The organic phase was washed (1 × 5 mL of H_2O, 1 × 5 mL of saturated aqueous NaCl), dried (K_2CO_3), filtered, and concentrated to give a dark brown oil. The residue was bulb-to-bulb distilled (50–100 °C, 2 mm) to give an impure mixture of pyrrolizidines **17**, **8**, and **18** (0.191 g, 2.8:1.2:1 ratio, respectively, by GC analysis). This mixture was separated by preparative gas chromatography (10% SP-2100 on Supelcoport, 6 ft × $^1/_4$ in.) to give pure pyrrolizidines **17** (0.084 g, 42%), **8** (0.040 g, 20%), and **18** (0.030 g, 15%).

***rac*-(1*S*,2*S*,8*S*)-4-Aza-2-(dimethoxymethyl)-1-methylbicyclo[3.3.0]octane (17)**: IR ($CHCl_3$) 1185, 1122, 1075, 1035 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$)[17] δ 4.78 (d, J = 7.5 Hz, MeOC*H*OMe), 3.45 (m, H-8), 3.35 (s, OCH_3), 3.30 (s, OCH_3), 3.12 (ddd, J = 3.1, 6.0, 10 Hz, H-5), 2.94 (dd, J = 8.0, 10.8 Hz, H-3), 2.68 (dd, J = 8.1, 10.8 Hz, H-3), 2.54 (ddd, J = 7.1, 8.2, 10 Hz, H-5), 2.20 (m, H-1), 2.08 (app quintet, J = 6.8 Hz, H-3), 1.90 (m, H-6), 1.68 (m, H-6 and H-7), 1.42 (m, H-7), 1.08 (d, J = 7.3 Hz, CH_3); MS (CH_4 CI), m/z 200.165 0 (200.165 0 calcd $C_{11}H_{22}NO_2$).

***rac*-(1*S*,2*R*,8*S*)-4-Aza-2-(dimethyoxymethyl)-1-methylbicyclo[3.3.0]octane (18)**: IR ($CHCl_3$) 1178, 1120, 1077, 1045 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$)[17] δ 4.40 (d, J = 7.7 Hz, MeOC*H*OMe), 3.80 (m, H-8), 3.35 (s, OCH_3), 3.30 (s, OCH_3), 3.20 (dd, J = 7.7, 9.8 Hz, H-3), 2.84 (m, H-2), 2.52 (m, H-5), 2.45 (dd, J = 8.5, 9.8 Hz, H-3), 2.25 (m, H-1), 1.98 (m, H-5 and H-7), 1.74 (m, 2 H-6 and H-7), 0.88 (d, J = 7.7 Hz, CH_3); MS (CH_4 CI), m/z 200.165 0 (200.165 0 calcd for $C_{11}H_{22}NO_2$).

Exploration of Possible Alkene Isomerization in the Rearrangement of *Z* Alkene Amino Acetal 15. Three reaction samples of **15** (1.0 mmol) were prepared as previously described, and the ampules were heated at 115 °C for 3, 4, or 8 h. At the end of each reaction period, the sample was cooled to 0 °C and quenched with an excess of solid $NaBH_4$ (0.04 g, 1 mmol). Comparison of these samples with authentic samples of the (*Z*)- and (*E*)-pyrrolidine alcohols **19** and **20**, by capillary GC (30 m DB-5 column) and 250-MHz ^1H NMR analyses indicated the presence of only **19**.

(*Z*)-1-Pyrrolidino-3-penten-2-ol (19). To a solution of amino alcohol **13** (0.301 g, 2.98 mmol) and dry THF (10 mL) was added anhydrous K_2CO_3 (2.5 g, 18 mmol). The slurry was stirred, and a solution of 1,4-dibromobutane (0.36 mL, 3.0 mmol) in dry THF (10 mL) was added via cannula over a 45-min period. The resulting mixture was stirred at 24 °C for 12 h and filtered, and the solvent was evaporated. Flash chromatography (30 g, SiO_2, 95:5 ether/Et_3N) gave **19** as a colorless oil (0.333 g, 72% yield): IR (film) 3600–3200, 1660, 1605, 1450, 1115 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 5.54 (dqd, J = 1.0, 6.7, 11.2 Hz, CH_3CH=), 5.35 (qdd, J = 1.6, 8.3, 11.2 Hz, =C*H*CH), 4.45 (pseudo dt, J = 4.0, 9.1 Hz, C*H*OH), 2.8–2.4 (m, 6 H, CH_2N), 1.66 (dd, J = 1.6, 6.7 Hz, CH_3CH), 1.45 (m, CH_2CH_2); MS (CH_4 CI), m/z 155.1311 (155.1310 calcd for $C_9H_{17}NO$).

