Experimental Section

1,3-Dihydroxy-2-acetylxanthone.[1]—Freshly fused $ZnCl_2$ (4 g) was dissolved in acetic acid (8 ml) by heating. Acetic anhydride (4 ml) and 1,3-dihydroxyxanthone (4 g) were added. The reaction mixture was heated at 145–150° for 1–5 hr, cooled, and poured into ice–water. Solid gradually separated out and was filtered and washed with water. The crude product was sublimed at 240–250° (8 mm). Crystallization from alcohol/acetic acid yielded **1** as pale yellow needles, mp 208–209° (1.4 g). It gave a blood red color with ethanolic ferric chloride.

Anal. Calcd for $C_{15}H_{10}O_5$: C, 66.67; H, 3.70. Found: C, 66.34; H, 3.98.

Its 2,4-dinitrophenylhydrazone formed tiny orange needles, mp 297° (acetic acid).

Anal. Calcd for $C_{21}H_{14}O_8N_4$: N, 12.45. Found: N, 12.24.

A mixture of salicylic acid (2 g), phloroacetophenone (3.5 g), freshly fused $ZnCl_2$ (6 g), and $POCl_3$ (20 ml) were heated at 70–80° for 2 hr. The reaction product was cooled and poured into ice–water. The yellow solid that separated was filtered and washed with 10% $NaHCO_3$ and water. The crude product was sublimed at 248–250° (8 mm). Crystallization from ethanol/acetic acid yielded **1** as pale yellow needles, mp and mmp (with the above sample) 208–209°, yield 0.8 g.

Anal. Calcd for $C_{15}H_{10}O_5$: C, 66.67; H, 3.70. Found: C, 66.43; H, 3.88.

1,2,3-Trihydroxyanthone (2a).—**1** (1.35 g) was dissolved in 10 ml of 4% NaOH, 10 ml of pyridine was added, and the mixture was cooled in an ice bath. Hydrogen peroxide (12 ml, 20 vol) was added dropwise with shaking during 5 min. The reaction mixture was left for 1 hr. Acidification yielded **2a** as a yellow solid. It crystallized from alcohol as yellow needles, mp 265°, yield 0.75 g. The ethanolic solution gave a dark green color with ferric chloride solution.

Anal. Calcd for $C_{13}H_8O_5$: C, 63.93; H, 3.28. Found: C, 64.17; H, 3.54.

1,2,3-Trimethoxyxanthone (2b).—**2a** (0.2 g) was refluxed with Me_2SO_4 (0.6 g) and anhydrous K_2CO_3 (2 g) for 10 hr. Potassium salts were filtered off and the filtrate after removal of solvent furnished **2b** as a colorless solid. It crystallized from alcohol as needles, mp 191°. It gave no color with $FeCl_3$ solution.

Anal. Calcd for $C_{16}H_{14}O_5$: C, 67.13; H, 4.89. Found: C, 66.97; H, 4.63.

1,3,4-Trimethoxyxanthone.—1,4-Dihydroxy-3-methoxyxanthone[4] (0.2 g) in anhydrous acetone (100 ml) was treated with anhydrous potassium carbonate (2.0 g) and dimethyl sulfate (2.5 ml), and the mixture was refluxed for 54 hr. The potassium salts were filtered and the solvent was removed. The residue on crystallization from alcohol gave 1,3,4-trimethoxyxanthone as needles, mp 164°, yield 0.12 g. It gave no color reaction with ethanolic ferric chloride.

Anal. Calcd for $C_{16}H_{14}O_5$: C, 67.1; H, 4.9. Found: C, 67.4; H, 5.0.

1,2,3-Triacetoxyxanthone (2d).—**2a** (0.1 g) with acetic anhydride (5 ml) and pyridine (a drop) gave **2d** as colorless shining cubes, mp 213° (alcohol), $FeCl_3$ test negative.

