1-Styrylimidazoles

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The reactions of imidazoles with styrene oxide to yield 1-(2-hydroxy-2-phenylethyl)derivatives are described. On treatment with thionyl chloride and potassium hydroxide the alcohols may be converted readily into 1-styrylimidazoles.

We have previously shown that 1-vinyl derivatives of pyrroles [1-3] and imidazoles [3,4] may be obtained directly from the reaction of the potassium salt of an α-alkoxycarbonyl derivative with an epoxide *via* a lactone intermediate. In view of the importance of hydroxyethyl and styryl substituents in biologically active imidazoles, *e.g.* 1-(2-hydroxyethyl)-2-methyl-5-nitroimidazole (Metronidazole) [5] and 2-styryl-5-nitroimidazoles,[6] we report the synthesis of some 1-(2-hydroxy-2-phenylethyl)imidazoles and their conversion into 1-styryl derivatives.

Potassium 2-methoxycarbonylbenzimidazolide (1) reacted with styrene oxide at 90 °C to give 1-(2-hydroxy-2-phenylethyl)benzimidazole (4). This result is in contrast to the reactions of pyrrole and imidazole esters which generally yield 1-styryl carboxylic acids. By analogy with the reactions of pyrrole- and imidazole-carboxylates,[2,4] the present reaction presumably involves a lactone intermediate (2) which is hydrolysed by hydroxide ion (formed from atmospheric moisture and methoxide) to the hydroxy-acid (3); this would be expected to undergo ready decarboxylation.[7] Attempts to obtain the 1-styryl derivative by repeating the reaction under nitrogen [1] yielded a mixture of the olefin (5) and the hydroxy-compound (4).

The reaction of imidazoles bearing only alkyl or aryl substituents with styrene oxide yielded 2-hydroxy-2-phenylethyl derivatives without complication. The

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[3] G. Cooper, W. J. Irwin, and D. L. Wheeler, *Tetrahedron Letters*, 1971, 4321.
[4] G. Cooper and W. J. Irwin, *J.C.S. Perkin I*, in the press.
[5] C. Cosar, C. Crisnar, R. Horclois, R. M. Jacob, J. Robert, S. Tchelitcheff, and R. Vaufre, *Arzneim. Forsch.*, 1966, **16**, 13; D. R. Hoff, U.S.P., 3,107,201/1963 (*Chem. Abs.*, 1963, **59**, 15,876); S. J. Powell, I. MacLeod, A. J. Wilmot, and R. Elson-Dew, *Lancet*, 1966, **2**, 1329.
[6] B. Cavalleri, R. Ballotta, V. Ariola, and G. Lancini, *J. Medicin. Chem.*, 1973, **16**, 557.
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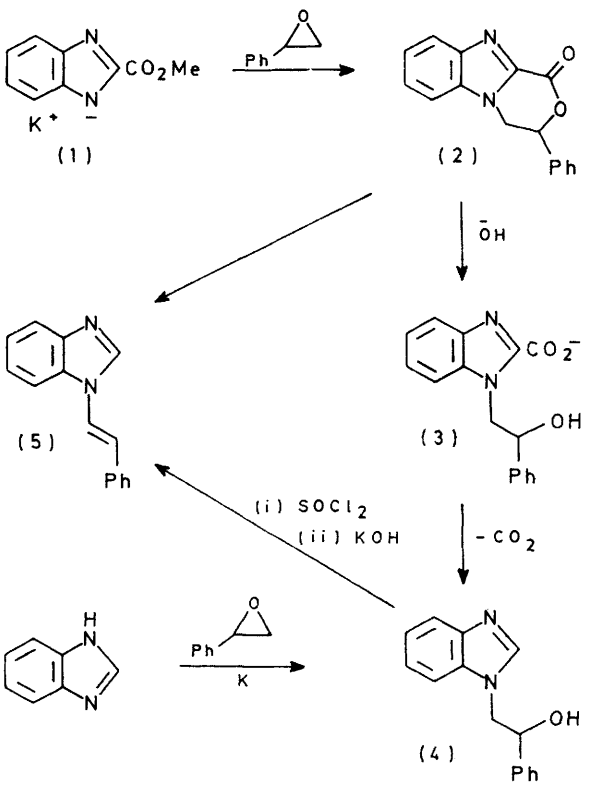
optimum conditions for the formation of 1-(2-hydroxy-2-phenylethyl)imidazole (6) involved the use of a catalytic amount of potassium with the reactants, in