(*E*)-1-Pyrrolidino-3-penten-2-ol (20). Amino alcohol **4** (1.002 g, 9.92 mmol) was alkylated with 1,4-dibromobutane (1.20 mL, 10.0 mmol) as described in the preparation of **19** to give **20** (1.169 g, 76%) as a colorless oil after purification on SiO_2: IR (film) 3600–3200, 1610, 1445, 1110, 1060 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) 5.70 (dqd, J = 15.2, 6.8, 1.0 Hz, CH_3CH=), 5.44 (qdd, J = 1.6, 6.5, 15.2 Hz, CH_3CH=C*H*), 4.10 (br m, C*H*OH), 2.8–2.4 (m, 6 H, CH_2N), 1.72 (dd, J = 1.6, 6.8 Hz, CH_3CH=), 1.45 (m, CH_2CH_2); MS (CH_4CI), m/z 155.1309 (155.1310 calcd for $C_9H_{17}NO$).

Crystallography. Single crystals were prepared by slow crystallization from hexane. The crystals were found to belong to the triclinic system with unit cell dimensions at 22 °C: a = 8.453 (3) Å, b = 9.803 (3) Å, c = 11.513 (4) Å; α = 74.25 (2)°, β = 94.18 (2)°, γ = 112.23 (2)°. Intensity statistics favored the centrosymmetric space group P_1^-. A density of 1.30 g cm^{-3} was calculated for Z = 2 formula units per unit cell. Three-dimensional intensity data were collected on a Syntex $P2_1$ automated diffractometer, using monochromatized Mo Kα radiation (λ = 0.709 30 Å). The $\theta/2\theta$ scan technique was used to measure the intensities of 2215 independent reflections within the range 0° < 2θ < 55°.[18] Of these, 1815 had $F^2 > 3\sigma(F^2)$ and were used in subsequent calculations.

The structure was solved in a straightforward fashion by direct methods, with MULTAN 77 system of programs.[19] The oxalic acid of crystallization was revealed at this stage. Refinement was by full-matrix least-squares methods.[20] Hydrogen atoms were included at their idealized positions (C–H = 0.95 Å) with fixed isotropic temperature factors of 5.0 Å^2. The final unweighted and weighted R values were 0.047 and 0.064, respectively. A final difference map showed no significant residue.

Acknowledgment. Financial support from the National Science Foundation (Grant CHE-82-03366) is gratefully acknowledged. We particularly thank R. Marquis for carrying out control experiments with **8**. NMR and mass spectra were determined with spectrometers purchased with the assistance of NSF Shared Instrumentation Grants. We thank also Dr. Joseph Ziller for assistance with the crystallographic data.

Registry No. **3**, 4388-56-1; (±)-**4**, 112069-72-4; (±)-**5**, 112069-73-5; (±)-**6**, 112069-74-6; (±)-**8**, 112069-75-7; (±)-**8**·oxalate, 112069-82-6; (±)-**9**, 112069-76-8; **10**, 1119-19-3; (±)-**11**, 112069-77-9; (±)-**12**, 112069-78-0; (±)-**13**, 112069-79-1; (±)-**14**, 112069-80-4; (±)-**15**, 112069-81-5; (±)-**17**, 112069-83-7; (±)-**18**, 112069-84-8; (±)-**19**, 112069-85-9; (±)-**20**, 112069-86-0; TMS-CN, 7677-24-9; $Br(CH_2)_4Br$, 110-52-1.

Supplementary Material Available: Experimental data for X-ray diffraction study of pyrrolizidine acetal **8** and tables of positional parameters, anisotropic temperature factors, bond angles, and interatomic distances (4 pages). Ordering information is given on any current masthead page.

(18) General procedures for data collection and processing have been given in: Sams, D. B.; Doedens, R. J. *Inorg. Chem.* **1979**, *18*, 153.

(19) Main, P.; Lessinger, L.; Woolfson, M. M.; Germain, G.; Declercq, J. P. MULTAN 77, University of York, England, 1977.

(20) All computations were carried out on a VAX 11/780 computer by use of a modified version of the UCLA Crystallographic Computing Package (C. E. Strouse, personal communication). Major programs in this package are derived from the MULTAN system and from the Oak Ridge ORFLS/ORFFE/ORTEP programs.

(21) The numbering of the bicyclo compounds in this paper does not follow IUPAC recommendations.

