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The Condensation of (Chlorocarbonyl)phenyl Ketene with Bisnucleophiles. Synthesis of 4-Hydroxy-5-phenylpyro-[2,3-*c*]pyrazol-6-ones and Formation of Pyrazolo[1,2-*a*] pyrazole-triones by Hydrogen Exchange in Unstable Mesoionic Compounds

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The addition of (chlorocarbonyl)phenyl ketene **2** to 5-alkylpyrazol-3(4*H*)-ones **1** led to the formation of 3-hydroxypyrazolo[1,2-*a*]pyrazole-dione/pyrazolo[1,2-*a*]pyrazole-trione derivatives **3**. This is ascribed to hydrogen exchange in initially formed unstable, mesoionic pyrazolo[1,2-*a*]pyrazol-4-ium-5-olates. In contrast, condensation of the same ketene with 3-alkyl-1-phenyl-2-pyrazolin-5-ones **4** afforded 4-hydroxy-3-alkyl-1,5-diphenylpyrano[2,3-*c*]pyrazol-6-one derivatives **5**. The latter reaction provides a new and rapid route to 4-hydroxy-2-pyrones fused to pyrazole rings, in good to excellent yields.

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Introduction

α-Oxoketenes are versatile bifunctional reagents for the synthesis of numerous heterocyclic compounds. The reactions of bisnucleophiles with these ketenes leads to cyclic compounds, as has been reported in several papers.[1–4] 5-Alkylpyrazol-3(4*H*)-ones have been found to be very effective 1,2- and 1,3-bisnucleophilic reagents, which react with a wide variety of electrophiles under mild experimental conditions; they have been used mainly for the synthesis of condensed polyfunctionally substituted pyrazolones.[5–7] These compounds have four electron rich centres at the two nitrogens, oxygen and C4, thus they can react with electrophiles.[8–10] In this paper, we describe the preparation of 4-hydroxy-2-pyrones fused to pyrazole rings by nucleophilic additions of pyrazolones **1** to (chlorocarbonyl)phenyl ketene **2**. The 2-pyrone moiety is an important structural motif present in many molecules that show a broad range of biological activities such as anti-HIV,[11] antimicrobial,[12] antifungal,[13] phytotoxic,[14] anti-leukemic,[15] anti-Alzheimer,[16] and anti-inflammatory[11] properties. 2-Pyrones are used in organic synthesis as building blocks for more complex chemical structures because they may participate in a variety of cycloaddition reactions to form bicyclic lactones. For example, they readily undergo Diels–Alder reactions with alkynes, producing substituted benzenes upon loss of carbon dioxide.[17] 2-Pyrone also forms the core structure of a variety of natural organic compounds. For example, the coumarins are an important class of benzo-fused 2-pyrones.

Results and Discussion

In order to access analogues of 4-hydroxy-2-pyrones fused to pyrazole rings, the condensation of (chlorocarbonyl)phenyl ketene **2** with 5-alkylpyrazol-3(4*H*)-ones was investigated. The pyrazolones can in principle react as 1,2- or 1,3-bisnucleophiles to form either pyrazolopyrone or pyrazolopyrazolone derivatives (Scheme 1). In the reactions with *N*-unsubstituted pyrazolones, specific 1,2-bisnucleophilic cycloaddition took place, and pyrazolo[1,2-*a*]pyrazole-trione derivatives **3a**–**c** were obtained in good to excellent yields in short experimental procedures (Scheme 1). They exist in two tautomeric forms **I** and **II** as described below.

