9; ethyl 4-bromo-3,5-dimethylpyrrole-2-carboxylate, 5408-07-1; ethyl 2-(hydroxyimino)-3-oxobutanoate, 5408-04-8; ethyl 3-oxo-4-phenylbutanoate, 718-08-1; ethyl 4-chloro-3,5-dimethylpyrrole-2-carboxylate, 58921-31-6.

(22) Gould, E. S. "Mechanismus und Struktur in der Organischen Chemie"; Verlag Chemie: Weinheim/Bergstr., Germany, 1964; p 679.

New Reaction of (Diethylamino)sulfur Trifluoride: Bis(diphenylmethyl) Ethers as Dehydration Products of (Diethylamino)sulfur Trifluoride and Diarylcarbinols†

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The use of (diethylaminosulfur)trifluoride [DAST, $(C_2H_5)_2NSF_3$] as a mild fluorinating agent has been described by Middleton[1a,b] and Markovskij.[1c] In particular, this reagent is useful for preparing monofluorides from alcohols and *gem*-difluorides from aldehydes and ketones under nonacidic conditions at low temperature. This represents an advance over the parent reagent, sulfur tetrafluoride (SF_4), which usually requires the presence of HF and higher temperatures than required for DAST for the successful replacement of oxygen functions by fluorine.[2]

In seeking a mild way to prepare fluorodiarylmethanes, I examined the reactions of some diarylcarbinols with DAST. As a result, I discovered a new reaction of DAST, namely, intermolecular dehydration of diarylcarbinols (**1**) to bis(diarylmethyl) ethers (**2**) in addition to, or instead of, replacement of OH by F to form fluorodiarylmethanes (**3**). The formation of ethers from aliphatic alcohols has been observed with SF_4[2b] and from halogenated acetaldehydes with both SF_4 and (dialkylamino)sulfur trifluorides at room temperature.[3] However, ether formation from diarylcarbinols and DAST occurs readily at −30 °C and represents a new and extremely simple way of forming these compounds from the parent alcohols. The ether-forming reaction appears to be limited to diarylcarbinols, both benzyl alcohol[1a] and triphenylcarbinol (see eq 1–3) giving the "normal" arylmethyl fluorides.

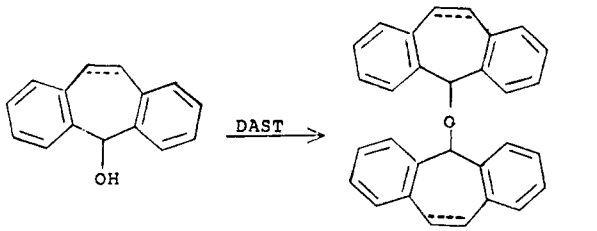
$$C_6H_5CH_2OH + (C_2H_5)_2NSF_3 \rightarrow C_6H_5CH_2F \quad (1)$$

$$\underset{\mathbf{1}}{Ar_2CHOH} + \underset{\text{DAST}}{(C_2H_5)_2NSF_3} \rightarrow \underset{\mathbf{2}}{Ar_2CHOCHAr_2} + \underset{\mathbf{3}}{Ar_2CHF} \quad (2)$$

$$(C_6H_5)_3COH + (C_2H_5)_2NSF_3 \rightarrow (C_6H_5)_3CF \quad (3)$$

Table I summarizes the overall results of four examples of this reaction which were examined in detail.

This reaction is an especially convenient way to prepare the ethers derived from dibenzsuberol and its 10,11-dehydro derivative, shown below. In the case of benzhydrol itself (**1d**; Ar = C_6H_5) essentially equal amounts of ether, **2d**, and fluoride, **3d**, are isolated after purification. 9-Fluorenol, **1c**, reacts in apparent good conversion to form a crude mixture of **2c** and **3c**, identified by NMR, but on further workup only **2c** was isolated pure in low yield, the fluoride fractions undergoing quite rapid decomposition at 25 °C.



