$ArCH_2$), 28.9, 24.3 and 23.1 (t, CH_2); IR (KBr) 2245 (CN) cm^{-1}; mass spectrum, m/e 237.128 (M^+, calcd, 237.127).

Anal. Calcd for $C_{15}H_{15}N_3$ (M_r 237.306): C, 75.92; H, 6.37; N, 17.71. Found: C, 75.93; H, 6.55; N, 17.75.

1,2,4,4a,5,6-Hexahydro[1,4]oxazino[4,3-*a*]quinoline-5,5-dicarbonitrile (18): yield, 84%; mp 110–160 °C dec (methanol); ^1H NMR δ 7.4–6.75 (m, 4 H, Ar H), 4.5–2.75 (m, 9 H, other hydrogen atoms); ^{13}C NMR δ 143.5 (s, C-10a), 129.3, 129.0, 120.3 and 113.1 (d, Ar C), 115.4 (s, C-6a), 113.5 and 112.8 (s, CN), 68.1 and 66.1 (t, OCH_2), 56.8 (d, NCH), 45.7 (t, NCH_2), 37.2 (t, $ArCH_2$), 33.1 [s, $C(CN)_2$]; IR (KBr) 2248 (CN) cm^{-1}; mass spectrum, m/e 239.109 (M^+, calcd, 239.106).

Anal. Calcd for $C_{14}H_{13}N_3O$ (M_r 239.279): C, 70.28; H, 5.48; N, 17.56. Found: C, 70.17; H, 5.53; N, 17.46.

Crystallographic Data and X-ray Structure Analysis of 13a. Crystals of **13a** are triclinic, space group $P\bar{1}$: a = 10.91 (1) b = 9.570 (3), c = 8.758 (8) Å; α = 65.15 (1), β = 66.94 (3), γ = 71.24 (1)°; Z = 2; d_c = 1.21 g cm^{-3}. Intensities were measured by using Mo Kα radiation [Philips PW1100 diffractometer, graphite monochromator, $\theta - 2\theta$ scan mode, $3 < \theta < 25°$, scan speed 0.025° s^{-1}, scan width deg 1.7 + 0.6 tan θ]. The total number of independent reflexions measured was 2384. The structure was solved by direct methods[31] and refined by full-matrix least-squares[32] to a final R-factor of 4.8%. In the refinements 1934 reflections with intensities greater than the standard deviation from counting statistics were used. Hydrogen atoms were found from difference Fourier syntheses. In the last cycles 263 parameters were refined (scale factor, extinction parameter, positional parameters of all atoms, thermal parameters of all atoms: anisotropic for non-hydrogen atoms, isotropic for hydrogen atoms). The drawing of the structure was made by ORTEP.[33]

Acknowledgment. We are grateful for the financial support of this work by the "Koningin Wilhelmina Fonds". We express our gratitude to T. Vos, G. M. de Boer, and R. J. Wijngaarden for their contributions to parts of the experimental work. We also acknowledge J. M. Visser and J. L. M. Vrielink for recording the NMR and T. W. Stevens for recording the mass spectra.

Registry No. **4a**, 87698-81-5; **4b**, 82212-00-8; **4c**, 87698-82-6; (*Z*)-**5a**, 83466-98-2; (*Z*)-**5b**, 87698-83-7; (*Z*)-**5c**, 87698-84-8; **6a**, 87698-85-9; **6b**, 40377-01-3; **6c**, 87698-86-0; (*Z*)-**7a**, 87698-87-1; (*E*)-**7a**, 87698-88-2; (*Z*)-**7b**, 87698-89-3; (*E*)-**7b**, 87698-90-6; (*Z*)-**7c**, 87698-91-7; (*E*)-**7c**, 87698-92-8; **8a**, 58028-74-3; **8b**, 34595-26-1; **8c**, 58028-76-5; **9**, 87698-93-9; **10**, 87698-94-0; **11a**, 87698-95-1; **11b**, 87698-96-2; **11c**, 87698-97-3; **12a**, 87698-98-4; **12b**, 87698-99-5; **13a**, 87699-00-1; **13b**, 87711-10-2; **14b**, 87699-01-2; **15a**, 87699-02-3; **15b**, 87699-03-4; **16a**, 87699-04-5; **16b**, 87699-05-6; **17**, 87699-06-7; 18, 87699-07-8; 2-bromoaniline, 615-36-1; 2-aminobenzeneacetonitrile, 2973-50-4; 1,4-dibromobutane, 110-52-1; 1,5-dibromopentane, 111-24-0; 1,1'-oxybis(2-bromoethane), 5414-19-7; benzaldehyde, 100-52-7; triethyl phosphonoacetate, 867-13-0; dimethyl malonate, 108-59-8; malononitrile, 109-77-3; DMAD, 762-42-5.

Supplementary Material Available: Tables of atomic coordinates, thermal parameters, bond distances, and bond angles (5 pages). Ordering information is given on any current masthead page.

(31) Germain, G.; Main, P.; Woolfson, M. M. *Acta Crystallogr., Sect. B* **1970**, *B26*, 274. Main, P. In "Computing in Crystallography"; Schenk, H., Ed.; Delft University Press: Delft, 1978, p 93.
(32) Busing, W. R.; Martin, K. O.; Levy, H. A. "ORFLS", Oak Ridge National Laboratory, Report ORNL-TM-305, 1962.
(33) Johnson, C. K. "ORTEP"; Oak Ridge National Laboratory, Oak Ridge, TN, 1965; Report ORNL-3794.

Regioselectivity Associated with the 1,3-Dipolar Cycloaddition of Nitrones with Electron-Deficient Dipolarophiles

Albert Padwa,* Lubor Fisera, Konrad F. Koehler, Augusto Rodriguez, and George S. K. Wong

Department of Chemistry, Emory University, Atlanta, Georgia 30322

Received February 28, 1983

A study of the cycloaddition behavior of a series of electron-deficient dipolarophiles with *C*-aryl-*N*-alkylnitrones has been carried out. The 1,3-dipolar cycloaddition proceeds in high yield to produce isoxazolidines. This [3 + 2] cycloaddition embodies a high degree of both regiochemical and stereochemical control and provides an efficient entry into such heterocyclic systems. The reactions follow frontier orbital predictions. Most dipolarophiles undergo cycloaddition to give 5-substituted isoxazolidines. The orientation has been explained in terms of maximum orbital overlap of the nitrone LUMO–dipolarophile HOMO. As the electron affinity of the dipolarophile increases, an increasing tendency toward formation of the 4-substituted isoxazolidine is encountered. In certain cases diastereomeric isoxazolidines were formed via different two-plane orientation complexes. The ratio of the diastereomers reflects the free energy difference of the two transition states. This difference comes from repulsive interactions caused by steric hindrance and attractive van der Waal forces associated with maximum π overlap of the substituent groups. The transition state which dominates in a particular case will depend on the nature of the groups attached to the N atom of the nitrone and to the dipolarophile π bond.

