# **646.** *Chemical and Physical Properties of Some Sydnones.*

By D. Ll. Hammick and Denys J. Voaden.

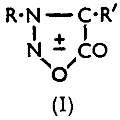
Some condensed-ring and related sydnones have been prepared, and their physical properties compared with those of the simpler derivatives.

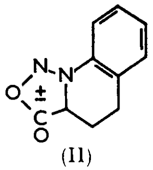
Many mono- and di-substituted derivatives of sydnone (I; R = R′ = H) have now been recorded, and several of their general chemical reactions have been described.[1,2] In the present investigation, the chemical and physical properties of condensed-ring [3,4] and other sydnones [4] are compared with those previously known.

3′,4′-Dihydroquinolino[1′,2′-3,4]sydnone (II; " dihydronaphthosydnone " [3]) has been made from 1,2,3,4-tetrahydro-1-nitrosoquinaldinic acid and acetic anhydride [3] or trifluoroacetic anhydride. The action of thionyl chloride and pyridine on a dioxan solution of the nitroso-acid gave a purple oil, however, and not the expected sydnone.[5]

The dihydroquinolinosydnone (II) was hydrolysed by dilute alkali and by dilute mineral acid [3] in the manner general for sydnones.

Dehydrogenation of the sydnone (II) at the 3′,4′ positions should give the fully aromatic compound, but this dehydrogenation could not be achieved, either catalytically with palladium on carbon alone, or in a solvent with ethyl cinnamate as hydrogen acceptor,[6] or chemically with sulphur, selenium dioxide, *N*-bromosuccinimide, or tetrachloro- or tetramethyl-*p*-benzoquinone.


(I)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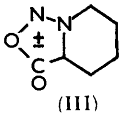
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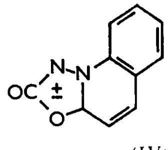
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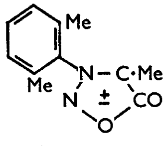
(V)

Further, it was not possible to remove the hydrogen atoms at an earlier stage of the

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[1] Eade and Earl, *J.*, 1946, 591.
[2] Baker and Ollis, *Quart. Rev.*, 1957, **11**, 15.
[3] Hammick, Roe, and Voaden, *Chem. and Ind.*, 1954, 251.
[4] Hammick and Voaden, *Chem. and Ind.*, 1956, 739.
[5] Cf. Baker, Ollis, and Poole, *J.*, 1950, 1543.
[6] Braude, Linstead, and Mitchell, *J.*, 1954, 3578.

synthesis, as tetrahydronitrosoquinaldinic acid was decomposed on reaction with *p*-benzoquinone in boiling tetrahydrofuran.

Kruger [7] has also described the sydnone (II), as well as the less easily accessible isomer derived from isoquinaldinic acid.

*N*-Nitrosopipecolic acid,[8] now isolated as a stable solid, readily gave 1′,2′,3′,4′-tetrahydropyridino[1′,2′-3,4]sydnone (III) on dehydration. This second condensed-ring sydnone possesses the ultraviolet spectrum of a typical alkylsydnone, and like 3-methylsydnone is soluble in cold water. It could not be dehydrogenated.

As sydnones unsubstituted at position 3 would rearrange to the isomeric 1,2,3-oxadiazol-5-ones [9] it seems that the simplest sydnone capable of existence is 3-methylsydnone [10] (I; R = Me, R′ = H), for which sarcosine is a readily available precursor. *N*-Nitrososarcosine can be dehydrated with acetic anhydride giving large, stable crystals of 3-methylsydnone.

When heated with benzylamine, some sydnones give the benzyl-amides of their parent nitroso-acids,[5] but it was not possible to effect this ring-opening with compounds (II) and (III), or with 4-methyl-3-*p*-tolylsydnone.

The ultraviolet spectra of simple sydnones possess a strong band ($\varepsilon$ *ca.* 8000) at 290 mμ (alkyl) to 310 mμ (aryl). Compound (II), in which the sydnone nitrogen is linked to an aryl residue, would be expected to show this band near 310 mμ. However, the band which must be attributed to a sydnone ring actually lies at 340 mμ in 3′,4′-dihydroquinolino[1′,2′-3,4]sydnone. This is very near the values reported for the extensively conjugated 3,4-diarylsydnones—*e.g.*, 333 mμ [12] or 340 mμ [11] for 3,4-diphenylsydnone, and 338 mμ for 3,4-di-*p*-tolylsydnone.[13]

The combustion analysis, behaviour on hydrolysis, and presence of methylene bands in the infrared spectrum confirmed the identity of the dehydration product of tetrahydro-*N*-nitrosoquinaldinic acid, and it is suggested that the extended spectrum is due to enhanced conjugation permitted by molecular geometry.