1a 10,11-single bond
b 10,11-double bond

2a 10,11-single bond
b 10,11-double bond

The reaction is conveniently carried out by stirring the appropriate secondary alcohol, **1**, in dichloromethane at −30 °C and adding DAST dropwise in an equimolecular amount. In the case of **1b**, a transient magenta color was

Table I. Formation of Bis(diarylmethyl) Ethers Using DAST

example	diarylcarbinol **1**	product yield, % **2**	**3**
a	dibenzsuberol	48–64	0
b	dibenzcycloheptenol	67	0
c	9-fluorenol	13	48[a]
d	benzhydrol	44	40

[a] The indicated products were obtained pure in the yields shown except **3c**, for which the crude yield is reported, this compound being unstable to storage and purification.

† Contribution No. 3035 from the Central Research and Development Department.

(1) (a) Middleton, W. J. *J. Org. Chem.* **1975**, *40*, 574. (b) Middleton, W. J.; Bingham, E. M. *Org. Synth.* **1977**, *57*, 50. (c) Markovskij, L. N.; Pashinnik, V. E.; Kirsanov, A. V. *Synthesis* **1973** 787.

(2) (a) Boswell, G. A.; Ripka, W. C.; Scribner, R. M.; Tullock, C. W. *Org. React.* **1974**, *21*, 1. (b) Hasek, W. R.; Smith, W. C.; Engelhardt, V. A. *J. Am. Chem. Soc.* **1960**, *82*, 543.

(3) Siegemund, G. *Liebigs Ann. Chem.* **1979**, 1280–1290.

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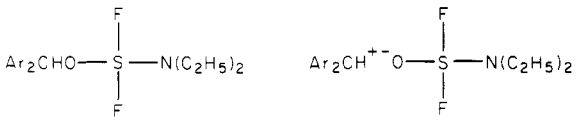
observed during the reaction process, which I attribute to the presence of a carbonium ion. Products **2** and **3** were separable by fractional crystallization. The use of hexane as the reaction solvent would be expected to improve the yield of **3** by discouraging ionization of the intermediate, but it is unsatisfactory because of the low solubility of the starting materials, **1**, in this medium.

Explanation of ether formation from these secondary alcohols must accommodate simultaneous fluoride formation and the observations that only fluoride products are formed from benzyl alcohol[1a] and triphenylcarbinol. Middleton[1a] has postulated that DAST reacts with alcohols to form an intermediate (dialkylamino)sulfonium difluoride, **4**, which undergoes internal collapse to fluoride



4 **5**

product **3**. The formation of olefins and Wagner–Meerwein rearrangement products observed by Middleton[1a] is explained by the carbonium ion character of **4**.

Benzyl fluoride is obviously a "normal" product arising from collapse of the complex **4**.[1a] For diaryl- and triarylcarbinols the increase in steric bulk of the starting materials and intermediates and the enhanced resonance stabilization of the carbonium ion in **5**, formed by autodecomposition of **4**, are important factors. The stabilities of a series of diarylcarbonium ions with respect to electronic effects have been studied by Volz and Mayer.[4] Rozhkov and Knunyants[10b] cite the formation of trityl fluoride as evidence for a radical cation in the anodic oxidation of triphenylmethane in the presence of fluoride ion. The mixed products obtained from DAST and diarylcarbinols are explained by competition between internal collapse of **4** to produce fluoride **3** and attack of the carbonium ion in **5** by unreacted diarylcarbinol to produce **2**. It is possible that some **3** arises indirectly by attack of the small F^- anion on **5** instead of by transfer within the ion pair. For triphenylcarbinol, the exclusive formation of fluorotriphenylmethane suggests that the product is formed by attack of F^- on triphenylmethyl cation, the bulky triphenylcarbinol being unable to compete effectively for the bulky cation.

Experimental Section

General Procedure. A mixture of alcohol **1** (25–50 mmol) in CH_2Cl_2 (75–150 mL) was stirred at −30 °C and treated dropwise with DAST (4.25–8.50 g, 25–50 mmol),[1b] equimolecular proportions of **1** and DAST being taken for each experiment. After 30 min at −30 °C, the mixture was washed with ice-cold 5% $NaHCO_3$ (100–200 mL) and brine (50–100 mL), dried over sodium sulfate, and evaporated at 25 °C to leave the crude product from which the bis(diarylmethyl) ether could be isolated by recrystallization. The fluoride product remained in the mother liquors for subsequent purification following filtration of the ether product.

