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Three Isomeric 1,3-Dimethyl-5-(dihydropyran-2′-yl)-2,4-pyrimidinediones. Palladium-Catalyzed Synthesis and Spectrometric Properties[1]

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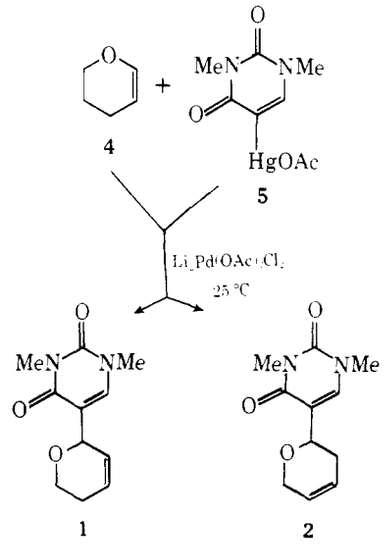
Reaction of 3,4-dihydro-2*H*-pyran with (1,3-dimethyl-2,4-pyrimidinedion-5-yl)mercury(II) acetate in the presence of 1 equiv of $Li_2Pd(OAc)_2Cl_2$ at 25 °C produced in high yield a mixture of two isomers, 1,3-dimethyl-5-(5′,6′-dihydro-2′*H*-pyran-2′-yl)-2,4-pyrimidinedione (**1**) and 1,3-dimethyl-5-(5′,6′-dihydro-2′*H*-pyran-6′-yl)-2,4-pyrimidinedione (**2**). Palladium-catalyzed [$(Ph_3P)_2Pd(OAc)_2$] reaction with 3,4-dihydro-2*H*-pyran with 1,3-dimethyl-5-iodo-2,4-pyrimidinedione at 100 °C yielded a third isomer, 1,3-dimethyl-5-(3′,4′-dihydro-2′*H*-pyran-2′-yl)-2,4-pyrimidinedione (**3**). The relationship between the three isomers was established by reduction of each to the same product, 1,3-dimethyl-5-(tetrahydropyran-2′-yl)-2,4-pyrimidinedione (**7**). Structure assignments of the three isomers based on mass and ^1H nuclear magnetic resonance spectra are discussed.

Reactions of cyclic enol ethers with palladium derivatives of nitrogen heterocycles[1] provide a potentially attractive synthetic route to C-nucleosides.[2,3] In preliminary investigations,[1] we have studied several reactions of enol ethers, including 3,4-dihydro-2*H*-pyran (**4**), with organopalladium reagents. In this report, we describe the preparation by this method and spectrometric properties of three isomeric 1, 3-dimethyl-5-(dihydropyran-2′-yl)-2, 4-pyrimidinediones (uracils) (**1**–**3**).

Two of the isomers, 1,3-dimethyl-5-(5′,6′-dihydro-2′*H*-pyran-2′-yl)-2,4-pyrimidinedione (**1**) and 1,3-dimethyl-5-(5′,6′-dihydro-2′*H*-pyran-6′-yl)-2,4-pyrimidinedione (**2**), result from reaction of 3,4-dihydro-2*H*-pyran (**4**) with an organopalladium reagent generated in situ by treatment of (1,3-dimethyl-2,4-pyrimidinedion-5-yl)mercury(II) acetate (**5**) with the palladium(II) salt $Li_2Pd(OAc)_2Cl_2$ in acetonitrile at room temperature during 1 day.[1] The reaction apparently involves regiospecific addition of an intermediate pyrimidinylpalladium species derived from **5** to the double bond of 3,4-dihydro-2*H*-pyran (**4**) followed by elimination of a hydridopalladium salt to yield the initial (major) product **1**. Readdition of the hydridopalladium salt to the double bond of the dihydropyranyl moiety of **1** and subsequent reelimination produced the double bond shifted minor product **2**.[1]