The 1,3-dipolar cycloaddition reaction is one of the most useful reactions for the synthesis of heterocyclic compounds.[1,2] It provides the chemist one of his best tools for constructing five-membered rings and has a nearly singular capability of establishing large numbers of stereochemical centers in one synthetic step. Nitrones represent a long-known and thoroughly investigated class of 1,3-dipoles.[3-7] The cycloaddition of nitrones with alkenes produces isoxazolidines in high yield.[8,9] The presence of a nitrogen atom within the isoxazolidine ring has made this heterocyclic moiety especially attractive for the synthesis

* Alexander von Humboldt Senior Scientist, 1983–1984.

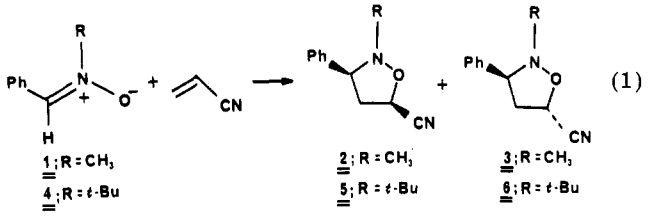
(1) Huisgen, R.; Grashey, R.; Sauer, J. In "The Chemistry of Alkenes"; Patai, S., Ed.; Interscience: London, 1964; pp 806–878.
(2) Huisgen, R. *Angew Chem., Int. Ed. Engl.* **1963**, *2*, 565, 633.
(3) Smith, L. I. *Chem. Rev.* **1938**, *23*, 193.
(4) Delpierre, G. R.; Lamchen, M. *Q. Rev., Chem. Soc.* **1965**, *19*, 329.
(5) Black, D. St. C.; Crozier, R. F.; Davis, V. C. *Synthesis* **1975**, *7*, 205.
(6) Padwa, A. *Angew. Chem., Int. Ed. Engl.* **1976**, *15*, 123.
(7) Oppolzer, W. *Angew. Chem., Int. Ed. Engl.* **1977**, *16*, 10.
(8) Grashey, R.; Huisgen, R.; Leitermann, H. *Tetrahedron Lett.* **1960**, 9.

0022-3263/84/1949-0276$01.50/0

of a number of alkaloids and related natural products.[10-16] The regioselectivity for nitrone cycloadditions onto monosubstituted ethylenes was originally believed to proceed in a unidirectional fashion, giving 5-substituted adducts regardless of the alkene substituent.[1,2] More recent work suggests that those ethylenes bearing electron-withdrawing groups show an increasing tendency to afford the corresponding 4-substituted isoxazolidine as the electron-withdrawing ability of the attached substituent increases.[17-22] The ability to utilize nitrone cycloadditions in organic synthesis depends heavily on understanding the factors which determine the regiochemistry of the reaction. Our interest in using nitrone cycloadditions for the synthesis of β-lactams[23,24] has focused our attention on the degree of regioselectivity observed for the reaction of several representative nitrones with electron-deficient dipolarophiles. In this paper we report on the mechanistic and regiochemical features associated with the cycloaddition of several nitrones onto nitro-substituted alkenes. In the following paper we describe in some detail the reorganization of the 5-nitro-substituted isoxazolidine to the β-lactam system.

Results

As our first model we chose to investigate the cycloaddition behavior of the *N*-alkyl-*C*-phenylnitrone system with several monosubstituted alkenes possessing electron-withdrawing substituents.[18] The reaction of *N*-methyl-*C*-phenylnitrone (1) with acrylonitrile (eq 1) pro-

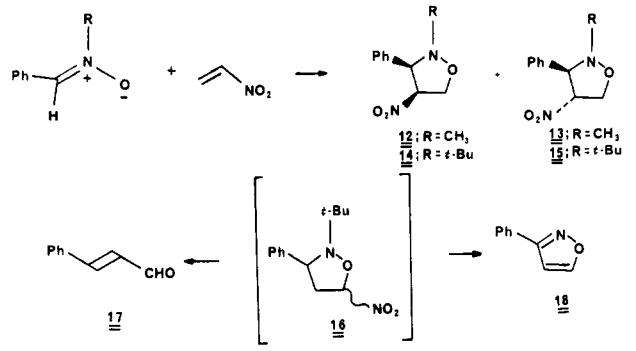


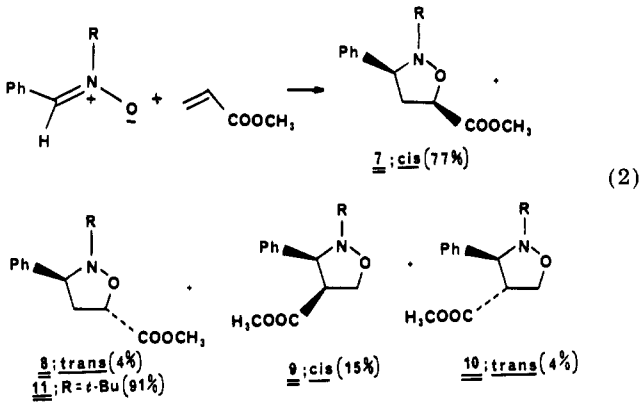
(1)

duced a mixture of cis (23%) and trans (77%) 5-cyano-substituted isoxazolidine. Similarly, treatment of the corresponding *tert*-butylnitrone 4 with acrylonitrile gave a mixture of cis (5) and trans (6) cycloadducts.

The reaction of methyl acrylate with nitrone 1 gave four adducts. The crude residue was chromatographically separated, and each cycloadduct could be obtained in pure form. NMR analysis of the crude thermolysate permitted a determination of the amount of the various regioisomers present in the original reaction mixture. Surprisingly, the reaction of *tert*-butylnitrone 4 with methyl acrylate gave trans adduct 11 (91%) as the major product (eq 2). This

Scheme I





(2)

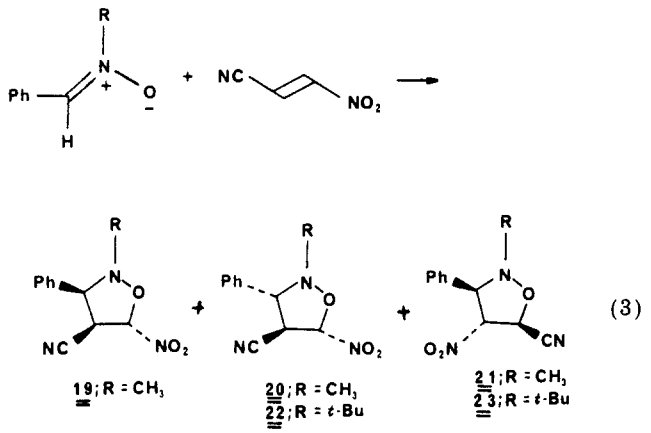
stands in marked contrast with the cycloaddition behavior of nitrone 1 which afforded the cis 5-substituted isoxazolidine 7 (77%) as the dominant adduct. The two-proton multiplet at ca. δ 3.30 (C_5) is characteristic of the diastereomeric mixture of 4-substituted isoxazolidines 9 and 10. With both systems the cycloaddition proceeds so as to afford the 5-substituted isoxazolidine with high regioselectivity. Thus, in the case of nitrone 1, the ratio of regioisomers is approximately 4:1.