5-Alkylpyrazol-3(4*H*)-ones exist in three tautomeric forms due to their keto-enol or lactam-lactime tautomerism, and this phenomenon is confirmed by spectral data (Scheme 2).[18,19]

Several high-level molecular orbital studies have been performed on the tautomeric structures of the unsubstituted pyrazolin-5-one ring in the gas phase and in solution.[20–22] Hillier and coworkers[20] reported that the most stable form in the gas phase is the CH tautomer, while Cao et al. and Luque et al.[21] considered the OH tautomer the most stable. When solvent effects were included, either the NH or CH tautomers were found to be the most stable, depending on the method used to simulate solvation. All three tautomeric forms have been observed experimentally.[22] There are several reports in the literature on the condensation of (chlorocarbonyl)phenyl ketene with pyrazoles, 1,2,3-triazoles, and 1,2,4-triazoles to generate mesoionic compounds.[23–25] When (chlorocarbonyl)phenyl ketene **2** was added to a refluxing tetrahydrofuran solution of the 5-alkylpyrazol-3(4*H*)-one derivative, the solution turned red, thus suggesting the generation of an unstable mesoionic compound (Scheme 3). The colours faded to yellow on continued heating. The short lifetimes of the red mesoionic pyrazolo[1,2-*a*]pyrazol-4-ium-5-olates would be due to their tautomerization to 3-hydroxypyrazolo[1,2-*a*]pyrazole-dione/pyrazolo

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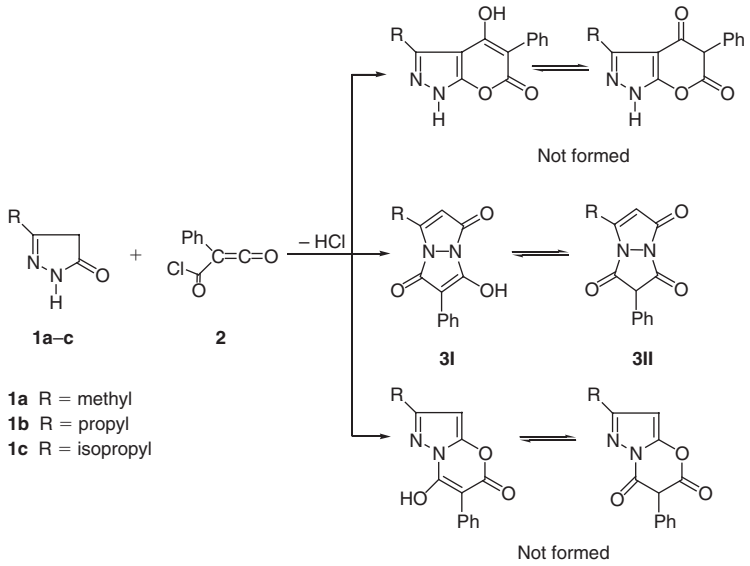
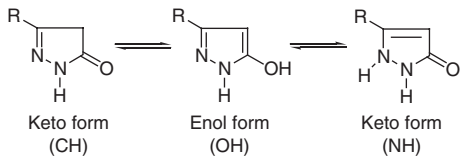
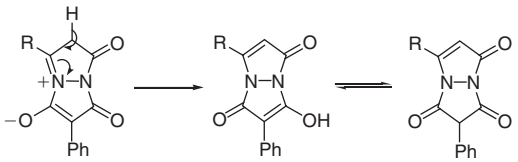


Scheme 1. Reaction of *N*-unsubstituted pyrazole-3(4*H*)-ones with (chlorocarbonyl)phenyl ketene.



Scheme 2. Tautomerism in pyrazole-3(4*H*)-ones.



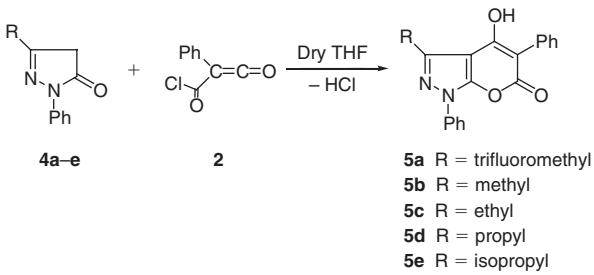
Scheme 3. Tautomerization of mesoionic pyrazolo[1,2-*a*]pyrazol-4-ium-5-olates to 3-hydroxypyrazolo[1,2-*a*]pyrazole-diones/pyrazolo [1,2-*a*] pyrazole-triones **3**.