5-[(10,11-Dihydro-5*H*-dibenzo[*a*,*d*]cyclohepten-5-yl)oxy]-10,11-dihydro-5*H*-dibenzo[*a*,*d*]cycloheptene (2a). The crude product (6.12 g) from dibenzsuberol (**1a**; 5.26 g, 25 mmol) was recrystallized from a mixture of EtOAc (25 mL) and hexane (5–10 mL) with recovery of 2.52–3.21 g (6.0–8.0 mmol, 48–64%) of **2a**; no evidence was seen for any fluoride product in the crude material by ^1H or ^{19}F NMR. The analytical sample was recrystalled once more and dried at 25 °C (0.1 mm). Pure 5-[(10,11-dihydro-5*H*-dibenzo[*a*,*d*]cyclohepten-5-yl)oxy]-10,11-dihydro-5*H*-dibenzo[*a*,*d*]cycloheptene has the following: mp 127–128 °C; IR ($CHCl_3$) ν_{max} 3020, 3000, 2920, 2880, 2600, 1480, 1110 cm^{-1}; UV (EtOH) λ_{max} 266 nm (ϵ 1300), 273 (1000); ^1H NMR ($CDCl_3/Me_4Si$) δ 7.03 (m, C_6H_4), 5.37 (br s, OCH), 5.40–4.27 (m, CH_2CH_2); ^{13}C NMR ($CDCl_3$) δ 139.32, 138.56, 130.24, 129.40, 127.97, 125.82; (C_6H_4) 82.02, 78.38, 76.95, 75.58, (CH_2CH_2); 32.17 (OCH); mass spectrum, *m/e* 402.1978 (calcd for $C_{30}H_{26}O$ parent 402.1982), 209.0966 (calcd for $C_{15}H_{13}O$ fragment 209.0966), 193.1019 (calcd for $C_{15}H_{13}$ fragment 193.1016). Anal. Calcd for $C_{30}H_{26}O$: C, 89.51; H, 6.51. Found: C, 89.76; H, 6.76.

5-[(5*H*-Dibenzo[*a*,*d*]cyclohepten-5-yl)oxy]-5*H*-dibenzo[*a*,*d*]cycloheptene (2b). Similar treatment of 10.4 g (50 mmol) of 5*H*-dibenzo[*a*,*d*]cyclohepten-5-ol with DAST (50 mmol) gave 6.68 g (17 mmol, 67%) of **2b** after recrystallization from 100 mL of 1:1 EtOAc/hexane. A transient magenta color was observed during the DAST addition, and no fluoride product was detected in the crude reaction product by ^{19}F or ^1H NMR. The analytical sample was recrystallized again and dried at 100 °C (0.1 mm). Pure 5-[(5*H*-dibenzo[*a*,*d*]cycloheptene-5-yl)oxy]-5*H*-dibenzo[*a*,*d*]cycloheptene has the following: mp 219–221 °C dec (lit.[5] mp 208–211 °C); IR (KBr) ν_{max} 3050, 3010, 1590, 1560, 1480, 1110 cm^{-1}; UV (EtOH) λ_{max} 282 nm (ϵ 26 140); ^1H NMR ($CDCl_3/Me_4Si$) δ 7.92 (br d, J = 7 Hz, ortho protons), 7.58–7.00 (m, meta and para protons), 6.83 (s, CH=CH), 4.98 (s, OCH); mass spectrum, *m/e* 398 ($C_{30}H_{22}O$, parent ion, too weak for accurate measurement), 207.0797 (calcd for $C_{15}H_{11}O$ fragment 207.0809), 191.0873 (calcd for $C_{15}H_{11}$ fragment 191.0860). Anal. Calcd for $C_{30}H_{22}O$: C, 90.42; H, 5.56. Found: C, 90.09; H, 5.71.

