(br s, 1 H), 9.73 (t, J = 2 Hz, 1 H); MS m/z 232 (M + H). Anal. Calcd for $C_{13}H_{13}NO_3$: C, 67.52; H, 5.67; N, 6.06. Found: C, 67.84; H, 5.75; N, 6.07.

(1′*S*,2″*S*,3″*R*,4″*S*)-2-[1-[*N*-(1-Cyclohexyl-3,4-dihydroxy-6-methyl-2-heptyl)carbamoyl]-2-(4-imidazolyl)ethyl]-1,2,3,4-tetrahydro-9*H*-pyrido[3,4-*b*]indol-1-one (10b). Reductive amination as above with **11** and the L-histidine amide of (2*S*,3*R*,4*S*)-2-amino-1-cyclohexyl-3,4-dihydroxy-6-methylheptane gave **12** in 89% yield as a 2:1 mixture of diastereomers after flash chromatography using 10% methanol in chloroform. A sample of **12** (325 mg, 0.55 mmol) in 8 mL of dioxane was treated with 4.5 mL (1.3 mmol) of 0.3 M aqueous NaOH and heated at 60 °C. After 6 h, the solvent was removed in vacuo, and the crude salt was cyclized as above, to give **10b** (mp 153–159 °C) in 13% yield after separation of diastereomers by silica gel chromatography using 7.5% methanol in chloroform: ^{1}H NMR ($CDCl_3$) δ 0.6–1.9 (br envelope), 0.78 (d, J = 7 Hz, 1 H), 0.93 (d, J = 7 Hz, 3 H), 3.0 (m, 3 H), 3.25 (m, 3 H), 3.5 (m, 1 H), 3.83 (m, 2 H), 4.33 (m, 2 H), 5.73 (dd, J = 10, 5 Hz, 1 H), 6.89 (s, 1 H), 7.16 (t, J = 8 Hz, 1 H), 7.32 (t, J = 8 Hz, 1 H), 7.43 (m, 2 H), 7.60 (d, J = 8 Hz, 1 H), 10.05 (br, 1 H); MS m/z 550 $(M + H)^+$. Anal. Calcd for $C_{31}H_{43}N_5O_4 \cdot 0.5H_2O$: C, 66.64; H, 7.94; N, 12.53. Found: C, 66.99; H, 7.94; N, 12.30.

Acknowledgment. The assistance of the Analytical Research Department at Abbott Laboratories in providing spectra is gratefully acknowledged.

Registry No. **2a**·Li, 124156-50-9; **2b**·Li, 124156-51-0; **2c**·Li, 124156-52-1; **2d**·Li, 124156-53-2; **2e**·Li, 124156-54-3; **2f**·Li, 124156-60-1; **3a**, 124156-40-7; **3b**, 18450-27-6; **4a**, 124156-42-9; **4b**, 124156-43-0; **4c**, 124175-16-2; **4d**, 124156-44-1; **4e**, 124156-45-2; **5a**, 124156-46-3; **5b**, 124175-17-3; **5c**, 124156-47-4; **5d**, 124156-48-5; **5e**, 124156-49-6; **6a**, 124156-55-4; **6a** (N^{im}-BOC deriv), 124156-61-2; **6a** (1′*R*-diastereomer), 124223-56-9; **6b**, 124156-56-5; **6b** (1′*R*-diastereomer), 124223-57-0; **6c**, 124156-57-6; **6c** (1′*R*-diastereomer), 124223-58-1; **6d**, 124156-58-7; **6e**, 124156-59-8; **7**, 17952-82-8; **8**, 124156-62-3; **9a**, 124156-63-4; **10a** (1′*S*-diastereomer), 124175-18-4; **10a** (1′*R*-diastereomer), 124262-95-9; **10b** (1′*S*-diastereomer), 124156-67-8; **10b** (1′*R*-diastereomer), 124223-60-5; **11**, 80364-01-8; **12** (1′*S*-diastereomer), 124156-66-7; **12** (1′*R*-diastereomer), 124223-59-2; H-His-OMe·2HCl, 7389-87-9; H-Leu-OMe, 2666-93-5; H-Gly-OMe, 616-34-2; (2*S*,3*R*,4*S*)-(c-C_6H_{11})CH_2CH(NH_2)CH(OH)CH(OH)CH_2CHMe_2·HCl, 104882-45-3; (2*S*,3*R*,4*S*)-(c-C_6H_{11})CH_2CH(NH_2)CH(OH)CH(OH)CH_2CHMe_2, 122621-77-6; (±)-$CH_3(CH_2)_3$CHBrCOOEt, 63927-44-6; $Ph_3(MeOCH_2)P^+Cl^-$, 4009-98-7; (2*S*,3*R*,4*S*)-(c-C_6H_{11})CH_2CH(H-His-NH)CH(OH)CH(OH)CH_2CHMe_2, 116183-33-6; indole-2-carboxylic acid, 1477-50-5; benzyl indole-2-carboxylate, 78277-27-7; methyl (±)-2-amino-3-(4-thiazolyl)propanoate, 119357-61-8; methyl (±)-2-amino-3-(3-pyrazolyl)propanoate, 124156-41-8; ethyl (*E*)-3-(2-methoxyethenyl)indole-2-carboxylate, 124156-64-5; ethyl (*Z*)-3-(2-methoxyethenyl)indole-2-carboxylate, 124156-65-6.

