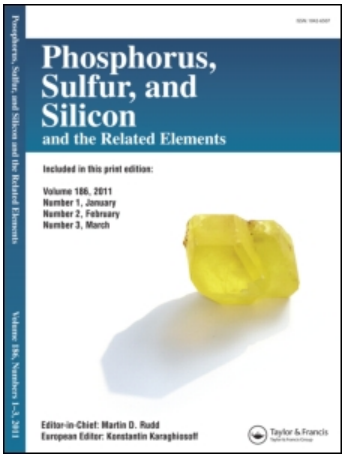

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THE REACTION OF ISATIN WITH ALKOXYCARBONYLMETHYLENE (TRIPHENYL)PHOSPHORANES

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Isatin **(1)** reacted with alkoxycarbonylmethylene(triphenyl)phosphoranes **(2)** in boiling benzene for about 3 h to give orange yellow crystalline products of alkyl (1,2-dihydro-2-oxo-3H-indol-3-yl)acetates **(3)**. The double bond of compounds **3** was reduced with zinc dust in boiling acetic acid to give **5**, which upon methylation with methyl iodide in dry acetone and anhydrous potassium carbonate yielded the corresponding products **6**. The structural assignments of the new compounds are based on the spectroscopic results.

Keywords: Isatin; Wittig reaction; NMR spectra (^1H; ^{13}C)

INTRODUCTION

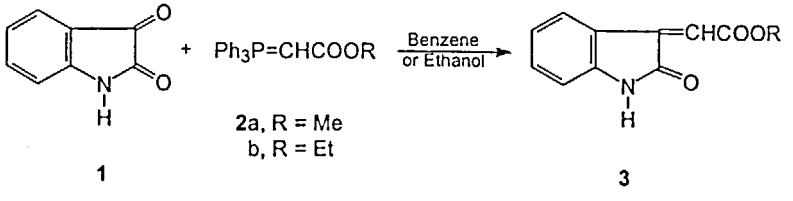
Generally, Wittig reaction of carbonyl compounds including α-diketones with alkoxycarbonylmethylene(triphenyl)phosphoranes affords the alkene formation at only one of the two possible carbonyl groups [1-23] to synthesis of (Z)- and (E)- isomeric esters. High selectivity for (Z)- or (E)- alkenes is available, depending on the particular circumstances, such as the type of ylide, type of carbonyl compound, or reaction conditions.[24-26] Recently, Takeuchi *et al.*[10] reported that the Wittig reaction of benzofuran-2,3-dione with ethoxycarbonylmethylene(triphenyl)phosphorane gave a mixture of 3-alkylidene-2(3H)-benzofuranone and 2-alkylidene-3(2H)-benzofuranone whereas (7-chloro-4,6-dimethoxy)benzofuran-2,3-dione with electron-donating substituent on the aromatic ring reacted with the same

* Corresponding author

ylide to go on Wittig olefination of the lactonic carbonyl and not of the keto carbonyl, forming 2-alkylidene-3(2H)-(7-chloro-4,6-dimethoxy)benzofuranone with high regioselectivity. In 1973 Brandman[16] reported that the reaction of isatin (**1**) with ylide **2b** in the usual solvents (benzene, dioxane, ethanol, tetrahydrofuran) provided the product in poor yield. In connection with these studies, we now report our results on the reaction of alkoxycarbonylmethylene(triphenyl)phosphoranes (**2**) with isatin (**1**) as an 1,2-dicarbonyl compound in which one of the two carbonyls is amidic in nature using usual solvents (benzene, ethanol). However, in some cases, it behaves as a potential α-diketone with both carbonyl groups taking part in the reaction. [27,28]

RESULTS AND DISCUSIONS

We have found that isatin (**1**) reacted with stabilized methylenephosphoranes (**2**) in boiling benzene or ethanol as usual solvents to give orange yellow crystalline products of (E)-isomer of alkyl (1,2-dihydro-2-oxo-3H-indol-3-yl)acetates (**3**) in good yields.