The results described above are in general accord with earlier observations that most electron-deficient dipolarophiles undergo cycloaddition with nitrones to give 5-substituted isoxazolidines.[1,2,18] Houk and co-workers reported in 1973 that very electron deficient dipolarophiles give significant amounts of, or even predominantly 4-substituted isoxazolidines.[19] In accord with his observations we find that nitroethylene reacts readily with *N*-methylnitrone 1 to give a mixture of cis (12) and trans (13) 4-substituted isoxazolidines (Scheme I). NMR analysis of the crude reaction mixture reveals the presence of 12 and 13 in a 2:1 ratio. When the mixture was submitted to flash column chromatography only the stable *trans*-4-nitro-3-phenyl-*N*-methylisoxazolidine (15) was obtained. The reaction mixture derived from *N*-*tert*-butylnitrone 4 and nitroethylene, on the other hand, displayed three *tert*-butyl singlets in the NMR spectrum. The two diastereomeric 4-substituted regioisomers (i.e., 14 + 15, 50%) could be isolated in pure form by silica gel chromatography while the labile 5-isoxazolidine 16 (24%) was converted to both cinnamaldehyde (17) and 3-phenylisoxazole (18) on chromatographic separation.

We have also studied the regiochemical aspects of the reaction of nitrones 1 and 4 with some additional 2-substituted nitroalkenes. Thus, cycloaddition of *N*-methyl-

(9) Huisgen, R.; Seidl, H.; Bruning, I. *Chem. Ber.* **1969**, *102*, 1102.
(10) Tufariello, J. J. *Acc. Chem. Res.* **1979**, *12*, 396.
(11) Confalone, P. N.; Pizzolato, G.; Confalone, D. L.; Uskokovic, M. R. *J. Am. Chem. Soc.* **1980**, *102*, 1954.
(12) Tufariello, J. J.; Mullen, G. B. *J. Am. Chem. Soc.* **1978**, *100*, 3638.
(13) Tufariello, J. J.; Tegeler, J. T.; Wong, S. C.; Ali, S. A. *Tetrahedron Lett.* **1978**, 1733.
(14) Monteiro, H. J. *Acad. Brasil Cienc.* **1980**, *52*, 493.
(15) Wovkulich, P. M.; Uskokovic, M. R. *J. Am. Chem. Soc.* **1981**, *103*, 3956.
(16) Schwartz, M. A.; Swanson, G. C. *J. Org. Chem.* **1979**, *44*, 953.
(17) Seidl, H.; Huisgen, R.; Knorr, R. *Chem. Ber.* **1969**, *102*, 904.
(18) For a related set of cycloadditions see: Huisgen, R.; Grashey, R.; Hauck, H.; Seidl, H. *Chem. Ber.* **1968**, *101*, 2568. Huisgen, R.; Seidl, H.; Wulff, J. *Ibid.* **1969**, *102*, 915.
(19) Sims, J.; Houk, K. N. *J. Am. Chem. Soc.* **1973**, *95*, 5798.
(20) Houk, K. N.; Bimanand, A.; Mukherjee, D.; Sims, J.; Chang, Y. M.; Kaufman, D. C.; Domelsmith, L. N. *Heterocycles* **1977**, *7*, 293.
(21) Joucla, M.; Tonnard, F.; Gree, D.; Hamelin, J. *J. Chem. Res.* **1978**, 3535.
(22) Ali, S. A.; Senaratnue, P. A.; Illig, C. R.; Meckler, H.; Tufariello, J. J. *Tetrahedron Lett.* **1979**, 4167.
(23) Padwa, A.; Koehler, K. F.; Rodriguez, A. *J. Am. Chem. Soc.* **1981**, *103*, 4974.
(24) Padwa, A.; Koehler, K. F.; Rodriguez, A. *J. Org. Chem.*, following paper in this issue.

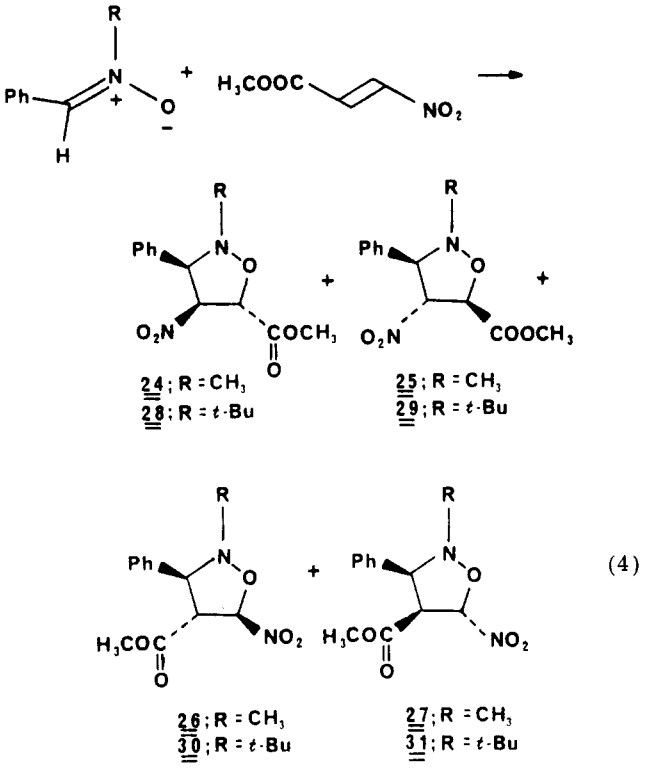
nitrone 1 with *trans*-1-nitro-2-cyanoethylene (eq 3) led to



(3)

a mixture of the 4-cyano-5-nitro-substituted regioisomers **19** and **20** as well as the isomeric 4-nitro-5-cyano-substituted isoxazolidine **21** (29%). *N*-*tert*-Butylnitrone **2** also adds to the same dipolarophile to give a mixture of two regioisomeric cycloadducts (i.e., **22** (57%) and **23** (43%)).

Thermolysis of *N*-methylnitrone **1** with methyl *trans*-3-nitroprop-2-enoate gave rise to four cycloadducts (eq 4)

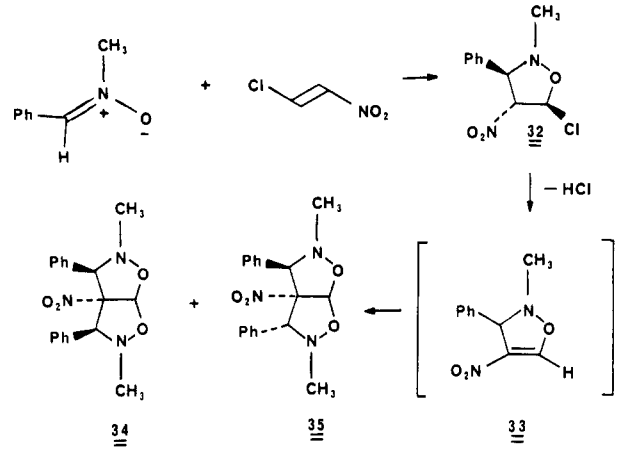


(4)

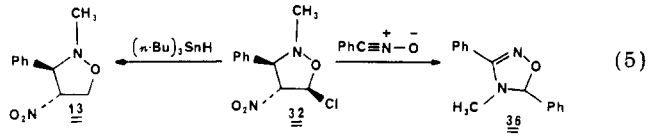
which could be isolated in pure form by silica gel chromatography. The reaction of *N*-*tert*-butylnitrone **4** with the same dipolarophile produced an analogous set of dipolar cycloadducts although in a slightly different ratio. The ratio of the two regioisomeric adducts is approximately 1:1 with both systems. The various cycloadducts were assigned on the basis of their spectral characteristics (see Experimental Section) and chemical behavior.[24]