Synthesis of *endo,endo*-2,5-Bis((diphenylphosphino)methyl)bicyclo[2.2.1]heptane, a Chelating Diphosphine with a Natural Bite Angle of 120°

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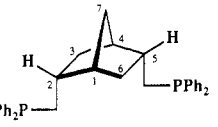
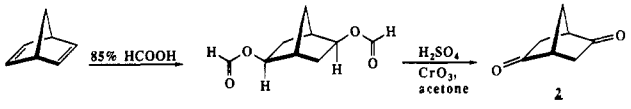
One of the key intermediates in rhodium-catalyzed hydroformylation reactions is a trigonal-bipyramidal rhodium diphosphine species. The two phosphines in $(R_3P)_2(CO)_2RhH$ have the possibility of occupying two axial, two equatorial, or one axial and one equatorial position. Use of chelating ligands such as DIPHOS, $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$, which has a preferred bite angle (P–M–P) of about 90° allows study of locked axial, equatorial diphosphine catalysts. However, few ligands are available to selectively occupy diequatorial positions in trigonal bipyramids. Because of our interest in hydroformylation, we have initiated a program to design and synthesize chelating diphosphines which have a constrained bite angle near 120° for selective diequatorial coordination.

Our design process begins with molecular models and progresses to molecular mechanics calculations. If molecular models indicate a promising candidate, we use molecular mechanics to predict the "natural bite angle" (β_n) of the chelate.[1] We define the natural bite angle as the preferred chelation angle determined only by ligand backbone constraints and not by metal valence angles.[2] Since the natural bite angle depends on the metal–phosphorus bond length, we input a typical Rh–P bond length of 2.30 Å. In addition, we calculate the potential energy well for distortions from the natural bite angle to estimate how flexible the chelate will be. Using this molecular mechanics procedure, we have calculated that Rh(I) complexes of *endo,endo*-2,5-bis((diphenylphosphino)methyl)bicyclo[2.2.1]heptane (**1**) will have a natural bite angle of 122.6°. It should be pointed out that **1** is a chiral molecule with a C_2 axis of symmetry and it may prove useful in enantioselective syntheses. Here we describe the synthesis and characterization of racemic diphosphine **1**.



The starting material for our four step synthesis of diphosphine **1** is bicyclo[2.2.1]heptane-2,5-dione (**2**). Diketone **2** is readily obtained by addition of formic acid to norbornadiene followed by Jones oxidation of the resulting diformate ester.[3]



Reaction of dione **2** with 2 equiv of $CH_2{=}PPh_3$ in DMSO led to the formation of 2,5-bis(methylene)bicyclo[2.2.1]heptane (**3**) in 66% yield. Use of DMSO as solvent gave substantially higher yields than diethyl ether (37%) as has been noted previously.[4]

Hydroboration–oxidation of diene **3** was anticipated to occur from the exo face since Brown had reported 85% exo selectivity in the hydroboration of 2-methylenenorbornane.[5] In practice, hydroboration–oxidation of diene **3** led to the selective formation of *endo,endo*-2,5-bis(hydroxymethyl)bicyclo[2.2.1]heptane (**4**) in 87% yield (Scheme I). No other isomer was detected by ^{1}H or ^{13}C NMR analyses. The presence of only five resonances in the ^{13}C NMR spectrum establishes the C_2 symmetry of diol **4**. In the ^{1}H NMR spectrum of **4**, the equivalent endo protons on C3 and C6 appear at δ 0.90 (dd, J_{gem} = 14.6 Hz, J = 5.7 Hz) upfield from the exo protons at δ 1.3; a similar

