# *N*-Oxides, *N*-Imides, and *N*-Ylides of Five-membered Heterocycles. Part 5.[1] Reactions of 2,4,5-Triphenyl-3*H*-pyrrol-3-one 1-Oxide

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2,4,5-Triphenyl-3*H*-pyrrol-3-one 1-oxide (1) undergoes a variety of cycloaddition reactions across the nitrone system. Adducts with alkene dipolarophiles are, in the main, stable, but other adducts rearrange to give pyridones, pyrimidones, or oxazinones. The adducts with dimethyl acetylenedicarboxylate and with dimethyl maleate undergo an unusual decarboxylative rearrangement. Complex metal hydrides reduce the title compound to the 1-hydroxypyrrole, whereas catalytic reduction or electron transfer gives the 3-hydroxy-isomer. Reactions with hydroxylamine and related compounds give derivatives of 5-benzoyl-3,4-diphenylisoxazole.

THE 3*H*-pyrrol-3-one 1-oxides are the monocyclic analogues of the isatogens. The synthesis of some 2,4,5-triaryl derivatives was described in 1930,[2] but their chemistry has received scant attention. We have studied a variety of reactions of 2,4,5-triphenyl-3*H*-pyrrol-3-one 1-oxide (1), particularly cycloadditions.

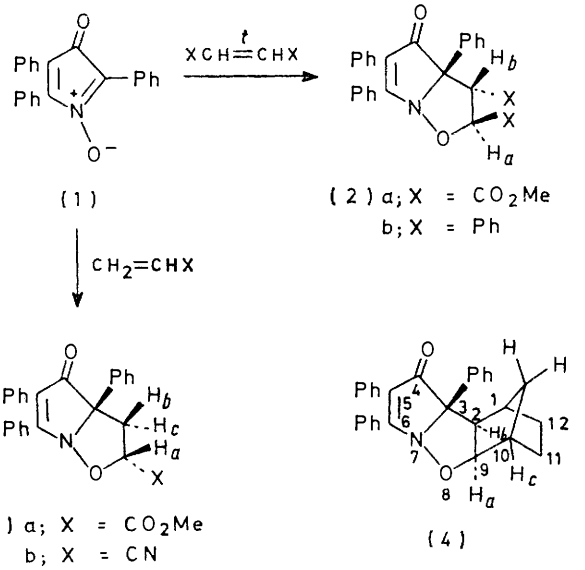
## RESULTS AND DISCUSSION

Cycloaddition across the nitrone system gives a bicyclic product, but this was isolable only for alkene dipolarophiles; other adducts underwent rearrangement with ring opening to give six-membered monocyclic products.

*Alkene Dipolarophiles.*—Dimethyl fumarate gave a single isolable product in good yield; its analytical and spectroscopic properties were in accord with the expected structure (2a), formed by stereospecific *syn*-addition controlled by steric interactions between the phenyl and methoxycarbonyl groups. *trans*-Stilbene similarly gave (2b), though this could not be isolated in a completely pure state.

Methyl acrylate and acrylonitrile reacted both stereospecifically and regiospecifically to give, respectively, compounds (3a) and (3b) as the only isolable products. The n.m.r. spectra unambiguously indicate that the methoxycarbonyl and cyano-groups are at position 2, as shown, rather than position 3.

The cycloaddition of norbornene also gave a single isolable adduct, though not in high yield. There are four possible stereoisomeric structures for the adduct: the *exo*- and *endo*-forms (with respect to the norbornane system), each of which could have the 2- and 3-position norbornyl hydrogen atoms either *cisoid* or *transoid* to the neighbouring phenyl group. The n.m.r. spectrum shows the characteristic coupling pattern of an *exo*-disubstituted norbornane, and the remarkably low frequency (δ *ca.* 0.3), of a signal which is probably derived from one of the methylene bridge protons shows that the




adduct must be the *exo*, *transoid*-isomer (4), since molecular models reveal that in the alternative *cisoid* form there are no protons near enough to either the phenyl ring or the carbonyl group to experience such abnormal shielding.

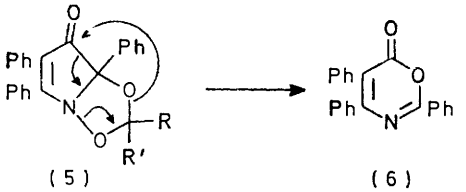
We also investigated cycloadditions with other alkenes. No reaction was observed with furan, 2,3-dihydropyran, or cyclopentadiene. Cyclopentadiene dimer and *cis*-stilbene both reacted but it was not possible to separate the complex mixtures formed. The reaction with dimethyl maleate was surprising: the product had the

[1] F. T. Boyle and R. A. Y. Jones, *J.C.S. Perkin I*, 1973, 170 is taken as Part 4.