The third isomer, 1,3-dimethyl-5-(3′,4′-dihydro-2′*H*-pyran-2′-yl)-2,4-pyrimidinedione (**3**), was produced as the sole isolable product of reaction of 3,4-dihydro-2*H*-pyran (**4**) with



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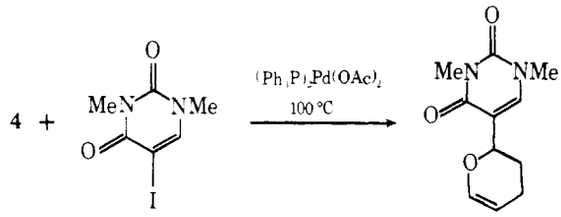

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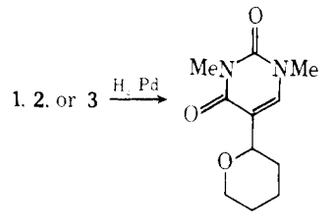
a similar pyrimidinylpalladium reagent formed from 1,3-dimethyl-5-iodouracil[4] (**6**) and a catalytic amount of diacetatobis(triphenylphosphine)palladium(II).[5] Presumably, the differing results of the two reactions are caused by the difference in reaction temperatures.[6] In the first reaction, the organopalladium species forms easily from the corresponding mercury derivative (**5**), and the reaction proceeds at room temperature.[1,6] In the second reaction, in which the organopalladium intermediate was generated from the 5-iodopyrimidine **6**, a temperature of 100 °C was required, resulting in the formation of **3** in which the double bond is stabilized by conjugation with the ring oxygen. It is not immediately clear why the fourth possible isomer, that in which the dihydropyranyl double bond is in conjugation with the pyrimidine rings, is not formed. Under similar conditions, palladium-catalyzed reaction of **6** with vinyl acetate readily yields the conjugated product, 1,3-dimethyl-5-vinyluracil.[7]

Isomers **1–3** were assigned structures after consideration of their chemical and spectrometric properties. Their isomeric nature was established by mass spectrometry. Further, reduction of isomers **1–3** produced a single compound, 1,3-dimethyl-5-(tetrahydropyran-2′-yl)-2,4-pyrimidinedione (**7**),



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establishing that the points of attachment of the pyrimidine and dihydropyranyl rings are the same for each isomer. The fact that isomers **1–3** exhibit identical ultraviolet spectra with absorption maxima at 270 nm (methanol) establishes that, in each compound, the dihydropyranyl double bond is not conjugated with the pyrimidine chromophore.[8]

The two structural features of each isomer remaining to be established, the point of attachment of the dihydropyranyl ring to the pyrimidine and the position of the dihydropyranyl double bond, were assigned from consideration of the respective ^{1}H nuclear magnetic resonance (^{1}H NMR) spectra (see Experimental Section).[9] Structure assignments are particularly straightforward upon comparison of the three spectra. Thus, in the spectra of **1** and **2**, dihydropyranyl resonances assignable to olefinic hydrogens (two) and hydrogens on oxygen-bearing carbon (three) are apparent, establishing the 2′ position for linkage of the dihydropyranyl moiety to the pyrimidine ring. The downfield position of the 2′-H resonance in **1** (δ 5.25) as compared with that in **2** (δ 4.59) is indicative of its allylic relationship to the dihydropyranyl double bond of **1**. These and other obvious resonance assignments permit unique structures to be designated for **1** and **2**. Similarly, assignment of a unique structure to compound **3** is facile from examination of its ^{1}H NMR spectra in deuteriochloroform and deuteriobenzene (Experimental Section).[9] In these spectra the olefinic resonances are essentially those of dihydropyran itself,[10] and the single proton assignable to oxygen-bearing carbon is consistent only with structure **3**.