(1) Casey, C. P.; Whiteker, G. T., submitted to *Isr. J. Chem.*
(2) This is accomplished by using a bending force constant of 0 kcal mol^{-1} rad^{-2} for the P–M–P angle.
(3) Hawkins, R. T.; Hsu, R. S.; Wood, S. G. *J. Org. Chem.* **1978**, *43*, 4648.
(4) Greenwald, R.; Chaykovsky, M.; Corey, E. J. *J. Org. Chem.* **1963**, *28*, 1128.
(5) Brown, H. C.; Kawakami, J. H. *J. Am. Chem. Soc.* **1970**, *92*, 1990.

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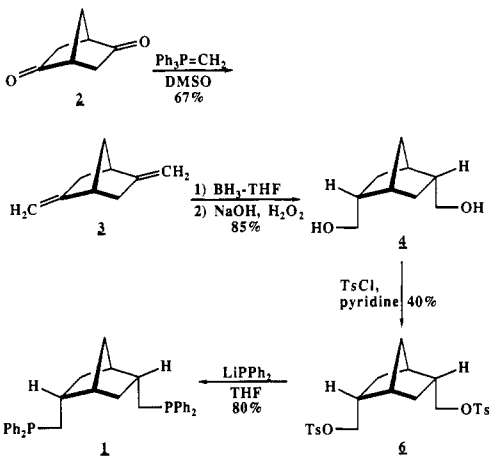
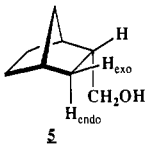
Scheme I

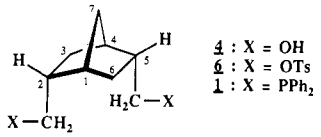


upfield shift of the endo protons is also seen for *endo*-2-(hydroxymethyl)bicyclo[2.2.1]heptane (**5**).[6] The 5.7-Hz coupling of the endo C3 (and C6) proton to a proton on C2 (and C5) requires the presence of an exo proton on C2 (and C5) and establishes the endo,endo stereochemistry of **4**. Similar coupling constants are seen for *endo*-hydroxymethyl compound **5**. The high selectivity for formation of endo,endo compound **4** may be due to enhanced selectivity in the hydroboration of a monohydroborated intermediate.



5

Diol **4** was converted to the crystalline ditosylate *6* in 40% yield by reaction with *p*-toluenesulfonyl chloride in pyridine at 0 °C. The ^{13}C NMR of the recrystallized ditosylate showed the presence of only one isomer. Reaction of ditosylate *6* with $LiPPh_2$ in THF produced diphosphine *1* as a white viscous oil in 80% yield.



The symmetry of **1** was demonstrated by the observation of a single ^{31}P NMR resonance at δ −17.8 and of five aliphatic ^{13}C NMR resonances. Four of these carbon resonances exhibit coupling to the ^{31}P nuclei allowing assignment of the uncoupled ^{13}C resonance at δ 41.5 as the unique bridging CH_2. The 2D ^{13}C–^{1}H NMR correlation spectrum of **1** indicates that the most upfield ^{13}C resonance at δ 29.6 is strongly coupled to two nonequivalent proton resonances at δ 1.25 and 1.65; this requires the assignment of this ^{13}C resonance to the equivalent methylene carbons C3 and C6.

We are currently involved in attempts to synthesize and structurally characterize metal complexes of diphosphine **1**. The effect of **1** and other chelating diphosphines with bite angles ≥120° upon rhodium-catalyzed alkene hydroformylation will be reported elsewhere. Resolution of C_2 chiral diphosphine, **1**, is also being pursued for use in enantioselective metal-catalyzed reactions.

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Experimental Section

General. ^{1}H NMR spectra were measured on a Bruker WP270 or AM500 spectrometer. ^{13}C NMR spectra were obtained on a Bruker AM500 spectrometer operating at 125.76 MHz. ^{31}P NMR spectra were obtained on an AM500 instrument (202.46 MHz) and were referenced to external H_3PO_4. Infrared spectra were measured on a Mattson Polaris (FT) spectrometer. Mass spectra were determined on a Kratos MS-80 mass spectrometer. Elemental analyses were performed by Galbraith Laboratories, Inc. (Knoxville, TN).