We also studied the cycloaddition behavior of *trans*-1-chloro-2-nitroethylene with *N*-methylnitrone **1**. When the reaction was carried out in refluxing benzene, a mixture of two diastereomeric *bis*-cycloadducts (i.e., **34** and **35**) was isolated in good yield (Scheme II). A distinction between the two isomers can be made on the basis of the chemical shift of the hydrogens in the NMR as well as their ^{13}C NMR data (see Experimental Section). The NMR spectrum of the trans isomer (mp 140–141 °C) showed signals at δ 2.65 (s, 3 H), 2.85 (s, 3 H), 4.15 (s, 1 H), 4.36 (s, 1 H), 6.30–6.40 (m, 2 H), 6.45 (s, 1 H), and 7.0–7.8 (m, 8 H). The cis isomer, which contains a plane of symmetry, showed both methyl signals at δ 2.60 (s, 6 H), the two equivalent hydrogens at the 4,6-positions at δ 4.45 (s, 2 H), a singlet at δ 6.60 (1 H), and a multiplet at δ 7.30 (10 H).

Scheme II



It should be noted that when the cycloaddition of **1** with chloronitroethylene was carried out for shorter periods of time or at room temperature, the expected 1,3-dipolar cycloadduct **32** could be isolated in 85% yield. This material was identified on the basis of its characteristic 90-MHz NMR spectrum ($CDCl_3$) which showed singlets at δ 2.97 (3 H), 6.60 (1 H), and 7.31 (5 H) and a pair of doublets at δ 4.84 (1 H, J = 6.0 Hz) and 5.62 (1 H, J = 6.0 Hz). This structure was further supported by its ready conversion to *trans*-4-nitro-3-phenyl-*N*-methylisoxazolidine (**13**) on treatment with tri-*n*-butyltin hydride (eq 5).



(5)

Heating a sample of **32** in the presence of excess nitrone gave rise to bis cycloadducts **34** and **35** in excellent yield. Clearly dihydroisoxazole **33** is initially formed by elimination of hydrogen chloride, and it then undergoes dipolar cycloaddition with *N*-methylnitrone **1**. In an attempt to induce an analogous cycloaddition, we treated cycloadduct **32** with benzonitrile oxide. Stirring a sample of **32** with benzohydroximoyl chloride in ether in the presence of excess triethylamine resulted, however, in the isolation of 4-methyl-3,5-diphenyl-1,2,4-oxadiazoline (**36**).[25] The formation of **36** probably proceeds via the intermediacy of benzaldehyde *N*-methylimine.

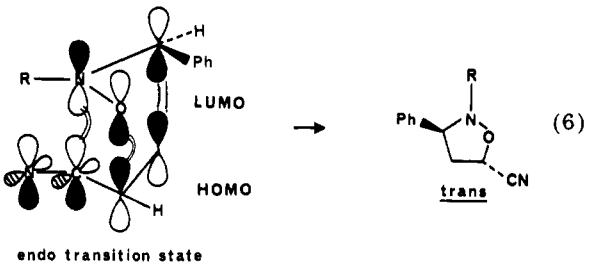
Discussion

The results described above are in general accord with the frontier orbital treatment of nitrone 1,3-dipolar cycloadditions.[26-29] This view suggests that most dipolaro-

(25) Bast, K.; Christl, M.; Huisgen, R.; Mack, W. *Chem. Ber.* **1972**, *105*, 2825.
(26) Fukui, K. *Acc. Chem. Res.* **1971**, *4*, 57.
(27) Houk, K. N.; Sims, J.; Duke, R. E.; Strozier, R. W.; George, J. K. *J. Am. Chem. Soc.* **1973**, *95*, 7287. Houk, K. N.; Sims, J.; Watts, C. R.; Luskus, L. J. *Ibid.* **1973**, *95*, 7301. Houk, K. N. *Acc. Chem. Res.* **1975**, *8*, 361.
(28) Sustmann, R. *Tetrahedron Lett.* **1971**, 2717. Sustmann, R.; Trill, H. *Angew. Chem., Int. Ed. Engl.* **1972**, *11*, 838.

philes should undergo cycloaddition to afford 5-substituted isoxazolidines with high regioselectivity. The orientation observed has been rationalized in terms of maximum orbital overlap of the nitrone LUMO–dipolarophile HOMO.[19,22] As the ionization potential of the nitrone decreases or the electron affinity of the dipolarophile increases,[20] an increasing tendency toward production of 4-substituted isoxazolidines is found.[19,27] At some point, there must be a switch over from LUMO to HOMO control as one increases the electron-withdrawing power of the substituent on the alkene. That point is apparently approached with methyl acrylate since regioisomeric mixtures of adducts are encountered with this dipolarophile. The lack of regiospecificity exhibited by 2-cyano- and 2-carbomethoxy-substituted nitroethylenes is not unreasonable since CNDO/2 calculations[30] indicate little difference in the size of the coefficients in the LUMO orbital or *trans*-β-nitroacrylonitrile. The CNDO/2 calculations with *trans*-1-nitro-2-chloroethylene reveals that there is a much larger difference in the coefficients of this dipolarophile.[30] This would account for the high regioselectivity encountered with this system.

There is strong evidence that nitrones derived from aromatic aldehydes possess a configuration in which the *C*-aryl and *N*-alkyl groups are in a trans relationship. This is based on a comparison of UV spectra of systems with fixed cis and trans geometry,[31] an X-ray crystal structure,[32] and intramolecular nuclear Overhauser enhancements. Dipolar cycloadditions, like the Diels–Alder reaction, proceed through exo or endo transition states.[33] The dominant interaction for the cycloaddition of acrylonitrile with *N*-methylnitrone **1** involves the LUMO dipole–HOMO dipolarophile interaction. An examination of the endo transition state in these terms reveals a secondary orbital interaction which favors this aproach over its exo counterpart, thereby accounting for the preferential formation of the trans isomer (eq 6). An alternate possibility

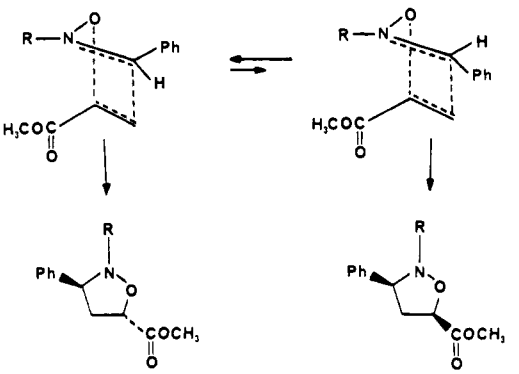


(6)

is that secondary orbital interactions are not significant and that repulsions between the alkyl group on nitrogen and the substituent on the dipolarophile are minimized in the exo transition state.

