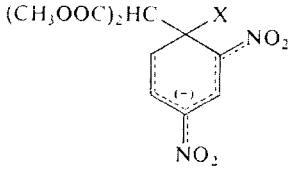
chlorobenzene in methanol faster than methoxide ion. Only the anion of acetoacetate *Ic*, which is a weaker base than methoxide ion by 4 orders of magnitude, reacts about 40 times more slowly. Methoxide ion in methanol is strongly bound to three molecules of the solvent[20]. At least one of these solvating methanol molecules must be split off in order that a bond may be formed to C-atom of dinitrochlorobenzene. In the case of the anions of C-acids *Ia–c* negatively charged oxygen atoms event. nitrogen atom are solvated, but the reacting carbon atom is "free". This fact represents obviously the main reason of the higher reactivity of carbanions. With proceeding reaction a new C—C resp. O—C bond is formed, and the negative charge is transferred to the aromatic nucleus. As a consequence the reacting nucleophile is gradually desolvated.

Reaction of the anion *Ia* with dinitroanisole is, according to preliminary experiments, slower than that with dinitrochlorobenzene almost by 3 orders of magnitude. As the methoxyl group activates the nuclear carbon atom to which it is bound, the activation being the same as or even slightly stronger than that by chlorine[21,22], the rate of formation of the intermediate *IVd* (X = OCH_3) is the same as or somewhat higher than that of *IVe* (X = Cl). It means that decomposition of the intermediate *IVd* is rate limiting, and splitting off of the carbanion is faster than that of methoxide ion by about 3 orders of magnitude.



IVd, e

A partial desolvation of methoxide ion in the activated complex of its reaction with dinitrochlorobenzene is the main reason of the acceleration of this reaction observed when methanol is gradually substituted by dimethyl sulphoxide[23]. In 70% (by vol.) dimethyl sulphoxide the reaction is about 400 times faster than that in methanol. Dependence of log *k* on the acidity function H_- (ref.[24]) (determined by measurements of dissociation constants of aromatic amines) is linear with the slope 0·72. Also the rate constants of reaction of dinitrochlorobenzene with anions of the C-acids *Ia–c* increase with increasing dimethyl sulphoxide concentration though somewhat more slowly (slope of the dependence log *k vs* H_- is about 0·5 for *Ia* and *Ib* and about 0·6 for *Ic*). Therefrom it can be deduced that, also in these reactions, formation of the activated complex is accompanied by considerable desolvation of the carbanion.

Although the reactivity of carbanions increases more slowly than that of methoxide ion, the ratio (dinitroanisole)/(*III*) decreases with increasing dimethyl sulphoxide

concentration, because the equilibrium constant of the reaction of C-acid *I* with methoxide ion considerably increases (Table III). This is obviously true also for weaker C-acids as acetone and cyclohexanone. Therefore, *e.g.* the observed rate constant of the reaction of arylpyridinium salt with cyclohexanone[25] increases with increasing dimethyl sulphoxide concentration faster than that with methoxide ion.

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ELECTROLYTIC REDUCTION OF QUATERNARY SALTS OF SOME ALCOHOLS OF THE PYRIDINE SERIES, OF CORRESPONDING ALKYLPYRIDINES AND ALKENYLPYRIDINES*

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The electrolytic reductions of methomethylsulfates or methiodides of dimethylpyridylmethanols, isopropylpyridines, isopropenylpyridines, 1-(2-pyridyl)-1-cyclopentanol, 1-(4-pyridyl)-1-cyclopentanol, 2-cyclopentylpyridine and 4-cyclopentylpyridine afford the corresponding 1-methyl-C-alkylpiperidine and 1-methyl-C-alkyl-3-piperideine. On electrolytic reduction of methomethylsulfate of dimethyl-3-pyridylmethanol or dimethyl-4-pyridylmethanol 1-methyl-3-isopropylidenepiperidine or 1-methyl-4-isopropenylpiperidine are also formed.