Dipole-moment studies have shown that the moment of the sydnone ring is coplanar (at least within a small angle) with the phenyl–nitrogen link in 3-phenylsydnone, and that there is evidence for inter-ring resonance in such arylsydnones.[14] Crystallographic investigation shows that solid arylsydnones are "almost coplanar," [15] but presumably there is a certain amount of free rotation of the rings in 3-phenylsydnone. However, models indicated that the ethylene bridge, by forming an extra ring, holds the three rings of compound (II) in a coplanar configuration. From the present evidence, we can deduce that the molecule of the sydnone (II) is somewhat more planar than that of 3-phenylsydnone, and therefore that conjugation *via* the C–N link would be increased. The general form of the phenylsydnone spectrum is retained in that of the isosteric and isoelectronic 3-3′-pyridylsydnone, which is similar in most respects.[16]

The extended ultraviolet spectrum of 3′,4′-dihydroquinolino[1′,2′-3,4]sydnone (II) possesses three maxima—at 240, 260, and 340 mμ, and the mesoionic compound (IV), which has a close structural similarity, exhibits a spectrum of very similar general appearance with maxima at 226, 270, and 325 mμ.[17] However, one must note that compound (IV) possesses a double bond in the interannular bridge; a comparison of the spectrum of (II) with those of (IV) and its reduction product, if accessible, would be valuable.

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[7] Kruger, *Chem. and Ind.*, 1954, 465.
[8] Willstätter, *Ber.*, 1896, **29**, 390.
[9] Baker and Ollis, *Quart. Rev.*, 1957, **11**, 21.
[10] Brookes and Walker, *J.*, 1957, 4416.
[11] Earl, Le Fèvre, and Wilson, *J.*, 1949, S105.
[12] Baker, Ollis, and Poole, *J.*, 1949, 311.
[13] Personal communication from Dr. Ollis.
[14] Hill and Sutton, *J.*, 1949, 746.
[15] Schmidt, *Bull. Res. Council Israel*, 1951, **1**, 121.
[16] Spectral data by courtesy of Prof. Hunsberger.
[17] Hoegerle, *Helv. Chim. Acta*, 1958, **41**, 553.

The ultraviolet spectrum of *m*-tolylsydnone did not differ markedly from those of phenylsydnone and pyridylsydnone, as might be expected from the position of the small substituent. However, the presence of *ortho*-substituents in the phenyl ring, or of groups replacing hydrogen at position 4 of the sydnone ring, modifies more or less profoundly the spectra in the 240—260 mμ region.

*Ultraviolet spectrophotometric data (mμ) for sydnones.*

| Sydnone | $\lambda_{max.}$ | $\varepsilon_{max.}$ | $\lambda_{min.}$ | $\varepsilon_{min.}$ |
|---|---|---|---|---|
| 3-Methyl- ......... | 290 | 6600 | 225 | 170 |
| 3-Butyl- ......... | 289·5 | 6450 | 225 | 800 |
| 3-Hexyl- ......... | 290·5 | 4950 | 242 | 500 |
| Tetrahydropyridino- ............ | 295 | 9100 | 240 | 260 |
| 3-Phenyl- ......... | 235 | 10,000 | 285 | 4250 |
| | 255 * | 7600 | | |
| | 310 | 5650 | | |
| 3-3′-Pyridyl-...... | 236 | 10,000 | 258 | 7100 |
| | 265 | 7300 | 295·5 | 4050 |
| | 312·5 | 4450 | | |
| 3-*m*-Tolyl- ...... | 223 | 8800 | 219 | 8700 |
| | 253 * | 6000 | 285·5 | 4000 |
| | 310 | 4700 | | |
| Dihydroquinolino- | 240 | 10,000 | 230 | 8250 |
| | 260 | 10,200 | 250 | 9650 |
| | 340 | 5400 | 305 | 2450 |
| 3-*o*-Tolyl- ............ | 300 | 6500 | 255 | 2100 |
| 3-*o*-Ethylphenyl-... | 225 * | 4250 | 255 | 2050 |
| | 298·5 | 6500 | | |
| 4-Ethyl-3-phenyl- | 307 | 6200 | 265 | 1450 |
| 4-Methyl-3-*o*-tolyl- | 235 * | 3400 | 255 | 2050 |
| | 302·5 | 9550 | | |
| 4-Methyl-3-*p*-tolyl- | 240 | 6200 | 225 | 4900 |
| | 307 | 6600 | 275 | 2750 |
| 4-Methyl-3-xylyl- | 305 | 7800 | 250 | 1250 |