Base-Induced Reactions of Polynitroarenes with Methyl 2-Chloropropionate

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The alkylation of nitro aromatic compounds by leaving group substituted carbanions, a method pioneered by Makosza and co-workers, has proven to be one of the few general processes accomplishing formal nucleophilic aromatic substitution for hydrogen.[1] Our study of one such system showed that methyl 2-chloropropionate (MCP) reacts with a variety of nitro aromatic compounds under the influence of 2 equiv of strong base (NaH, Me_3COK, or Me_3CONa) in *N*,*N*-dimethylformamide (DMF) to give methyl 2-(4-nitrophenyl)propionates in good yields.[2] While reactions of this type are thought to proceed via Meisenheimer salt formation followed by elimination of HCl to give product anion (Scheme I), there is little direct evidence to support this mechanism.

Makosza and Glinka studied the mechanism of nitrobenzene alkylation by chloromethyl phenyl sulfone and

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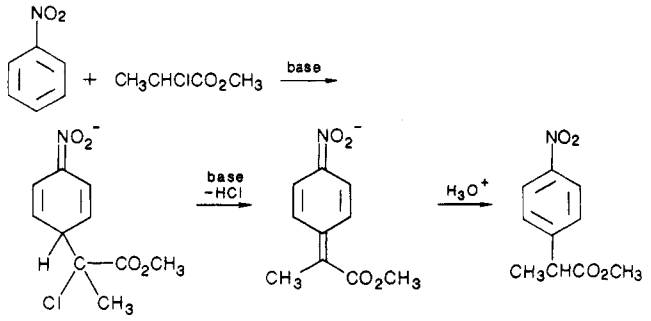
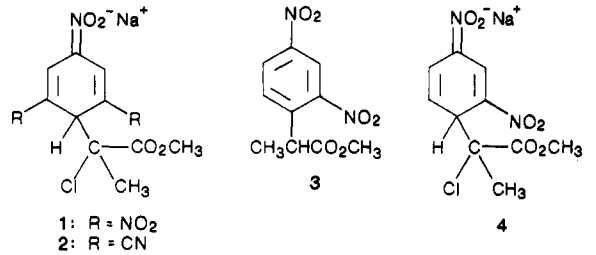
0022-3263/88/1953-0690$01.50/0

Scheme I



concluded that the reaction proceeds by a fast, reversible carbanion addition followed by a slower base-induced elimination.[3] However, while the proposed mechanism calls for breaking of the Ar–H bond during the rate-determining step, only a secondary kinetic isotope effect could be derived from experiments using nitrobenzene-*4-d*. Makosza suggested that the small isotope effect is due to a nonsymmetrical transition state in the elimination step.

We have studied the reactions of 1,3,5-trinitrobenzene (TNB), 3,5-dicyanonitrobenzene (DCNB), and 1,3-dinitrobenzene (DNB) with MCP in DMF, with Me_3CONa as base. The expected alkylation product did not form in the TNB reaction. Instead a stable red oil was isolated by chromatography and was identified by spectral means as Meisenheimer complex 1. Isolated Meisenheimer



complexes containing α-halogen atoms are rare.[4] The reaction of DCNB with MCP under similar conditions also failed to yield an alkylated product. Spectral (UV–vis λ_{max} 422, 521 nm)[5] and TLC evidence suggested the presence of a Meisenheimer complex in the reaction mixture, but attempts to isolate the complex were unsuccessful. A likely structure for the complex is **2**, where addition has occurred para to the nitro group. This proposed regiochemistry is supported by the reported para selectivity of methoxide addition to DCNB.[6]

In contrast to the TNB and DCNB results, DNB was alkylated by MCP to give compound **3** in 37% yield. Presumably, Meisenheimer salt **4** is an intermediate in this reaction.

These data suggest that hydrogen chloride is readily eliminated from complex **4** but not from complexes **1** or **2**. In fact, treatment of isolated **1** with several bases led only to recovery of unreacted **1** (Me_3COK, DMF, 25 °C; DBU, THF, reflux) or decomposition to intractable products (NaH, DMF, 25 °C; LDA, THF, –78 °C). This resistance to HCl elimination could be explained by the steric argument that rotation of the side chain of **1** is restricted by the flanking aromatic nitro groups, preventing an antiperiplanar arrangement of the hydrogen and chlorine atoms. However, such an explanation will not comfortably account for the observed lack of elimination from complex **2**, where the flanking substituents are the much smaller cyano groups.