[2] E. P. Kohler and C. R. Addinall, *J. Amer. Chem. Soc.*, 1930, **52**, 1590.

molecular formula $C_{27}H_{21}NO_4$, corresponding to the loss of $CH_2O_2$ from the simple adduct, and appeared to be identical with the product of the reaction with dimethyl acetylenedicarboxylate. This is discussed further below.

*Carbonyl Dipolarophiles.*—A number of carbonyl compounds catalysed the rearrangement of the pyrrolone oxide to the isomeric 1,3-oxazin-6-one (6). The mechanism presumably involves an intermediate cycloadduct (5) which can rearrange as shown. There seems to be



(5) (6)

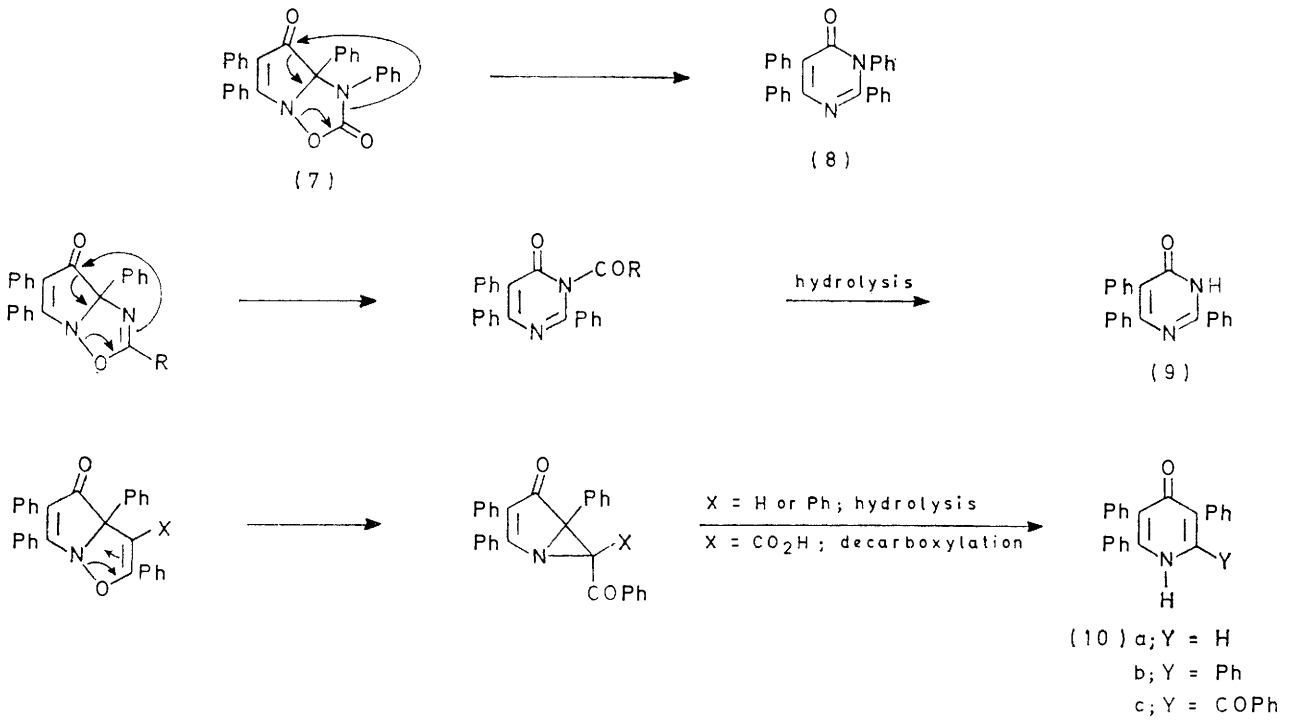
no obvious rationale for the behaviour of different carbonyl compounds: benzaldehyde, chloral, benzophenone, and cyclopentanone all led to rearrangement; acetaldehyde, acetone, acetophenone, and acetylacetone did not react.

*Phenyl Isocyanate as Dipolarophile.*—Phenyl isocyanate reacted to give the known 2,3,5,6-tetraphenylpyrimidin-4(3*H*)-one (8); this could be formed by a decarboxylative rearrangement of the intermediate cycloadduct (7), mechanistically similar to the carbonyl-induced rearrangement. Phenyl isothiocyanate gave an inseparable mixture of products.

