Photochemistry of cyclobutanones with nitrogen acids. A potential nucleoside synthesis

IVY E. E. HAYES, LINDA JANDRISITS, AND EDWARD LEE-RUFF[1]
Department of Chemistry, York University, Toronto, Ont., Canada M3J 1P3

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Irradiation of 3-methylene-2,2,4,4-tetramethylcyclobutanone with nitrogen acids such as succinimide, phthalimide, imidazole, 3,5-dimethylpyrazole, purine, and benzimidazole gives 1:1 adducts that were identified as cyclic α-aminoacetals structurally related to nucleosides.

Key words: photochemistry of cyclobutanones, oxacarbene, nucleoside analogs.

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L'irradiation de la 3-méthylène-2,2,4,4-tétraméthylcyclobutanone en présence d'acides azotés comme la succinimide, la phtalimide, l'imidazole, la 3,5-diméthylpyrazole, la purine et la benzimidazole fournit des adduits 1:1 que l'on a identifiés comme étant des α-aminoacétals cycliques reliés d'un point de vue structural à des nucléosides.

Mots clés : photochimie de cyclobutanones, oxacarbène, analogues nucléoside.

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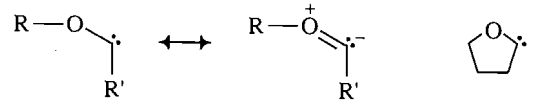
The strain associated with small ring systems promotes reactivity that contrasts that of larger ring strain-free molecules. Cyclobutanones undergo a variety of regio and stereoselective ring expansion reactions useful in the preparation of medicinally important compounds (1). Photochemical ring expansion to furanyl derivatives has also been reported to occur stereoselectively (2).

The photochemistry of cyclobutanones proceeds by three competitive pathways, decarbonylation, cycloelimination, and ring expansion to an oxacarbene **3** (3). The distribution of the photoproducts is largely dependent on the nature of the ring substituent and recently we have shown that it is also dependent on solvent effects (4). To date, the cyclic oxacarbene has been trapped by intra or intermolecular reactions with OH functional groups (5, 6).[3] In one case of a related cyclic oxacarbene generated photochemically, a small amount of product derived from olefin insertion was observed (8). Recent studies by Kesselmayer and Sheridan (9) on acyclic oxacarbenes indicate that these can be represented by zwitterionic resonance contributors such as **2** to account for their somewhat unusual reactivity when compared to dialkylmethylenes. The nucleophilic character of such oxacarbenes can readily account for the commonly observed OH insertion products and the lack of olefin insertion, in contrast to the behavior of dialkylmethylenes. We were interested whether oxacarbenes **3** generated from cyclobutanones would be sufficiently basic to insert into N—H bonds. Such a reaction would be of synthetic use in the direct preparation of α-aminofuranosides related to nucleosides. Pirrung and DeAmicis recently reported that N—H insertion of selected acidic amines occurs with an oxacarbene generated from a cyclobutanone (10). Preliminary investigation with cyclobutanone and ketone **4** showed that mixtures with aniline, diethylamine, and cyclohexylamine were photostable under uv irradiation based on gc and ^{1}H nmr analysis of reaction mixtures.[4] On the other hand, more acidic nitrogen acids with pK_a values comparable to, or smaller than, those of alcohols form 1:1 adducts with **4** under uv irradiation. Ketone **4** was chosen since ring expansion to oxacarbene **5** represents the dominant pathway in its photoreaction with alcohols (3). Both photodecarbonylation and cycloelimination are insignificant for **4**.



1 **2** **3**

SCHEME 1

Results and discussion

Mixtures of **4** (0.01–0.20 M) and the nitrogen acid (succinimide, phthalimide, imidazole, 3,5-dimethylpyrazole, purine, and benzimidazole) in dry tetrahydrofuran, dichloromethane, or acetonitrile were irradiated using a high pressure mercury lamp for 17.5–78 h. In most cases a single product was obtained and identified as the 1:1 photoadduct(s) **6***a–f* by spectroscopic and analytical data. The proton nmr spectra of **6** were characteristic in the separation of all four methyl protons, the observation of an acetal proton (δ 5.5–6.2 ppm), an almost equivalent AB pattern for the methylene protons (4.8–5.0 ppm), and signals associated with the nitrogen acid. In some cases, when the solvent was not rigorously dried, a 2:1 adduct of ketone **4** with water was obtained and identified as the bis-acetal **7**. This product most likely arises from the photoreaction of the water adduct **6** (X = OH) with oxacarbene **5**. Confirmation of this was obtained by photolysis of **5** in a solution containing 1 equivalent of **6** (X = OH) and the observation of **7** as a major product. Further confirmation of **6***a* was obtained from its acid methanolysis using saturated HCl solutions to give acetal **6** (X = OCH_3), identical in all respects to the photoadduct obtained when **4** was irradiated in methanol solution.