* Inflexion.

The effects of *ortho*-substitution on the ultraviolet spectra of biphenyls are now well known.[18] Qualitatively, similar effects, especially the steric resistance to interannular conjugation, are observed for the substituted 3-phenylsydnones.

A single methyl group immediately creates a pronounced minimum at 255 mμ in 3-*o*-tolylsydnone; the spectra of 3-*o*-ethylphenyl-, 4-ethyl-3-phenyl-, and 4-methyl-3-*o*-tolyl-sydnone are also very similar. Flexing of the ethyl groups may cause them to show no more steric effect than the methyl substituent.

4-Methyl-3-*p*-tolylsydnone possesses a maximum at 240 mμ, which may be related to a red shift which is favoured by *para*-substituents in benzene derivatives;[19] the sydnone-ring peak is 4 mμ further towards longer wavelength than in the *ortho*-isomer.

However, when the molecule is made more crowded by substitution in both *ortho*-positions, rotation and resonance must be much more hindered, and in the trisubstituted 4-methyl-3-(2,6-dimethylphenyl)sydnone (V), the ultraviolet spectrum begins to resemble that of an alkylsydnone, such as 3-n-butylsydnone.[16] The sydnone peak is conspicuous and in a normal position, but the minimum at 250 mμ is very weak.

The simple alkylsydnone spectrum is of course shown by 3-methylsydnone, 3-hexylsydnone,[16] and the tetrahydropyridinosydnone (III), and is of the form earlier reported for 3-benzylsydnone.[11]

The half-wave potentials for polarographic reduction of these sydnones are in accordance with the suggested steric effects, and will be discussed in relation to the spectra in a separate publication.[20]

## EXPERIMENTAL

All (capillary) m. p.s are corrected unless otherwise stated.

*3′,4′-Dihydroquinolino*[1′,2′-3,4]*sydnone*.[3,7]—(*a*) A solution of 1,2,3,4-tetrahydro-1-nitrosoquinaldinic acid (2 g.) in acetic anhydride (20 ml.) was left for 6 days in the dark, then poured into dilute ammonia and worked up, giving the dihydroquinolinosydnone (1·19 g., 66%), m. p. 146°, and unchanged nitroso-acid (0·58 g.).

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[18] See, *e.g.*, Beaven, in Gray (Ed.), " Steric Effects in Conjugated Systems," Butterworth, London, 1958, Ch. 3.

[19] Gillam and Stern, " An Introduction to Electronic Absorption Spectroscopy in Organic Chemistry," 2nd Edn., Arnold, London, 1957, p. 143.

[20] Zuman and Voaden, to be published; cf. Zuman, *Acta Chim. Acad. Sci. Hung.*, 1959, **18**, 143.

(*b*) Tetrahydronitrosoquinaldinic acid (1·4 g.), stirred in dry ether (25 ml.) at −10°, was treated with trifluoroacetic anhydride (1·5 g.). The suspension, poured into 3N-ammonia (16 ml.) at room temperature, gave the dihydroquinolinosydnone (0·75 g.; 59%), m. p. 145—145·5°, and unchanged nitroso-acid (0·42 g.).

The sydnone was stable to *ca.* 180°. A mull in paraffin showed an infrared C=O band at 5·80μ; a mull in bromonaphthalene showed a $CH_2$ band at 3·40μ.

*Quinaldinic Acid Trifluoroacetate.*—Quinaldinic acid (1·25 g.) in chloroform (25 ml.) at 45° was treated with trifluoroacetic acid (1 ml.). The white precipitate of *quinaldinic acid trifluoroacetate* (2·02 g.; 91%), m. p. 165—166°, crystallised as needles from acetone (Found: C, 50·1; H, 5·4. $C_{12}H_8NO_4F_3$ requires C, 50·2; H, 4·9%).