*Nitrile Dipolarophiles.*—Trichloroacetonitrile and tetracyanoethylene both gave 2,5,6-triphenylpyrimidin-4-one (9; one tautomer only shown). This behaviour is analogous to that reported for isatogens [3,4] and is mechanistically similar to the two rearrangements described above.

*Acetylenic Dipolarophiles.*—Isatogens react with acetylenes to give, after rearrangement of the initial adduct, a variety of quinolones.[4,5] Analogous reactions with the pyrrolone oxide would give the pyridones (10) *via* the three-membered cyclic intermediate shown. Phenylpropiolic acid did indeed give the corresponding pyridone (10c), but the products with phenylacetylene and diphenylacetylene proved hard to characterise. Microanalyses were inconsistent, and although the high resolution mass spectra (discussed below) were compatible with the structures (10a and b) they were not unambiguous. The proposed mechanism requires the formation of benzoic acid as a by-product in these two reactions and this was indeed isolated.

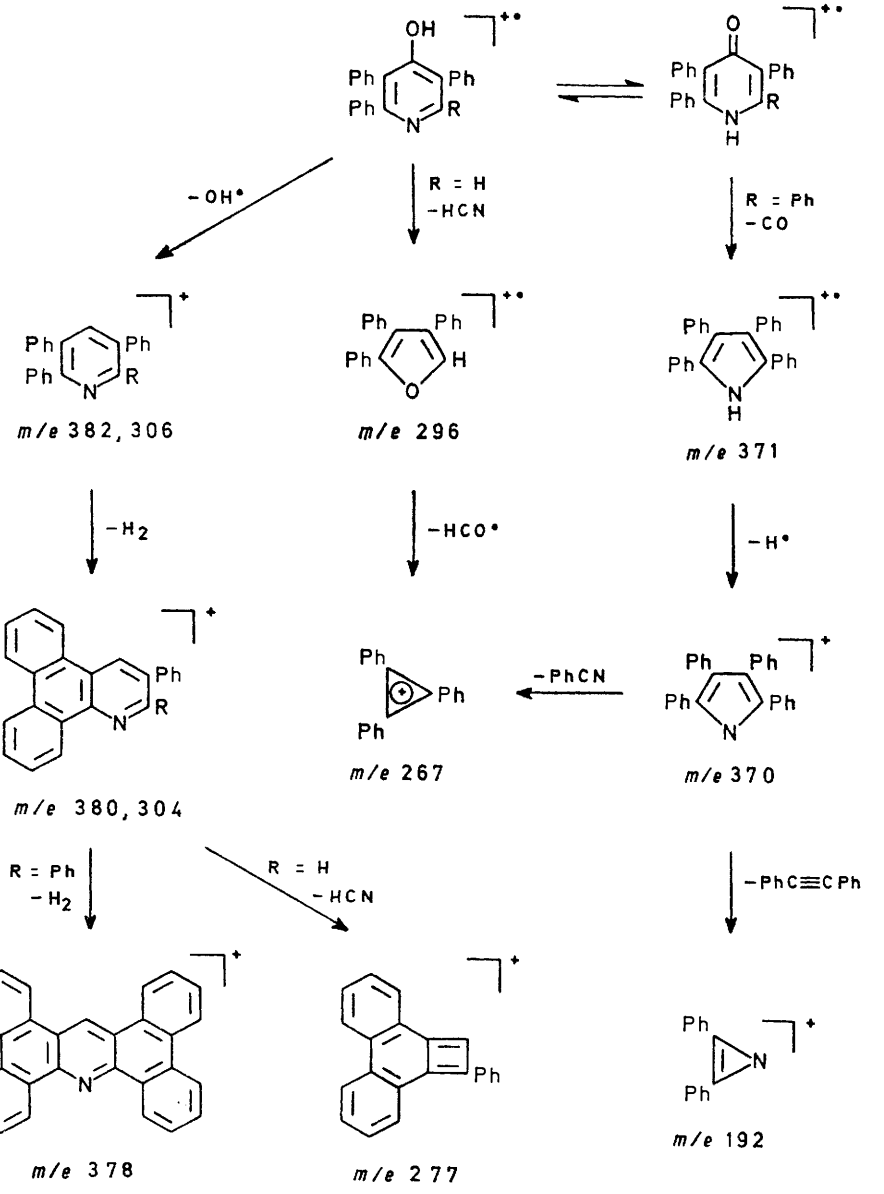
The mass spectra of both the triphenyl- and tetraphenyl-pyridones contained abundant ions with *m/e* values greater than those of the molecular ions. In the spectrum of the triphenyl compound the molecular ion was actually absent, but there was a peak at *m/e* 399 corresponding to a tetraphenylpyridone. In the tetraphenylpyridone spectrum a peak at *m/e* 503 corresponds to a benzoylated parent molecule, and a pair of ions at *m/e* 459 and 458 may be derived from pentaphenylpyridine. The immediate conclusion is that the products cannot be pure, and for the tetraphenylpyridone this seems to be supported by the appearance of the benzoyl cation as the base peak; in the spectra of the