The mass spectra[9] reveal that the three isomers exhibit interesting differences in fragmentation modes. The mass spectrum of each isomer exhibits a different base ion. In the spectrum of **1** the base ion (*m/e* 167) corresponds to the pyrimidine moiety (B) plus a CO fragment derived from the dihydropyranyl ring. The mass spectrum of **2** exhibits a prominent B + 28 ion at *m/e* 167; however, in this case the B + H ion at *m/e* 140 is the base ion. An abundant B + H ion is characteristic of N- rather than C-nucleosides[11–13] and apparently arises in the spectrum of **2** because for this isomer transfer of a 5′-H of the dihydropyranyl ring to the pyrimidine moiety with rupture of the C_5–$C_{6'}$ bond and formation of 2*H*-pyran is particularly favored. The favored fragmentation mode for isomer **3** is still different. In this case the base ion at *m/e* 166 arises by retro-Diels–Alder reaction of the dihydropyranyl moiety, yielding acrolein and the 5-vinylpyrimidine. In the spectrum of each isomer an ion at *m/e* 81 corresponding to the heteroaromatic $(C_5H_5O)^+$ ion is observed.

Experimental Section

General Comments. Melting points were determined with a hot stage microscope and are uncorrected. Ultraviolet spectra were recorded with a Cary-15 spectrophotometer, and ^{1}H NMR spectra were obtained on deuteriochloroform and deuteriobenzene solutions using a Varian Associates HA-100 spectrometer. Mass spectra were obtained using a CEC 21-110B mass spectrometer (direct insertion probe, high-resolution data) and a DuPont 21-491 mass spectrometer (gas chromatography–mass spectrometry). Elemental analyses were provided by Dr. R. Wielesek, University of Oregon.

(1,3-Dimethyl-2,4-pyrimidinedion-5-yl)mercury(II) Acetate (5). A mixture of 14 g (0.1 mol) of 1,3-dimethyluracil[8] and 31.8 g (0.1 mol) of mercury(II) acetate in 200 mL of methanol containing several drops of 70% perchloric acid was stirred at room temperature for 12 h. The resulting precipitate was collected and washed with cold methanol to yield 36.4 g (93%) of (1,3-dimethyl-2,4-pyrimidinedion-5-yl)mercury(II) acetate (**5**), which was suitable for use without further purification.

1,3-Dimethyl-5-(5′,6′-dihydro-2′*H*-pyran-2′-yl)-2,4-pyrimidinedione (1) and 1,3-Dimethyl-5-(5′,6′-dihydro-2′*H*-pyran-6′-yl)-2,4-pyrimidinedione (2). A mixture consisting of 4.0 g (0.01 mol) of (1,3-dimethyl-2,4-pyrimidinedione-5-yl)mercury(II) acetate (**5**), 2.3 g (0.01 mol) of palladium acetate, 0.8 g (0.02 mol) of lithium chloride, and 1.6 g (0.02 mol) of 3,4-dihydro-2*H*-pyran (**4**) in 100 mL of anhydrous acetonitrile was stirred for 12 h at room temperature. During this time a black precipitate (presumably palladium) formed. Hydrogen sulfide was passed through the reaction mixture, and the resulting precipitated metal salts were removed by filtration. Evaporation of the filtrate left a residue (2.0 g, 91%) consisting of a mixture of **1** and **2** (3:1). The mixture of **1** and **2** was difficult to separate; silica gel chromatography with dichloromethane elution yielded in early fractions, 1,3-dimethyl-5-(5′,6′-dihydro-2′*H*-pyran-6′-yl)-2,4-pyrimidinedione (**2**) as colorless crystals from hexane: mp 134–135 °C UV λ_{max} (MeOH) 270 nm (ϵ 7900); MS *m/e* 222.1029 (M^+·; calcd for $C_{11}H_{14}N_2O_3$, 222.1004), 167.0470 (calcd for $C_7H_7N_2O_3$, 167.0457); ^{1}H NMR ($CDCl_3$) δ 1.8–2.8 (m, 2 H, 5′-H), 3.38 and 3.43 (s and s, N–Me), 4.35 (brd, 2′-H), 4.59 (d of d, J = 5 and 10 Hz, 6′-H), 5.6–6.1 (m, 2 H, 3′- and 4′-H), 7.29 (s, 6-H).

