

data (CDCl₃): δ 6.47 (s, 1 H); 3.97 (s, 3 H); 2.50 (s, 4 H).

3-Methoxy-5-isoxazolecarboxaldehyde (4). To the above ether solution of crude 3 was added at 0 °C lithium aluminium hydride (722 mg; 21 mmol). After stirring the mixture at 0 °C for 2 h an aqueous solution of sulfuric acid (40 ml; 20 %) was added. The organic phase was isolated and the aqueous phase extracted with three 40 ml portions of ether. The combined ether phases were dried and concentrated *in vacuo* to give 2.1 g of crude 4. An analytical sample was distilled *in vacuo* in a "kugelrohr" to give 4, the IR and ¹H NMR data of which were consistent with those published for 4.⁹

3-Methoxy-5-hydroxyiminomethylisoxazole (5). A solution of crude 4 (2.1 g; ca. 17 mmol), sodium acetate trihydrate (2.5 g; 18 mmol), and hydroxylammonium chloride (1.3 g; 18 mmol) in aqueous ethanol (50 ml; 50 %) was refluxed for 30 min. The solution was evaporated *in vacuo* and upon addition of water (10 ml) the mixture was extracted with three 20 ml portions of ether. The combined ether phases were dried and evaporated *in vacuo* to give 1.8 g of crude 5, which was shown by TLC to consist of two compounds with $R_F=0.05$ and $R_F=0.03$ (eluent: methylene chloride). ¹H NMR data of crude 5 (DMSO-*d*₆), which are consistent with a mixture (ca. 2:3) of the isomeric oximes: δ 12.5 and 12.0 (two broad signals, 0.4 H and 0.6 H, respectively); 8.08 and 7.60 (two s, 0.6 H and 0.4 H, respectively); 6.72 and 6.38 (two s, 0.4 H and 0.6 H, respectively); 3.93 and 3.91 (two s, a total of 3 H).

3-Methoxy-5-aminomethylisoxazole hydrochloride (6). To a solution of crude 5 (1.8 g; ca. 13 mmol) in aqueous methanol (80 ml; 50 %) was added aluminium amalgam, prepared by treatment of aluminium strips (3.9 g; 144 mmol) with an aqueous solution of mercuric chloride (200 ml; 5 %) for 30 s followed by washing with ethanol. After stirring for 4 h at 25 °C the mixture was filtered. Upon addition of hydrochloric acid (4 ml; 4 N) the filtrate was evaporated *in vacuo* to give 2.5 g of crude 6. Recrystallization (2-propanol) of an analytical sample afforded 6, m.p. 176–177 °C (decomp.) (Ref. 3, m.p. 175–177 °C). $\lambda_{\max} < 210$ nm (CH₃OH). IR data (KBr) cm⁻¹: 3500–3350 (m), 3130 (m), 3050–2400 (s), 1625 (s), 1580 (w), 1510 (s). ¹H NMR data (DMSO-*d*₆): δ 9.6–8.4 (broad s, 3 H); 6.45 (s, 1 H); 4.17 (broad s, 2 H); 3.94 (s, 3 H).

5-Aminomethyl-3-isoxazolol hydrobromide (7). A solution of crude 6 (2.5 g; ca. 15 mmol) in glacial acetic acid (30 ml) containing 43 % of hydrogen bromide was refluxed for 10 min. The solution was evaporated *in vacuo*. Recrystallization (methanol-ether) afforded 1.49 g of 7, m.p. 179–181 °C (decomp.). Anal. C₄H₇BrN₂O₂: C, H, N, Br. $\lambda_{\max} < 210$ nm (CH₃OH). IR data (KBr) cm⁻¹: 3530–3300 (m), 3200–2300 (s), 1635 (s), 1595 (m), 1580 (m), 1545 (s), 1505 (s). ¹H NMR data (DMSO-*d*₆):

δ 10.3–9.0 (broad s, 4 H); 6.25 (s, 1 H); 4.15 (s, 2 H).

5-Aminomethyl-3-isoxazolol (Muscimol) (8). An analytical sample of 7 was passed through a column containing ion exchange resin [Amberlite IRA 400, (OH), 5 ml] using acetic acid (1 M) as an eluent. The fractions containing 8 were concentrated *in vacuo*, and recrystallization (water-ethanol) of the residue gave muscimol (8), m.p. 171–172 °C (decomp.) [Ref. 3, m.p. 172–174 °C (decomp.)]. The IR and mass spectra of 8 were identical with those of an authentic sample.

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