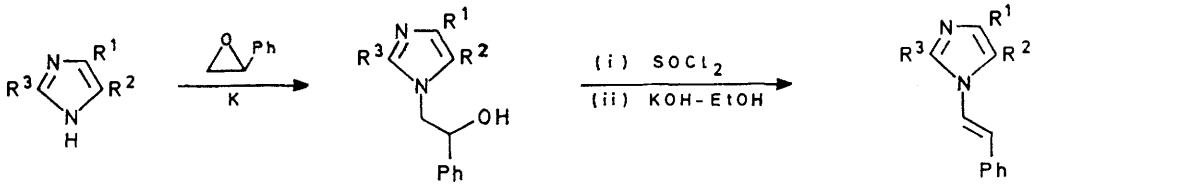


dimethylformamide, at room temperature. The reactions of the substituted imidazoles and of benzimidazole with styrene oxide under these conditions, and even with 1 mol. equiv. of potassium, were slow, probably owing to the increased bulk of the nucleophile and decreased nucleophilicity of the anion, but the reactions of 2-methyl-, 2-phenyl-, and 4,5-diphenyl-imidazole at elevated temperatures proceeded in excellent yield to give the hydroxy-compounds (6)—(9). Benzimidazole and styrene oxide yielded 1-(2-hydroxy-2-phenylethyl)-benzimidazole (4), identical with that obtained from 2-methoxycarbonylbenzimidazole, but in greater yield.

The reaction of potassium imidazolide with *cis*- or *trans*-stilbene oxide at room temperature only gave *ca.* 15% yield of the *threo*- (10) or *erythro*- (11) alcohol, respectively. The stereospecificity of the reaction is consistent with inversion at the site of nucleophilic attack. In an attempt to obtain higher yields of the alcohols (10) and (11) the reaction was conducted at 90 °C, but only *N*-benzylimidazole (12) was obtained in each case.

Compound (12) was also formed by the reaction of either the *threo*- or the *erythro*-alcohol with sodium hydride in dimethylformamide at 90 °C, thus suggesting the intermediacy of these alcohols as illustrated. The driving force for the elimination of benzaldehyde from the alkoxide ion is, presumably, the production of a stabilised benzylic carbanion, and a reduction in the amount of anion formed in the reaction of the potassium imidazolide with the stilbene oxides, at elevated temperatures, might be expected to decrease the rate of elimination. However, use of less potassium was not successful; only a catalytic amount is necessary to complete the reaction cycle to produce 1-benzylimidazole and more potassium imidazolide. The reaction was, however, quenched at the alcohol stage by forming the potassium salt from imidazole and potassium hydroxide. The water then present in the reaction mixture is a stronger acid than the alkoxide ion and so protonates it and stops the reaction at that stage.

The reaction of imidazole with propene oxide in dimethylformamide with a catalytic amount of potassium gave, in almost quantitative yield, a water-miscible viscous oil, the n.m.r. spectrum of which showed the presence of 1-(2-hydroxypropyl)imidazole. The mass spectrum showed a molecular ion at *m/e* 126, consistent with this but also contained a small peak at *m/e* 184. On the basis of n.m.r. data this impurity was considered to be 1-[2-(2-hydroxypropoxy)propyl]imidazole rather than a disubstituted imidazole.



(6) $R^1 = R^2 = R^3 = H$
(7) $R^1 = R^2 = H, R^3 = Me$
(8) $R^1 = R^2 = H, R^3 = Ph$
(9) $R^1 = R^2 = Ph, R^3 = H$

(15) $R^1 = R^2 = R^3 = H$
(16) $R^1 = R^2 = H, R^3 = Me$
(17) $R^1 = R^2 = H, R^3 = Ph$
(18) $R^1 = R^2 = Ph, R^3 = H$

The conversions of 1-(2-hydroxyethyl) derivatives of pyrrole,[8] indole,[9] carbazole,[8] and imidazole[10] into 1-vinyl compounds under pyrolytic conditions have been described. However, the main by-products of these reactions are polymeric; we were interested in defining milder conditions for the reaction of the 1-(2-hydroxy-2-phenylethyl)imidazoles to yield the thermo-