(7) (8) (9) (10) a; Y = H b; Y = Ph c; Y = COPh

[3] W. E. Noland and D. A. Jones, *J. Org. Chem.*, 1962, **27**, 341.
[4] W. E. Noland and R. F. Modler, *J. Amer. Chem. Soc.*, 1964, **86**, 2086.
[5] C. C. Bond and M. Hooper, *J. Chem. Soc.* (*C*), 1969, 2453.

other pyridones (even the benzoylpyridone) and the pyrimidones this ion is of only low abundance. However, all attempts to demonstrate the presence of a second component in either product failed. It is certainly possible that an alternative explanation for the high *m/e* ions is that they arise from ion–molecule interactions. In the spectrum of the tetraphenylpyridone two main modes of fragmentation can be discerned: the first initiated by loss of carbon monoxide and the second by loss of a hydroxyl radical. The latter occurs in the fragmentation of the triphenylpyridone, but the loss of hydrogen cyanide takes precedence over that of carbon monoxide. The Scheme illustrates the proposed fragmentation paths.



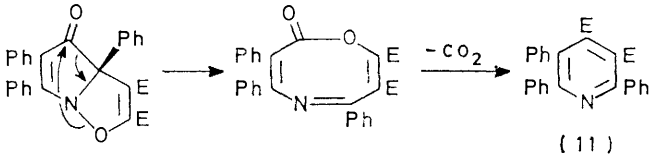

SCHEME

With dimethyl acetylenedicarboxylate the cycloaddition took a different course. A single product was isolated in high yield. Microanalysis, confirmed by high resolution mass spectrometry,[6] indicated the molecular formula $C_{27}H_{21}NO_4$, showing that the initial adduct had lost the elements of carbon dioxide. The n.m.r. spectrum revealed the presence of two methoxycarbonyl groups, so the decarboxylation must have involved the carbonyl group and the *N*-oxide oxygen atom from the pyrrolone oxide. We suggest that the product is dimethyl 2,5,6-triphenylpyridine-3,4-dicarboxylate (11), which could be formed by the route shown. This compound is thought by Eicher and his co-workers[7] to be the product of a cycloaddition to

[6] R. A. Y. Jones and N. Sadighi, *J.C.S. Perkin II*, in the press.

[7] T. Eicher, F. Abdesaken, G. Franke, and J. L. Weber, *Tetrahedron Letters*, 1975, 3915.

2,4,5-triphenyl-3*H*-pyrrol-3-one [*i.e.* the *N*-deoxygenated analogue of (1)]. Such a reaction would have to entail a Diels–Alder addition across the azacyclopentadienone system followed by a decarbonylation, and it could be that our reaction follows a similar path with an additional deoxygenation step. However, reaction across the nitrone system seems the more likely. The physical properties of our product are similar to, but not identical with, those reported by Eicher;[7] we have not yet had the opportunity of making a direct comparison.