Anal. Calcd for $C_{19}H_{14}O_8$: C, 61.62; H, 3.78. Found: C, 61.88; H, 3.95.

1-Hydroxy-2,3-diacetoxyxanthone (2c).—**2a** (1.0 g) was refluxed with boron triacetate (1.5 g) and acetic anhydride (8 ml) for 10 min. The yellow diacetoborate that separated on cooling was filtered and washed with anhydrous ether. Subsequently it was suspended in water (50 ml) and heated to boiling when it decomposed. **2c** thus obtained crystallized from alcohol as pale yellow needles, mp 203–204°, yield 0.8 g. It gave a dark brown ferric chloride test.

Anal. Calcd for $C_{17}H_{12}O_7$: C, 62.19; H, 3.66. Found: C, 62.55; H, 3.91.

1-Methoxy-2,3-diacetoxyxanthone (2e).—**2c** (0.5 g) in acetone (100 ml) was refluxed with methyl iodide (2 ml) and active Ag_2O (1 g) for 20 hr. The reaction product was filtered and the filtrate on removal of solvent yielded **2e** which crystallized from alcohol as colorless needles, mp 111°, yield 0.45 g. It gave a negative ferric chloride test.

Anal. Calcd for $C_{18}H_{14}O_7$: C, 63.15; H, 4.09. Found: C, 63.37; H, 3.90.

1-Methoxy-2,3-dihydroxyxanthone (2f).—**2e** (0.25 g) was refluxed with alcoholic potash (5%, 10 ml) for 1 hr. Subsequent acidification yielded **2f** which crystallized from alcohol as colorless pale yellow needles, mp 176°. With $FeCl_3$ it gave a green color immediately changing to reddish brown.

Anal. Calcd for $C_{14}H_{10}O_5$: C, 65.11; H, 3.88. Found: C, 65.58; H, 3.67.

Ethyl 1-Hydroxy-2-acetyl-9-oxo-3-xanthyloxyacetate (2g).—**1** (1.35 g) in acetone (300 ml) was refluxed with ethyl bromoacetate (0.85 g) and anhydrous K_2CO_3 (6 g) for 10 hr. The potassium salts were filtered off and the solvent was removed from the filtrate. As no residue was obtained, the potassium salts were suspended in water and decomposed with dilute HCl. The solid that separated was filtered and washed with water. **2g** thus obtained crystallized from alcohol/acetic acid as colorless plates, mp 210°. It gave a reddish brown color with $FeCl_3$, yield 0.9 g.

Anal. Calcd for $C_{19}H_{16}O_7$: C, 64.04; H, 4.49. Found: C, 64.22; H, 4.63.

1-Hydroxy-2-acetyl-9-oxo-3-xanthyloxyacetic Acid (2h).—**2g** (0.75 g) in acetone (300 ml) was refluxed with aqueous Na_2CO_3 (60 ml, 5%) for 3 hr. Removal of acetone and acidification yielded **2h** which crystallized from acetic acid as colorless plates, mp 253°. It gave a reddish brown color with $FeCl_3$, yield 0.6 g.

Anal. Calcd for $C_{17}H_{12}O_7$: C, 62.19; H, 3.66. Found: C, 62.51; H, 3.84.

1-Acetoxy-3-methylfurano[4,5-*b*]xanthone (3a).—**2h** (0.55 g) was refluxed with NaAc (0.6 g) and Ac_2O (6 ml) for 2 hr. Subsequent work-up gave **3a** which crystallized from aqueous alcohol as pale yellow needles, mp 173–174°, yield 0.45 g. It gave a negative $FeCl_3$ test.

Anal. Calcd for $C_{18}H_{12}O_5$: C, 70.13; H, 3.89. Found: C, 70.59; H, 4.01.