The absence of photocarbene insertion into N—H bonds of simple amines suggests that the basicity of the carbene may be a factor in such reactions. Although C—H insertion by aryl and alkyl carbenes is a common reaction (11), these proceed by

[1]Author to whom correspondence may be addressed.

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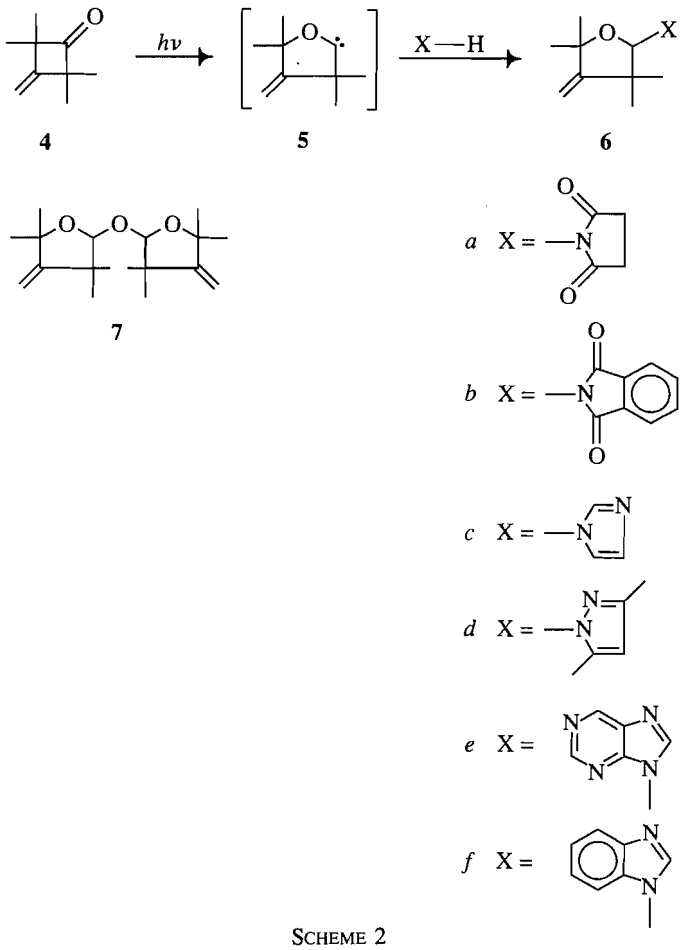
[3]There are some examples of 1,2-hydrogen shifts leading to enol ethers. See, for example, ref. 7.

[4]Solutions containing 0.1 g of ketone **4** in 25 mL of CH_2Cl_2 with 5 equivalents of amine were irradiated for 17–20 h. Unreacted cyclobutanone and amine were detected by gc and nmr. Some photodecomposition of **4** to volatile products occurred (presumably 1,1,2,2-tetramethyl-3-methylenecyclopropane and 1,1-dimethyl-2-isopropylidenecyclopropane (19)) but these could not be isolated. However, no reaction products with the amines could be detected.



SCHEME 2

mechanisms not involving proton transfer. Evidence for proton transfer reactions to aryl carbenes has been obtained for the O—H insertions of these species at temperatures slightly below ambient (12). Although oxacarbenes are considered to be nucleophilic carbenes relative to alkyl and aryl carbenes, the proton affinities of methoxylmethylene are about 10 kcal/mol lower than those of methylmethylene (13). This would suggest that if the proton transfer mechanism is involved in the formation of X—H insertion photoproducts, then the X—H acidity would have to be a contributing factor in the efficiency of this reaction. An alternate mechanism for X—H insertion involving electrophilic attack by the carbene at X and formation of an ylide (14) can be ruled out in our case since the more nucleophilic aniline, diethylamine, and cyclohexylamine did not undergo photoreaction with **4**. Although it has been reported that simple amines undergo N—H insertions with a photocarbene generated from a cyclobutanone (10), these photoadducts were not unambiguously characterized and could have been misinterpreted, with their actual structures being the hemiacetal produced from cyclobutanone photolysis with adventitious water present. The nmr data and gc retention times of the amine adducts and the hemiacetal are quite similar.