N-*Nitrosopipecolic Acid.*—Sodium nitrite (11·5 g.) solution was slowly added beneath the surface of stirred aqueous pipecolic acid hydrochloride (30 g.), and the liquid was extracted with ether for 40 hr. After removal of solvent, *N*-nitrosopipecolic acid (14·6 g.; 51%) remained as a viscous yellow oil, which solidified to prisms, m. p. 95—97°, after being dried *in vacuo* ($P_2O_5$). After being thoroughly drained, and washed with a little warm benzene, the nitroso-acid had m. p. 99° (decomp.) (Found: C, 45·4; H, 6·2; N, 17·8. Calc. for $C_6H_{10}N_2O_3$: C, 45·6; H, 6·4; N, 17·7%).

Mixed aqueous solutions of *N*-nitrosopipecolic acid and cupric sulphate slowly (2—3 months) deposited rosettes of bright green needles of the *monohydrated cupric salt* (Found: C, 36·5, 36·3; H, 5·4, 5·5; N, 13·7. $C_{12}H_{20}CuN_4O_7$ requires C, 36·4; H, 5·1; N, 14·2%).

1′,2′,3′,4′-*Tetrahydropyridino*[1′,2′-3,4]*sydnone.*—*N*-Nitrosopipecolic acid (13 g.), warmed with acetic anhydride (30 ml.), left 6 days in the dark, filtered, and evaporated *in vacuo* at 60°, gave a mass of white needles in brown tar (11·4 g.). This was stirred with light petroleum, as much as possible of the tar drained away, and recrystallised twice from ethyl acetate (41% overall recovery), giving a pure, Liebermann-negative *pyridinosydnone*, m. p. 104·5° [Found: C, 51·5; H, 5·6; N, 19·6, 20·2%; *M* (Rast), 162. $C_6H_8N_2O_2$ requires C, 51·4; H, 5·8; N, 20·0%; *M*, 140].

This sydnone began to decompose above 235°. In paraffin mull $\lambda_{CO}$ was at 5·80 μ.

o-*Ethylanilinoacetic Acid.*—*o*-Ethylaniline (4 g.) and chloroacetic acid (2 g.) were boiled under reflux for 2½ hr. with water (25 ml.). After filtration, the solution rapidly deposited o-*ethylanilinoacetic acid* (1·85 g., 49%) as small needles, m. p. 147·5° not raised by further crystallisation (Found: C, 67·2; H, 7·1; N, 8·0. $C_{10}H_{13}NO_2$ requires C, 67·0; H, 7·3; N, 7·8%).

*Preparation of* N-*Nitroso-acids.*—These intermediates were all obtained by dissolving the appropriate imino-acid in N-hydrochloric acid and adding the calculated quantity of sodium nitrite solution with cooling. Initially, the products were often oily but they slowly solidified. They all gave a strong Liebermann test.

The following were thus obtained: o-*ethyl*-N-*nitrosoanilinoacetic acid*, m. p. 64° (Found: C, 57·4; H, 5·8. $C_{10}H_{12}N_2O_3$ requires C, 57·7; H, 5·8%); α-N-*nitrosoanilinobutyric acid* (98%) (from α-anilinobutyric acid [21]), m. p. 90—90·5° (decomp.) (from ether–light petroleum) (Found: N, 13·8. $C_{10}H_{12}N_2O_3$ requires N, 13·5%); N-*nitroso*-o-*toluidinoacetic acid* (69%) (from *m*-toluidinoacetic acid [22]), m. p. 80·5—82·5° (Found: N, 14·4. $C_9H_{10}N_2O_3$ requires N, 14·4%); α-(N-*nitroso*-o-*toluidino*)*propionic acid* (89%) (from α-*o*-toluidinopropionic acid [23]), m. p. 93—95° (decomp.) (Found: C, 57·6; H, 5·9; N, 13·4. $C_{10}H_{12}N_2O_3$ requires C, 57·7; H, 5·8; N, 13·5%); α-(N-*nitroso*-p-*toluidino*)*propionic acid* (88%) (from α-*p*-toluidinopropionic acid [24]), m. p. 110° (decomp.) (as needles from benzene) (Found: C, 57·5; H, 5·9; N, 13·1%); and α-(2,6-*dimethyl*-N-*nitroso*)*propionic acid* by nitrosation of the solution from refluxing of 2,6-dimethylaniline and α-bromopropionic acid in water for 21 hr. (Found: N, 12·0. $C_{11}H_{14}N_2O_3$ requires N, 12·6%).