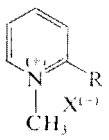
In connection with the study of mixed electrolytic reductions of quaternary pyridinium salts with ketones[1] we considered it useful to investigate electrolytic reductions of quaternary salts of some alcohols of the pyridine series and of quaternary salts of corresponding alkylpyridines, or alkenylpyridines. On electrolytic reduction of dimethyl-2-pyridylmethanol methiodide (*Ia*) and 2-isopropylpyridine methomethylsulfate (*Ib*) on lead electrodes in dilute sulfuric acid a mixture of two substances was obtained in which we demonstrated the presence of 1-methyl-2-isopropylpiperidine (*IVb*) and 1-methyl-6-isopropyl-3-piperideine (*Vb*). The electrolytic reduction of 1-(2-pyridyl)-1-cyclopentanol methomethylsulfate (*Ic*) and methomethylsulfate of 2-cyclopentylpyridine (*Id*) took place in a similar manner under formation of the piperidine derivative *IVd* and the piperideine derivative *Vd*. Electrolytic reductions of methomethylsulfates of dimethyl-4-pyridylmethanol (*IIIa*) and 4-isopropylpyridine (*IIIb*), 1-(4-pyridyl)-1-cyclopentanol methiodide (*IIIc*) and 4-cyclopentylpyridine methiodide (*IIId*) afforded mixtures of derivatives of 1-methylpiperidine (*IXb* or *IXd*, resp.) and 1-methyl-3-piperideine (*Xb* or *Xd*, resp.). The same products as those obtained by electrolytic reduction of the quaternary salts of *IIIa*, and *IIIb* were also obtained by electroreduction of methomethylsulfate of 4-isopropenylpyridine (*IIIe*). Electrolytic reduction of methomethylsulfates of dimethyl-3-pyridylmethanol (*IIa*) and 3-isopropenylpyridine (*IIe*) gave a mixture of 1-methyl-3-isopropylpiperidine (*VIb*), 1-methyl-5-isopropyl-3-piperideine (*VIIb*), 1-methyl-3-iso-

* Part L in the series Studies in the Pyridine Series; Part XLIX: This Journal *40*, 2183 (1975).

propyl-3-piperideine (*VIIIb*) and 1-methyl-3-isopropylidenepiperidine (*VIg*). A similar reduction of methomethylsulfate of 3-isopropylpyridine (*IIb*) gave a mixture of *VIb*, *VIIb* and *VIIIb*, as expected.

1-Methyl-6-cyclopentyl-3-piperideine (*Vd*) was also prepared by reduction of 2--cyclopentylpyridine methiodide (*Id*) with sodium borohydride. Its hydrogenation gave 1-methyl-2-cyclopentylpiperidine (*IVd*). In a similar manner we also prepared 1-methyl-4-cyclopentyl-3-piperideine (*Xd*), 1-methyl-4-cyclopentylpiperidine (*IXd*) and 1-methyl-4-isopropylpiperidine (*IXb*). On reduction of 3-isopropylpyridine methiodide (*IIb*) with lithium aluminum hydride we obtained a mixture of piperideines *VIIb* and *VIIIb* from which the required 1-methyl-5-isopropyl-3-piperideine (*VIIb*) was isolated by gas chromatography.

1-Methyl-3-isopropenylpiperidine (*VIe*) and 1-methyl-3-isopropylidenepiperidine (*VIg*), as well as 1-methyl-4-isopropenylpiperidine (*IXe*) and 1-methyl-4-isopropy-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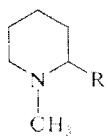
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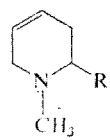
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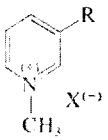
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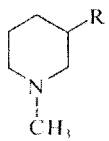
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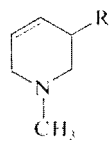
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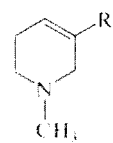
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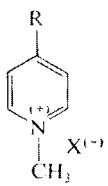
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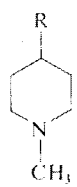
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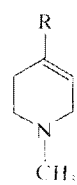
X

For *I–X* *a*) R = $(CH_3)_2C(OH)-$ *b*) R = $(H_3C)_2CH-$ *c*) R =  *d*) R =  *e*) R = $CH_2=C(CH_3)-$ *f*) R =  *g*) R = $(H_3C)_2C=$

lidenepiperidine (*IXg*) were prepared from corresponding tertiary alcohols *VIa* or *IXa*, resp., with thionyl chloride and subsequent dehydrohalogenation. The required alcohols *VIa*, *IXa* were obtained from the corresponding pyridylmethylmethanol by its conversion to methiodide, its reduction with sodium borohydride and subsequent hydrogenation.

EXPERIMENTAL

Gas chromatography was carried out on a Chrom II apparatus (column length 170 cm, diameter 0·6 cm, 20% Tridox on porovina, carrier gas nitrogen). Preparative gas chromatography was carried out on a non-commercial instrument[2]. ^{1}H-NMR spectra were measured on a Varian XL-100-15 instrument at 100·1 MHz in deuteriochloroform; temperature 37°C. The IR spectra were measured with a Perkin-Elmer Model 325 spectrophotometer. The temperature data are not corrected.