The possibility of using chiral, stereospecifically substituted cyclobutanones (15) with appropriate substituents in such a photoreaction in the preparation of nucleoside analogues is under investigation. It is known that photolysis of unsymmetric cyclobutanones proceeds in a stereoselective manner (2). The question of stereochemistry at the anomeric center in unsymmetrically substituted furanosides will be investigated.

Experimental

Melting points (mp) were determined on a Reichert melting point apparatus and were uncorrected. Infrared (ir) spectra were recorded on Pye Unicam SP-1000 and SP3-200 spectrophotometers as thin films or KBr pellets. Nuclear magnetic resonance (nmr) spectra were recorded on a Bruker AM-300 (300 MHz) spectrometer using samples dissolved in $CDCl_3$ containing 1% Me_4Si as internal standard. All nmr values are reported as chemical shift δ in ppm downfield from Me_4Si. Mass spectra were recorded on a VG Micromass 16F spectrometer. High resolution mass spectrometry was performed at the McMaster Regional Centre for Mass Spectrometry using a VG ZAB-E instrument in the EI mode at 70 eV. Elemental analyses were performed by Guelph Chemical Laboratories Limited. Photolyses were carried out using a Hanovia 450-W high pressure mercury arc lamp in a water cooled quartz immersion well with a Pyrex filter. The quartz sample tube was strapped around this well and immersed in an ice–water bath. Reaction mixtures were degassed with N_2 for 1 h prior to photolyses. Column chromatography utilized Terochem silica gel (flash chromatography type, 20–45 μm) or Merck silica gel 60 (70–270 mesh). Analytical thin-layer chromatography (tlc) was conducted on precoated plastic sheets (silica gel 60 F_{254}, layer thickness 0.2 mm) manufactured by E. Merck and Co. The sheets were visualized by examination with ultraviolet light (254 nm) or by dipping with a phosphomolybdic acid solution (20% in ethanol) followed by heating the sheet to 75°C. Anhydrous ether was obtained from Fisher Scientific Ltd. Reagent grade tetrahydrofuran (Fisher) was dried over NaOH and distilled from Na before use. Gold label acetonitrile was distilled from CaH_2 and stored over molecular sieves. Glass distilled hexane and dichloromethane were obtained from BDH Chemicals and used without distillation. Nitrogen acids were obtained from Aldrich Chemical Co., Inc. and not further purified unless otherwise stated.

3-Methylene-2,2,4,4-tetramethylcyclobutanone **4**

This ketone was prepared by a modification of the method of Hamon (16) and Lee-Ruff (17). The monophenylimine of 2,2,4,4-tetramethylcyclobutane-1,3-dione (24.2 g) (18) in 121 mL ether was added in one lot to a stirred suspension of dry triphenylphosphonium methyl iodide (50.9 g) and potassium *tert*-butoxide (14.5 g) in ether (606 mL) under an atmosphere of nitrogen. The canary-yellow coloured mixture was stirred overnight. After filtration of the triphenylphosphine oxide, the filtrate was diluted with water and extracted with ether. The ether layer was then dried over anhydrous magnesium sulfate and the ether distilled using a short Vigreux column. The crude imine of **4** (16.1 g) was stirred and refluxed with 50% acetic acid (100 mL). A solid collected in the reflux condenser in a few minutes. This was washed back into the solution and refluxing continued for 1 h. After cooling, the excess acetic acid was destroyed by addition of solid bicarbonate. The mixture was then extracted with ether and the ether layer was washed with water until neutral, dried over anhydrous magnesium sulfate, and distilled through a short column to give an oil. The oil mixture was sublimed at room temperature at 25 Torr (1 Torr = 133.3 Pa) pressure to give a crystalline material **4**, mp 43–44°C (lit. (16) mp 42–43°C) (6.3 g, 40%; 2 steps).