Anal. Calcd for $C_{11}H_{14}N_2O_3$: C, 59.5; H, 6.31; N, 12.6. Found: C, 59.1; H, 6.09; N, 12.6.

Further elution yielded middle fractions containing both **1** and **2** and finally late fractions containing pure 1,3-dimethyl-5-(5′,6′-dihydro-2′*H*-pyran-2′-yl)-2,4-pyrimidinedione (**1**): mp 123–124 °C (from hexane); UV λ_{max} (MeOH) 270 nm (ϵ 8100); MS *m/e* 222.1000 (M^+·; calcd for $C_{11}H_{14}N_2O_3$, 222.1004); ^{1}H NMR ($CDCl_3$) δ 1.8–2.5 (m, 2 H, 5′-H), 3.37 and 3.42 (s and s, N–Me), 3.7–4.2 (m, 2 H, 6′-H), 5.25 (brd, 1 H, 2′-H), 5.7–6.1 (m, 2 H, 3′- and 4′-H), 7.27 (s, 1 H, 6-H).

Anal. Calcd for $C_{11}H_{14}N_2O_3$: C, 59.5; H, 6.31; N, 12.6. Found: C, 59.4; H, 6.15; N, 12.6.

1,3-Dimethyl-5-(3′,4′-dihydro-2′*H*-pyran-2′-yl)-2,4-pyrimidinedione (3). A mixture consisting of 0.27 g (1 mmol) of 1,3-dimethyl-5-iodouracil (**6**), 0.2 g (2 mmol) of triethylamine, 0.7 mg (0.01 mmol) of diacetatobis(triphenylphosphine)palladium(II),[5] and 10 mL of 3,4-dihydro-2*H*-pyran (**4**) contained in a sealed tube was heated at 100 °C for 5 h. The cooled reaction mixture was poured into water and extracted with chloroform. The chloroform extract was chromatographed on silica gel using dichloromethane–ether (9:1) for

elution to yield 0.14 g (64%) of 1,3-dimethyl-5-(3′,4′-dihydro-2′*H*-pyran-2′-yl)-2,4-pyrimidinedione (**3**) which exhibited λ_{max} (MeOH) 270 nm (ϵ 8000) and MS *m/e* 222.1008 (M^+; calcd for $C_{11}H_{14}N_2O_3$, 222.1004) and 166.0737 (calcd for $C_8H_{10}N_2O_2$, 166.0742); ^1H NMR ($CDCl_3$) δ 1.5–2.5 (m, 4 H, 3′- and 4′-H), 3.37 and 3.45 (s and s, NMe), 5.7–5.95 (m, 2 H, 2′- and 5′-H), 6.45 (d, J = 6 Hz, 1 H, 6′-H), 7.27 (s, 1 H, 6-H). In deuteriobenzene solution, the resonances for 2′-H and 5′-H are separated,[9] permitting facile assignment.

Anal. Calcd for $C_{11}H_{14}N_2O_3$: C, 59.5; H, 6.31; N, 12.6. Found: C, 59.3; H, 6.22; N, 12.5.

1,3-Dimethyl-5-(2′-tetrahydropyranyl)-2,4-pyrimidinedione (7). To a solution of 0.14 g of 1,3-dimethyl-5-(3′,4′-dihydro-2′*H*-pyran-2′-yl)-2,4-pyrimidinedione (**3**) in 50 mL of tetrahydrofuran was added 14 mg of 5% palladium on carbon. The resulting mixture was shaken under 2 atm of hydrogen pressure for 2 h. The catalyst was removed, and the solvent was evaporated to yield 0.10 g (71%) of 1,3-dimethyl-5-(2′-tetrahydropyranyl)-2,4-pyrimidinedione (**7**): mp 105–106 °C; UV λ_{max} (MeOH) 270 nm (ϵ 7900); MS *m/e* 224.1224 (M^+; calcd for $C_{11}H_{16}N_2O_3$, 224.1191); ^1H NMR ($CDCl_3$) δ 3.35 and 3.40 (NMe), 4.35 (d, J = 11 Hz, 2′-H), 7.24 (6-H).