E = $CO_2Me$

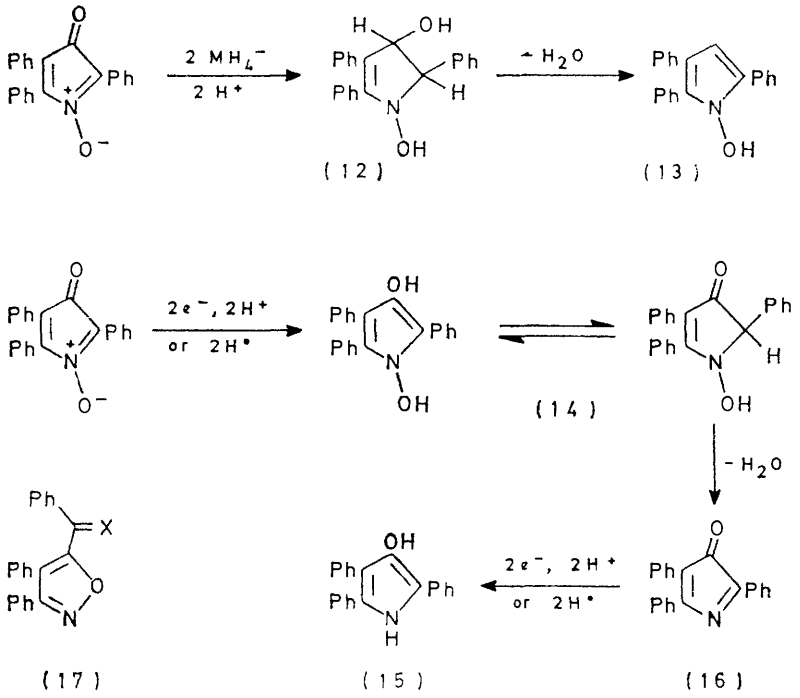
The same product was formed in the reaction between the pyrrolone oxide and dimethyl maleate. This could be rationalised by supposing that the reactions follow similar paths, with oxidation of the dihydropyridine which would be formed in the maleate reaction. However, there seems to be no sensible explanation for the dramatic difference between the maleate and the fumarate.

*Diethyl Azodicarboxylate as Dipolarophile.*—Only intractable resins were obtained from this reaction.

In addition to the cycloaddition reactions we have also investigated the reactions of the pyrrolone oxide (1) with reducing agents and with hydroxylamine, 2,4-dinitrophenylhydrazine, and semicarbazide.

*Reduction.*—Lithium aluminium hydride and sodium borohydride both reduced the pyrrolone oxide to 1-hydroxy-2,3,5-triphenylpyrrole (13). Catalytic reduction or reduction by electron transfer (Zn–HOAc, Na–EtOH), on the other hand, gave the *C*-hydroxy-isomer (15). The two isomers could be distinguished by their mass spectra:[6] the *N*-hydroxy-compound was characterised by an abundant fragment ion at $M - 16$ which was absent from the *C*-hydroxy-compound spectrum. This reductive behaviour can be explained by the Schemes shown: the direct, two-stage reduction by the metal hydrides gives a dihydroxy-intermediate (12) which is dehydrated by preferential loss of the *C*-hydroxy-group. The stepwise reduction by catalytic hydrogenation or electron transfer involves the tautomeric intermediate (14), which can undergo dehydration only with loss of the *N*-hydroxy-group.

Reduction with triethyl phosphite led to an intractable tar, but treatment with phosphorus trichloride gave a yellow-green oil which, though it could not be purified, appeared to be principally 2,4,5-triphenyl-3*H*-pyrrol-3-one (16) (*cf.* reference 7).



*Isomerisation to Benzoylisoxazole Derivatives.*—Reactions of the pyrrolone oxide (1) with hydroxylamine hydrochloride, with 2,4-dinitrophenylhydrazine, and with semicarbazide hydrochloride gave in each case the appropriate derivative of the isomeric 5-benzoyl-3,4-diphenylisoxazole (17). The reactions are analogous to the oximation of isatogens investigated by Pinkus and his co-workers.[8]

## EXPERIMENTAL

Elemental analyses were performed with a Technicon instrument. M.p.s were measured with a Kofler hot-stage

[8] J. L. Pinkus, T. Cohen, M. Sundaralingam, and G. A. Jeffrey, *Proc. Chem. Soc.*, 1960, 70; J. L. Pinkus, G. G. Woodyard, and T. Cohen, *J. Org. Chem.*, 1965, **30**, 1104.

apparatus. I.r. spectra were obtained on a Perkin-Elmer 257 spectrophotometer, n.m.r. spectra on a Perkin-Elmer R12 or Varian HA-100 spectrometer, u.v. spectra on a Unicam SP 800A spectrophotometer, and mass spectra on a Hitachi RMU-6E or A.E.I. MS902 instrument. The n.m.r. spectra of compounds (2)—(4) are recorded in the Table. A discussion of the high resolution mass spectra of some of the compounds reported here will be published elsewhere.[6]