2-Succinimidyl-3,5-tetramethyl-4-methylene tetrahydrofuran **6**a

A solution of 0.25 g (1.81 mmol) of ketone **4** and 0.90 g (9.08 mmol) of succinimide (recrystallized from EtOH) in 150 mL of CH_2Cl_2 was irradiated for 25.25 h. Removal of the CH_2Cl_2 by rotary evaporation and flash chromatography on silica gel (10% tetrahydrofuran/hexane) gave 0.11 g (26%) of **6***a* (recrystallized from pentane); mp 48–49°C; ir (KBr), cm^{-1}: 3090 (=C—H), 2980 (CH), 1790, 1710 (C=O), 1090 (C—O); ^1H nmr δ: 5.68 (s, acetal H), 4.84 (AB *exo* CH_2), 2.67 (s, CH_2 of succinimidyl), 1.51, 1.38, 1.31, 1.10 (s, methyl H's); ^{13}C nmr δ: 176.1 (CO), 163.1 (quat. C of C=C), 101.6 (*exo* CH_2), 88.9 (acetal C), 83.8 (quat. C α to O), 45.7 (quat. C), 30.5, 30.2, 27.8, 23.3 (CH_3's), 27.5 (CH_2 of succinimidyl); ms, *m/e*: 237 (M^+), 222 ($M^+ - C_{12}H_{16}NO_3$), 139 ($M^+ - C_4H_4NO_2$), 123 ($M^+ - C_9H_{15}$). *Anal.* calcd. for $C_{13}H_{19}NO_3$: C 65.80, H 8.07, N 5.90; found: C 65.76, H 8.04, N 5.68.

2-Phthalimidyl-3,5-tetramethyl-4-methylene tetrahydrofuran **6**b

A solution of 0.50 g (3.62 mmol) of ketone **4** and 0.48 g (3.26 mmol) of phthalimide in 20 mL of tetrahydrofuran was irradiated for 62 h. Solvent evaporation by rotary evaporation and column chromatography (10% tetrahydrofuran/hexane) gave 0.13 g (14%) of **6***b* (recrystallized from hexane); mp 114–115°C; ir (thin film), cm^{-1}: 3070 (=C—H), 2970 (C—H), 1775, 1720 (C=O), 1610 (C=C), 1050 (C—O); ^1H nmr δ: 7.65 (dd, J = 5.5 Hz, 3.0 Hz, CH of phthalimidyl), 7.77 (dd, J = 5.5 Hz, 3.0 Hz, CH of phthalimidyl), 5.84 (s, acetal H), 4.91 (AB *exo* CH_2), 1.57, 1.42, 1.36, 1.17 (s, methyl H's); ^{13}C nmr δ: 168.0 (CO), 160.1 (quat. C of C=C), 134.3 (phthalimide C-5, C-6), 132.5 (phthalimide C-3a, C-7a), 123.5 (phthalimide C-4, C-7), 105.7 (*exo* CH_2), 89.1 (acetal C), 85.2 (quat. C α to O), 43.3 (quat. C), 29.8, 29.6, 29.5, 29.4 (CH_3's); *m/e*: 286 $(M + H)^+$, 139 $(M^+ - C_8H_4NO_2)$; high resolution ms, *m/e* calcd.: 286.1444; found: 286.1438. *Anal.* calcd. for $C_{17}H_{19}NO_3$: C 71.56, H 6.71, N 4.91; found: C 71.29, H 7.14, N 4.46.

2-Imidazolyl-3,5-tetramethyl-4-methylene tetrahydrofuran **6**c

A solution of 0.23 g (1.66 mmol) of ketone **4** and 0.066 g (0.97 mmol) of imidazole (recrystallized from benzene) in 10 mL of tetrahydrofuran was irradiated for 26.5 h. Solvent removal by rotary evaporation, followed by two flash columns (15% tetrahydrofuran/hexane) gave 0.19 g (95%) of white crystalline **6***c* (recrystallized from hexane); mp 64.5–65.5°C; ir (KBr), cm^{-1}: 3120 (=C—H), 2990 (C—H), 1805 ($R_2C{=}CH_2$), 1520, 1490, 1465, 1378 (azole), 1080 (C—O); ^1H nmr δ: 7.66 (s, H of C-2 of imidazole), (7.04, 7.07) (s, H of C-4 and C-5 of imidazole), 5.48 (s, acetal H), 4.94 (AB *exo* CH_2), 1.53, 1.39, 1.22, 0.80 (s, methyl H's); ^{13}C nmr δ: 162.8 (quat. C of C=C), 135.7 (imidazole C-2), (129.0, 117.1) (imidazole C-4, C-5), 104.5 (*exo* CH_2), 93.1 (acetal C), 82.4 (quat. C α to O), 46.7 (quat. C), 29.9, 28.7, 24.9, 24.1 (CH_3's); ms, *m/e*: 206 (M^+), 139 $(M^+ - C_3H_3N_2)$. *Anal.* calcd. for $C_{12}H_{18}N_2O$: C 69.87, H 8.79, N 13.58; found: C 70.22, H 8.93, N 13.76.