5-[(5*H*-Dibenzo[*a*,*d*]cyclopent-5-yl)oxy]-5*H*-dibenzo[*a*,*d*]cyclopentane (2c) and 9-Fluorofluorene (3c). Similar treatment of 9-fluorenol (4.60 g, 25 mmol) with DAST (25 mmol) gave 6.24 g of crude product as a gummy residue which appeared to be a 3:2 mixture of **2c** and **3c** by ^1H NMR by using the ratio of signal areas at 6.10 (d, J = 48 Hz, CHF) and 5.95 ppm (s, CHO); there was a signal at −187.75 ppm (d, J = 48 Hz) in the ^{19}F NMR spectrum. Attempted removal of the fluoride product, **3c**, by sublimation at 75 °C (0.1 mm) gave a greenish sublimate (0.69 g) which quickly decomposed on storage. The nonvolatile residue from the sublimation was stirred with hot EtOAc (30 mL) and treated wth Darco, and the filtrate was diluted with hexane (20 mL) to give 0.52 g of crude **2c**. Further recrystallization of this material from EtOAc (25 mL) gave 0.22 g (0.64 mmol, 2.5%) of pure **2c** after drying at 25 °C (0.1 mm). 5-[(5*H*-Dibenzo[*a*,*d*]-cyclopent-5-yl)oxy]-5*H*-dibenzo[*a*,*d*]cyclopentane has the following: mp 228–229 °C (lit. mp 235.5–236.5,[6a] 227–228 °C[6b]); IR (KBr) ν_{max} 3070, 3040, 3020, 1605, 1575, 1100 cm^{-1}; UV (THF) λ_{max} 238 nm (ϵ 37 760), 245 (39 150), 276 (23 100); ^1H NMR ($CDCl_3/Me_4Si$) δ 7.9–7.12 (m, C_6H_4), 5.95 (s, OCH) (lit.[6b] δ 6.97–7.49, 5.74); mass spectrum, *m/e* 346.1329 (calcd for $C_{26}H_{18}O$ parent 346.1301), 181.0622 (calcd for $C_{13}H_9O$ fragment 181.0591), 165.0694 (calcd for $C_{13}H_9$ fragment 165.0684). Anal. Calcd for $C_{26}H_{18}O$: C, 90.14; H, 5.24. Found: C, 90.13; H, 5.39.

In another experiment, the crude reaction product was treated with EtOAc (50 mL) and hexane (200 mL) to give 1.10 g (3.18 mmol; 13%) of **2c**. The syrup left upon evaporation of the mother liquors (2.23 g) crystallized on standing. The latter product was 9-fluorofluorene (**3c**), characterized by a ^1H NMR signal at 6.10 ppm and an ^{19}F NMR signal at −187.67 ppm (both d, J = 48 Hz). This material decomposed to an orange gum after 1–2 days at 25 °C.

(Diphenylmethoxy)diphenylmethane (2d) and Fluorodiphenylmethane (3d). Similar treatment of benzhydrol (9.2 g, 50 mmol) with DAST (50 mmol) gave the crude product as a colorless oil (10.06 g) which appeared to be a 5:3 mixture of **3d**/**3c** by ^1H NMR. The crude product was recrystallized from hexane (40 mL) with Darco treatment to produce ether **2d**; fluoride **3d** remained in the mother liquors. Crystalline **2d** was recrystallized twice more from hexane and dried at 25 °C (0.1 mm). Pure (diphenylmethoxy)diphenylmethane has the following: mp 107–109 °C (lit.[7] mp 109–110.5 °C); sublimes at 105 °C (0.05 mm); IR (KBr) ν_{max} 3080, 3060, 3020, 2850, 1590, 1480, 1450, 1075 cm^{-1}; UV (EtOH) λ_{max} 252 nm (ϵ 1320), 258 (1320), 264 (890); ^1H NMR

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(6) (a) *Beilstein, 4th ed., Suppl. 3* **1967**, *6*, 3491. (b) M. Minabe and K. Suzuki, *Bull. Chem. Soc. Jpn.*, 48, 1301 (1975).

(7) *Beilstein, 4th ed., Suppl. 3* **1967**, *6*, 3369.

($CDCl_3/Me_4Si$) δ 7.27 (s, C_6H_5), 5.38 (s, OCH); mass spectrum, *m/e* 349.1570 (calcd for deprotonated parent $C_{26}H_{20}O$ 349.1591), 272.1179 (calcd for $C_{20}H_{16}O$ fragment 272.1200), 183.0794 (calcd for $C_{13}H_{11}O$ fragment 183.0809), 167.0849 (calcd for $C_{13}H_{11}$ fragment 167.0861). Anal. Calcd for $C_{26}H_{27}O$: C, 89.11; H, 6.33. Found: C, 88.98; H, 6.36.