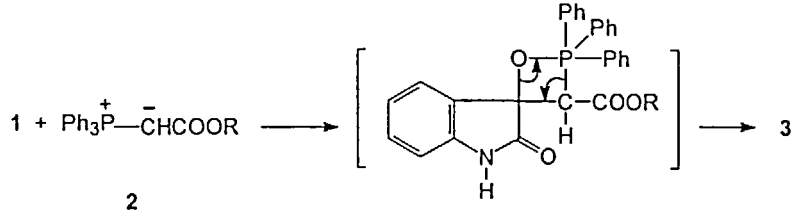


1 **3**

These compounds were separated by column chromatography on silica gel. Triphenylphosphine oxide was also isolated and identified. The assigned structure of the products **3** was established from the elemental analyses, IR, ^{1}H NMR and mass spectra. The IR spectra of compounds **3** showed the presence of band at 3196 cm^{-1}, due to the NH group and very strong absorption band around 1711 cm^{-1}, corresponding to the carbonyl of the ester and the amidic carbonyl. The ^{1}H NMR spectrum of **3a**, taken as an example, showed a singlet at δ 3.88 ppm, due to the methoxyl group and a singlet at δ 6.89 ppm, for the exocyclic vinyl proton. The phenyl proton at C-7 appeared as a doublet centered at δ 6.87 ppm with coupling constant J_{HH} = 7.8 Hz whereas the chemical shift of the proton at C-4 is

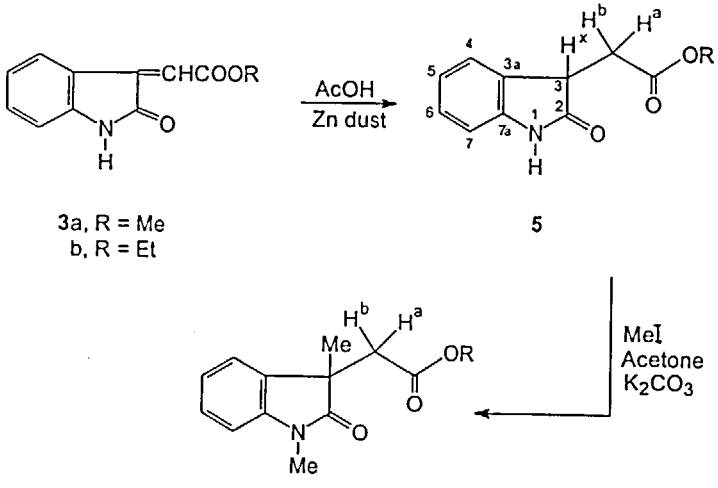
deshielded at δ 8.56 ppm, due to the anisotropic effect of the carbonyl group of the ester[29] and split into doublet of doublets with J_{HH} = 7.8 and 1.2 Hz . The other two phenyl protons at C-5 and C-6 appeared as two *di-ortho/meta* triplet of doublets[30] at δ 7.06 and 7.33 ppm, respectively with J_{HH} = 7.8 and 1.2Hz. The broad singlet at δ 7.89 ppm corresponding to NH which is exchangeable with D_2O.

The formation of the α,β-unsaturated ketones **3** by reaction of the stabilized ylides **2** with the α-diketone **1** can be explained in Scheme 1 by initial nucleophilic attack by the carbanion in the phosphoranes **2** on the reactive carbonyl-carbon of the α-diketone **1** to give the oxaphosphetane intermediate **4** which undergo preferential four-centered ring cleavage [26] *via* ejection of triphenylphosphine oxide to form the mono-alkene products **3.**



SCHEME 1

The double bond of alkyl (1,2-dihydro-2-oxo-3H-indol-3-yl)acetates (**3**) was reduced with zinc dust in boiling acetic acid to give colorless crystals of alkyl 2,3-dihydro-2-oxo-lH-indole-3-acetates (**5**) in quantitative yields (Scheme 2). Their assigned structure were established from the elemental analyses and spectral properties which are consistent with expectation. The ^1H NMR spectra of compounds **5a** and **5b** show that the three protons $-CH_2-CH-$ give rise to an ABX pattern, due to the mutual coupling between the geminal methylene protons (H^a and H^b) and the vicinal methine proton (H^x). The chemical shift at the higher field can be assigned to the proton H^a. Each proton is split by the other ($J_{H}a_{H}b$ = 17 Hz) and unequally by the vicinal proton H^x($J_{H}a_{H}x$ = 8 Hz and $J_{H}b_{H}x$ = 4.6 Hz). The proton H^x on the asymmetric carbon appeared as doublet of doublets and its chemical shift is located at lower field . These data were supported by ^{13}C NMR results *(cf.* Experimental).