3,5-Dimethylpyrazole adduct **6**d

A solution of 0.50 g (3.62 mmol) of ketone **4** and 1.74 g (18.1 mmol) of 3,5-dimethylpyrazole (recrystallized from EtOH) in 30 mL of CH_2Cl_2 was irradiated for 20.5 h. Removal of the CH_2Cl_2 by rotary evaporation followed by flash column chromatography (10% tetrahydrofuran/hexane) yielded 0.36 g (43%) of a colourless oil **6***d*; ir (thin film), cm^{-1}: 3070 (=C—H), 2960 (C—H), 1655 (C=C), 1555, 1465, 1365 (azole), 1065 (C—O); ^1H nmr δ: 5.73 (s, pyrazole H-4), 5.58 (s, acetal H), 4.85 (AB *exo* CH_2), (2.28, 2.16) (s, pyrazole CH_3's), 1.61, 1.41, 1.32, 0.89 (s, methyl H's); ^{13}C nmr δ: 163.8 (quat. C of C=C), (147, 139.5) (C-3 and C-5 of pyrazole), 105.5 (C-4 of pyrazole), 102.3 (*exo* CH_2), 94.0 (acetal C), 83.8 (quat. C α to O), 47.5 (quat. C), (30.3, 29.3) (pyrazole CH_3's), 29.2, 22.9, 13.5, 11.6 (CH_3's); ms, *m/e*: 234 (M^+), 139 $(M^+ - C_5H_7N_2)$; high resolution ms, *m/e* calcd.: 234.1734; found: 234.1742.

Purine adduct **6**e

A solution of 1.05 g (7.60 mmol) of ketone **4** and 0.52 g (4.33 mmol) of purine in 500 mL of acetonitrile was irradiated for 78 h. Solvent removal by rotary evaporation and column chromatography (20% tetrahydrofuran/hexane) gave 0.35 g (31%) of a yellow oil **6***e*; ir (thin film), cm^{-1}: 2960 (C—H), 1660 (C=C), 1593, 1580, 1495, 1410, 1370 (purine), 1060 (C—O); ^1H nmr δ: 9.16 (s, H-6 of purine), 8.98 (s, H-2 of purine), 8.37 (s, H-8 of purine), 6.23 (s, acetal H), 5.00 (AB *exo* CH_2), 1.59, 1.46, 1.39, 0.88 (s, methyl H's); ^{13}C nmr δ: 162.5 (quat. C of C=C), 152.3 (C-2 of purine), 151.0 (C-4 of purine), 148.3 (C-8 of purine), 143.6 (C-6 of purine), 128.0 (C-5 of purine), 104.6 (*exo* CH_2), 90.0 (acetal C), 82.9 (quat. C α to O), 46.7 (quat. C), 29.6, 28.4, 25.1, 24.4 (CH_3's); ms, *m/e*: 258 (M^+), 139 $(M^+ - C_5H_3N_4)$; high resolution ms, *m/e* calcd.: 258.1482; found: 258.1493.