[1,2-*a*]pyrazole-trione derivatives by intermolecular hydrogen exchange (Scheme 3).

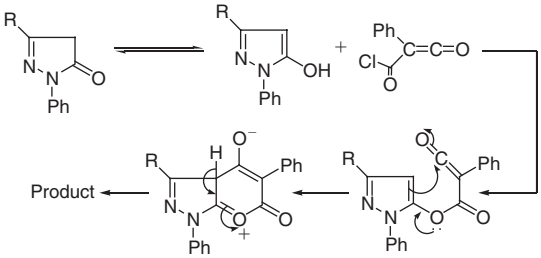
The spectral data for the isolated compounds **3** demonstrate that they exist as mixtures of the hydroxydione **I** and trione **II** tautomers (Schemes 1 and 3). Thus, the IR spectra of compounds **3a**–**c** revealed a strong absorption at 3150–2870 cm^{-1} attributed to the OH group in the dione tautomer **3I** (Scheme 1). In the ^{1}H NMR spectra, the OH proton appears at 13–12 ppm as a singlet. The spectrum of the trione tautomer **3II** exhibited a singlet at 5.4–5.0 ppm, which is attributed to the malonyl-type proton at C3.

In contrast to the reaction described in Schemes 1 and 3, the cycloaddition reactions described in Scheme 4 were accomplished by mixing equimolar quantities of (chlorocarbonyl)phenyl ketene **2** and *N*-substituted pyrazolones **4** in a dry solvent at ambient temperature. 4-Hydroxypyrano[2,3-*c*]pyrazol-6-ones **5** were the only products.

The 2-phenylpyrazol-3(4*H*)-ones **4a**–**e** can exist in either the CH or OH tautomeric forms, as shown in Scheme 5. A plausible mechanism for the formation of **5** therefore involves the reaction of the ketene with the enol 5-alkyl-2-phenylpyrazol-3-ol (Scheme 5).[26,27]



Scheme 4. Reaction of *N*-substituted pyrazole-3(4*H*)-ones with (chlorocarbonyl)phenyl ketene.



Scheme 5. Proposed mechanism for the formation of pyranopyrazolones **5**.

The structures of **5a**–**e** were determined on the basis of their elemental analyses, mass spectra, ^{1}H- and ^{13}C-NMR and IR spectral data. Only one product was obtained in each case. The ^{1}H NMR spectrum of **5a** indicated two kinds of protons along with one signal at low field (10.90 ppm) attributed to the OH group. This assignment is consistent with literature precedents for related compounds.[1,3,22]

Conclusions

In summary, we have shown that the condensation of (chlorocarbonyl)phenyl ketene **2** with 1,2- and 1,3-bisnucleophilic pyrazol-3-one derivatives provides a convenient and rapid synthesis of pharmacologically interesting compounds in high yields. Furthermore, no cumbersome apparatus is needed; the

precipitate from the reaction mixtures, and their purification is straightforward.

Experimental

General Procedures

(Chlorocarbonyl)phenyl ketene **2** was prepared according to the literature procedure.[28] The 5-alkyl-pyrazol-3(4*H*)-ones **1a**–**c** and 5-alkyl-2-phenylpyrazol-3-ones **4a**–**e** were known and prepared according to the reported procedure.[29] Solvents were dried over sodium and distilled before use. Melting points were determined on a Gallenkamp melting point apparatus and are uncorrected. IR spectra were measured on a Mattson 1000 FT-IR spectrophotometer. ^{1}H NMR and ^{13}C NMR spectra were determined on a Bruker DRX-500 AVANCE spectrometer at 500 and 125.77 MHz, respectively. Mass spectra were recorded on a Shimadzu QP 1100EX mass spectrometer operating at an ionization potential of 70 eV. Elemental analyses were performed using a Heracus CHN-O-Rapid analyzer.