The hexane mother liquors were filtered and evaporated, and the residue was distilled in a Kugelrohr apparatus at 75 °C (0.1 mm) to give colorless liquid product **3d**. Fluorodiphenylmethane has the following: bp 75 °C (0.1 mm) [lit. bp 105–108 °C (2 mm),[8a] 110–113 °C (1 mm[8b])]; IR ($CHCl_3$) ν_{max} 3040, 3000, 2870, 1650, 1590, 1505 cm^{-1}; UV (isooctane) λ_{max} 252 nm (ε 465), 258 (515), 264 (395); ^1H NMR ($CDCl_3/Me_4Si$) δ 7.20 (m, C_6H_5), 6.35 (d, *J* = 48 Hz, CHF) [lit.[8b] δ 7.3, 6.4 (d, *J* = 48 Hz)]; ^{19}F NMR ($CDCl_3/CFCl_3$) δ −167.34 (d, *J* = 48 Hz, CHF). Anal. Calcd for $C_{13}H_{11}F$: C, 83.84; H, 5.95. Found: C, 83.54; H, 6.40.

Fluorotriphenylmethane. Similar treatment of triphenylcarbinol (6.5 g, 25 mmol) with DAST (25 mmol) gave 6.5 g of crude fluoride; no trace of bis(triphenylmethyl) ether[9] was detected by ^1H NMR. The crude product was recrystallized from 50 mL of hexane; yield 5.57 g (21.2 mmol, 85%). The analytical sample was recrystallized again and dried at 25 °C (0.1 mm). Pure fluorotriphenylmethane has the following: mp 102–103 °C (lit. mp 103–104,[10a] 105–106 °C[10b]); IR ($CHCl_3$) ν_{max} 3040, 1590, 1570, 1480, 1440 cm^{-1}; UV (EtOH) λ_{max} 252 nm (ε 560), 258 (710), 264 570); ^1H NMR ($CDCl_3/Me_4Si$) δ 7.25 (s) C_6H_5; ^{19}F NMR ($CDCl_3/CFCl_3$) δ −126.67 (s, C–F; lit.[10b] δ 49.6 from CF_3CO_2H); mass spectrum, *m/e* 262.1150 (calcd for $C_{19}H_{15}F$ parent 262.1157), 185.0759 (calcd for $C_{13}H_{10}F$ fragment 185.0766), 165.0695 (calcd for $C_{13}H_9$ fragment 165.0704). Anal. Calcd for $C_{19}H_{15}F$: C, 86.99; H, 5.76. Found: C, 86.92; H, 5.81.

Registry No. **1a**, 1210-34-0; **1b**, 10354-00-4; **1c**, 1689-64-1; **1d**, 91-01-0; **2a**, 83693-20-3; **2b**, 35066-77-4; **2c**, 31859-93-5; **2d**, 574-42-5; **3c**, 20825-90-5; **3d**, 579-55-5; DAST, 38078-09-0; fluorotriphenylmethane, 427-36-1; triphenylcarbinol, 76-84-6.

(8) (a) Mindl, J.; Pivoňka, P.; Večeřa, M. *Collect. Czech. Chem. Commun.* **1972**, *37*, 2568. (b) Zieger, H. E.; Angres, I.; Mathisen, D. *J. Am. Chem. Soc.* **1976**, *98*, 2580.

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(11) Correspondence should be addressed to the Biochemicals Department, Pharmaceuticals Division, Building 353, Experimental Station, E. I. du Pont de Nemours and Co., Wilmington, DE 19898.

Regiospecific Synthesis of 3-Substituted 5-Alkylisoxazoles by Modification of the Dilithio Oxime Route to Isoxazoles