2,5-Bis(methylene)bicyclo[2.2.1]heptane (3). Addition of a solution of methyltriphenylphosphonium bromide (17.3 g, 48.3 mmol) in 50 mL of DMSO to a 0 °C solution of dimsyl anion, prepared by addition of *n*-BuLi (27.3 mL, 1.77 M in hexane, 48.3 mmol) to 25 mL of DMSO, produced a bright yellow solution of $Ph_3P{=}CH_2$. Bicyclo[2.2.1]heptane-2,5-dione[3] (3.00 g, 24.2 mmol) in 20 mL of DMSO was added, and the mixture was stirred overnight at room temperature. Water (200 mL) was added, and the solution was extracted with pentane (5 × 100 mL). The combined pentane extracts were washed with H_2O (100 mL) and saturated brine (100 mL) and dried ($MgSO_4$). Removal of pentane by distillation gave a yellow oil, which was purified by filtration through 60 g of alumina and elution with 1 L of pentane. Removal of pentane by distillation gave **3** as a colorless liquid (1.92 g, 66%): ^{1}H NMR ($CDCl_3$, 270 MHz) δ 4.89 (s, 2 H, C=C*H*H), 4.83 (s, 2 H, C=CH*H*), 2.79 (br s, 2 H, bridgehead H), 2.28 (d, J = 16 Hz, 2 H, allylic C*H*H), 1.97 (d, J = 16 Hz, 2 H, allylic CH*H*), 1.47 (s, 2 H, CH_2 bridge); ^{13}C NMR ($CDCl_3$) δ 154.5 (s, *C*=CH_2), 103.0 (t, J_{CH} = 165 Hz, C=*C*H_2), 46.2 (d, J_{CH} = 123 Hz, CH bridgehead), 40.1 (t, J_{CH} = 125 Hz, CH_2 bridge), 38.6 (t, J_{CH} = 125 Hz, allylic CH_2); HRMS calcd for C_9H_{12} 120.0939, found 120.0946.

***endo,endo*-2,5-Bis(hydroxymethyl)bicyclo[2.2.1]heptane (4).** BH_3·THF (40 mL, 1 M in THF) was added to a solution of **3** (7.68 g, 63.9 mmol) in 10 mL of THF at 0 °C. Excess diborane was destroyed by addition of 10 mL of H_2O at room temperature. Aqueous NaOH (20 mL, 3 M) and 30% H_2O_2 (20 mL) were added. The aqueous layer was extracted with THF (4 × 100 mL), and the combined organic layers were washed with 100 mL of saturated brine and dried ($MgSO_4$). Rotary evaporation of solvent gave **4** as a pale yellow oil (8.65 g, 87%): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.3 (m, 4 H, *C*H_2OH), 3.0 (s, 2 H, OH), 2.1 (m, 2 H, CH_2 bridge), 1.9 (m, 2 H, *C*HCH_2OH), 1.4 (m, 2 H, bridgehead), 1.3 (m, 2 H, exo H on C3), 0.90 (dd, J = 14.6, 5.7 Hz, endo H on C3); $^{13}C\{^{1}H\}$ NMR ($CDCl_3$) δ 63.6 (CH_2OH), 42.9 (*C*HCH_2OH), 38.2 (CH bridgehead), 29.7 (CH_2 bridge), 25.1 (C3 and C6); IR (neat) 3400 (br) cm^{-1}.