A concerted, kinetically controlled cycloaddition of *C*-phenyl-*N*-methylnitrone to methyl acrylate would also be expected to produce the trans 5-substituted regioisomer. Surprisingly, the major cycloadduct formed corresponded to the cis isomer (i.e., **7**). In order to account for the preferential formation of adduct **7**, we assume that the trans form of the nitrone is in equilibrium with a small amount of the cis isomer and that the major adduct is that derived from the minor rotamer of the nitrone. There is good literature precedence for this suggestion. Earlier work by Whitham and co-workers has shown that there is a significant barrier to rotation in nitrones but that this barrier is not sufficient to prohibit cis–trans interconversion under the conditions of dipolar cycloaddition.[35] The two-plane orientation complex leading to **7** permits efficient π overlap of the phenyl and ester groups which are located one above the other (see Scheme III). In this case the attractive van der Waals forces associated with maximal π overlap are responsible for the preferred cycloaddition stereochemistry. The stereochemical outcome of the cycloaddition reaction involving the *N*-*tert*-butylnitrone **4** with methyl acrylate is markedly different. Only a single adduct was detected, and this was shown to possess the trans structure **11**. Although the transition state leading to the cis isomer benefits from attractive van der Waal forces, it simultaneously suffers repulsive interactions caused by steric hindrance. When a *tert*-butyl group is present on the nitrogen atom, the steric factors dominate. This nicely accounts for the exclusive formation of the trans isomer.

Scheme III



The reaction of *tert*-butylnitrone **4** with *trans*-1-cyano-2-nitroethylene gave rise to a mixture of two regioisomeric isoxazolidines **22** (60%) and **23** (40%). Cycloaddition of this same dipolarophile with *N*-methylnitrone **1**, on the other hand, produced a mixture of three isomeric cycloadducts. Two of these (i.e., **19** and **20**) derive from one regiochemical mode of cycloaddition of **1** to the π bond, while the other (i.e., **21**) derives from the alternate mode of addition. To account for the formation of the two diastereomeric cycloadducts **19** and **20**, we assume that the trans isomer of *C*-phenyl-*N*-methylnitrone is in equilibrium with a small amount of the cis form and that the two transition states leading to **19** and **20** are of comparable energy. This is not the case with the corresponding *tert*-butylnitrone **4**, presumably as a consequence of steric factors.

In conclusion, we have shown that the 1,3-dipolar cycloaddition of nitrones with electron-deficient olefins is a type II process. That interaction which dominates in a particular case will depend on the nature of the substituent group on the dipolarophile. In the following paper the ring-contraction reaction of the 5-nitroisoxazolidine cycloadduct to a β-lactam ring is described in some detail.

(29) Bastide, J.; El Gandour, N.; Henri-Rousseau, O. *Tetrahedron Lett.* **1973**, 4225; *Bull. Soc. Chem. Fr.* **1973**, 2290. Bastide, J.; Henri-Rousseau, O. *Bull. Soc. Chem. Fr.* **1973**, 2294.

(30) Verbicky, J. Ph.D. Thesis, Georgie Institute of Technology, Atlanta, GA, 1979.

(31) Thesing, J.; Sirrenberg, W. *Chem. Ber.* **1958**, *91*, 1978.

(32) Folting, K.; Lipscomb, W. N.; Jerslev, B. *Acta Chem. Scand.* **1963**, *17*, 2138.

(33) Gree and Carrie have studied exo/endo ratios in nitronate ester cycloadditions and have reached similar conclusions. See: Gree, R.; Tonnard, F.; Carrie, R. *Tetrahedron* **1976**, *32*, 675; **1976**, *32*, 683.

(34) Boyd, D. R.; Jennings, W. B.; Spratt, R.; Jerina, D. M. *Chem. Commun.* **1970**, 745.

(35) Boyle, L. W.; Peagram, M. J.; Whitham, G. H. *J. Chem. Soc. B* **1971**, 1728.

Experimental Section[36]

Reaction of *N*-*tert*-Butyl-*C*-phenylnitrone (4) with *trans*-1-Nitro-2-cyanoethylene. In a 250-mL round-bottomed flask was added 1.80 g of freshly prepared *N*-*tert*-butyl-*C*-phenylnitrone (4)[37] followed by 150 mL of dry benzene. To this solution was added dropwise 1.0 g of *trans*-1-cyano-2-nitroethylene[38] in 50 mL of dry benzene. The resulting mixture was heated at reflux for 24 h until the nitrone had been totally consumed as judged by thin-layer analysis. The solvent was removed under reduced pressure, and the residue was immediately diluted with a 10% acetone–hexane solution and was cooled to 0 °C. Recrystallization of the resulting solid afforded *trans*-4-cyano-5-nitro-3-phenyl-*N*-*tert*-butylisoxazolidine (**22**): 57% yield; white crystalline solid; mp 76–77 °C (10% acetone–hexane); IR (neat) 3050, 2950, 1540, 1450, 1340 cm^{-1}; NMR (90 MHz, $CDCl_3$) δ 7.30 (s, 5 H), 5.65 (d, J = 2.7 Hz, 1 H), 4.25 (*d*, J = 7.5 Hz, 1 H), 4.20 (dd, J = 7.5, 2.7 Hz, 1 H), 1.00 (s, 9 H); UV (cyclohexane) 220 nm (ϵ 3400); MS, *m/e* 275, 173, 145 (base), 130, 129; ^{13}C NMR (20 MHz, $CDCl_3$) δ 129.9, 127.42 (aromatic), 116.06, 102.48, 69.19, 59.92, 50.20, 26.03. Anal. Calcd for $C_{14}H_{17}N_3O_3$: C, 61.08; H, 6.27; N, 15.26. Found: C, 61.09; H, 6.22; N, 15.23.

The mother liquors were concentrated under reduced pressure, and the resulting residue was subjected to medium-pressure silica gel chromatrography with an 8% acetone–hexane mixture as the eluent to give *trans*-5-cyano-4-nitro-3-phenyl-*N*-*tert*-butylisoxazolidine (**23**): 43% yield; mp 56–57 °C (methanol); IR (KBr) 3090, 3000, 2990, 2220, 1600, 1520, 1340, 790 cm^{-1}; NMR (90 MHz, $CDCl_3$) δ 7.20 (m, 5 H), 5.48 (d, J = 1.5 Hz, 1 H), 5.19 (dd, J = 6.0, 1.5 Hz, 1 H), 4.60 (*d*, J = 6.0 Hz, 1 H), 1.00 (s, 9 H); UV (95% ethanol) 210 nm (ϵ 6200); MS, *m/e* 275 (M^+, base), 256, 220, 219, 172, 142, 143; ^{13}C NMR (20 MHz, $CDCl_3$) δ 129.39, 129.13, 127.81, 115.84, 98.85, 68.25, 66.72, 59.58, 26.09. Anal. Calcd for $C_{14}H_{17}N_3O_3$: C, 61.08; H, 6.22; N, 15.26. Found: C, 61.00; H, 6.26; N, 15.22.