3a, R = Me
b, R = Et

5

6

SCHEME 2

Reaction of alkyl 2,3-dihydro-2-oxo-1H-indole-3-acetates **(5)** with methyl iodide in presence of acetone and anhydrous potassium carbonate led to the formation of alkyl 2,3-dihydro-1,3-dimethyl-2-oxo-1H-indole-3-acetates **(6)** as shown in Scheme 2.The structure of compounds **6a** and **6b** was elucidated by correct elemental analyses, molecular weight determination (MS) and compatible spectroscopic results. The ^{1}H NMR spectra show the methylene protons H^a and H^b are coupled to one another to form two doublets ($J_{H^aH^b}$ = 16.2 Hz) with slightly different chemical shift Also, the presence of two singlets at δ 1.37 and 3.25 ppm, corresponding to the methyl protons at C-3 and N-CH_3, respectively. Further evidence supporting structure **6**, the spectral data of ^{13}C NMR *(cf.* Experimental).

CONCLUSION

From the results of this investigation, it has been shown that isatin **(1)** reacted with alkoxycarbonylmethylene(triphenyl)phosphoranes **(2)** in benzene or ethanol to give alkyl (1,2-dihydro-2-oxo-3H-indol-3-yl)acetates

(3) in good yields and not as reported by Brandman[16] that the reaction in the usual solvents (benzene, dioxane, ethanol, tetrahydrofuran) provided the product in poor yield.

EXPERIMENTAL

Melting points were determined on Electrothermal digital-melting-point apparatus and are uncorrected. The IR spectra were recorded in KBr disks, on a Jasco Fourier Transform Infrared spectrophotometer Model FT/IR-3000E. The NMR spectra were measured in $CDCl_3$, on a Varian Gemini-200 spectrometer for ^1H and on a Jeol EX-270 spectrometer for ^{13}C, using tetramethylsilane as an internal reference. The mass spectra (MS) were determined at 70 eV on a Finnigan MAT SSQ 7000 spectrometer.

Reaction of Isatin (1) with Methoxycarbonylmethylene(triphenyl)phosphorane (2a)

a) In benzene solution

To a solution of isatin (1) (0.33 g, 2.2 mmole) in dry benzene (15 ml), ylide **2a**[31] (0.74 g, 2.2 mmole) was added in small portions. Then, the reaction mixture was heated under reflux for about 3 h. After cooling, the orange yellow crystals, thus formed, were filtered off and crystallized from benzene to give methyl (1,2-dihydro-2-oxo-3H-indol-3-yl)acetate (**3a**) (0.22 g), mp 190–191 °C. The benzene filtrate was evaporated under reduced pressure and the residue was chromatographed on silica gel, using petroleum ether (bp 60–80°C) containing increasing amounts of ethyl acetate. The first fraction (85–80% petroleum ether) gave an additional amount of **3a** (0.17 g) (the total amount 0.39 g, 87% yield). Anal. Calcd. for $C_{11}H_9NO_3$: C, 65.02; H, 4.46; N, 6.90. Found : C, 65.11; H, 4.39; N, 6.83%. IR cm^{-1} : 3196 (NH); 1711 (C=O, ester and C=O, amide); 1614 (C=C). ^1H NMR: δ 3.88 (s, 3H, ester CH_3); 6.87 (d, J_{HH}= 7.8 Hz, 1H, ArH at C-7) ; 6.89 (s, 1H, =CH-); 7.06 (dt, J_{HH} = 7.8 and 1.2 Hz, 1H, ArH at C-5); 7.33 (dt, J_{HH}= 7.8 and 1.2 Hz, 1H, ArH at C-6); 7.89 (s, 1H, NH); 8.56 (dd, J_{HH}= 7.8 and 1.2 Hz, 1H, ArH at C-4). MS : m/z (relative intensity) 203 (M^+, 42%), 188 (7), 172 (37), 144 (76), 132 (8), 117 (23), 116 (100), 89 (46) and 59 (16). The second fraction (75–50% petroleum ether)

gave colorless crystals (0.5 g, 83% yield), identified as triphenylphosphine oxide by mp and mixed mp 151 °C.