Electrolytic Reduction of Dimethyl-4-pyridylmethanol Methomethylsulfate (*IIIa*)

A solution of 25·3 g (0·1 mol) of the quaternary salt (prepared by refluxing a methanolic solution of dimethyl-4-pyridylmethanol[3] with dimethyl sulfate) in 200 ml of a 20% sulfuric acid was reduced on lead electrodes with a 32 Ah current (8 A/h). The catholyte was alkalized and steam distilled. Working up of the distillate gave 7 g (49·9%) of product, b.p. 66–68°C/30 Torr, which according to gas chromatography and comparison with standards represents a mixture of 1-methyl-4-isopropylpiperidine (see below), 1-methyl-4-isopropyl-3-piperideine[4] and 1-methyl-4-isopropenylpiperidine. Electroreduction of the remaining quaternary salts was carried out in a similar manner. The results and the characteristics of the products are listed in Table I.

1-Methyl-4-isopropylpiperidine (*IXb*)

A solution of 2 g of 1-methyl-4-isopropyl-3-piperideine[4] in 150 ml of methanol was hydrogenated on 0·1 g of Adams catalyst. When the consumption of hydrogen ceased (350 ml instead of the calculated 320 ml) platinum was filtered off and a base was isolated from the filtrate which boiled at 73°C/25 Torr. For $C_9H_{19}N$ (141·3) calculated: 76·53% C, 13·56% H, 9·91% N; found: 76·70% C, 13·78% H, 10·28% N.

Reduction of 3-Isopropylpyridine Methiodide (*IIb*) with Lithium Aluminum Hydride

3-Isopropylpyridine methiodide[5] (30 g, 0·115 mol) was added to a mixture of 180 ml of diethyl ether and 9 g lithium aluminum hydride and the mixture was refluxed under stirring for 11 hours. It was then decomposed with dilute hydrochloric acid, the ethereal layer was separated, the aqueous layer alkalized and steam distilled. Working up of the distillate gave 9·8 g (65%) of a mixture boiling at 47–51°C/14 Torr, containing 77% of 1-methyl-3-isopropyl-3-piperideine[5] and 23% of 1-methyl-5-isopropyl-3-piperideine (see below). The last product was isolated by GLC, b.p. 141°C/760 Torr. For $C_9H_{17}N$ (139·2) calculated: 77·63% C, 12·31% H, 10·06% N; found: 77·75% C, 12·48% H, 9·86% N. ^{1}H-NMR spectrum (p.p.m.): $C(CH_3)_2$ 0·91 and 0·93 (two doublets, 7 Hz), N—CH_3 2·32 (s), CH= 5·70 (m), $(CH_3)_2C\underline{H}$ 1·60 (m); corresponds to *VIIb*.

Reduction of 2-Cyclopentylpyridine Methiodide (*Id*) with Sodium Borohydride

A solution of the quaternary salt (7 g) obtained by refluxing of a methanolic solution of 2-cyclopentylpyridine[7] with methyl iodide in 15 ml of water was mixed with a solution of sodium hydroxide (1 g) in water (15 ml) and sodium borohydride (1 g) in 7 ml of water and the mixture was steam distilled. Working up of the distillate gave 2·7 g (67%) of a yellow liquid, b.p. 95°C/10 Torr, which is according to GLC a mixture of three compounds identified as 2-cyclopentylpyridine[7] (9%), 1-methyl-2-cyclopentyl-3-piperideine (31%), and 1-methyl-6-cyclopentyl-3-piperideine (60%).

1-*Methyl*-2-*cyclopentyl*-3-*piperideine*, b.p. 82°C/10 Torr. For $C_{11}H_{19}N$ (165·3) calculated: 79·94% C, 11·59% H, 8·47% N; found: 79·97% C, 11·85% H, 8·32% N. ^{1}H-NMR spectrum (p.p.m.): N—CH_3 2·40 (s), N—CH_2—CH_2 2·58—2·74 (m), N—CH 2·80—3·0 (m), CH= 5·50—5·70 (m) and 5·75—5·95 (m), CH_2—C= 1·95—2·2 (m), other 1·15—1·85.