1-Hydroxy-3-methylfurano[2,3-*b*]xanthone (3b).—**3a** (0.4 g) was refluxed with alcoholic potash (5%, 15 ml) for 1 hr. Subsequent acidification gave **3b** which crystallized from alcohol as yellow needles, mp 232°, yield 0.3 g. It gave a green color with $FeCl_3$.

Anal. Calcd for $C_{16}H_{10}O_4$: C, 72.18; H, 3.76. Found: C, 72.66; H, 3.81.

Registry No.—**1**, 27460-08-8; **1** 2,4-DNP, 27460-09-9; **2a**, 27519-51-3; **2b**, 27460-10-2; **2c**, 27460-11-3; **2d**, 27460-12-4; **2e**, 27460-13-5; **2f**, 20362-26-9; **2g**, 27460-14-6; **2h**, 27460-15-7; **3a**, 27460-16-8; **3b**, 27460-17-9; 1,3,4-trimethoxyxanthone, 27460-18-0.

(4) V. V. Kane, A. B. Kulkarni, and R. C. Shah, *J. Sci. Ind. Res., Sect. B*, **18**, 75 (1959).

The Cyclization of *cis*- and *trans*-2-(2-Methoxycyclohexyl)ethanol to *cis*- and *trans*-Perhydrobenzofurans[1]

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We have reported[2] that compounds of type **1** undergo cyclization with tosyl chloride–pyridine to form perhydrobenzofurans **2**, with loss of a methoxyl group. This reaction, which we first observed in degradations of the antibiotic fumagillin,[3] involves a methoxonium ion intermediate.[4]

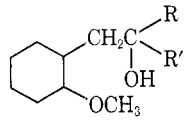
(1) Aided by Grant 2252-C from the Petroleum Research Fund of the American Chemical Society, for which we express our appreciation.

(2) S. E. Cantor and D. S. Tarbell, *J. Amer. Chem. Soc.*, **86**, 2902 (1964).

(3) D. D. Chapman, S. E. Cremer, R. M. Carman, M. Kunstmann, J. G. McNally, A. Rosowsky, and D. S. Tarbell, *ibid.*, **82**, 1009 (1960); D. S. Tarbell, *et al.*, *ibid.*, **83**, 3096 (1961).

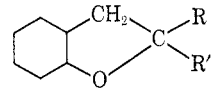
(4) S. Winstein, E. Allred, R. Heck, and G. Glick, *Tetrahdron*, **3**, 1 (1958); E. Allred and S. Winstein, *J. Amer. Chem. Soc.*, **89**, 3991, 3998, 4008, 4012 (1967); E. R. Novak and D. S. Tarbell, *ibid.*, **89**, 73 (1967); A. W. Friederang and D. S. Tarbell, *J. Org. Chem.*, **33**, 3797 (1968); J. R. Hazen and D. S. Tarbell, *Tetrahedron Lett.*, 5927 (1968); J. R. Hazen, *ibid.*, 1897 (1969); R. J. Gargiulo and D. S. Tarbell, *Proc. Nat. Acad. Sci. U. S.*, **62**, 52 (1969).

Further examination of this reaction has shown that the reported[2] results require amplification and correction, for the cis and trans primary alcohols **3** and **4**.



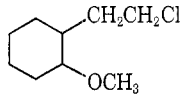
1

3, R = R′ = H, *cis*
4, R = R′ = H, *trans*

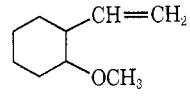


2

5, R = R′ = H, *cis*
9, R = R′ = H, *trans*



6, *cis*
7, *trans*



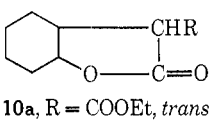
8, *trans*

The fact that the cyclization of **1** to **2** does occur with the secondary alcohol (**1**, R = CH_3; R′ = H) and the tertiary alcohol (**1**, R = R′ = CH_3), giving the corresponding 2-substituted perhydrobenzofuran **2**, is indicated by additional work,[5] as well as by the original observations.[2]

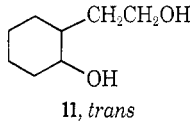
Treatment of the cis alcohol **3** with tosyl chloride–pyridine yielded 3% of *cis*-perhydrobenzofuran (**5**), identified by vpc and by its correspondence in spectral characteristics with a sample prepared by a different method.[5b] The other product, in addition to starting material, was the chloro compound **6**, identified by analysis and nmr spectrum.