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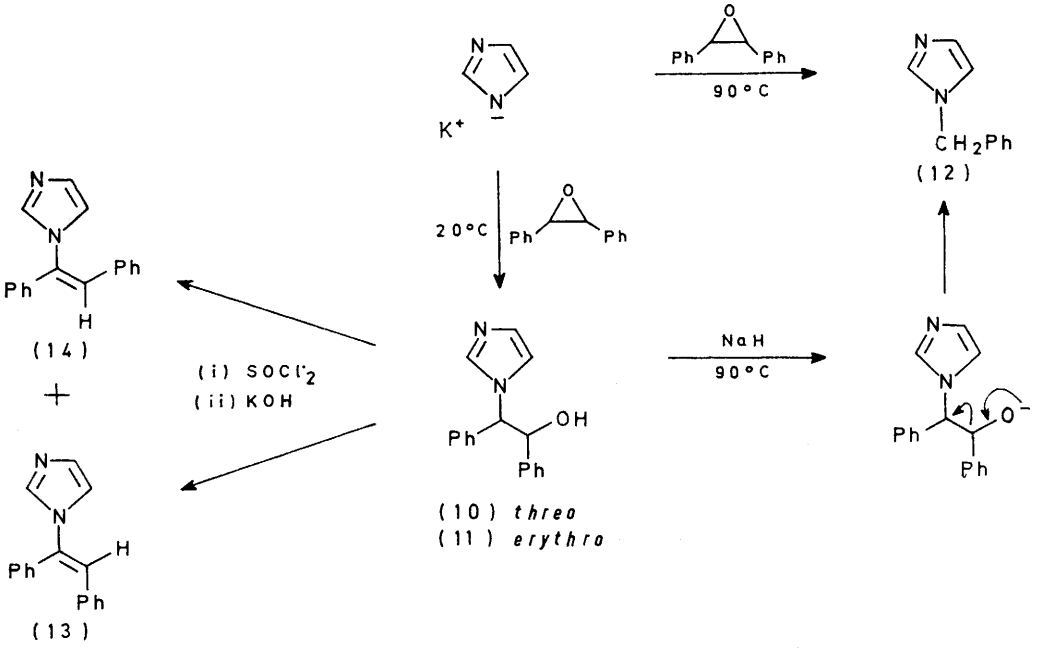
[9] N. Hiroyuki, H. Masaniro, and K. Shu, *J. Polymer Sci.*, 1966, **4**, 623.
[10] C. Schuster, G.P., 854,955/1952 (*Chem. Abs.*, 1952, **58**, 15,592).

labile 1-styryl derivatives.[4] Traynelis has reported [11] the dehydration of secondary and tertiary benzylic alcohols by hot dimethyl sulphoxide; however attempted dehydration of the secondary benzylic alcohol (6) to the olefin (15) afforded only starting material. The alcohol (6) was recovered from an attempted reaction in dimethylformamide with an excess of potassium ethoxide or t-butoxide, heating with dicyclohexylcarbodi-imide [12] in dimethyl sulphoxide was ineffective, and only the



toluene-*p*-sulphonate salt was obtained when the alcohol was treated with toluene-*p*-sulphonic acid.

In view of the ready reaction of lactones [2,4] to yield *N*-styryl compounds, acetate derivatives of the alcohols were examined. *trans*-1-Styrylimidazole (15) was isolated in 8% yield on treatment of the acetate of (6) with sodium methylsulphinylmethanide.[12] A much greater amount of the alcohol (6) was also isolated, presumably owing to contamination of the mixture with atmospheric moisture. It has been demonstrated [13] that the fine suspension of sodium hydroxide produced by adding an equimolar amount of water to sodium methylsulphinylmethanide in dimethyl sulphoxide can hydrolyse esters at 10^4—10^5 times faster than in hydroxylic solvents. A similar attempt to convert the acetate of (4) into the olefin (5) was likewise unsuccessful.