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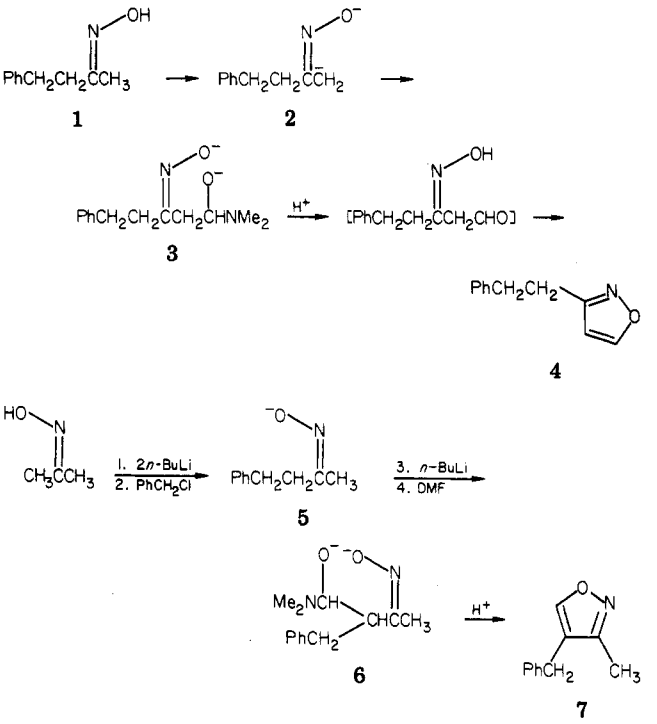
Received June 7, 1982

A useful regiospecific synthesis of unsymmetrical[1] isoxazoles from oximes is described in a recent publication from this laboratory.[2] The method is illustrated by the conversion of benzylacetone oxime (**1**) to the isoxazole (**4**, 91% overall yield distilled) by deprotonation of **1** with *n*-BuLi to form the *syn*-dilithio salt (**2**), which is acylated by DMF to give **3**. The latter subsequently is cyclized to **4** with hot aqueous acid. In a variant of the above route, the isomeric isoxazole (**7**) has been obtained in 82% pure



yield by successive treatment of acetone oxime with (1) 2*n*-BuLi, (2) $PhCH_2Cl$, (3) *n*-BuLi, (4) DMF, and (5) H^+.

Crucial to the utility and success of these processes are the following facts: (A) Of the two possible oxime geometrical isomers, only the less hindered (e.g., **1**) usually is obtained on reaction of ketones with hydroxylamine.[3] However, products from the more hindered isomer are available by the second scheme depicted (i.e., via **5**). (B) Oxime anions and dianions retain their stereochemical integrity, and deprotonation affords syn products exclusively (e.g., **2**, **3**, and **6**).[4] (C) Adducts such as **3** (**6**) do not split off Me_2NH until acid treatment.[5]

The methodology above is especially valuable for the synthesis of many kinds of historically difficult to obtain unsymmetrical isoxazoles (e.g., 3-substituted, 4-substituted/unsubstituted, 5-unsubstituted or -aryl[6]). Thus, its availability substantially extends the utility of isoxazoles as precursors, intermediates, and reagents in preparative organic chemistry, an area in which this ring system already is preeminent vs. other heteroaromatic systems.[7]

5-Alkylisoxazoles with 3-substituents (4-substituted/unsubstituted) are the most important class of unsymmetrical isoxazoles not accessible by the processes of Barber and Olofson and which cannot be made easily by older routes. Here, the oxime schemes fail because the dianion preferentially abstracts a proton from the amide reactant to yield an enolate instead of adding to the amide carbonyl (→ **3**). In this paper, we report variants of the Barber–Olofson methods that largely avoid this problem and afford 5-alkylisoxazoles in moderate yield.

Success requires reversing the selectivity of the dianion to favor carbonyl addition (b in **8**) instead of deprotonation (a in **8**). We thought this could be achieved by significantly increasing the electrophilicity of the carbonyl carbon of the acylating agent. Then attack at that carbon should

(1) Defined as when the substituents on C_3 and C_5 are different. Classical isoxazole syntheses from $RCOCH_2COR^1$ usually yield isomer mixtures.

(2) Barber, G. N.; Olofson, R. A. *J. Org. Chem.* **1978**, *43*, 3015.

(3) Without stereospecificity, oximes would be poor derivatives in classical qualitative organic analysis; see ref 2.

(4) For rationalizations and evidence, see ref 2.

(5) Otherwise an equivalent of **2** would be destroyed by reaction with the more acidic β-dicarbonyl-type species thus formed.

(6) For recent review and references to earlier reviews, see Wakefield, B. J.; Wright, D. J. *Adv. Heterocycl. Chem.* **1979**, *25*, 147.

(7) For highlights, see ref 6 and footnote 2 in ref 2.

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