Similar result was obtained by carrying out the same reaction at room temperature for 6 h to give **3a** in 73% yield.

b) In ethanol

A mixture of **1** (0.29 g, 2.0 mmole) and ylide **2a** (0.67 g, 2.0 mmole) in ethanol (20 ml) was heated under reflux for about 30 h. Then, the sc!vent was removed under reduced pressure and the residue chromatographed on silica gel. Elution with ethyl acetate-petroleum ether (bp 60–80 °C) afforded two fractions. The first fraction yielded orange yellow product (0.28 g, 68% yield), identified as methyl (1,2-dihydro-2-oxo-3H-indol-3-yl)acetate (**3a**) (mp and mixed mp, comparative IR and ^{1}H NMR spectra) *(vide supra).* The second fraction gave colorless crystalline product of triphenylphosphine oxide (0.45 g, 80% yield).

Reaction of Isatin (1) with Ethoxycarbonylmethylene(triphenyl)phosphorane (2b)

a) In benzene solution

A mixture of the diketone **1** (0.33 g, 2.2 mmole) and ylide **2b**[31] (0.77 g, 2.2 mmole) in dry benzene (15 ml) was heated under reflux for 3 h. The reaction mixture was allowed to cool to room temperature. The orange yellow precipitate was filtered off and crystallized from benzene to give ethyl (1,2-dihydro-2-oxo-3H-indol-3-yl)acetate (**3b**) (0.20 g). mp 168–169 C. The filtrate was evaporated under reduced pressure and the residue was chromatographed on silica gel; using system: petroleum ether (bp 60–80 °C), then petroleum ether containing increasing amounts of ethyl acetate. The first fraction gave an additional amount of **3b** (0.18 g) (the total yield 0.38 g, 79%). Anal. Calcd. for C_{12} H_{11} NO_3 : C, 66.33; H, 5.11; N, 6.45. Found: C, 66.12; H, 5.03; N, 6.64%. IR cm^{-1} ; 3192 (NH); 1716 (C=O, ester and C=O, amide); 1612 (C=C). ^{1}H NMR: δ 1.37 (t, J_{HH} = 7.2 Hz, ester CH_3); 4.34 (q, J_{HH} = 7.2 Hz, 2H, ester CH_2); 6.87 (d, J_{HH}= 7.6 Hz, 1H, ArH at C-7); 6.88 (s, 1H, =CH-); 7.04(dt, J_{HH}= 7.4 and 1.2 Hz, 1H, ArH at C-5); 7.32 (dt, J_{HH} = 7.4 and1.2 Hz,1H, ArH at C-6); 8.54 (d, J_{HH} = 7.6 Hz, 1H, ArH at C-4); 8.77 (s, 1H, NH). MS: m/z (relative intensity) 217 (M^+, 100%), 188 (3), 172 (51), 161 (3), 145 (21), 116 (35), 89

(23), 75 (3) and 63 (6). The second fraction yielded colorless crystals of triphenylphosphine oxide (0.52 g, 87% yield) (mp and mixed mp with an authentic sample) .

Methyl 2,3-dihydro-2-oxo-1H-indole-3-acetate (5a)