It is possible that relative Meisenheimer complex stabilities are particularly important in understanding these reactions. Equilibrium constants have been determined or estimated for complexes of methoxide ion with TNB (23 L mol^{-1}),[7] DCNB (1.4×10^{-3} L mol^{-1}),[6] and DNB ($\sim 10^{-6}$ L mol^{-1})[8] in methanol. The TNB and, to a lesser degree, DCNB complexes are more stable than the DNB complex and do not readily lose HCl to form alkylated nitrobenzenes. On the other hand, mononitroarenes, which afford enduring complexes only in certain systems at low temperatures,[9] are readily alkylated by MCP in yields that exceed that obtained by using DNB.[2] However, this Meisenheimer complex stability argument is not applicable in all cases. Apparently the character of the nucleophile is an important variable, as Makosza and Ludwiczak observed trisubstitution of TNB by chloromethyl phenyl sulfone.[10]

In search of deuterium isotope effects, we conducted experiments in which MCP was allowed to compete for nondeuteriated and perdeuteriated nitroarenes. Product mixtures were examined by gas chromatography/mass spectrometry. Control experiments were used to determine the extent of hydrogen (or deuterium) exchange occurring under the conditions used (Me_3CONa, DMF, 0 °C). For example, reaction of nitrobenzene-d_5 with MCP gave a single product containing four deuterons. Reaction of DNB-d_4 with MCP afforded products containing either two or three deuterons. The d_2 product presumably derived from exchange of the acidic deuteron situated between the two nitro groups. This type of exchange in the DNB system has been studied extensively.[11]

Reaction of equimolar amounts of MCP, nitrobenzene, and nitrobenzene-d_5 afforded a 1:1 ratio of products arising from MCP attack at protonated and deuteriated positions. Therefore, as in Makosza's system,[3] no primary deuterium isotope effect was evident. However, reaction of MCP with a mixture of DNB and DNB-d_4 gave a preponderance of the product arising from MCP attack on DNB. The ratio of $d_0 + d_1$ species to $d_2 + d_3$ species afforded a k_H/k_D value of 3.9, corresponding to a primary isotope effect.

Makosza and Winiarski recently reported similar isotope effects in DNB reactions and offered a reasonable mechanistic explanation.[12] They maintained that the elimination step is rate determining. Variations in the symmetry of the elimination transition state due to factors such as Meisenheimer complex stability and steric interactions are responsible for variations in the magnitude of the observed isotope effect. Thus, intermediacy of a less stable Meisenheimer complex such as a mononitroarene complex would result in an unsymmetrical elimination transition state and a small isotope effect. On the other hand, a more stable complex such as that from a dinitroarene would proceed through a more symmetrical elimination transition state and result in an observable primary isotope effect.

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(5) The UV–vis spectrum is consistent with the spectra of similar complexes. See: Strauss, M. J. *Chem. Rev.* **1970**, *70*, 667.
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An alternative explanation, at least for our systems, is that the rate-determining step changes with Meisenheimer complex stability.[13] In the DNB–MCP reaction, the rate-determining transformation may be elimination from the relatively stable DNB Meisenheimer complex. In this case the observed primary isotope effect would be anticipated. However, in the nitrobenzene–MCP reaction, formation of the unstable Meisenheimer complex may be rate determining, a situation that is consistent with the absence of a primary isotope effect. Similar multistep reaction pathways have been examined in detail and are believed to involve rate-determining Meisenheimer complex formations.[14]

It is evident that these reactions are complex, particularly when the diversity of possible nucleophiles, substrates, and reaction conditions is considered. A definitive clarification of the actual pathways involved awaits further experimentation.

Experimental Section

NMR spectra were recorded on a Varian EM-390 or a GE/NIC NT-360 spectrometer. Chemical shifts are reported in parts per million relative to tetramethylsilane. Infrared spectra were recorded on a Perkin-Elmer 983 spectrophotometer. UV–visible spectra were obtained on a Varian Cary 219 spectrophotometer. Mass spectra were obtained on a Finnigan 4023 gas chromatograph/mass spectrometer equipped with a 50-M SE-52 fused silica capillary column. Preparative thin-layer chromatography (PTLC) was carried out on commercially prepared silica gel plates (Analtech), and visualization was by ultraviolet light. Elemental analyses were performed by Galbraith Laboratories, Knoxville, TN.