The trans alcohol **4** with tosyl chloride–pyridine under several sets of conditions yielded the corresponding *trans*-chloro compound **7** and the unsaturated compound **8**. Distillation of the *trans*-chloro compound **7** or passage through a vpc column at 200° did yield a small amount of the *trans*-perhydrobenzofuran (**9**), but it is clear that no detectable amount of **9** is formed by the tosyl chloride–pyridine treatment itself. Previously,[2] the reaction product from the trans alcohol **4** was distilled at atmospheric pressure which may have formed some of the trans cyclized product **9**.

We have prepared a pure sample of **9** by treating cyclohexene oxide with malonic ester[6] which yields the *trans*-carbethoxylactone **10a**; hydrolysis and decarboxylation gives the trans lactone[6] **10b** which is converted by lithium aluminum hydride reduction to the trans diol **11**. Treatment of this diol with tosyl chloride–pyridine yielded *trans*-perhydrobenzofuran (**9**) which was characterized and shown to be different from the cis compound **5**. The synthesis of a mixture of the cis and trans compounds **5** and **9** (by cyclization of a cis–trans mixture of the diols **11**), followed by vpc separation[7] gave **9**, with properties in reasonable agreement with those of our product. A mixture of **5** and **9** was also prepared by lead tetraacetate oxidation[7] of cyclohexylethanol. Detailed ir and nmr spectral data for **5** and **9** are given in the Experimental Section.



10a, R = COOEt, *trans*
b, R = H, *trans*



11, *trans*

(5) (a) D. P. Brust and D. S. Tarbell, *J. Org. Chem.*, **31**, 1251 (1966); (b) W. E. Harvey and D. S. Tarbell, *ibid.*, **32**, 1679 (1967).

(6) S. Coffey, *Rec. Trav. Chim. Pays-Bas*, **42**, 387 (1923). The trans ring opening of an epoxide by malonate was demonstrated by W. E. Grigsby, J. Hind, J. Chanley, and F. H. Westheimer, *J. Amer. Chem. Soc.*, **64**, 2606 (1942).

Experimental Section[8]

trans-**2-Allylcyclohexanol.**[9]—Allylmagnesium bromide was prepared[10] from 195 g of magnesium turnings in 2.4 l. of ether and 400 g of allyl bromide in an equal volume of ether. To this solution, cooled in ice bath, was added dropwise 150 g of cyclohexene oxide in ether (100 ml) over a 5-hr period. The reaction complex was hydrolyzed by the slow addition of saturated ammonium chloride solution. After decanting the organic layer and thoroughly washing the salt cake with ether, the combined solution was dried, concentrated, and distilled to give 190 g (90%) of *trans*-2-allylcyclohexanol, bp 86–88° (9 mm), $n^{25}D$ 1.4751 [reported[11] bp 94° (14 mm), $n^{25}D$ 1.4758]. Conversion of the product to the methyl ether as below and analysis by vpc showed no trace of the cis isomer. The 3,5-dinitrobenzoate melted at 69–70° after recrystallization from methanol as reported.[11] When the cyclohexene oxide was added to the Grignard at such a rate as to cause gentle refluxing, the isolated alcohol contained 3–5% of the cis isomer.

trans-**2-Allyl-1-methoxycyclohexane** was prepared in 73% yield by methylation of the above trans alcohol with NaH and methyl iodide in DMF, bp 75–75.5° (14 mm), $n^{25}D$ 1.4535.

trans-**2-(Methoxycyclohexyl)ethanol** (**4**) was prepared much as before[2] by oxidation with osmium tetroxide–periodate in aqueous THF, followed without isolation by sodium borohydride reduction of the aldehyde. The overall yield was 50%, and the product showed bp 114–115° (25 mm), $n^{26}D$ 1.4640 (reported[2] $n^{25}D$ 1.4598).