The elimination reaction of *N*-(2-chloroethyl)carbazole to give *N*-vinylcarbazole with methanolic potassium hydroxide has been reported.[14] The reaction of the alcohol (6) with thionyl chloride [15] gave crude 1-(2-chloro-2-phenylethyl)imidazole, whose n.m.r. spectrum closely resembled that of the alcohol (6) and showed the presence of very little impurity. Treatment of the crude chloro-compound with ethanolic potassium hydroxide, at room temperature, almost immediately yielded a white precipitate of potassium chloride, and *trans*-1-styrylimidazole (15) was isolated in high yield. To avoid the possibility of hydrolysis of the chloro-compound to the alcohol the free base was not isolated but rather an excess of ethanolic potassium hydroxide was added to the crude hydrochloride salt of the chloroethylimidazole to effect the elimination. In this way the alcohols derived from 2-methyl- (7), 2-phenyl- (8), and 4,5-diphenyl-imidazole (9) gave the 1-styrylimidazoles (16)—(18) in high yield. The synthesis of *trans*-1-styrylbenzimidazole (5) from 1-(2-hydroxy-2-phenylethyl)benzimidazole (4) was also successful.

Evidence for the occurrence of stereospecific *trans*-elimination in the reaction of potassium 2-methoxycarbonylpyrrolide with *cis*- and *trans*-stilbene oxides has been presented.[1] Dehydration of the *threo*- (10) or *erythro*- (11) alcohol *via* the chloro-derivative, however, gave an oil having the same spectroscopic properties in each case. The n.m.r. spectrum showed no absorptions due to aliphatic protons and the u.v. spectrum ($\lambda_{max.}$ 290 nm) confirmed the olefinic nature of the product. Only one spot was observed on t.l.c. but the fact that both alcohols gave the same mixture of olefins [(13) and (14)] indicates that the dehydration was non-stereospecific. Replacement of a hydroxy-group by a chlorine

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[12] L. F. Fieser and M. Fieser, ' Reagents for Organic Synthesis,' Wiley, New York, 1967.
[13] W. Roberts and M. C. Wiley, *J. Chem. Soc.*, 1965, 1290.
[14] G. R. Coemo and W. H. Perkin, *J. Chem. Soc.*, 1924, **125**, 1804.
[15] D. E. Welch and R. D. Vatne, *J. Medicin. Chem.*, 1968, **11**, 370.

atom with thionyl chloride is known [16] to proceed *via* an S_Ni mechanism with retention of configuration only in the absence of base. The imidazole reactant may thus act as a base for the liberation of chloride ion, and therefore initiate a non-stereospecific nucleophilic substitution to give a mixture of *threo*- and *erythro*-alkyl chlorides and thence a mixture of *cis*- (13) and *trans*- (14) olefins.

1-(2-Hydroxypropyl)imidazole also underwent an elimination on treatment of the oil with thionyl chloride followed by alcoholic potassium hydroxide. The n.m.r. spectrum of the product showed that it was *trans*-1-(2-methylvinyl)imidazole.

EXPERIMENTAL

I.r. spectra were determined for Nujol mulls, unless otherwise stated, with a Unicam SP 200 spectrophotometer. N.m.r. spectra were determined for solutions in deuteriochloroform, unless otherwise stated, with tetramethylsilane as internal standard on a Varian A60-A spectrometer, or if indicated on a Varian HR220 spectrometer by the Physicochemical Measurements Unit, Harwell. Mass spectra were determined on an A.E.I. MS9 spectrometer, operating at 100 uA and 70 eV. U.v. spectra were determined for solutions in methanol on a Beckmann Acta V spectrophotometer. Reaction temperatures quoted are those of an external oil-bath. Light petroleum refers to the fraction with boiling range 60—80°. Spectroscopic data for compounds marked with an asterisk are available as Supplementary Publication No. SUP 21649 (11 pp., 1 microfiche).†

General Method for the Reaction of Imidazoles with Epoxides.—The imidazole was dissolved in dimethylformamide (DMF) and potassium (1.0 or 0.1 equiv.), cut under light petroleum, was added in small portions with stirring. When all the potassium had dissolved the epoxide (1.1 equiv.) was added and the mixture was stirred at the stated temperature for the time indicated. Water was then added (10 times the volume of DMF) to precipitate the basic products, which were either filtered off or extracted into chloroform. The chloroform solutions were purified to remove neutral compounds by extraction with 5N-hydrochloric acid. The acidic extracts were basified with sodium hydroxide (40%). The basic products were then extracted into chloroform and the solution was washed with water, dried ($MgSO_4$), and evaporated to yield the products. The initial aqueous solutions were washed with ether and were acidified with concentrated hydrochloric acid to yield the acidic products.