*Preparation of Arylsydnones.*—The appropriate *N*-nitroso-acid was dehydrated with acetic anhydride, sometimes for *ca.* 1 hr. at 100°, but more often by leaving the reactants for 1—3 days at room temperature, and then working them up with dilute ammonia. The following were obtained: 3-o-*ethylphenylsydnone*, small white needles, m. p. 69·5°, after two crystallisations

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[21] Nastvogel, *Ber.*, 1889, **22**, 1794.
[22] A. Ehrlich, *Ber.*, 1882, **15**, 2011.
[23] Steppes, *J. prakt. Chem.*, 1900, **170**, 498.
[24] Steppes, *J. prakt. Chem.*, 1900, **170**, 495.

from water (Found: C, 63 1; H, 5·6. $C_{10}H_{10}N_2O_2$ requires C, 63·2; H, 5·3%), $\lambda_{CO}$ 5·69 μ; 4-*ethyl*-3-*phenylsydnone* (75% yield), m. p. 75°, $\lambda_{CO}$ 5·78 μ, after 3 crystallisations from ether [Found: C, 63·6; H, 5·5; N, 14·4, 14·6%; *M* (Rast), 212. $C_{10}H_{10}N_2O_2$ requires C, 63·2; H, 5·3; N, 14·7%; *M*, 190]; 3-*o*-tolylsydnone (74%), needles, m. p. 100°, $\lambda_{CO}$ 5·69 μ, from boiling water (lit., m. p. 89°,[25] 96° [26]) (Found: C, 61·3; H, 4·4; N, 15·8. Calc. for $C_9H_8N_2O_2$: C, 61·4; H, 4·6; N, 15·9%); 3-m-*tolyl compound* (61%), small white needles, m. p. 79° (Kofler), $\lambda_{CO}$ 5·66 μ, after 3 recrystallisations from water (Found: C, 61·3; H, 4·8; N, 15·5%); 4-*methyl*-3-o-*tolylsydnone* (90%), m. p. 72°, $\lambda_{CO}$ 5·70 and 5·80 μ [Found: 63·2; H, 5·5; N, 14·6%; *M* (Rast), 170. $C_{10}H_{10}N_2O_2$ requires C, 63·2; H, 5·3; N, 14·7%; *M*, 190]; 4-*methyl*-3-p-*tolylsydnone* (74%), prisms, m. p. 166·5°, $\lambda_{CO}$ 5·67 and 5·76 μ after two recrystallisations from methanol [Found: C, 63·4; H, 5·3; N, 15·2%; *M* (Rast), 191]; 3-(2,6-*dimethylphenyl*-4-*methylsydnone* (crude yield 55%), small prisms, m. p. 120·5—121°, $\lambda_{CO}$ 5·71 and 5·79 μ, after three recrystallisations from water (Found: C, 65·0; H, 6·2; N, 13·5. $C_{11}H_{12}N_2O_2$ requires C, 64·7; H, 5·9; N, 13·7%).

Acid hydrolysis of an ethanolic solution of 4-methyl-3-*p*-tolylsydnone gave *p*-tolylhydrazine hydrochloride, m. p. 222° (decomp.) (lit.,[27] m. p. 222—235°).

α-m-*Toluidinopropiono*-m-*toluidide*.—*m*-Toluidine (20 g.), α-bromopropionic acid (11 g.), and water (100 ml.) were boiled overnight; after free amine and some resin had been removed, and the liquid refrigerated, white crystals slowly appeared, which had m. p. 115° after two recrystallisations from aqueous methanol. These appeared to be α-m-*toluidinopropiono*-m-*toluidide* (Found: C, 76·1; H, 7·3; N, 10·6, 10·7. $C_{17}H_{20}N_2O$ requires C, 76·1; H, 7·5; N, 10·4%). By nitrosating the filtrate from this amide, a very small amount of α-(*N*-nitroso-*m*-toluidino)propionic acid, m. p. 99·5° (decomp.), was obtained.