2-(2-Methoxyphenyl)ethanol was prepared in 76% yield by the above method from *o*-allylanisole.[12] Reduction of 7 g of this material with hydrogen in 30 ml of acetic acid and 3.3 g of 5% rhodium on alumina gave, after the usual work-up and distillation, the following fractions: (1) bp 50–52° (10 mm), 0.7 g; (2) bp *ca.* 117° (10 mm), 1.0 g; (3) bp 117° (10 mm), 3.5 g, $n^{25}D$ 1.4655. Examination by vpc showed that cut 1 was mainly *cis*-perhydrobenzofuran, cut 2 was a mixture of *cis*-perhydrobenzofuran (**5**) and *cis*-2-(2-methoxycyclohexyl)ethanol (**3**), and cut 3 was pure *cis*-2-(2-methoxycyclohexyl)ethanol. The retention time of *cis*-perhydrobenzofuran was identical with that of an authentic sample.

Reaction of *cis*-2-(2-Methoxycyclohexyl)ethanol (3) with Tosyl Chloride in Pyridine.—A mixture of 3.5 g of *cis*-2-(2-methoxycyclohexyl)ethanol, pure from vpc examination, 4.7 g of tosyl chloride, and 31 ml of anhydrous pyridine was stirred for 5.5 hr at 63–65°, poured onto 50 g of cracked ice, and extracted with ether. The ether layer was washed with 10% aqueous hydrochloric acid and saturated sodium chloride solution and dried. After solvent was removed, 2 g of a crude liquid was obtained. Distillation gave these cuts: (1) bp 92–100° (15 mm), 0.15 g; (2) bp 102–103° (15 mm), $n^{25}D$ 1.4651, 1.6 g (41%). Vpc analysis of cut 1 showed a mixture of *cis*-perhydrobenzofuran (**5**, about 3%) and the chloro compound **6** (1:1 ratio). The retention time of **5** corresponded to that of a known sample. Cut 2 showed

(7) S. Moon and B. H. Waxman, *J. Org. Chem.*, **34**, 288 (1969).

(8) Analyses were by Galbraith Laboratories; nmr spectra were taken on a Varian A-60 spectrometer in CCl_4 and $CDCl_3$, with $(CH_3)_4Si$ as an internal standard, and are reported in ppm (δ units); ir spectra were determined with a Beckman IR-10 spectrometer. Vpc analyses were done on an Aerograph Model A90, using 5 ft × 0.25 in. columns with 25% UCON Polar or 20% Carbowax 20M packing.

(9) Method of H. Felkin and G. Roussi, *Tetrahedron Lett.*, 4153 (1965), who give no experimental detail. This procedure gives a purer product stereochemically than the one used earlier.[2]

(10) O. Grummitt, E. P. Budewitz, and C. C. Chudd, "Organic Syntheses," Collect. Vol. IV, Wiley, New York, N. Y., 1963, p 748.

(11) J. Cologne and F. Collomb, *Bull. Soc. Chim. Fr.*, **18**, 241 (1951).

(12) R. Adams and R. E. Rindfusz, *J. Amer. Chem. Soc.*, **41**, 648 (1919).

one peak on vpc, and the nmr spectrum was identical with that of the analytical sample of the chloro compound **6** (prepared in another run).

The chloro compound **6** showed the following nmr spectrum in $CDCl_3$: 3.41 (t, J = 6 cps, CH_2Cl, 2 H), 3.3 (s, OCH_3, 3 H), 3.2–3.4 (m, OCH, 1 H), 0.8–2.3 (CH and CH_2, 11 H).