1-(2-Hydroxy-2-phenylethyl)imidazole (6),* obtained as prisms (24 g, 70%) from imidazole (14 g, 0.2 mol), potassium (1 g, 0.025 mol), styrene oxide (26.4 g, 0.22 mol), and DMF (50 cm³) at room temperature for 17 h, had m.p. 151—152° (from methanol) (lit.,[17] 151—152°) (Found: C, 70.3; H, 6.3; N, 15.0. Calc. for $C_{11}H_{12}N_2O$: C, 70.2; H, 6.4; N, 14.9%). Acetylation with acetic anhydride in pyridine yielded the acetate * (5.1 g, 80%), b.p. 170° at 1 mmHg, as a viscous oil.

1-(2-*Hydroxy*-2-*phenylethyl*)-2-*methylimidazole* (7),* obtained as needles (3.0 g, 50%) from 2-methylimidazole (2.46 g, 0.03 mol), potassium (0.12 g, 0.003 mol), styrene oxide (4.0 g, 0.033 mol), and DMF (15 cm³) at 90 °C for 17 h, had m.p. 117—118° (from benzene) (Found: C, 71.05; H, 7.05; N, 13.7. $C_{12}H_{14}N_2O$ requires C, 71.3; H, 6.95; N, 13.85%).

1-(2-*Hydroxy*-2-*phenylethyl*)-2-*phenylimidazole* (8),* obtained as needles (28 g, 54%) from 2-phenylimidazole (2.9 g, 0.02 mol), potassium (0.08 g, 0.002 mol), styrene oxide (2.6 g, 0.022 mol), and DMF (15 cm³) at 95 °C for 17 h, had m.p. 162—163° (from benzene) (Found: C, 77.0; H, 5.95; N, 10.35. $C_{17}H_{16}N_2O$ requires C, 77.25; H, 6.05; N, 10.6%).

1-(2-*Hydroxy*-2-*phenylethyl*)-4,5-*diphenylimidazole* (9),* obtained as microprisms (6.7 g, 98%) from 4,5-diphenylimidazole (4.4 g, 0.02 mol), potassium (0.08 g, 0.002 mol), styrene oxide (2.64 g, 0.022 mol), and DMF (15 cm³) at 80 °C for 17 h, had m.p. 236—237° (from methanol) (Found: C, 81.35; H, 5.95; N, 8.2. $C_{23}H_{20}N_2O$ requires C, 81.2; H, 5.9; N, 8.25%).

1-(2-*Hydroxy*-2-*phenylethyl*)*benzimidazole* (4),* obtained as needles (10 g, 45%) from benzimidazole (11.8 g, 0.1 mol), potassium (0.5 g, 0.0125 mol), styrene oxide (2.1 g, 0.11 mol), and DMF (40 cm³) at room temperature for 17 h, had m.p. 106—107° (from chloroform–light petroleum) (Found: C, 75.4; H, 6.0; N, 11.6. $C_{15}H_{14}N_2O$ requires C, 75.65; H, 5.9; N, 11.75%). 2-Methoxycarbonylbenzimidazole [18] (0.9 g, 0.005 mol), potassium (0.2 g, 0.005 mol), styrene oxide (0.66 g, 0.0055 mol), and DMF (10 cm³) at 90—100 °C for 2 h also yielded the alcohol (4) (0.4 g, 27%). No acidic products were isolated, and when this reaction was repeated in a dry-box, olefin was detected (*ca.* 50%) in the product by ^1H n.m.r. Acetylation of the alcohol (4) with acetic anhydride and pyridine yielded the acetate * (3.2 g, 52%) as a viscous oil, b.p. 200° at 1 mmHg.