*Ethyl* α-(2,6-*Dimethylanilino*)*propionate*.—2,6-Dimethylaniline (10 g.) and ethyl α-bromopropionate (5 g.) in benzene (25 ml.) were refluxed 96 hr. Crystals of 2,6-*dimethylanilinium bromide* (3·26 g.; 58%) (Found: C, 47·1; H, 6·2. $C_8H_{12}BrN$ requires C, 47·5; H, 6·0%) were removed, and the filtrate fractionated. *Ethyl* α-(2,6-*dimethylanilino*)*propionate* (2·26 g., 37%) after two distillations had b. p. 146—148°/20—21 mm., 166°/45 mm. (Found: C, 70·2; H, 8·6; N, 6·6. $C_{13}H_{19}NO_2$ requires C, 70·6; H, 8·7; N, 6·3%).

2,6-*Dimethylanilinium bromide monohydrate*, forming laths from dilute hydrobromic acid, had m. p. 232° (slight decomp., loss of water at *ca.* 100°) (Found: Br, 36·3. $C_8H_{12}BrN,H_2O$ requires Br, 36·3%).

N-*Nitrososarcosine*.—Commercial sarcosine solution (450 ml.; *ca.* 12% w/v) was made alkaline with sodium hydroxide, boiled to expel methylamine, and acidified with 10N-hydrochloric acid. After removal of sodium chloride, the filtrate was treated with sodium nitrite (37 g.) solution, and extracted for 36 hr. with ether. This gave *N*-nitrososarcosine (64 g., 88%) as a viscous yellow oil.

The acid exploded (mildly) on attempted distillation at 160°/0·18 mm. Another sample, heated for a time at 40°/15 mm., set on cooling to a yellow slush, which was drained and crystallised thrice from chloroform. This gave solid *N*-nitrososarcosine with m. p. 73—74° (Found: C, 30·8; H, 5·2; N, 23·9. Calc. for $C_3H_6N_2O_3$: C, 30·5; H, 5·2; N, 23·8%).

3-*Methylsydnone*.—*N*-Nitrososarcosine (38 g.) in acetic anhydride (120 ml.), warmed for a few minutes on a steam-bath, became very hot. The mixture was left for 3 days and the excess of anhydride was then removed *in vacuo*. Two distillations of the residue gave a liquid, b. p. 147—148°/0·15 mm., which solidified, m. p. 35—36° (9·8 g.; 31%). The pure, Liebermann-negative sydnone, obtained after two further distillations, had b. p. 125—128°/0·12 mm. (lit.,[10] b. p. 140—142°/0·2 mm.), m. p. 36°, $\lambda_{CO}$ at 5·82 μ (film) (Found: C, 36·1; H, 4·3; N, 28·2. Calc. for $C_3H_4N_2O_2$: C, 36·0; H, 4·0; N, 28·0%).

*Methylhydrazine from* 3-*Methylsydnone*.—3-Methylsydnone (4 g.) and 10N-hydrochloric acid (8 ml.) were heated for 4 hr. at 100°. On addition of a further 3 ml. of acid, and refrigeration overnight, glistening white platelets of *methylhydrazine sesquihydrochloride sesquihydrate* (3·1 g.; 62%) were obtained, with m. p. 118° (sealed tube, lost water from 60°) (Found: C, 9·8; H, 7·2. $CH_6N_2,1\frac{1}{2}HCl,1\frac{1}{2}H_2O$ requires C, 9·5; H, 7·2%). When dried *in vacuo* ($P_2O_5$) the hygroscopic *methylhydrazine sesquihydrochloride* (Found: C, 11·5; H, 7·5; Cl, 54·0; loss, 21·8. $CH_6N_2,1\frac{1}{2}HCl$ requires C, 11·9; H, 7·5; Cl, 53·0; loss, 19·2%) was obtained.

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[25] Hadaček and Švehla, *Publ. Fac. Sci. Univ. Masaryk*, 1954, **357**, 257.
[26] Hashimoto and Ohta, *Bull. Chem. Soc., Japan*, 1958, **31**, 1049.
[27] Hunsberger *et al.*, *J. Org. Chem.*, 1956, **21**, 396.

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