*Cycloaddition Reactions of* 2,4,5-*Triphenyl*-3H-*pyrrol*-3-*one* (1).—The general procedure for these reactions was as follows. The pyrrolone oxide [2] (typically 0.5 or 1 g) was heated for 4—6 h under reflux, either with an equimolar quantity of the dipolarophile in xylene (15—20 ml) or, if it had a convenient b.p., with the dipolarophile in excess. The solvent was then removed under reduced pressure and the resulting oil usually solidified on being triturated with a little ethanol. In this way the following were prepared: *dimethyl* 2,3,3a,4-*tetrahydro*-4-*oxo*-c-3a,5,6-*triphenylpyrrolo*-[1,2-b]*isoxazole*-r-2,t-3-*dicarboxylate* [from dimethyl fumarate (0.4 g)], as yellow needles (0.35 g, 49%), m.p. 185° (from propan-2-ol) (Found: C, 71.3; H, 5.2; N, 2.9. $C_{28}H_{23}NO_6$ requires C, 71.6; H, 4.9; N, 3.0%), $\nu_{max.}$ (KBr) 1 730 and 1 690 $cm^{-1}$, *m/e* 469; 3,3a-dihydro-*r*-2,*t*-3,*c*-3a,5,6-pentaphenylpyrrolo[1,2-*b*]isoxazol-4(2*H*)-one (2b) (from *trans*-stilbene (0.75 g)], which despite several recrystallisations, could not be completely purified but which was characterised spectroscopically [$\nu_{max.}$ (Nujol) 1 685 $cm^{-1}$]; *methyl* 2,3,3a,4-*tetrahydro*-4-*oxo*-t-3a,5,6-*triphenylpyrrolo*-[1,2-b]*isoxazole*-r-2-*carboxylate* (3a) [from methyl acrylate in excess (20 ml) stabilised with hydroquinone (0.1 g)], as pale yellow plates (0.6 g, 49%), m.p. 152—153° (from ethanol) (Found: C, 75.6; H, 5.3; N, 3.4%; *m/e*, 411.147 3. $C_{26}H_{21}NO_4$ requires C, 75.9; H, 5.1; N, 3.4%; *M*, 411.147 0), $\nu_{max.}$ (KBr) 1 730 and 1 690 $cm^{-1}$; 2,3,3a,4-*tetrahydro*-4-*oxo*-t-3a,5,6-*triphenylpyrrolo*[1,2-b]*isoxazole*-r-2-*carbonitrile* (3b) [from acrylonitrile in excess (20 ml) stabilised with hydroquinone (0.1 g)], as yellow plates (0.44 g, 39%), m.p. 180° (decomp.) (from ethanol) (Found: C, 79.4; H, 5.2; N, 7.4. $C_{25}H_{18}N_2O_2$ requires C, 79.3; H, 4.8; N, 7.4%), $\nu_{max.}$ (KBr) 2 240 and 1 710 $cm^{-1}$ *m/e* 378; cis-2(endo-2H,9H)transoid-2,3-3,5,6-*triphenyl*-8-*oxa*-7-*azatetracyclo*[8.2.1.$0^{2,9}$.$0^{3,7}$]*tridec*-5-*en*-4-*one* (4), [from norborn-2-ene (0.47 g)], as yellow plates (0.12 g, 15%), m.p. 190—192° (from ethyl acetate) (Found: C, 83.4; H, 6.3; N, 3.5. $C_{29}H_{25}NO_2$ requires C, 83.0; H, 6.0; N, 3.3%), $\nu_{max.