General Procedure for Compounds *3*

5-Methylpyrazol-3(4*H*)-one (2 mmol) was dissolved in boiling anhydrous THF (20 mL), and a mixture of (chlorocarbonyl)phenyl ketene (2 mmol) in 5 mL dry THF was added drop wise over 2 min, which caused the colour of the solution to change to red. The reaction mixture was heated at reflux for 1/2 h, during which time it turned yellow. The reaction mixture was cooled, and the solid product was collected and recrystallized from dry *n*-hexane/THF (2:3).

*7-Methyl-2-phenyl-1*H*,5*H*-pyrazolo[1,2-*a*]pyrazolone **3a***

Yield 0.17 g (87%), pale green crystals, mp 165–166°C (Found: C 64.32, H 3.83, N 11.46%; $C_{13}H_{10}N_2O_3$ requires C 64.46, H 4.16, N 11.56%). ν_{max}(KBr)/cm^{-1} 3105 (broad, OH), 1765, 1742, 1661, 1617. δ_H (500 MHz, d$_6$-DMSO) 11.13 (1H, s, OH), 8.03 (2H, d, $^3J_{HH}$ 7.04, ArH), 7.15 (2H, t, $^3J_{HH}$ 7.62, ArH), 6.88 (1H, t, $^3J_{HH}$ 6.70, ArH), 6.06 (1H, s, H$_6$), 5.31 (1H, s, malonyl-H on C$_2$ keto), 2.37 (3H, s, CH$_3$). δ_C (125 MHz, d$_6$-DMSO) 170.57, 162.29, 161.40, 150.95 (C=O and C7), 136.22, 128.33, 124.07, 123.14, 101.40 (C$_6$), 85.21 (C$_2$ enol), 67.87 (C$_2$ keto), 13.36. *m/z* (ES-MS): 242 (80%, [M]$^+$), 186 (8), 145 (35), 118 (100), 89 (65), 77 (10), 63 (22), 51 (12).

*2-Phenyl-7-propyl-1*H*,5*H*-pyrazolo[1,2-*a*]pyrazolone **3b***

Yield 0.20 g (80%), pale yellow crystals, mp 113–116°C (Found: C 66.32, H 5.46, N 10.65%; $C_{15}H_{14}N_2O_3$ requires C 66.66, H 5.22, N 10.36%). ν_{max}(KBr)/cm^{-1} 3050 (broad, OH), 1760, 1713, 1659, 1647. δ_H (500 MHz, d$_6$-DMSO) 12.56 (1H, s, OH), 8.05 (2H, d, $^3J_{HH}$ 6.77, ArH), 7.14 (2H, t, J_{HH} 7.52, ArH), 6.87 (1H, t, $^3J_{HH}$ 6.68, ArH), 5.72 (1H, s, H$_6$), 5.30 (1H, s, malonyl-H on C$_2$ keto), 2.75 (2H, t, $^3J_{HH}$ 7.40, CH$_2$), 1.62 (2H, m, CH$_2$), 0.91 (3H, t, $^3J_{HH}$ 7.30 Hz, CH$_3$). δ_C (125 MHz, d$_6$-DMSO) 162.13, 161.61, 158.16, 154.98 (C=O and C$_7$), 136.30, 131.03, 129.07, 128.29, 127.99, 124.03, 123.05, 100.44 (C$_6$), 90.24 (C$_2$ enol), 67.87 (C$_2$ keto), 28.88, 21.91, 14.40. *m/z* (ES-MS): 270 (82%, [M]$^+$), 145 (15), 118 (100), 98 (11), 90 (45), 89 (55).