***endo,endo*-2,5-Bis((*p*-tolylsulfonoxy)methyl)bicyclo[2.2.1]heptane (6).** Solid *p*-toluenesulfonyl chloride (4.08 g, 21.4 mmol) was slowly added to a solution of **4** (1.67 g, 10.7 mmol) in 16.0 mL of pyridine at 0 °C. After 3 h at 0 °C, 1 M HCl (60 mL) was added, and the solution was extracted with Et_2O (4 × 50 mL). The Et_2O extracts were washed with 50 mL of saturated brine, dried ($MgSO_4$), and concentrated. The resulting white solid was washed with 10 mL of cold methanol to give **5** (1.67 g, 34%) as a white, microcrystalline solid, mp 99.5–100.5 °C. Analytically pure material was obtained by recrystallization from methanol: ^{1}H NMR ($CDCl_3$, 270 MHz) δ 7.8 (d, J = 10 Hz, 4 H), 7.3 (d, J = 10 Hz, 4 H), 4.8 (m, 4 H, CH_2OTs), 2.4 (s, 6 H, CH_3), 2.0–2.2 (m, 4 H), 1.6–1.3 (m, 8 H), 0.75 (dd, 2 H, J = 13.6, 4.5 Hz, endo CH); ^{13}C INEPT ($CDCl_3$) δ 144.8, 132.9 (para and ipso), 129.9, 127.8 (ortho and meta), 70.5 (CH_2O), 40.6 (CH_2 bridge), 38.9, 38.1 (*C*HCH_2O and bridgehead CH), 24.2 (CH_2), 21.4 (CH_3). Anal. Calcd for $C_{23}H_{28}O_6S_2$: C, 59.46; H, 6.07. Found: C, 59.53; H, 6.17.

***endo,endo*-2,5-Bis((diphenylphosphino)methyl)bicyclo[2.2.1]heptane (1).** A solution of $LiPPh_2$, prepared from lithium metal (0.46 g, 66 mmol) and chlorodiphenylphosphine (0.91 mL, 5.1 mmol) in 50 mL of THF was added under N_2 via syringe to a solution of **6** (1.16 g, 2.5 mmol) in 20 mL of THF. After 1 h, methanol (2 mL) was added and the solution was evaporated under vacuum. The residue was dissolved in 50 mL of dichloromethane, filtered through a 5-cm plug of Celite and alumina, and evaporated to give **1** (0.98 g, 80%) as a viscous, white liquid: ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.5–7.2 (m, 20 H), 2.16 (m, 4 H, CH_2P), 2.15 (m, 2 H, bridgehead), 1.91 (m, 2 H, C*H*CH_2P), 1.65

(td, J = 12.8, 5.1 Hz, 2 H, exo H) 1.37 (s, 2 H, bridge), 1.25 (dd, J = 12.8, 5.5 Hz, 2 H, endo H); $^{13}C\{^1H\}$ NMR ($CDCl_3$), assignments aided by INEPT experiment, δ 139.1 (d, J_{CP} = 12 Hz, ipso), 133.0, 128.3 (ortho and meta), 125.1 (para), 42.1 (d, J_{CP} = 9 Hz, *C*H bridgehead), 41.5 (s, CH_2 bridge), 37.5 (d, J_{CP} = 13 Hz, *C*H β to P), 31.0 (d, J_{CP} = 13 Hz, CH_2P), 29.6 (d, J_{CP} = 7 Hz, C3 and C6); $^{31}P\{^1H\}$ NMR ($CDCl_3$) δ −17.81 (s); HRMS Calcd for $C_{33}H_{34}P_2$ 492.2136, found 492.2193.

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Ozonolysis of Substituted Uracils

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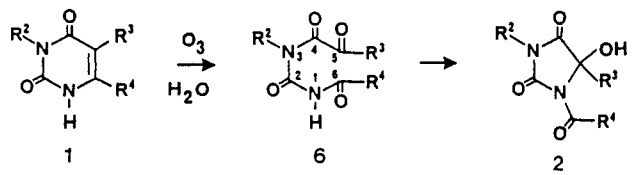
The reactions of cellular substances with ozone is one of the most important subjects in ozone chemistry. Although the ozonization reactions of DNA and RNA are of interest in connection with damage to biological systems, these reactions have received only limited attention.[1,2] With pyrimidine nucleotides, ozone preferentially attacks the base moieties.[3] Recently, the ozonolysis of uracils to give 1-acyl-5-hydroxyhydantoins has been reported.[4] Since some hydantoins have been used as anticonvulsants, their synthesis is also of interest for medicinal applications. Hydantoins are generally prepared by heating carbonyl compounds with potassium cyanide and ammonium carbonate.[5] However, little is known concerning the synthesis of 1-acyl-5-hydroxyhydantoins.[6] In order to obtain new substituted 1-acyl-5-hydroxyhydantoins, substituent effects on the ozonolysis of uracils were examined.