}$ ($CCl_2{:}CCl{\cdot}CCl{:}CCl_2$) 1 690 $cm^{-1}$, *m/e* 419; 2,4,5-triphenyl-6*H*-1,3-oxazin-6-one (6) [from various carbonyl compounds (see text)], as pale yellow needles, m.p. 207° (lit.,[9] 207—208°) (yields 13—53%; satisfactory analyses), $\lambda_{max.}$ (EtOH) 340 (log ε 4.08) and 265 nm (4.34), $\nu_{max.}$ (KBr) 1 730 and 1 610 $cm^{-1}$, δ ($CDCl_3$) 7.0—7.2 (10 H, m), 7.4 (3 H, m), and 8.2—8.3 (2 H, m), *m/e* 325; 2,3,5,6-tetraphenylpyrimidin-4(3*H*)-one (8) [from phenyl isocyanate in excess (15 ml)], as yellow plates (0.64 g, 53%), m.p. 295° (from acetone) (lit.,[10] 295°) (Found: C, 84.1; H, 5.1; N, 7.0. Calc. for $C_{28}H_{20}N_2O$; C, 84.0; H, 5.0, N, 7.0%), $\nu_{max.}$ (KBr) 1 670, 1 590, 1 560, and 1 540 $cm^{-1}$, δ ($CF_3{\cdot}CO_2H$) 7.3—8.3 (m), *m/e* 400; 2,5,6-triphenylpyrimidin-4-one (9) [from trichloroacetonitrile or tetracyanoethylene], as white needles, m.p. 290—293° (from ethanol) (lit.,[11] 288—293°) (yields 53 and 25% respectively; satisfactory analyses), $\nu_{max.}$ (KBr) 3 280, 1 640, and 1 610 $cm^{-1}$, δ ($CF_3{\cdot}CO_2H$) 7.1—7.5 (13 H, m) and 8.1—8.3 (2 H, m), *m/e* 324; 2,3,5-*triphenyl*-4-*pyridone* (10a) [from phenylacetylene (0.4 g) with propanoic acid (2 ml)], as yellow needles, (0.46 g, 47%), m.p. 218° (decomp.) (from ethanol) (Found: H, 5.4; N, 4.4. $C_{23}H_{17}NO$ requires H, 5.3, N, 4.3%), $\nu_{max.}$ (KBr) 3 300, 1 610, and 1 535 $cm^{-1}$, δ ($CF_3{\cdot}CO_2H$) 3.8br (1 H, s), 6.9—7.8 (15 H, m), and 8.0 (1 H, s); 2,3,5,6-*tetraphenyl*-4-*pyridone* (10b) [from diphenylacetylene (1.6 g) with propanoic acid (2 ml)], as white needles (0.49 g, 41%), m.p. 239—240° (from ethanol) (Found: *m/e*, 398.157 7. $C_{29}H_{21}NO$ requires 398.154 5 for $M^+ - H$), $\nu_{max.}$ (KBr) 3 300, 1 610, and 1 530 $cm^{-1}$; δ ($CDCl_3$) 3.6br (1 H, s) and 6.7—7.6 (20 H, m); 2-*benzoyl*-3,5,6-*triphenyl*-4-*pyridone* (10c) [from phenylpropiolic acid (2 g)], as white needles (1.25 g, 32%), m.p. 286° (from ethanol) (Found: C, 84.3; H, 5.2; N, 3.2%; *m/e* 427.155 6. $C_{30}H_{21}NO_2$ requires C, 84.3; H, 5.0; N, 3.3%; *M*, 427.157 2); $\nu_{max.}$ (KBr) 3 200, 1 670, 1 610, and 1 535 $cm^{-1}$, δ ($CF_3{\cdot}CO_2H$) 7.1—7.6 (m); dimethyl 2,5,6-triphenylpyridine-3,4-dicarboxylate (11) [from dimethyl acetylenedicarboxylate (1 g) in chloroform (15 ml)], as white plates (0.74 g, 58%), m.p. 231° (from methanol) (lit.,[7] 222—223°) (Found: C, 76.5; H, 5.2; N, 3.2%; *m/e*, 423.145 3. Calc. for $C_{27}H_{21}NO_4$; C, 76.6; H, 5.0; N, 3.3%; *M*, 423.147 0).