*7-Isopropyl-2-phenyl-1*H*,5*H*-pyrazolo [1,2-*a*]pyrazolone **3c***

Yield 0.21 g (84%), yellow crystals, mp 134–136°C (Found: C 66.28, H 5.49, N 10.47%; $C_{15}H_{14}N_2O_3$ requires C 66.66, H 5.22, N 10.36%). ν_{max}(KBr)/cm^{-1} 3080 (broad, OH), 1762, 1742, 1685. δ_H (500 MHz, d$_6$-DMSO) 11.83 (1H, s, OH), 8.02 (2H, d, $^3J_{HH}$ 6.78, ArH), 7.15 (2H, t, $^3J_{HH}$ 7.50, ArH), 6.87 (1H, t, $^3J_{HH}$ 6.79, ArH), 5.70 (1H, s, H$_6$), 5.34 (1H, s, malonyl-H on C$_2$ keto), 3.37 (1H, m, CH), 1.20 (6H, d, $^3J_{HH}$ 6.78, 2CH$_3$). δ_C (125 MHz, d$_6$-DMSO) 161.82, 161.46, 160.92, 158.09 (C=O and C$_7$), 135.99, 131.05, 130.05, 129.14, 126.05, 124.22, 123.30, 98.25 (C$_6$), 88.57 (C$_2$ enol), 67.87 (C$_2$ keto), 26.82, 21.72. *m/z* (ES-MS): 270 (100%, [M]$^+$), 242 (10), 214 (8), 145 (38), 125 (10), 118 (98), 111 (12), 89 (37), 67 (10), 53 (12).

General Procedure for Compounds *5*

(Chlorocarbonyl)phenyl ketene (2 mmol) was added to 2-phenyl-5-(trifluoromethyl)pyrazol-3(4*H*)-one (2 mmol) in dry THF (20 mL) with stirring at ambient temperature over the course of 20 min. The solid product was collected and recrystallized from *n*-hexane/THF (2:3).

*4-Hydroxy-1,5-diphenyl-3-(trifluoromethyl)pyrano [2,3-c]pyrazol-6-one **5a***

Yield 0.37 g (82%), grey crystals, mp 214–216°C (Found: C 61.11, H 2.69, N 7.41%; $C_{19}H_{11}F_3N_2O_3$ requires C 61.30, H 2.98, N 7.52%). ν_{max}(KBr)/cm^{-1} 3204 (broad, OH), 1706, 1682, 1659. δ_H (500 MHz, d$_6$-DMSO) 10.90 (1H, s, OH), 7.96–7.31 (10H, m, ArH). δ_C (125 MHz, d$_6$-DMSO) 161.44 (C=O), 160.84, 151.41, 136.70, 135.45 (q, $^2J_{C\text{-}F}$ 39.25, C$_3$), 135.28, 132.20, 130.53, 129.58, 129.34, 128.68, 122.91, 121.26 (q, $^1J_{C\text{-}F}$ 267.75, CF$_3$), 101.39, 97.97. *m/z* (ES-MS): 372 (100%, [M]$^+$), 352 (13), 255 (21), 235 (25), 118 (90), 90 (20), 77 (42), 51 (13).

*4-Hydroxy-3-methyl-1,5-diphenylpyrano[2,3-c]pyrazol-6-one **5b***

Yield 0.33 g, (97%) white crystals, mp 232–234°C (Found: C 71.32, H 4.07, N 8.81%; $C_{19}H_{14}N_2O_3$ requires C 71.69, H 4.43, N 8.80%). ν_{max}(KBr)/cm^{-1} 3150 (broad, OH), 1710, 1682, 1659. δ_H (500 MHz, d$_6$-DMSO) 11.63 (1H, s, OH), 7.78–7.29 (10H, m, ArH), 2.42 (3H, s, CH$_3$). δ_C (125 MHz, d$_6$-DMSO) 163.43 (C=O), 162.20, 150.87, 144.92, 137.61, 132.86, 132.34, 130.39, 129.20, 128.31, 127.96, 121.33, 99.39, 98.67, 14.81. *m/z* (ES-MS): 318 (28%, [M]$^+$), 200 (100), 132 (13), 118 (15), 91 (28), 77 (25), 67 (12), 51 (11).