1-*Methyl*-6-*cyclopentyl*-3-*piperideine*, b.p. 87°C/10 Torr. For $C_{11}H_{19}N$ (165·3) calculated: 79·94% C, 11·59% H, 8·47% N; found: 80·11% C, 11·82% H, 8·50% N. ^{1}H-NMR spectrum (p.p.m.): N—CH_3 2·36 (s), N—CH 3·12—3·25 (m), CH= 5·50—5·90 (m), N—CH_2—CH_2 1·90—2·60 (m), other 1·1—2·68.

TABLE I
Review of Electrolytic Reductions

Quaternary salt		Piperidine, % (ref.)		3-Piperideine, % (ref.)	
Ia	(X=I)	*IVb*	66 (6)	*Vb*	34 (6)
Ib	($X=OSO_3CH_3$)	*IVb*	53 (6)	*Vb*	47 (6)
Ic	(X=I)	*IVd*	63[a]	*Vd*	37[a]
Id	(X=I)	*IVd*	83[a]	*Vd*	17[a]
IIa	($X=OSO_3CH_3$)	*IVb*	51 (6)	*VIIb*	9[a,b]
IIb	($X=OSO_3CH_3$)	*VIb*	69 (6)	*VIIb*	13·5[a,c]
IIe	($X=OSO_3CH_3$)	*VIb*	34 (6)	*VIIb*	17[a,d]
IIIa	($X=OSO_3CH_3$)	*IXb*	4·5[a]	*Xb*	32·5 (4)[e]
IIIb	($X=OSO_3CH_3$)	*IXb*	36[a]	*Xb*	64 (4)
IIIc	(X=I)	*IXd*	25[a]	*Xd*	75[a]
IIId	(X=I)	*IXd*	16[a]	*Xd*	84[a]
IIIe	($X=OSO_3CH_3$)	*IXb*	34[a]	*Xb*	66 (4)
IIIf	(X=I)	*IXd*	19[a]	*Xd*	81[a]

[a] This paper, for properties see Experimental; [b] *VIg* 32[a], *VIIIb* 8 (ref.[5]); [c] *VIIIb* 17·5 (ref.[5]); [d] *VIg* 44[a], *VIIIb* 5 (ref.[5]); [e] *IXe* 63[a].

1-Methyl-2-cyclopentylpiperidine (*IVd*)

A solution of 0·8 g of a mixture of bases, obtained by reduction of 2-cyclopentylpyridine methiodide with sodium borohydride, in 13 ml of acetic acid was hydrogenated on 13 mg of Adams catalyst. The conventional work-up of the mixture gave 0·62 g of product, b.p. 95–97°C/12 Torr, which contained 58% of the required compound and 42% of 2-cyclopentylpiperidine[1]. This mixture was refluxed with 2 ml of formic acid and 1 ml of 40% formaldehyde for 7 hours. The conventional work-up gave, 0·41 g of pure product, b.p. 102°C/18 Torr. For $C_{11}H_{21}N$ (167·3) calculated: 79·02% C, 12·66% H, 8·32% N; found: 79·09% C, 12·73% H, 8·30% N. Compounds *Xd* and *IXd* were prepared in a similar manner.

1-*Methyl-4-cyclopentyl-3-piperideine* (Xd), b.p. 91°C/10 Torr. For $C_{11}H_{19}N$ (165·3) calculated: 79·94% C, 11·59% H, 8·47% N; found: 79·95% C, 11·61% H, 8·39% N. ^{1}H-NMR spectrum (p.p.m.): N—CH_3 2·43 (s), N—CH_2 (t; 6 Hz), N—CH_2—C= 2·83–2·97 (m), CH= 5·33–5·46 (m), CH_2—C= 2·0–2·26 (m), 1·20–1·90.

1-*Methyl-4-cyclopentylpiperidine* (IXd), b.p. 96°C/10 Torr. For $C_{11}H_{21}N$ (167·3) calculated: 79·02% C, 12·66% H, 8·32% N; found: 79·15% C, 12·68% H, 8·26% N.

Dehydration of 1-Methyl-4-(1-hydroxy-2-propyl)piperidine (*IXa*)

Thionyl chloride (3·64 g, *i.e.* 0·03 mol) in diethyl ether (20 ml) was added to a solution of *IXa* (ref.[8]) (3·92 g, 0·025 mol) in diethyl ether (110 ml) and the mixture stirred at room temperature for 20 minutes. After decomposition with 5 ml of water 10 ml of a 40% sodium hydroxide solution were added and the mixture extracted with diethyl ether. The solvent was distilled off, leaving a mixture of substances, b.p. 68–71°C/10 Torr (2·18 g, 63%). Using GLC (preheater 140°C, column 120°C, flow 150 ml of nitrogen/min) the following compounds were isolated:

1-*Methyl-4-isopropenylpiperidine* (IXe), b.p. 60·5–61·5°C/10 Torr (57%). For $C_9H_{17}N$ (139·2) calculated: 77·63% C, 12·30% H, 10·06% N; found: 77·50% C, 12·49% H, 10·02% N. IR spectrum: (values of characteristic bands only): γ(=C—H) 890 vs, ν(C=C) 1644 s, ν(=CH_2)$_{as}$ 3075 m (in cm^{-1}). ^{1}H-NMR spectrum (p.p.m.): CH_3—N 1·07 (s), CH_e—N 1·64–1·94 (m), CH_2= 3·55 (m), other 0·62.

1-*Methyl-4-isopropylidenepiperidine* (IX g), b.p. 66–67·5°C/10 Torr (43%). For $C_9H_{17}N$ (139·2) calculated: 77·63% C, 12·30% H, 10·06% N; found: 77·79% C, 12·43% H, 10·26% N. ^{1}H-NMR spectrum (p.p.m.): CH_3—C 1·55 (s), CH_3—N 2·17 (s), other 2·25.

1-Methyl-3-(1-hydroxy-2-propyl)piperidine (*VIa*)

A solution of 127 g of methyl iodide in 60 ml of methanol was added to a solution of 54·3 g of 3-pyridyldimethylmethanol[9] in 125 ml of methanol and the mixture refluxed for 22 hours. After evaporation 105·4 g (92%) of crude methiodide of 3-pyridyldimethylmethanol were obtained, which was dissolved in 260 ml of water and additioned with 16·5 g of sodium hydroxide dissolved in 260 ml of water and a solution of 16·5 g of sodium borohydride in 130 ml of water. The mixture was stirred under a reflux condenser at room temperature for one hour, then extracted with chloroform, and the extract dried over potassium carbonate. After evaporation of the solvent 42·7 g (76%) of a mixture of reduced amino alcohols were obtained, b.p. 106–109°C/11 Torr, which was hydrogenated in 600 ml of ethanol on 1·28 g of Adams catalyst. From the hydrogenation experiment 33·4 g (77%) of *VIa* were obtained, b.p. 106–108°C/12 Torr. The product was purified by GLC (preheater 150°C, column 130°C, flow of nitrogen 200 ml/min); b.p. 107 to 108°C/14 Torr, m.p. 39–40·5°C. For $C_9H_{19}NO$ (157·25) calculated: 68·74% C, 12·18% H,

8·91% N; found: 68·69% C, 12·28% H, 8·82% N. ^{1}H-NMR spectrum (p.p.m.): CH_3—C 1·12 (s), CH_3—N 2·23 (s), N—CH_eCH_a—CH_2 2·62—2·86 (m), N—CH_eCH_a—CH 2·86—3·08 (m), other 1·35—2·03.

Dehydration of 1-Methyl-3-(1-hydroxy-2-propyl)piperidine (*VIa*)

The reaction was carried out analogously as dehydration of *IXa*. A mixture of substances with b.p. 78—94°C/10 Torr (65% yield) was thus obtained which was submitted to GLC (preheater 155°C, column 130°C, nitrogen flow 135 ml/min) to give:

1-*Methyl*-3-*isopropenylpiperidine* (VIe), b.p. 78—79°C/10 Torr (69%). For $C_9H_{17}N$ (139·2) calculated: 77·63% C, 12·30% H, 10·06% N; found: 77·50% C, 12·45% H, 9·96% N. ^{1}H-NMR spectrum (p.p.m.): CH_3—C 1·70 (bs), CH_3—N 2·23 (s), CH_e—N 2·65—3·10 (m), CH_2= 4·68 (m), other 1·55—2·55.

1-*Methyl*-3-*isopropylidenepiperidine* (VIg), b.p. 85—86°C/10 Torr (31%). For $C_9H_{17}N$ (139·2) calculated: 77·63% C, 12·30% H, 10·06% N; found: 77·38% C, 12·25% H, 10·30% N. ^{1}H-NMR spectrum (p.p.m.): CH_3—C 1·65 (s) and 1·66 (s), CH_3—N 2·24 (s), CH_2—CH_eH_a—N 2·6—2·8 (m), =CH_2—N 2·90 (m), other 1·80—2·52.

The analyses were carried out in the analytical laboratories of our Department (head Dr L. Helešic) and the measurement of ^{1}H-NMR *spectra was carried out under the direction of Dr P. Trška.*

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