The orange yellow solution of **3a** (0.2 g, 0.1 mmole) in acetic acid (7 ml) was heated under reflux. Then, zinc dust (0.1 g) was added in small portions. The color of solution disappeared during 15 minutes. Heating was continued for 30 minutes at reflux temperature and filtered off to remove the inorganic residue. The solution was concentrated under reduced pressure, followed by addition of small amount of water and extracted with chloroform (4×15 ml). The extract was dried over anhydrous sodium sulfate and concentrated under reduced pressure with addition of few drops of petroleum ether (bp 40–60 °C). The colorless crystalline product, thus formed, was filtered off to give **5a** (0.19 g, 95% yield), mp 171–172 °C. Anal. Calcd. for C_{11} $H_{11}NO_3C$, 64.38 ; H, 5.40 ; N, 6.83. Found: C, 64.46 ; H, 5.36; N, 6.85%. IR cm^{-1} :3147 (NH); 1725, 1701 (2 C=O, amide and ester). ^1H NMR: δ 2.83 (dd, J_Ha_Hb = 17 Hz, J_Ha_Hx = 8 Hz, 1H, H^a); 3.10 (dd, J_Hb_Ha = 17 Hz, J_Hb_Hx = 4.6 Hz, 1H, H^b) ; 3.70 (s, 3H, ester CH_3) ; 3.83 (dd, J_Hx_Ha = 8.2 Hz, J_Hx_Hb = 4.6 Hz, 1H, H^x); 6.91 (d, J_{HH}= 7.6 Hz, 1H, ArH at C-7) ; 7.01 (t, J_{HH}= 7.5 Hz, 1H, ArH at C-5) ; 7.226 (d overlaped, J_{HH}= 7.4 Hz, 1H, ArH at C-4); 7.227 (t overlaped, J_{HH}= 7.4 Hz, 1H, ArH at C-6); 8.75 (s, 1H, NH). ^{13}C NMR: δ 34.5 (CH_2); 42.3 (C-3); 52.0 (OCH_3); 109.8 (C-7); 122.5 (C-5); 124.0 (C-4); 128.3 (C-6); 128.6 (C-3a); 141.5 (C-7a); 171.5 (C-2); 179.2 (C=O, ester). MS: m/z (relative intensity) 205 (M^+, 100%), 173 (22), 145 (73), 132 (7), 117 (28), 104 (2), 91 (4), and 76 (2).

Similarly, compound **5b** was obtained as a colorless crystalline product by the reduction of **3b** with zinc dust in acetic acid.

Ethyl 2,3-dihydro-2-oxo-lH-indole-3-acetate (5b)

(96% yield), crystallized from chloroform-petroleum ether (bp 40–60 °C), mp 90–91°C . Anal. Calcd. for C_{12} $H_{13}NO_3$; C, 65.74 ; H, 5.98; N, 6.39. Found: C, 65.79; H, 5.91; N, 6.33%. IR cm^{-1}: 3184 (NH); 1730 1705 (2 C=O, amide and ester). ^1H NMR: δ 1.21 (t, J_{HH}= 7.2 Hz, 3H, ester CH_3); 2.84 (dd, J_Ha_Hb= 17 Hz, J_Ha_Hx = 8 Hz, 1H, H^a) ; 3.08 (dd, J_Hb_Ha = 17 Hz, J_Hb_Hx = 4.6 Hz, 1H, H^b) ; 3.81 (dd, J_Hx_Ha = 8 Hz, J_Hx_Hb = 4.6 Hz, 1H,

H^x) ; 4.14 (q, J_{HH} = 7.2 Hz, 2H, ester CH_2) ; 6.89 (d, J_{HH} = 7.6 Hz, 1H ArH at C-7) ; 7.01 (t, J_{HH} = 7.6 Hz, 1H, ArH at C-5) ; 7.225 (t overlaped, J_{HH} = 7.4 Hz, 1H, ArH at C-6) ; 7.24 (d overlaped, J_{HH} = 7.6 Hz, 1H, ArH at C-4); 8.26 (s, 1H, NH). ^{13}C NMR: δ 14.3 (CH_3, ester); 35.1 (CH_2); 42.4 (C-3); 61.4 (OCH_2) ; 110.4 (C-7); 122.8 (C-5); 124.4 (C-4); 128.6 (C-6); 129.2 (C-3a); 142.2 (C-7a); 171.5 (C-2); 180.2 (C=O, ester). MS : m/z (relative intensity) 219 (M^+, 100%), 173 (15), 145 (31), 117 (27), 89 (6), 63 (3) and 51 (4).