Reaction of *N*-Methyl-*C*-phenylnitrone with *trans*-1-Nitro-2-cyanoethylene. A 250-mL, one-necked, round-bottomed flask was charged with 1.23 g of *N*-methyl-*C*-phenylnitrone[39] followed by 100 mL of dry benzene. To this mixture was added dropwise 1.0 g of *trans*-1-nitro-2-cyanoethylene in 50 mL of dry benzene. The reaction mixture was heated at reflux for 24 h. At the end of this time the solvent was removed under reduced pressure, and the residue was diluted with 5 mL of methanol and cooled to 0 °C. The solid which precipitated was recrystallized several times from a 10% acetone–hexane mixture to give *cis*-4-cyano-5-nitro-3-phenyl-*N*-methylisoxazolidine (**19**): 60% yield; mp 126–127 °C (methanol); IR (KBr) 3080, 2980, 2900, 1540, 1500, 1460, 1366, 1310, 1200, 1140 cm^{-1}; NMR (90 MHz, CD_3CN) δ 7.50 (s, 5 H), 6.16 (br s, 1 H), 4.67 (dd, J = 6.0, 1.5 Hz, 1 H), 4.40 (*d*, J = 6.0 Hz, 1 H), 2.88 (s, 3 H); UV (95% ethanol) 210 nm (ϵ 9400), 243 (1900); ^{13}C NMR (20 MHz, $CDCl_3$) δ 124.79, 124.63, 124.24, 123.94, 122.88, 99.15, 72.08, 64.51, 42.76, 38.85; MS, *m/e* 233, 159 (base), 144, 118, 117, 77. Anal. Calcd for $C_{11}H_{11}N_3O_3$: C, 56.65; H, 4.72; N, 18.02. Found: C, 56.69; H, 4.75; N, 17.92.

The mother liquors were concentrated under reduced pressure, and the resulting residue was diluted with 10 mL of absolute methanol and cooled to 0 °C. Repetitive crystallization of the solid gave *trans*-4-cyano-5-nitro-3-phenyl-*N*-methylisoxazolidine (**20**): 16% yield; mp 108–109 °C; IR (KBr) 3020, 2960, 2900, 2320, 1580, 1400, 1360, 1170, 840, 810, 740 cm^{-1}; NMR (90 MHz, $CDCl_3$) δ 7.45 (br s, 5 H), 5.80 (*d*, J = 3.0 Hz, 1 H), 4.34 (dd, J = 9.0, 3.0 Hz, 1 H), 3.89 (*d*, J = 9.0 Hz, 1 H), 2.76 (s, 3 H); UV (95% ethanol) 210 nm (ϵ 6320), 240 (1580); MS, *m/e* 233, 159, 144, 130, 118, 103, 77. Anal. Calcd for $C_{11}H_{11}N_3O_3$: C, 56.65; H, 4.72; N, 18.02. Found: C, 56.60; H, 4.71; N, 18.10.

The oily residue obtained from the mother liquors was purified by medium-pressure liquid chromatography on a silica gel column with an 8% ethyl acetate–hexane solution as the eluent to give *trans*-5-cyano-4-nitro-3-phenyl-*N*-methylisoxazolidine (**21**): 29% yield; mp 87–88 °C; IR (KBr) 3100, 3070, 2250, 1625, 1580, 1460, 1380, 1060 cm^{-1}; NMR (90 MHz, $CDCl_3$) δ 7.40 (s, 5 H), 5.43 (*d*, J = 1.5 Hz, 1 H), 5.25 (dd, J = 6.0, 1.5 Hz, 1 H), 3.85 (*d*, J = 6.0 Hz, 1 H), 2.65 (s, 3 H); UV (95% ethanol) δ 210 (ϵ 6600), 240 (2700); MS, *m/s* 233, 186 (base), 118, 115, 109; ^{13}C NMR (20 MHz, $CDCl_3$) δ 133.33, 129.92, 129.59, 128.11, 97.33, 77.08, 67.61, 42.44. Anal. Calcd for $C_{11}H_{11}N_3O_3$: C, 56.65; H, 4.72; N, 18.02. Found: C, 56.45; H, 4.79; N, 17.96.

Reaction of 4 with Methyl *trans*-3-Nitroprop-2-enoate. A 500-mL, two-necked, 2 round-bottomed flask was charged with 3.54 g of **4** followed by 150 mL of dry benzene. To this solution was added dropwise 2.96 g of methyl *trans*-3-nitroprop-2-enoate in 100 mL of dry benzene. This mixture was heated at reflux for 24 h, after which time the solvent was removed under reduced pressure. The oily residue was purified via flash column chromatography with a 10% acetone–hexane mixture as the eluent. Four major fractions were obtained. The first component isolated was assigned as *cis*-3-phenyl-4-nitro-5-carbomethoxy-N-*tert*-butylisoxazolidine (**28**): 50% yield; mp 87–87 °C; IR (KBr) 3050, 2900, 2850, 1775, 1560, 1380, 1200 cm^{-1}; NMR (60 MHz, $CDCl_3$) δ 7.40 (s, 5 H), 5.95 (dd, J = 6.0, 2.0 Hz, 1 H), 5.20 (d, J = 2.0 Hz, 1 H), 4.65 (*d*, J = 6.0 Hz, 1 H), 3.90 (s, 3 H), 1.0 (s, 9 H); MS, *m/e* 308, 293, 252, 147, 121, 115, 104, 91, 77. Anal. Calcd for $C_{15}H_{20}N_2O_5$: C, 58.43; H, 6.54; N, 9.09. Found: C, 58.41; H, 6.55; N, 9.06.

The second component obtained was assigned as *trans*-3-phenyl-4-nitro-5-carbomethoxy-N-*tert*-butylisoxazolidine (**29**): 5% yield; mp 67–68 °C; IR (KBr) 2900, 1720, 1540, 1360, 1240 cm^{-1}; NMR (90 MHz, $CDCl_3$) δ 7.30 (m, 5 H), 5.58–5.28 (m, 2 H), 4.65 (d, J = 8.7 Hz, 1 H), 3.80 (s, 3 H), 1.03 (s, 9 H); MS, *m/e* 308, 293, 252, 147, 121, 115, 104, 91, 77. Anal. Calcd for $C_{15}H_{20}N_2O_5$: C, 58.43; H, 6.54; N, 9.05. Found: C, 58.42; H, 6.55; N, 9.02.

The third component isolated from the column was a clear oil whose structure was assigned as *trans*-3-phenyl-4-carbomethoxy-5-nitro-N-*tert*-butylisoxazolidine (**30**): 37% yield; IR (KBr) 3020, 2900, 1740, 1560, 1440, 1370, 1240, 760 cm^{-1}; NMR (60 MHz, $CDCl_3$) δ 7.40 (s, 5 H), 5.90 (d, J = 1.8 Hz, 1 H), 4.45 (d, J = 8.0 Hz, 1 H), 4.25 (dd, J = 8.0, 1.8 Hz, 1 H), 3.85 (s, 3 H), 1.08 (s, 9 H); MS, *m/e* 308, 262, 206, 178, 146, 131, 103, 77. Anal. Calcd for $C_{15}H_{20}N_2O_5$: C, 58.43; H, 6.54; N, 9.05. Found: C, 58.51; H, 6.50; N, 9.06.

The fourth material isolated from the column was assigned as *cis*-3-phenyl-4-carbomethoxy-5-nitro-*N*-*tert*-butylisoxazolidine (**31**): 13% yield; mp 104–105 °C (acetone–hexane); IR (KBr) 3500 (enol), 3010, 3000, 2950, 1740, 1560, 1425, 1420, 1360, 1200, 1120 cm^{-1}; NMR (60 MHz, $CDCl_3$) δ 7.30 (s, 5 H), 6.05 (*d*, J = 3.0 Hz, 1 H), 4.79(*d*, J = 8.0 Hz, 1 H), 4.25 (dd, J = 8.0, 3.0 Hz), 3.30 (s, 3 H), 1.00 (s, 9 H); MS, *m/e* 308, 262, 206, 178, 146, 131, 103, 77. Anal. Calcd for $C_{15}H_{20}N_2O_5$: C, 58.43; H, 6.54; N, 9.05. Found: C, 58.50; H, 6.55; N, 9.07.