1-(2-Hydroxypropyl)imidazole,* obtained as a pale yellow viscous oil (6.3 g, 90%) from imidazole (3.5 g, 0.05 mol), potassium (0.2 g, 0.005 mol), propene oxide (3.5 g, 0.06 mol), and DMF (5 cm³) at room temperature for 17 h, had b.p. 200—205° at 1 mmHg. Treatment of the hydroxy-compound with thionyl chloride and then base yielded *trans*-1-(prop-1-enyl)imidazole * (0.4 g, 43%) as a colourless oil.

threo-1-(2-*Hydroxy*-1,2-*diphenylethyl*)*imidazole* (10),* obtained as prisms (0.25 g, 14%) from imidazole (0.35 g, 0.005 mol), potassium (0.25 g, 0.05 mol), *cis*-stilbene oxide (1.5 g, 0.0065 mol), and DMF (5 cm³) at room temperature for 2 days, had m.p. 171—172° (from ethyl acetate) (Found: C, 77.3; H, 6.15; N, 10.5. $C_{17}H_{16}N_2O$ requires C, 77.25 H, 6.05; N, 10.6%). This alcohol (10) was also obtained (1.6 g, 60%) from imidazole (0.7 g, 0.01 mol), potassium hydroxide (0.6 g, 0.01 mol), *cis*-stilbene oxide (2.2 g, 0.011 mol), and DMF (10 cm³) at 80 °C for 17 h.

erythro-1-(2-*Hydroxy*-1,2-*diphenylethyl*)*imidazole* (11) * was obtained similarly from *trans*-stilbene oxide in similar yields as prisms, m.p. 171—172° (from ethyl acetate) (Found: C, 77.2; H, 6.15; N, 10.4%). A mixture of the *threo*- and *erythro*-alcohols had m.p. 145—150°.

† For details of Supplementary Publications see Notice to Authors No. 7, *J.C.S. Perkin I*, 1975, Index issue.

[16] E. S. Lewis and C. E. Boozer, *J. Amer. Chem. Soc.*, 1952, **74**, 308; C. C. Lee and A. J. Finlayson, *Canad. J. Chem.*, 1961, **39**, 260; C. C. Lee, J. W. Clayton, D. G. Lee, and A. J. Finlayson, *Tetrahedron*, 1962, **18**, 1395.

[17] Beilsteins Handbuch der Organischen Chemie, Springer–Verlag, 1954, 23, II, 37.

[18] J. Rokach, Y. Girard, and J. G. Atkinson, *Canad. J. Chem.* 1973, **51**, 3765.

N-Benzylimidazole (12). (*a*) Imidazole (0.35 g, 0.0005 mol), potassium (0.2 g, 0.005 mol), *cis*-stilbene oxide (1.5 g, 0.0065 mol), and DMF (10 cm³) at 90 °C for 3 h yielded the product * (0.3 g, 38%), m.p. 71—72° (lit.,[19] 71—72°) as needles (from light petroleum) (Found: C, 75.65; H, 6.15; N, 17.5. Calc. for $C_{10}H_{10}N_2$: C, 75.95; H, 6.35; N, 17.7%).

(*b*) Repetition of the reaction on the same scale with *trans*-stilbene oxide gave an identical product in similar yield.

(*c*) *threo*-1-(2-Hydroxy-1,2-diphenylethyl)imidazole (0.11 g) was stirred with sodium hydride (0.02 g of a 50% dispersion in oil; 1 equiv.) in DMF (5 cm³) at 90 °C for 3 h. The product was precipitated with water and purified as above to yield *N*-benzylimidazole (0.04 g, 60%), identical with that described above.

(*d*) *erythro*-1-(2-Hydroxy-1,2-diphenylethyl)imidazole (0.11 g) when treated as in (*c*) gave an identical product in similar yield.

Attempted Dehydration of 1-(2-*Hydroxy*-2-*phenylethyl*)-*imidazole* (6).—(*a*) The alcohol (0.5 g, 0.0025 mol), heated with potassium ethoxide (0.46 g, 0.005 mol) and DMF (5 cm³) under reflux for 16 h, was unchanged.

(*b*) The alcohol (0.4 g, 0.002 mol) was heated under reflux for 17 h with sodium t-butoxide (0.4 g, 0.004 mol) in DMF (5 cm³). A chloroform extract of the diluted mixture yielded starting material.

(*c*) The alcohol (0.5 g, 0.0025 mol) was heated in dry dimethyl sulphoxide (10 cm³) with dicyclohexylcarbodiimide (redistilled; 2.5 g, 0.0125 mol) at 170—180 °C for 16 h. Starting material was isolated after dilution of the mixture with water.

Attempted Preparation of trans-1-*Styrylimidazole from* 1-(2-*Acetoxy*-2-*phenylethyl*)*imidazole*.—(*a*) The acetyl derivative (1.2 g) was heated under reflux in dry methanol (20 cm³) with potassium methoxide (1.1 g, 2 equiv.) for 2 h. Evaporation and extraction into chloroform yielded 1-(2-hydroxy-2-phenylethyl)imidazole (0.5 g).