1-*Hydroxy*-2,3,5-*triphenylpyrrole* (13).—The pyrrolone oxide [2] (1 g) in dry ether (10 ml) was added to a stirred suspension of lithium aluminium hydride (0.3 g) in dry ether (15 ml) at a rate such as to maintain gentle reflux. The mixture was then stirred 1 h more and the excess of hydride was destroyed by adding wet ether (30 ml) and water (20 ml). The mixture was filtered and separated and the ethereal layer was dried ($MgSO_4$) and evaporated. The residue was recrystallised from methanol–acetone (1 : 1) to give 1-*hydroxy*-2,3,5-*triphenylpyrrole* as light yellow needles (0.72 g, 78%), m.p. 291° (Found: C, 84.6; H, 5.5; N, 4.3%; *m/e*, 311.131 7. $C_{22}H_{17}NO$ requires C, 84.9; H, 5.5; N, 4.5%; *M*, 311.131 0), $\nu_{max.}$ (KBr) 3 220 $cm^{-1}$. The same product was obtained in 66% yield from reduction of the pyrrolone with sodium borohydride in ethanol.

3-*Hydroxy*-2,4,5-*triphenylpyrrole* (15).—The pyrrolone oxide [2] (2.5 g) in glacial acetic acid (30 ml) and water (3 ml) was heated under gentle reflux for 5 h during which time zinc dust (6 g) was added in small portions. The solution was filtered and evaporated to dryness under reduced pressure. The residue was slurried in water (30 ml) and filtered to give a greenish yellow solid. Recrystallisation gave needles of 3-*hydroxy*-2,4,5-*triphenylpyrrole*. A further crop was obtained from the aqueous filtrate when this was

N.m.r. spectra of adducts with alkene dipolarophiles *

| Compound | $H_a$ | $H_b$ | $H_c$ | $J_{ab}$ | $J_{bc}$ | $J_{ac}$ | Other resonances ¶ |
|---|---|---|---|---|---|---|---|
| (2a) † | 4.55 | 3.93 | | 9 | | | 3.76, 3.90 (OMe) |
| (2b) ‡ | 4.68 | 4.20 | | 9 | | | |
| (3a) ‡ | 4.69 | 2.96 | 3.52 | 9 | 14 | 3 | 3.30 (OMe) |
| (3b) § | 4.26 | ~3.1 | 3.58 | 9 | 14 | 3 | |
| (4) § | 3.92 | 2.72 | | 5 | | 1 | 1.4—0.25 (aliphatic) |

* δ Values; *J* in Hz. † In $(CD_3)_2CO$. ‡ In $CDCl_3$. § In $(CD_3)_2SO$. ¶ All five compounds also showed resonances for the aromatic protons.

[9] T. Sasaki, K. Kanematsu, and A. Kakehi, *J. Org. Chem.*, 1971, **36**, 2451.

[10] L. Giammanco and F. P. Invidiata, *Ann. Chim. (Italy)*, 1970, **60**, 188.

[11] J. F. M. Wajon and F. Arens, *Rec. Trav. chim.*, 1957, **76**, 79.

made alkaline and kept at 0 °C for several hours (total yield 1.7 g, 73%), m.p. 285° (Found: C, 84.6; H, 5.4; N, 4.5%; *m/e*, 311.130 2. $C_{22}H_{17}NO$ requires C, 84.9; H, 5.5; N, 4.5%; *M*, 311.131 0), $\nu_{max.}$ (KBr) 322 0 $cm^{-1}$. The same product was obtained when the pyrrolone was reduced with sodium in absolute ethanol (yield 68%), and with hydrogen over a palladium–charcoal in dioxan at 60 lb $in^{-2}$ for 20 h (yield 89%).

*5-Benzoyl-3,4-diphenylisoxazole Oxime* (17; X = NOH). —A solution of hydroxylamine hydrochloride (0.75 g) in ethanol (25 ml) was added to the pyrrolone oxide [2] in ethanol (10 ml) and the solution was heated under reflux for 15 h. Hot aqueous acetic acid (20 ml) was added and the solution was left at room temperature for 1 day. A white crystalline precipitate formed, and on recrystallisation from ethanol–water (1 : 1) gave the *oxime* as long white needles (0.43 g, 84%), m.p. 181—182° (Found: C, 77.5; H, 5.0; N, 8.0%; *m/e*, 340.122 8. $C_{22}H_{16}N_2O_2$ requires C, 77.6; H, 4.7; N, 8.2%; *M*, 340.121 2), $\nu_{max.}$ (KBr) 3 300—3 160, 1 660, and 980 $cm^{-1}$, δ ($CDCl_3$) 7.0—7.4 (15 H, m), and 7.8br (1 H, s). In a similar manner were prepared: *5-benzoyl-3,4-diphenylisoxazole 2,3-dinitrophenylhydrazone* [17; X = $NNHC_6H_3(NO_2)_2$] as orange-yellow needles (81%), m.p. 245—246° (from acetonitrile) (Found: C, 66.9; H, 4.1; N, 13.9. $C_{28}H_{19}N_5O_5$ requires C, 66.6; H, 3.8; N, 13.9%), δ ($CDCl_3$) 6.98 (1 H, d, *J* 4 Hz), 7.2—7.6 (15 H, m), 8.05 (1 H, dd, *J* 4 and 1 Hz), and 8.98 (1 H, d, *J* 1 Hz), *m/e* 323 ($M^+ - C_6H_4N_3O_4$); and *5-benzoyl-3,4-diphenylisoxazole semicarbazone* (17; X = $NNHCONH_2$) as pale yellow needles (77%), m.p. 185—187° (Found: C, 72.0; H, 4.5; N, 14.2. $C_{23}H_{18}N_4O_2$ requires C, 72.2; H, 4.7; N, 14.7%), $\nu_{max.}$ (Nujol) 3 480—3 100 $cm^{-1}$, δ [$(CD_3)_2CO$] 7.1—7.6 (m) (Found: *m/e*, 364.131 8. *M* − $H_2O$ requires 364.132 4).

[6/652 *Received, 5th April,* 1976]