Meisenheimer Complex 1. A mixture of 100 mg (1.04 mmol) of sodium *tert*-butoxide, 107 mg (0.502 mmol) of 1,3,5-trinitrobenzene (TNB), 57 μL (0.50 mmol) of methyl 2-chloropropionate (MCP), and 1 mL of *N,N*-dimethylformamide (DMF) was heated at 60 °C for 1 h and poured into 10 mL of water. The resulting red, aqueous mixture was extracted with three 5-mL portions of dichloromethane, treated with 3 g of sodium chloride, and extracted with eight 10-mL portions of diethyl ether. Combination, drying ($MgSO_4$), and concentration of the ether layers afforded a residue, which was purified by PTLC (20% methanol in dichloromethane eluent), giving 15 mg of **1** as a dark red oil: ^1H NMR (DMSO-d_6) δ 1.40 (s, 3 H), 3.58 (s, 3 H), 5.68 (m, 1 H), 8.44 (m, 2 H); ^{13}C NMR (DMSO-d_6) 24.9 (q), 44.6 (d), 53.4 (q), 75.0 (s), 122.9 (s), 126.7 (s), 128.2 (d), 128.2 (s), 128.8 (d), 169.8 (s); UV-vis (DMF) λ_{max} 448, 502 nm.

Methyl 2-(2,4-Dinitrophenyl)propionate (3). An ice-cold mixture of 100 mg (1.04 mmol) of sodium *tert*-butoxide and 0.5 mL of DMF was treated dropwise with a solution of 84 mg (0.50 mmol) of 1,3-dinitrobenzene (DNB) and 57 μL (0.50 mmol) of methyl 2-chloropropionate (MCP) in 0.5 mL of DMF. The purple mixture was allowed to warm to room temperature and poured into 10 mL of 1 N HCl. Extraction with three 10-mL portions of diethyl ether followed by combination, drying ($MgSO_4$), and concentration of the ether layers afforded a residue, which was purified by PTLC (50% petroleum ether in dichloromethane eluent), giving 47 mg (37% yield) of **3** as an oil: IR (neat) 3099, 2952, 1738, 1605, 1534, 1349 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.68 (d, 3 H, J = 7 Hz), 3.72 (s, 3 H), 4.45 (q, 1 H, J = 7 Hz), 7.78 (d, 1 H, J = 8 Hz), 8.47 (dd, 1 H, J = 8, 2 Hz), 8.81 (d, 1 H, J = 2 Hz); mass spectrum (70 eV), m/e (relative intensity) 208 (22), 102 (27), 77 (45), 76 (22), 65 (25), 59 (100), 51 (30), 43 (98). Anal. Calcd for $C_{10}H_{10}N_2O_6$: C, 47.25; H, 3.97; N, 11.02. Found: C, 47.62; H, 3.90; N, 11.29.

Acknowledgment. Acquisitions of mass spectral data by Dr. V. O. Brandt and ^{13}C NMR data by Dr. L. S. Simeral are gratefully acknowledged. We thank the Callery Chemical Co. for a gift of sodium *tert*-butoxide.

Registry No. 1, 111870-36-1; 2, 111902-85-3; 3, 93742-87-1; DCNB, 33224-18-9; TNB, 99-35-4; MCP, 17639-93-9; DNB, 99-65-0; deuterium, 7782-39-0.

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An Asymmetric Synthesis of Carbapenem Antibiotic (+)-PS-5 from Ethyl 3-Hydroxybutanoate

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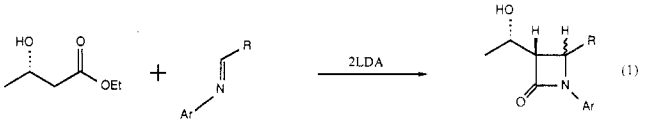
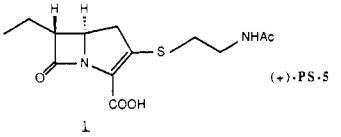
Recently, we[1] and others[2] have demonstrated that readily available esters of optically active 3-hydroxybutyric acid are of great utility in the convergent synthesis of thienamycin and related β-lactam antibiotics.[3] As detailed in eq 1, dianion arylaldimine condensation produces 3-



(1)

Ar = p-methoxyphenyl

(hydroxyethyl)-2-azetidinones with the correct absolute stereochemistry at position 3 of the β-lactam ring system as needed for the elaboration of the total synthesis of thienamycin, antibiotic PS-5 (**1**), and related penems. We now report the full details of an extension of this methodology toward the asymmetric synthesis of carbapenem antibiotic (+)-PS-5 (**1**).[4,5] Our strategy relies on the



(+)-PS-5

1

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0022-3263/88/1953-0692$01.50/0