

matograph-mass spectrometer unit. The effluent gas (He) from the gas chromatograph was pumped out in a molecular separator made from Teflon tubing<sup>9</sup> (ID 0.3 mm, 10 m long, at 280°C). The mass spectra were determined using an exponential voltage scan<sup>3</sup> and recorded in 20 sec with a Visicorder 1706 oscillograph.

4-Methyl-5-hydroxyhexanoic acid lactone was synthesized according to the method of Dew and Rai,<sup>10</sup> starting from ethyl acrylate and ethyl 2-methyl-3-ketobutanoate.

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## Synthesis of Mesyloxyacetic Acid and some Derivatives

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In connection with studies concerning the relations of the structures and physical properties of alkylating agents, especially alkyl alkanesulfonates, to their mutagenic and toxic activities,<sup>1,2</sup> attention was given to mesyloxyacetic acid and some of its derivatives. Ethyl mesyloxyacetate<sup>3</sup> has been obtained from the bromoacetate and silver mesylate in acetonitrile but mesyloxyacetic acid,<sup>4</sup> prepared *via* glycolonitrile, has only recently been briefly mentioned. The acid, its amide and its methyl ester have now been prepared from suitable halogen derivatives. Mesyloxyacethydroxamic acid was obtained, in low yield, from mesyloxyacetyl chloride.

*Experimental.* Melting points were determined on a Kofler Micro hot stage. IR-spectra were measured on a Perkin-Elmer IR 221 spectrophotometer. The solvents used were of analytical grade.

*Mesyloxyacetic acid.* A solution of bromoacetic acid (35 g) and an excess of silver mesylate (56 g) in acetonitrile (175 ml) was refluxed for 5 h. The solution was filtered, the precipitate was washed with acetonitrile and the combined solutions were evaporated to dryness *in vacuo*. The residue was extracted with acetone, the extract was evaporated to dryness and the crude acid (32 g) was crystallised from ethyl acetate to give mesyloxyacetic acid as needles, m.p. 114–115° (lit.<sup>4</sup> m.p. 113–114°) (Found: C 23.4; H 3.92; S 20.8. C<sub>3</sub>H<sub>6</sub>O<sub>3</sub>S requires C 23.3; H 3.78; S 22.6)  $\nu_{\max}^{\text{KBr}}$  1740, 1360, 1170 cm<sup>-1</sup>.

*Mesyloxyacetyl chloride.*<sup>4</sup> Thionyl chloride (18 g) was added to mesyloxyacetic acid (9 g). The mixture was refluxed for 3 h and excess thionyl chloride was distilled off in an atmosphere of dry argon. The acid chloride (7.5 g) was distilled, b.p. 99–100°/2 mm,  $n_D^{25}$  1.4608. (Found: C 21.0; H 2.90; S 18.6; Cl 20.4. C<sub>3</sub>H<sub>6</sub>O<sub>2</sub>SCl requires C 20.9; H 2.92; S 18.6; Cl 20.6.)  $\nu_{\max}^{\text{pure}}$  1820, 1360, 1180 cm<sup>-1</sup>.

*Mesyloxyacetamide.* A solution of silver mesylate (12 g) in acetonitrile (50 ml) was added to a solution of iodoacetamide (9 g) in

acetonitrile (35 ml). The mixture was kept for 2 h at 80° and was then worked up as described above. The crude amide (5.7 g) was crystallised from ethyl acetate to give mesyloxyacetamide as prisms, m.p. 118–119°. (Found: C 23.7; H 4.68; N 9.24; S 21.0.  $C_5H_7O_4NS$  requires C 23.5; H 4.61; N 9.15; S 20.9).  $\nu_{max}^{KBr}$  1680, 1350, 1180  $cm^{-1}$ .

Lichtenberger and Faure<sup>5</sup> report m.p. 86° for a substance claimed to be prepared from mesyloxyacetonitrile and formulated as mesyloxyacetamide, but their analytical figures agree with values calculated for ethansulfonyloxyacetamide,<sup>6</sup> m.p. 86°.

*Mesyloxyacethydroxamic acid.* Mesyloxyacetyl chloride (5.2 g, 0.03 mole) was added with stirring and external cooling, to a solution of hydroxylamine prepared by mixing solutions of hydroxylammonium chloride (4.9 g, 0.07 mole) in methanol (50 ml) and of sodium methoxide (sodium, 1.4 g, 0.06 mole) in methanol (40 ml). The reaction mixture was kept for 2 days at room temperature. The solution was filtered and evaporated to dryness *in vacuo*. The residue was extracted with ethyl acetate and the extract was evaporated to dryness. The product (3.8 g) was chromatographed on a column (4 × 50 cm) of polyamide (Woelm) (200 g), using acetone as eluent. The fractions (10 ml) were analysed by TLC as described below. The appropriate fractions (Nos. 70–115) were combined and evaporated to dryness; yield, 0.63 g (13 %). Crystallisation from ethyl acetate gave mesyloxyacethydroxamic acid (0.2 g) as needles, m.p. 103–104°. (Found: C 21.2; H 4.08; N 8.18; S 18.8.  $C_5H_7O_4NS$  requires C 21.3; H 4.17; N 8.28; S 19.0).  $\nu_{max}^{KBr}$  3300, 3175, 1670, 1350, 1180  $cm^{-1}$ .

TLC was performed on Polyamide (Merck) using acetone as eluent. The spots were made visible by spraying with solutions containing iron(III) chloride and an equimolar mixture of iron(III) chloride and potassium hexacyanoferrate(III), respectively, in 50 % aqueous methanol.

*Methyl mesyloxyacetate.* Methyl bromoacetate (b.p. 144–145°/760 mm, prepared analogously to ethyl bromoacetate<sup>7</sup>) (27.6 g) was added to a solution of silver mesylate (41 g) in acetonitrile (200 ml). The mixture was refluxed for 5 h and was worked up by the procedures described above. Distillation gave methyl mesyloxyacetate (20.6 g), b.p. 148–149°/14 mm,  $n_D^{25}$  1.4400 (Found: C 28.8; H 4.81; S 19.1.  $C_5H_7O_4S$  requires C 28.6; H 4.80; S 19.1).  $\nu_{max}^{CHCl_3}$  1760; 1360; 1170.

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## Determination of the Succinic Dehydrogenase Activity of Beef and Human Heart Mitochondrial Acetone Powder with the Help of the Cartesian Diver Technique

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During an investigation of the metabolism of the human heart there was a great need of a micromethod for the determination of succinic dehydrogenase activity. Recently, a biopsy was obtained at a heart operation which made it possible to prepare mitochondrial acetone powder according to Bernath and Singer.<sup>1a</sup> This material was used for measuring the succinic dehydrogenase activity with the Warburg method as well as with the Cartesian diver technique. The assay method was that used by Bernath and Singer,<sup>1b</sup> using phenazine methosulfate as an electron acceptor. A preparation according to the above method was made from beef heart and was used as a reference. For a detailed description of