Anal. Calcd for $C_9H_{17}ClO$: C, 61.18; H, 9.63. Found: C, 61.38; H, 9.57.

cis-Perhydrobenzofuran (**5**) which was pure by vpc examination showed the following ir spectrum (cm^{-1}): 2940 (s), 2870 (s), 1445 (m), 1380 (w), 1360 (w, b), 1290 (w), 1240 (w), 1180 (w), 1155 (w), 1120 (w, b), 1080 (m), 1050 (m), 1025 (s), 1000 (w), 985 (m), 930 (w), 870 (w), 805 (w), 680 (w, b). The nmr spectrum in $CDCl_3$ showed 0.8–2.3 (b, 11 H), 3.6–4.2 (m, 3 H).

Reaction of *trans*-2-(2-Methoxycyclohexyl)ethanol (4) with Tosyl Chloride in Pyridine.—A mixture of 5 g of *trans*-2-(2-methoxycyclohexyl)ethanol, 6.3 g of tosyl chloride, and 44 ml of anhydrous pyridine was stirred for 5.5 hr at 63–65°, poured onto 70 g of cracked ice, and extracted with ether (seven 20-ml portions). The usual work-up gave 2.3 g of crude product. Distillation at 7 mm gave the following cuts: (1) bp 75–90°, 0.1 g; (2) bp 93–95°, $n^{25}D$ 1.4650, 1.7 g (30%). Vpc analysis of 1 showed a mixture of the unsaturated compound **8**, and the *trans*-chloro compound **7**. Fraction 2 showed only one peak, the chloro compound **7**; the nmr spectrum of this compound (taken in CCl_4 on a sample from a different run) showed 1.5 (m, 11 H), 2.8 (m, $CHOCH_3$), 3.22 (s, OCH_3, 3 H), and 3.45 (t, J = 6.5 cps, CH_2-CH_2Cl, 2 H).

Anal. Calcd for $C_9H_{17}ClO$: C, 61.18; H, 9.63; Cl, 20.06. Found: C, 61.31; H, 9.68; Cl, 19.76.

The structure of the unsaturated compound **8** was based on the following nmr spectrum ($CDCl_3$): 3.27 (s, OCH_3, 3 H), 3.7 (m, CHO, 1 H), 4.7–5.7 (m, vinyl H, 3 H). Ir bands appeared at 3090 and 1645 cm^{-1}.

Lactone of *trans*-(2-Hydroxycyclohexyl)acetic Acid (10b).—Coffey's procedure[7] was modified as follows. To a solution of 57 g of ethyl malonate and 9 g of sodium in 200 ml of absolute ethanol was added 33 g of cyclohexene oxide in 100 ml of absolute ethanol. The reaction mixture became semisolid after reflux for a few minutes; reflux was continued for additional 30 min. Solvent was removed under vacuum. The residual semisolid material was dissolved in 200 ml of 10% NaOH, refluxed for 3 hr, concentrated under vacuum, acidified with HCl, and extracted with $CHCl_3$. The $CHCl_3$ layer was dried, solvent was removed, and the remaining oil was heated at 170–190° for 1 hr. Evolution of gas was observed. Distillation of resulting oil gave the lactone of cyclohexanolacetic acid, bp 97–98° (2 mm), C=O band at 1785 cm^{-1}.

***trans*-2-(2-Hydroxycyclohexyl)ethanol (11).**—To a suspension of 1.8 g of lithium aluminum hydride in 50 ml of ether was added dropwise 6.1 g of the lactone **10b**; the mixture was stirred for 30 min at 0° and then for 4 hr at room temperature. Work-up in the usual way and distillation yielded 4.1 g of the diol **11** as a viscous colorless liquid, bp 104–105° (1 mm). A sample was treated with bis(trimethylsilyl)trifluoroacetamide; vpc showed a single peak. The ir (liquid film) showed bands at 3300 (b), 1450, 1070, 1055, and 1035 cm^{-1}. The nmr in $CDCl_3$ showed 0.9–2.2 (m, CH_2 and CH, 11 H), 2.9–3.4 (m, 1 H), 3.5–3.8 (m, 2 H), 4.7 (OH, 2 H).