*3-Ethyl-4-hydroxy-1,5-diphenylpyrano[2,3-c]pyrazol-6-one **5c***

Yield 0.35 g (94%), white crystals, mp 214–216°C (Found: C 72.08, H 4.62, N 8.26%; $C_{20}H_{16}N_2O_3$ requires C 72.28, H 4.85, N 8.43%). ν_{max}(KBr)/cm^{-1} 3100 (broad, OH), 1706, 1682, 1600. δ_H (500 MHz, d$_6$-DMSO) 11.36 (1H, s, OH), 7.79–7.30 (10H, m, ArH), 2.82 (2H, q, $^3J_{HH}$ 7.42, CH$_2$), 1.25 (3H, t, $^3J_{HH}$ 7.42, CH$_3$). δ_C (125 MHz, d$_6$-DMSO) 163.59 (C=O), 162.20, 150.93, 150.30, 137.68, 132.99, 132.35, 130.37, 129.18, 128.24, 127.93, 121.38, 99.24, 98.08, 22.35, 13.76. *m/z* (ES-MS): 332 (34%, [M]$^+$), 214 (100), 146 (10), 118 (12), 91 (22), 77 (24), 51 (8).

*4-Hydroxy-1,5-diphenyl-3-propylpyrano[2,3-c]pyrazol-6-one **5d***

Yield 0.36 g (90%), white crystals, mp 182–183°C (Found: C 72.58, H 5.03, N 8.05%; $C_{21}H_{18}N_2O_3$ requires C 72.82, H 5.24, N 8.09%). ν_{max}(KBr)/cm^{-1} 3120 (broad, OH), 1729, 1682, 1612. δ_H (500 MHz, d$_6$-DMSO) 11.45 (1H, s, OH), 7.79–7.29 (10H, m, ArH), 2.78 (2H, t, $^3J_{HH}$ 7.30, CH$_2$), 1.72 (2H, m,

CH_2), 0.93 (3H, t, $^3J_{HH}$ 7.28, CH_3). δ_C (125 MHz, d_6-DMSO) 163.38 (C=O), 162.14, 150.87, 149.01, 137.66, 132.83, 132.36, 130.36, 129.22, 128.33, 127.92, 121.36, 99.36, 98.17, 30.75, 22.34, 14.60. *m/z*(ES-MS): 346 (32%, $[M]^+$), 318 (7), 228 (100), 200 (18), 118 (17), 91 (35), 77 (89), 65 (18), 51 (29).

4-Hydroxy-3-isopropyl-1,5-diphenylpyrano [2,3-c]pyrazol-6-one ***5e***

Yield 0.37 g (92%), white crystals, mp 171–172°C (Found: C 72.76, H 5.11, N 7.99%; $C_{21}H_{18}N_2O_3$ requires C 72.82, H 5.24, N 8.09%). ν_{max}(KBr)/cm^{-1} 3157 (broad, OH), 1706, 1682, 1589. δ_H (500 MHz, d_6-DMSO) 11.56 (1H, s, OH), 7.79–7.29 (10H, m, ArH), 3.31 (1H, m, CH), 1.31 (6H, d, $^3J_{HH}$ 6.76, $2CH_3$). δ_C (125 MHz, d_6-DMSO) 163.33 (C=O), 162.06, 154.25, 150.96, 137.68, 132.85, 132.40, 130.36, 129.26, 129.16, 128.36, 127.96, 121.48, 99.33, 97.39, 28.71, 22.21. *m/z*(ES-MS): 346 (30%, $[M]^+$), 228 (100), 213 (10), 160 (5), 118 (7), 91 (16), 77 (22), 65 (5), 51 (7).

Acknowledgements

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