Methyl 2,3-dihydro-1,3-dimethyl-2-oxo-1H-indole-3-acetate (6a)

A mixture of **5a** (0.45 g, 2.2 mmole) and anhydrous potassium carbonate (2 g) in dry acetone (30 ml) was stirred at room temperature for 30 minutes. Then, freshly distilled methyl iodide (5 ml) was added dropwise and the mixture was gently heated under reflux for about 20 h. After removal of the inorganic residue, the solution was evaporated under reduced pressure to give an oily product, triturated with benzene/ n-hexane to form colorless crystals of **6a** (0.29 g, 54% yield), mp 79–80 °C. Anal. Calcd. for C_{13} H_{15} NO_3: C, 66.94; H, 6.48; N, 6.00. Found: C, 66.83 ; H, 6.42; N, 5.95%. IR cm^{-1}: 1741 1699 (2 C=O, amide and ester). ^1H NMR : δ 1.37 (s, 3H, CH_3 at C-3) ; 2.84 (d, $J_{H^a H^b}$ = 16.6 Hz, 1H, H^a); 3.01 (d, $J_{H^b H^a}$=16.2 Hz, 1H, H^b) ; 3.25 (s, 3H, N-CH_3) ; 3.45 (s, 3H, ester CH_3) ; 6.86 (d, J_{HH}= 7.6 Hz, 1H, ArH at C-7) ; 7.03 (t, J_{HH}= 7.4 Hz, 1H, ArH at C-5) ; 7.19 (d, J_{HH}= 7.2 Hz, 1H, ArH at C-4); 7.27 (t, J_{HH}= 7.4 Hz, 1H, ArH at C-6). ^{13}C NMR: δ 24.0 (CH_3); 26.2 (N-CH_3); 41.1 (CH_2); 45.2 (C-3); 51.4 (OCH_3); 107.9 (C-7); 122.0 (C-5); 122.2 (C-4); 127.9 (C-6); 132.7 (C-3a); 143.4 (C-7a); 170.1 (C-2); 179.7 (C=O, ester). MS : m/z (relative intensity) 233 (M^+, 100%), 218 (2), 202 (4), 174 (14), 160 (69), 144 (7), 130 (9), 117 (3) and 103 (2).

In a similar manner, compound **6b** was obtained from the reaction of **5b** with methyl iodide as an oily product could not be solidified. It is purified by column chromatography.

Ethyl 2,3-dihydro-1,3-dimethyl-2-oxo-lH-indole-3-acetate (6b)

(65% yield), Anal. Calcd. for C_{14} H_{17} NO_3: C, 68.00 ; H, 6.93; N, 5.66. Found: C, 67.94; H, 6.88 ; N, 5.68%.IR cm^{-1}: 1714 (C=O, ester and C=O, amide) ; 1614 (C=C). ^1H NMR : δ 0.995 (t, J_{HH}= 7.1 Hz, 3H, ester CH_3);

1.385 (s, 3H, CH_3 at C-3); 2.85 (d, $J_{H^a H^b}$= 16.4 Hz, 1H, H^a) ; 3.05 (d, $J_{H^b H^a}$= 16.2 Hz, 1H, H^b); 3.265 (s, 1H, N-CH_3); 3.876 (m, 2H, ester CH_2); 6.88 (d, J_{HH}=7.4 Hz, 1H, ArH at C-7) ; 7.05 (t, J_{HH} = 7.4 Hz, 1H, ArH at C-5) ; 7.23 (d, J_{HH}= 7.2 Hz, 1H, ArH at C-4) ; 7.28 (t, J_{HH} = 7.6 Hz, 1H, ArH at C-6). ^{13}C NMR: δ 13.6 (CH_3, ester); 24.1 (CH_3); 26.1 (N-CH_3); 41.2 (CH_2); 45.3 (C-3); 60.6 (OCH_2); 107.8 (C-7); 122.0 (C-5); 122.1 (C-4); 127.9 (C-6); 132.6 (C-3a); 143.3 (C-7a); 169.4 (C-2); 179.6 (C-O, ester). MS : m/z (relative intensity) 247 (M^+, 100%), 233 (6), 218 (1), 202 (8), 174 (10), 160 (17), 144 (2), 130 (4) and 116 (1).

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