***trans*-Perhydrobenzofuran (9).**—The trans diol **11** (3.3 g) was heated with 6.6 g of tosyl chloride in 35 ml of dry pyridine at 95–100° for 2 hr. Distillation of the product resulting from the usual work-up gave 2.4 g of the *trans*-perhydrobenzofuran, bp 72° (25 mm), $n^{25}D$ 1.4632. This material was homogeneous when examined by vpc; addition of a pure sample of *cis*-perhydrobenzofuran (**5**) showed two peaks. Molecular weight by mass spectroscopy was 126 (calcd 126). The nmr spectrum in $CDCl_3$ showed 0.8–2.3 (11 H), 3.6–4.2 (3 H). The ir in liquid film showed the following bands, clearly different from the cis compound above: 2940 (s), 2870 (s), 1455 (m), 1390 (w), 1355 (w), 1340 (w), 1308 (w), 1290 (w), 1270 (w, b), 1190 (w), 1145 (m), 1110 (w), 1065 (s), 1055 (sh), 1020 (w), 980 (s), 930 (m), 925 (sh), 915 (sh), 857 (m), 830 (w), 660 (w, b). These properties supplant those previously reported by us.[2]

Registry No.—**3**, 27345-66-0; **4**, 27345-67-1; **5**, 10198-29-5; **6**, 27384-94-7; **7**, 27345-69-3; **9**, 27345-70-6; **10b**, 27345-71-7; **11**, 27345-72-8.

Reactivities and Electronic Aspects of Nucleic Acid Heterocycles. II. Diazomethane Methylation of Uracil and Its Methyl Derivatives[1]

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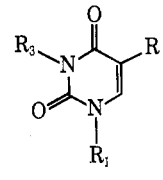
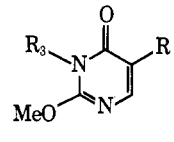
The methylation with diazomethane of nucleic acid constituents has been extensively studied[2] in connection with the plausible relationship[3] between the mechanism of mutagenesis and carcinogenesis. The action of diazomethane on uracil (**1**) and thymine (**1a**) was reported earlier[4] to afford only the 1,3-dimethyl derivatives, and reaction of diazomethane with uridines or the uracil residue in dinucleoside phosphates yielded exclusively the 3-N-methylation products.[5] However, in the case of diazomethane methylation of 1-β-D-arabinofuranosyl-5-fluorouracil, a minor amount of 4-O-methylation was also observed.[6]

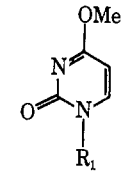
We have found that uracil (**1**), upon treatment with diazomethane, gave rise to four dimethyl compounds: 1,3-dimethyluracil (**2**), 2-methoxy-3-methyl-4-pyrimidone (**3**), 4-methoxy-1-methyl-2-pyrimidone (**4**), and 2,4-dimethoxypyrimidine (**5**). These products were isolated by preparative thin layer and gas–liquid phase



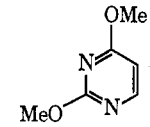
1, R = R_1 = R_3 = H
1a, R = Me; R_1 = R_3 = H
2, R = H; R_1 = R_3 = Me
6, R = R_1 = H; R_3 = Me
6a, R = R_3 = Me; R_1 = H
7, R = R_3 = H; R_1 = Me



3, R = H; R_3 = Me
3a, R = R_3 = Me
8, R = R_3 = H



4, R_1 = Me
9, R_1 = H



5

chromatography. The previously unreported dimethyluracil (**3**) was identified by its nmr spectrum

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