Table I summarizes the results of the ozonolyses of uracils. Ozonolysis of uracil (**1a**) gave 1-formyl-5-hydroxyhydantoin (**2a**) in 29% yield. This compound has previously been obtained by the radiolysis of uracil.[6] Ozonolysis of 3-methyluracil (**1b**) gave 1-formyl-5-hydroxy-3-methylhydantoin (**2b**) in 59% yield. 5-Alkyluracils **1c,d** gave the corresponding 5-alkyl-1-formyl-5-hydroxyhydantoins **2c,d** in low yields. While that of 5-phenyluracil (**1e**) did not give the 1-acyl derivative. 5-(Trifluoromethyl)uracil (**1f**), bearing a strongly electron attracting substituent, did not readily react with ozone and gave 5-hydroxy-5-(trifluoromethyl)hydantoin (**3**) in 98% yield. The ozonolyses of 5,6-dialkyl- and 6-alkyluracils **1g–j** gave the corresponding 1-acyl-5-hydroxyhydantoins **2g–j** in 32, 50, 20, and 23% yields, respectively, while 6-(trifluoromethyl)uracil (**1k**) afforded 5-hydroxyhydantoin (**4**) in 85% yield. The ozonolyses of 6-aryluracils **1l,m** gave the corresponding 1-aroyl-5-hydroxyhydantoins **2l,m**, accompanied by the formation of *N*-aroylureas. 1-Substituted uracils **1n,o** gave small amounts of parabanic acids. 5-Halouracils **1p,q** gave parabanic acid (**5**) quantitatively.

Scheme I



1 6 2

a: $R^2=R^3=R^4=H$
b: $R^2=Me$, $R^3=R^4=H$
c: $R^2=H$, $R^3=Me$, $R^4=H$
d: $R^2=H$, $R^3=Et$, $R^4=H$
g: $R^2=H$, $R^3=Me$, $R^4=Me$
h: $R^2=H$, $R^3=H$, $R^4=Me$
i: $R^2=H$, $R^3=H$, $R^4=CH_2Cl$
j: $R^2=H$, $R^3=H$, $R^4=CH_2OMe$
l: $R^2=H$, $R^3=H$, $R^4=Ph$
m: $R^2=H$, $R^3=H$, $R^4=$ *p*-$MeOC_6H_4$

Scheme I presents a reasonable reaction path for the formation of 1-acyl-5-hydroxyhydantoins. The ozonolyses of the 5–6 bond of uracils **1** gives the key intermediates **6**, followed by intramolecular cyclization to afford 1-acyl-5-hydroxyhydantoins **2**. In the ozonolyses of 5-substituted uracils, the reaction time to ozonize the substrate completely varied drastically with substituent, and increased in the following order: H < Br, F < CF_3. This reactivity order suggests an electrophilic ozone attack. The yield of **2** increased in the following order based on substituent: Ph < Et < Me < H. These results indicate that a bulky 5-substituent depresses the cyclization of **6**. The substituent at the 5-position affects the reaction time with ozone much more so than does one at the 6-position. The steric bulk of the 6-substituent does not affect the cyclization of **6**. An electron-releasing methyl group increases the electron density at the N-1 position of **6**, accelerating the cyclization. 1-Acyl-5-hydroxyhydantoins thus obtained showed no optical activity. It is well known that amides are easily hydrolyzed to give amines. Among simple amides, hydrolysis stability is substituent dependent, increasing in the order HCO < Ac < Bz. The lability of the haloacetyl derivatives to mild acid hydrolysis increases in the order Ac < $ClCH_2CO$ < Cl_2CHCO < Cl_3CCO < F_3C-CO.[7] Since a small amount of water is contained in acetic acid, it is likely that amides are easily hydrolyzed to give amines during the ozonolysis reaction. Other hydrolysis processes of N-1–C-2, C-2–N-3, and N-3–C-4 bonds of the key intermediates **6** to form urea derivatives also compete with the cyclization process.

In the ozonolyses of 1-methyluracils **1n,o**, parabanic acids can be produced by the mechanism proposed in the literature.[2]

Scheme II shows a probable mechanism in the ozonolyses of 5-halouracils **1p,q**. Key intermediates **6** can be converted into parabanic acid (**5**) by way of three processes: (a) a hydrolysis of the N-1–C-6 bond of **6** to give **7**, which in turn undergoes an intramolecular cyclization to give **8**, followed by the elimination of hydrogen halide to afford

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