Reaction of *N*-Methyl-*C*-phenylnitrone with Methyl *trans*-3-Nitroprop-2-enoate. A 250-mL round-bottomed flask was charged with 4.0 g of *N*-methyl-*C*-phenylnitrone and 100 mL of dry benzene. This mixture was stirred under a nitrogen atmosphere and 3.88 g of methyl *trans*-3-nitroprop-2-enoate in 50 mL of dry benzene was added dropwise. After the addition was complete, the reaction mixture was heated at reflux for 3 h. The solvent was then removed under reduced pressure, and the oily residue was chromatographed on a flash silica gel column with an 8% acetone–hexane mixture as the eluent. Four products were obtained from the chromatography. The first material to elute from the column was identified as *trans*-*N*-methyl-3-phenyl-4-nitro-5-carbomethoxyisoxazolidine (**25**): 30% yield; mp 82–83 °C (10% acetone–hexane); IR (KBr) 3600–3400, 3010–2800, 1730, 1540, 1450–1430, 1380, 1220, 1060, 1000, 800 cm^{-1}; NMR (60 MHz, $CDCl_3$) δ 7.24 (s, 5 H), 5.49 (dd, J = 7.0, 2.0 Hz, 1 H), 4.95 (d, J = 2.0 Hz, 1 H), 3.80 (*d*, J = 7.0 Hz, 1 H), 3.75 (s, 3 H), 2.55 (s, 3 H); MS, *m/e* 266 (M^+), 156, 144, 134, 118, 105, 103, 91, 77. Anal. Calcd for $C_{12}H_{14}N_2O_5$: C, 54.13; H, 5.30; N, 10.52. Found: C, 54.15; H, 5.34; N, 10.51.

(36) All melting points and boiling points are uncorrected. Elemental analyses were performed by Atlantic Microlabs, Atlanta, GA. The infrared absorption spectra were determined on a Perkin-Elmer 467 infrared spectrophotometer. The ultraviolet absorption spectra were measured with a Cary Model 14 recording spectrophotometer by using 1-cm matched cells. The proton magnetic resonance spectra were determined at 90 MHz by using a Varian EM-390 spectrometer. Mass spectra were determined with a Finnigan 4000 mass spectrometer at an ionizing voltage of 70 eV.

(37) Emmons, W. D. *J. Am. Chem. Soc.* **1957**, *79*, 5739.

(38) Shechter, H.; Conrad, F.; Daulton, A. L.; Kaplan, R. B. *J. Am. Chem. Soc.* **1952**, *74*, 3052.

(39) Shindo, H.; Umezowa, B. *Chem. Pharm. Bull.* **1962**, *10*, 492.

The second material obtained was assigned as *cis*-*N*-methyl-3-phenyl-4-nitro-5-carbomethoxyisoxazolidine (**24**): 30% yield; clear oil; IR (neat) 3700–3300, 3020, 2950, 2900, 2890, 1750, 1560, 1440, 1380, 1240, 1220, 1100 cm^{-1}; NMR (60 MHz, CCl_4) δ 7.25 (s, 5 H), 5.50–5.20 (m, 2 H), 3.85 (d, J = 6.0 Hz, 1 H), 3.75 (s, 3 H), 2.60 (s, 3 H); MS, m/e 266, 192, 150, 118, 103, 91, 84, 77. Anal. Calcd for $C_{12}H_{14}N_2O_5$: C, 54.13; H, 5.30; N, 10.52. Found: C, 54.15; H, 5.28; N, 10.50.

The third material isolated from the column was assigned as *cis*-*N*-methyl-3-phenyl-4-carbomethoxy-5-nitroisoxazolidine (**27**): 25% yield; mp 94–95 °C, IR (KBr) 3500, 3080, 3000, 2950, 1740, 1560, 1430, 1380, 1200, 1020, 740 cm^{-1}; NMR (60 MHz, $CDCl_3$) δ 7.40 (s, 5 H), 6.15 (d, J = 2.0, 1 H), 4.55 (*d*, J = 8.0 Hz, 1 H), 4.30 (dd, J = 8.0, J = 2.0 Hz, 1 H), 3.40 (s, 3 H), 2.85 (s, 3 H); MS, m/e 266, 192, 150 (base), 118, 103, 91, 84, 77. Anal. Calcd for $C_{12}H_{14}N_2O_5$: C, 54.13; H, 5.30; N, 10.52. Found: C, 54.20; H, 5.35; N, 10.48.

The last material eluted from the column was identified as *trans*-*N*-methyl-3-phenyl-4-carbomethoxy-5-nitroisoxazolidine (**26**) 15% yield; oily material; IR (neat) 3075, 3000, 1740, 1560, 1380, 1210, 760 cm^{-1}; NMR (90 MHz, $CDCl_3$) δ 7.35 (s, 5 H), 5.75 (d, J = 3.0 Hz, 1 H), 4.1 (dd, J = 910 Hz, 3.0 Hz, 1 H), 3.8 (s, 3 H), 3.75 (*d*, J = 9.0 Hz, 1 H), 2.7 (s, 3 H); MS, m/e 266 (M^+), 192, 160 (base), 150, 131, 103, 91, 77.

Reaction of *N*-Methyl-*C*-phenylnitrone with Chloronitroethylene. A solution containing 3.44 g of chloronitroethylene in 8 mL of benzene was added dropwise to a solution containing 3.0 g of *N*-methyl-*C*-phenylnitrone in 50 mL of benzene at 25 °C under a nitrogen atmosphere. After the mixture was stirred for 18 h, the solvent was removed under reduced pressure. The resulting brown oil was subjected to flash chromatography on silica gel with a 10% acetone–hexane mixture as the eluent. The first component isolated contained 0.4 g of benzaldehyde (17%). The second component isolated from the column contained 0.76 g (20%) of a white solid (mp 140–141 °C) whose structure was assigned as 3,7-diaza-*N*,*N*′-dimethyl-4,6-*trans*-diphenyl-5-nitro-2,8-dioxabicyclo[3.3.0]octane (**35**) on the basis of its spectral properties: IR (KBr) 3050, 3000, 2980, 2940, 2890, 1540, 1480, 1440, 1360, 1300 cm^{-1}; NMR ($CDCl_3$, 90 MHz) δ 2.65 (s, 3 H), 2.85 (s, 3 H), 4.15 (s, 1 H), 4.36 (s, 1 H), 6.30–6.40 (m, 2 H), 6.45 (s, 1 H), 7.0–7.8 (m, 8 H); ^{13}C NMR ($CDCl_3$, 20 MHz) δ 42.79 (q), 43.20 (q), 76.52 (d), 76.77 (d), 104.96 (d), 111.38 (s), 127.84, 128.39, 128.96, 130.09, 131.46, 131.61; MS, m/e 341 (M^+), 220, 144, 134, 118 (base), 91, 77. Anal. Calcd for $C_{18}H_{19}N_3O_4$: C, 63.33; H, 5.61; N, 12.31. Found: C, 63.58; H, 5.57; N, 12.37.