2-Benzimidazolyl-3,5-tetramethyl-4-methylene tetrahydrofuran **6**f

A solution of 0.56 g (4.05 mmol) of ketone **1** and 0.46 g (3.89 mmol) of benzimidazole in 45 mL of tetrahydrofuran was irradiated for 17.5 h. Solvent removal by rotary evaporation and flash column chromatography gave 0.93 g (93%) of crystalline **6***f* (recrystallized from hexane); mp 138–139°C; ir (KBr), cm^{-1}: 3060 (=C—H), 2980 (C—H), 1662 (C=C), 1615, 1490, 1468, 1370, 1320 (azole), 1060 (C—O); ^1H nmr δ: 8.18 (s, H-2 of benzimidazole), (7.45, 7.82) (m, H-7, H-4 of benzimidazole), 7.28 (m, H-5, H-6 of benzimidazole), 5.89 (s, acetal H), 4.98 (AB *exo* CH_2), 1.60, 1.45, 1.32, 0.87 (s, methyl H's); ^{13}C nmr δ: 163.0 (quat. C of C=C), (143.6, 133.2) (quat. C's of benzimidazole), 141.3 (benzimidazole C-2), (123.0, 122.3) (benzimidazole C-5, C-6), (120.3, 110.5) (benzimidazole C-4, C-7), 104.5 (*exo* CH_2), 91.7 (acetal C), 82.4 (quat. C α to O), 47.2 (quat. C), 29.8, 28.4, 25.3, 24.5 (CH_3's); ms, *m/e*: 256 (M^+), 139 $(M^+ - C_7H_5N_2)$. *Anal.* calcd. for $C_{16}H_{20}N_2O$: C 74.97, H 7.86, N 10.93; found: C 74.61, H 7.92, N 11.10.

*3,3,5,5-Tetramethyl-4-methylene-2-methoxytetrahydrofuran (***6***) (X = OCH_3)*

A solution of 0.64 g (4.63 mmol) of ketone **4** in 20 mL of methanol was irradiated for 24 h. Solvent evaporation and column chromatography (5% tetrahydrofuran/hexane) gave **6** (X = OCH_3), whose ^1H nmr spectrum was similar to the literature (19): ^1H nmr δ: 4.82 (s, 1), 4.78 (s, 1), 4.54 (s, 1), 3.34 (s, 3), 1.37 (s, 6), 1.14 (s, 3), 1.11 (s, 3).

A solution of 0.28 g (2.03 mmol) of **6***a* in 2 mL of a saturated HCl/MeOH solution was stirred under N_2 for 1 h. The product obtained was identical to **6** (X = OCH_3).

*3,3,5,5-Tetramethyl-4-methylene-2-hydroxytetrahydrofuran (***6***) (X = OH)*

A solution of 0.50 g (3.63 mmol) of ketone **4** in 20 mL of tetrahydrofuran and 1 mL of water was irradiated for 12 h. Solvent evaporation was followed by addition of ether (50 mL) to the crude oil. The ether fraction was washed with water (2 × 50 mL), dried with magnesium sulfate, and concentrated by rotary evaporation to give 0.33 g (58%) of a yellow oil; ir (thin film), cm^{-1}: 3390 (OH), 2970 (C—H), 1665 (C=C), 1380, 1365 (*gem* dimethyls), 1080, 1035 (C—O); ^1H nmr δ: 5.06 (s, acetal H), 4.83, 4.78 (AB *exo* CH_2), 4.20 (bs, OH), 1.42, 1.32, 1.11, 1.10 (s, methyl H's); ^{13}C nmr δ: 164.0 (quat. C of C=C), 103.9 (acetal C), 102.8 (*exo* CH_2), 83.3 (quat. C α to O), 47.2 (quat. C), 31.6, 30.5, 28.1, 21.5 (CH_3's); ms, *m/e*: 139 $(M^+ - OH)$.

Bis-acetal **7**

A solution of 0.33 g (2.11 mmol) of the crude alcohol **6** (X = OH) and 0.24 g (1.74 mmol) of ketone **4** in 20 mL of tetrahydrofuran was irradiated for 7 h. Solvent evaporation and column chromatography (5% ethyl acetate/hexane) gave 0.12 g (24%) of a colourless oil **7**; ir (thin film), cm^{-1}: 2960 (C—H), 1375, 1360 (*gem* dimethyls), 1060 (C—O); ^1H nmr δ: 4.87 (s, acetal H), 4.79, 4.76 (AB *exo* CH_2), 1.41, 1.36, 1.14, 1.10 (s, methyl H's); ^{13}C nmr δ: 164.6 (quat. C of C=C), 108.9 (acetal C), 101.8 (*exo* CH_2), 83.8 (quat. C α to O), 47.7 (quat. C), 31.1, 30.6, 29.2, 22.1 (CH_3's); ms, *m/e*: 294 $(M^+$, 8%), 279 $(M^+ - CH_3$, 18%), 139 $(M^+ - C_9H_{15}O$, 100%).

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