(*b*) The acetyl derivative was unchanged after heating overnight in dimethyl sulphoxide at 145 °C.

(*c*) The acetyl derivative (2.4 g) was dissolved in dry dimethyl sulphoxide (10 cm³) and sodium hydride (0.5 g of a 50% dispersion in oil; 1 equiv.) was added. The mixture was gently heated to dissolve the hydride and hydrogen was evolved, and after 5 min the solution was cooled. Dilution and extraction with chloroform yielded an oil; extraction of this with boiling light petroleum left a residue of 1-(2-hydroxy-2-phenylethyl)imidazole (0.2 g) and the cooled solution yielded crystals of the *olefin* (0.12 g, 5%), identical with that synthesized from 4-methoxycarbonyl-*trans*-1-styrylimidazole-5-carboxylic acid [4] and that obtained from the chloro-derivative.

Similar treatment of 1-(2-acetoxy-2-phenylethyl)benzimidazole gave only starting material, sometimes mixed with the corresponding alcohol.

General Method for Dehydration of 1-(2-*Hydroxy*-2-*phenylethyl*)*imidazoles*.—The alcohol (1 g) was added, in portions, with care, to thionyl chloride (20 cm³); the solution was heated under reflux for 1 h, then evaporated under reduced pressure to a solid or gummy residue, which was the hydrochloride salt of the chloro-derivative. Ethanolic potassium hydroxide (10%; 50 cm³) was then added, in small portions, with cooling (ice) and the mixture was stirred for 16 h. Water (50 cm³) was added and the ethanol was evaporated off under reduced pressure to leave the olefin as an oil or a solid which was filtered off or extracted with chloroform.

1-(2-Hydroxy-2-phenylethyl)imidazole (1 g) yielded a solid (0.84 g) which was extracted with boiling light petroleum, from which trans-1-*styrylimidazole* (15) * crystallised (0.72 g, 70%; m.p. 85—86°) as pale yellow plates (Found: C, 77.4; H, 6.0; N, 16.4. $C_{11}H_{10}N_2$ requires C, 77.65; H, 5.9; N, 16.5%), which polymerise on prolonged contact with hot solvents.

1-(2-Hydroxy-2-phenylethyl)benzimidazole (1 g) yielded trans-1-*styrylbenzimidazole* (5) * (0.85 g, 90%), m.p. 95—96°, as prisms (from ether) (Found: C, 81.9; H, 5.55; N, 12.9. $C_{15}H_{12}N_2$ requires C, 81.85; H, 5.45; N, 12.75%).

1-(2-Hydroxy-2-phenylethyl)-2-methylimidazole (1.3 g) yielded 2-*methyl*-trans-1-*styrylimidazole* (16) * (0.6 g, 51%), m.p. 89—90°, as pale yellow rhombic prisms (from light petroleum) (Found: C, 78.15; H, 6.7; N, 15.3. $C_{12}H_{12}N_2$ requires C, 78.25; H, 6.5; N, 15.2%).

1-(2-Hydroxy-2-phenylethyl)-2-phenylimidazole (1.5 g) yielded an oil which was extracted with boiling light petroleum. While the mixture was still warm, a small amount of dark oil separated, from which the supernatant solution was decanted. The solution was allowed to cool to yield 2-*phenyl*-trans-1-*styrylimidazole* (17) * (0.93 g, 77%), m.p. 83—84°, as white rosettes (from light petroleum) (Found: C, 82.65; H, 5.85; N, 11.45. $C_{17}H_{14}N_2$ requires C, 82.95; H, 5.7; N, 11.4%).

1-(2-Hydroxy-2-phenylethyl)-4,5-diphenylimidazole (2.0 g) gave, on addition of water to the alcoholic potassium hydroxide solution, a precipitate of 4,5-*diphenyl*-trans-1-*styrylimidazole* (18) * (1.5 g, 87%), m.p. 217—218° as felted needles (from ethyl acetate) (Found: C, 85.9; H, 5.7; N, 8.6. $C_{23}H_{18}N_2$ requires C, 85.7; H, 5.6; N, 8.7%).

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