Anal. Calcd for $C_{11}H_{16}N_2O_3$: C, 58.9; H, 7.14; N, 12.5. Found: C, 58.9; H, 7.16; N, 12.4.

Similar reductions of both **1** and **2** in methanol yielded **7**. Prolonged (>5 h) contact with the reducing conditions resulted in reduction of the pyrimidinedione ring, producing a tetrahydro product of M_r 226.

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Registry No.—**1**, 67464-93-1; **2**, 67464-94-2; **3**, 67464-95-3; **4**, 110-87-2; **5**, 65904-27-0; **6**, 40738-83-8; **7**, 67464-96-4; 1,3-dimethyluracil, 874-14-6; mercury(II) acetate, 1600-27-7.

Supplementary Material Available: Complete ^1H NMR and electron ionization mass spectra for compounds 1–3 (2 pages). Ordering information is given on any current masthead page.

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Reactions of Heteroaromatic Cations with Nucleophilic Reagents. Addition of Methoxide Ion to 2,6-Diphenyl- and 4-Methoxy-2,6-diphenylpyrylium Cations

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The title reactions have been studied mainly in methanol and 9:1 acetonitrile–methanol. 2,6-Diphenylpyrylium cation yields a 4*H*-pyran as the kinetically favored product and diphenylpentadienone **7**. The latter, which forms upon ring cleavage of a 2*H*-pyran, is the product of a thermodynamically favored pathway. In methanol the reaction of the methoxy-substituted cation yields comparable amounts of the isomeric 2*H*- and 4*H*-pyranic adducts; in this case the 2*H*-pyran apparently does not undergo ring cleavage. In 9:1 acetonitrile–methanol the only primary product is the 4*H*-pyran. The proticity of the medium seems to have an important role in promoting the interconversion of 4*H*-pyrans to other reaction products.

The pyrylium cation, one of the fundamental heteroaromatic systems, reacts easily with nucleophilic reagents. The nucleophilic attack occurs preferentially at the α or γ position;[1] in the absence of a good leaving group the reaction yields nonaromatic adducts (2*H*- or 4*H*-pyrans). The formation of a 2*H*-pyran is often followed by a ring-opening reaction, yielding a dienonic valence tautomer of the 2*H*-pyran.[2] If, on the other hand, the attacked position is bound to a good leaving group, the formation of the pyran is followed by the loss of this group and formation of a substituted pyrylium cation. Owing to the high reactivity of the pyrylium ring, substitution occurs easily, even with such poor leaving groups as alkoxy groups.[3,4]

Nucleophilic substitutions of pyrylium cations are similar to nucleophilic substitutions of pyridinium cations[5] and of activated benzenoid substrates,[6] where the intermediacy of σ adducts seems a general common feature.

While the equilibrium reactions of formation of Meisenheimer adducts from nitro-activated substrates[7] and of dihydropyridines from pyridinium cations[8] have been intensively investigated, reversible reactions of formation of pyrans from pyrylium cations have received limited attention so far.[9]

In view of the interest in the nucleophilic substitutions of pyrylium and related cations,[4,10,11] and in connection with our studies on the formation of adducts from heteroaromatic substrates,[12] we present the results concerning the course of the reaction of methoxide ion with 2,6-diphenylpyrylium (**1**) and 4-methoxy-2,6-diphenylpyrylium (**2**) cations and the structure assignment of the reaction products. Hydrogen and methoxyl groups are known to affect, in a different way, the structure and stability of Meisenheimer adducts.[13] The phenyl groups at the α positions were expected to have some hindering effect[14] toward attack at such positions and to decrease therein the reactivity, leaving virtually unaffected the reactivity of the γ position.

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