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SHORT COMMUNICATIONS

Pyrolysis of Enaminone Derivatives of 1,2-Ethylenediamine

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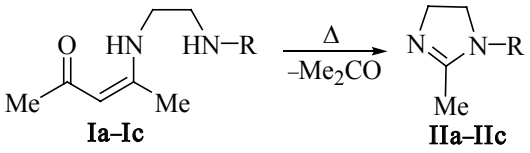
Enaminones are applied as synthons in the preparation of heterocyclic compounds [1, 2]. Their properties depend essentially on the character of the amine fragment of the molecule. In this study we showed that the enaminones obtained from acetylacetone and monosubstituted 1,2-ethylenediamines at 200–250°C suffer a thermal cleavage to give acetone and N-substituted imidazolines.

The reaction apparently proceeds along a mechanism similar to that of the ester cleavage of β-dicarbonyl compounds.



Ia–Ic **IIa–IIc**

Enaminones **Ia–Ic** were heated at 200–250°C and pressure of 15 mm Hg collecting a wide fraction of the decomposition products. Imidazolines **IIa–IIc** were separated by subsequent distillation.

2-(2-Methylimidazolin-1-yl)ethanol (IIa). Yield 65%, bp 166–170°C (10 mm Hg), mp 40–41°C, n_D^{20} 1.514 (of supercooled liquid). IR spectrum, ν, cm^{-1}: 3290, 3079, 2958, 2932, 2776, 1667, 1605, 1499, 1454, 1433, 1362, 1316, 1268, 1203, 1177, 1114, 1078, 1054, 1013, 976, 940, 866, 814, 679, 602, 590, 512, 418. ^{1}H NMR spectrum, δ, ppm: 1.84 s (3H, CH_3), 3.15 t (2H, $NC\underline{H}_2CH_2O$, ^3J 5.4 Hz), 3.30 m (2H, NCH_2), 3.56 m (4H, CH_2N=, CH_2O), 5.78 br.s (1H, OH). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 14.10 (CH_3), 49.29 ($N\underline{C}H_2CH_2O$), 49.89 (NCH_2), 51.46 (CH_2N=), 59.17 (OCH_2), 165.10 (C=N). Found, %: C 56.34; H 9.53; N 21.71. $C_6H_{12}N_2O$. Calculated, %: C 56.22; H 9.44; N 21.86.

1-(2-Vinyloxyethyl)-2-methylimidazoline (IIb). Yield 74%, oily substance, bp 90–92°C (3 mm Hg), d_4^{20} 0.9840, n_D^{20} 1.4942. IR spectrum, ν, cm^{-1}: 3105, 3030, 2915, 2855, 1620, 1605, 1475, 1410, 1350, 1300, 1250, 1180, 1105, 1065, 995, 950, 920, 805, 685, 575, 475. ^{1}H NMR spectrum, δ, ppm: 1.89 s (3H, CH_3), 3.33 m (4H, CH_2NHCH_2), 3.62 m (2H, CH_2N=), 3.72 t (2H, CH_2O, ^3J 5.4 Hz), 3.99 d.d (1H, *cis*-HC=CO, $^2J_{zem}$ 2.2, $^3J_{cis}$ 6.7 Hz), 4.14 d.d (1H, *trans*-HC=CO, $^2J_{gem}$ 2.2, $^3J_{trans}$ 14.3 Hz), 6.41 d.d (1H, OCH=C, $^3J_{cis}$ 6.7, $^3J_{trans}$ 14.3 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 13.97 (CH_3), 45.67 ($N\underline{C}H_2CH_2O$), 49.86 (NCH_2), 51.87 (CH_2N=), 65.37 (OCH_2), 86.53 (=CH_2), 150.99 (OCH=), 163.37 (C=N). Found, %: C 62.47; H 9.23; N 18.05. $C_8H_{14}N_2O$. Calculated, %: C 62.31; H 9.15; N 18.17.

3-Vinyloxy-1-(2-methylimidazolin-1-yl)-2-propanol (IIc). Yield 69%, oily substance, bp 155–158°C (2 mm Hg), n_D^{20} 1.5117. IR spectrum, ν, cm^{-1}: 3309, 3116, 3080, 2928, 2868, 2745, 2582, 1664, 1613, 1494, 1431, 1376, 1321, 1269, 1201, 1117, 1077, 1021, 1004, 975, 943, 819, 746, 704, 647, 595, 554, 470. ^{1}H NMR spectrum, δ, ppm: 1.89 s (3H, Me), 3.19–3.44 m (4H, NCH_2CH_2N=), 3.60 m (2H, $CHC\underline{H}_2N$), 3.67 m (2H, OCH_2), 3.95 m (2H, CHOH), 4.01 d.d (1H, *cis*-HC=CO, $^2J_{gem}$ 2.1, $^3J_{cis}$ 6.7 Hz), 4.17 d.d (1H, *trans*-HC=CO,

$^2J_{gem}$ 2.1, $^3J_{trans}$ 14.2 Hz), 6.46 d.d (1H, OCH=C, $^3J_{cis}$ 6.7, $^3J_{trans}$ 14.2 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 13.89 (Me), 49.80 ($CH\underline{C}H_2N$), 50.62 ($N\underline{C}H_2CH_2N$=), 51.32 ($NCH_2\underline{C}H_2N$=), 67.04 (OCH_2), 69.25 (CHOH), 86.76 (=CH_2), 151.29 (OCH=), 164.87 (NC=N). Found, %: C 58.97; H 8.18; N 15.27. $C_9H_{16}N_2O_2$. Calculated, %: C 58.67; H 8.75; N 15.21.

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