The third component isolated from the column contained 2.2 g (41%) of a white solid (mp 146–147 °C) whose structure was assigned as *trans*-5-chloro-4-nitro-3-phenyl-*N*-methylisoxazolidine (**32**) on the basis of its spectral and chemical properties: IR (KBr) 3050, 2950, 2900, 2800, 1530, 1440, 1420, 1360, 1300, 1100, 1040, 1020, 740 cm^{-1}; NMR ($CDCl_3$, 90 MHz) δ 2.97 (s, 3 H), 4.84 (d, 1 H, J = 6.0 Hz), 5.62 (d, 1 H, J = 6.0 Hz), 6.60 (s, 1 H), 7.31 (s, 5 H); ^{13}C NMR ($CDCl_3$, 20 MHz) δ 45.98 (q), 72.19 (d), 90.22 (d), 99.58 (d), 127.77, 129.14, 129.79, 130. 42; MS, m/e 242 (M^+), 161, 160, 136, 136 (base), 119, 118, 115, 91, 77. Anal. Calcd for $C_{10}H_{11}ClN_2O_3$: C, 49.49; H, 4.57; N, 11.54; Cl, 14.61. Found C, 49.53; H, 4.59; N, 11.55; Cl, 14.64.

The structure of this material was further established by reduction of **32** to give *trans*-4-nitro-3-phenyl-*N*-methylisoxazolidine (**13**). A solution containing 0.39 g of tri-*n*-butyltin hydride in 30 mL of benzene was added to a solution containing 0.3 g of **32** in 30 mL of benzene. The mixture was stirred at 25 °C for 44 h. Removal of the solvent followed by flash chromatography on silica gel gave 0.17 g of *trans*-4-nitro-3-phenyl-*N*-methylisoxazolidine (**13**).[19]

The fourth component isolated from the column contained 0.57 g (15%) of a white solid (mp 137–138 °C) whose structure was assigned as 3,7-diaza-*N*,*N*-dimethyl-4,6-*cis*-diphenyl-5-nitro-2,8-dioxabicyclo[3.3.0]octane (**34**) on the basis of its spectral characteristics: IR (KBr) 3070, 3030, 2990, 1545, 1495, 1457, 1435, 1379; NMR ($CDCl_3$, 90 MHz) δ 2.60 (s, 6 H), 4.45 (s, 2 H), 6.60 (s, 1 H), 7.30 (br s, 10 H); ^{13}C NMR ($CDCl_3$, 20 MHz) δ 41.60 (q), 103.35 (d), 114.28 (s), 128.25, 128.98, 129.58, 131.84; MS, m/e 341 (M^+), 295, 267, 250, 220, 144, 134 (base), 118, 91. Anal. Calcd for $C_{18}H_{19}N_3O_4$: C, 63.33; H, 5.61; N, 12.31. Found: C, 63.17; H, 5.67; N, 12.27.

The cycloaddition was repeated with 3.0 g of *N*-methyl-*C*-phenylnitrone, 7.17 g of chloronitroethylene, and 3.28 g of stronitium carbonate. Under these conditions only 5-chloro-4-nitro-3-phenyl-*N*-methylisoxazolidine (**32**) was isolated in 85% yield. When 3.0 g of *N*-methyl-*C*-phenylnitrone and 1.19 g of chloronitroethylene were used, a 37% yield of the two isomeric bis adducts (1:1) were the only products that could be isolated and identified. Treatment of 0.27 g of the nitrone with 0.24 g of chloronitroethylene in 20 mL of benzene at 25 °C for 72 h gave rise to a mixture of the two isomeric bis adducts **34** and **35**.

Reaction of 5-Chloro-4-nitro-3-phenyl-*N*-methylisoxazolidine (32) with Benzohydroximoyl Chloride. To a solution containing 0.65 g of benzohydroximoyl chloride in 300 mL of ether at 0 °C was added 195 mL of triethylamine. To this mixture was added a solution containing 1.0 g of 5-chloro-4-nitro-3-phenyl-*N*-methylisoxazolidine (**32**) in 100 mL of ether. The mixture was allowed to stir at 25 °C for 24 h. The solution was filtered and concentrated under reduced pressure. Flash chromatography of the residue on a silica gel column with a 10% ethyl acetate–hexane mixture gave 0.15 g of the nitrile oxide dimer followed by 0.24 g (24%) of a white solid (mp 104–105 °C) whose structure was assigned as 4-methyl-3,5-diphenyl-1,2,4-oxadiazoline (**36**):[25] IR (KBr) 1580, 1540, 1490, 1450, 1370, 1280, 1210, 1050, 1000, 830, 750 cm^{-1}; NMR ($CDCl_3$, 90 MHz) δ 2.63 (s, 3 H), 6.03 (s, 1 H), 7.13–7.77 (m, 10 H); UV (95% ethanol) 274 nm (ϵ 2300), 216 (17 100); MS, m/e 238 (M^+), 161, 134, 118, 105, 77. Anal. Calcd for $C_{15}H_{14}N_2O$: C, 75.60; H, 5.92; N, 11.76. Found: C, 75.65; H, 5.97; N, 11.74.

Acknowledgment. We gratefully acknowledge the National Cancer Institute for generous support of this research. A.P. thanks the organic faculty at the University of Wurzburg for their gracious hospitality during his stay at the Institute as an Alexander von Humboldt Senior Awardee. Use of the high-field NMR spectrometer used in these studies was made possible through a NSF equipment grant.

Registry No. 1, 3376-23-6; **2**, 87190-43-0; **3**, 87190-44-1; **4**, 3376-24-7; **5**, 87190-45-2; **6**, 87190-46-3; **7**, 87190-47-4; **8**, 87190-48-5; **9**, 87190-49-6; **10**, 87190-50-9; **11**, 87190-51-0; **12**, 43044-79-7; **13**, 43044-80-0; **14**, 87190-52-1; **15**, 87190-53-2; **16**, 87190-54-3; **19**, 78759-39-4; **20**, 78759-40-7; **21**, 87190-55-4; **22**, 78759-34-9; **23**, 78759-35-0; **24**, 87190-56-5; **25**, 87190-57-6; **26**, 87190-65-6; **27**, 87190-58-7; **28**, 87190-59-8; **29**, 87190-60-1; **30**, 87190-61-2; **31**, 87190-62-3; **32**, 87190-64-5; **34**, 87247-57-2; **35**, 87190-63-4; **36**, 37716-09-9; acrylonitrile, 107-13-1; methyl acrylate, 96-33-3; nitroethylene, 3638-64-0; *trans*-1-nitro-2-cyanoethylene, 76954-16-0; *trans*-methyl 3-nitroprop-2-enoate, 52745-92-3; chloronitroethylene, 61404-99-7; benzohyroximoyl chloride, 698-16-8.

Supplementary Material Available: Experimental details are given for the reactions of *N*-methyl-*C*-phenylnitrone and *N*-*tert*-butyl-*C*-phenylnitrone with acrylonitrile, methyl acrylate, and nitroethylene (6 pages). Ordering information is given on any current masthead page.