## 164. *The Degradation of Mannich Base Oximes.*

By Zu-Yoong Kyi and Walter Wilson.

Numerous oximes are made from Mannich bases; alkaline degradation of the oxime methiodides yields αβ-unsaturated oximes and $\Delta^2$-*iso*oxazolines, in proportions depending on the nature of the compound and of the solvent. In aqueous alkali, αβ-unsaturated oximes are produced, but, in alcoholic alkali, $\Delta^2$-*iso*oxazolines often predominate. The ultra-violet absorption properties of the unsaturated oximes and $\Delta^2$-*iso*oxazolines are discussed.

$\Delta^2$-Pyrazolines are formed in the reaction between Mannich bases and hydrazines (*e.g.*, Blicke, *Org. Reactions*, 1942, **1**, 319; Nisbet, *J.*, 1945, 126; *J. Pharm. Pharmacol.*, 1952, **4**, 294; Beech, Turnbull, and Wilson, *J.*, 1952, 4686). With hydroxylamine, Mannich bases form normal oximes (Mannich *et al.*, *Ber.*, 1920, **53**, 1876; 1922, **55**, 359, 3515; *Arch. Pharm.*, 1917, **255**, 261; 1926, **264**, 164; 1927, **265**, 589, 598). It seemed that under suitable conditions, $\Delta^2$-*iso*oxazolines might be formed instead of Mannich base oximes, or that the latter could be transformed into *iso*oxazolines.

The series of Mannich bases (I; R′ = Me) and (II; R′ = $CHPh_2$ or Ph) with hydroxylamine under a variety of conditions, yielded the expected oximes (III) and (IV); these were much more stable than the parent bases. The Mannich bases (I; R′ = $CH_2Ph$) derived from dibenzyl ketone behaved less simply; normal oximes were obtained from the morpholino-base ($NR_2$ = morpholino) under the above variety of conditions, and from

the dimethylamino- or diethylamino-bases (R = Me or Et) and hydroxylamine hydrochloride alone or in pyridine. However, with hydroxylamine hydrochloride–sodium acetate, the two latter bases afforded the same unidentified neutral compound, m. p. 101—102°, and small amounts of the expected normal oximes. The compound, m. p. 101—102°, had the composition of the αβ-unsaturated oxime or the *iso*oxazoline of this series, but its properties were not consistent with either structure.

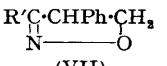
(I) $R'\cdot CO\cdot CHPh\cdot CH_2\cdot NR_2$ $R'\cdot CO\cdot CH_2\cdot CH_2\cdot NR_2$ (II)

(III) $R'\cdot C(:N\cdot OH)\cdot CHPh\cdot CH_2\cdot NR_2$ $R'\cdot C(:N\cdot OH)\cdot CH_2\cdot CH_2\cdot NR_2$ (IV)

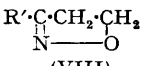
The Mannich base oximes were usually isolated as hydrochlorides, then converted through the free bases into the methiodides. It seemed possible that *iso*oxazolines could arise from the methiodides by internal *O*-alkylation [cf. the formation of *iso*oxazolines from β-chloro-ketones and hydroxylamine (von Auwers and Müller, *J. pr. Chem.*, 1933, **137**, 102) which probably proceeds *via* the β-chloro-oximes].

Only the corresponding αβ-unsaturated oximes (V) were obtained by treating the methiodides of (III; R′ = Me and $CH_2Ph$) with aqueous or alcoholic alkali. The methiodides of (IV; R′ = $CHPh_2$ and Ph) yielded mixtures of the αβ-unsaturated oximes and the *iso*oxazolines on alkaline degradation; the composition of the mixture depended on the solvent. The methiodides of the oximes (IV; R′ = Ph, $NR_2$ = $NMe_2$ or morpholino) with aqueous alkali afforded the αβ-unsaturated oxime (VI; R′ = Ph), but with alcoholic alkali 3-phenyl-$\Delta^2$-*iso*oxazoline (VIII; R′ = Ph) was obtained. The

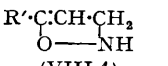
$R'\cdot C(:N\cdot OH)\cdot CPh:CH_2$ (V) $R'\cdot C(:NOH)\cdot CH:CH_2$ (VI)



(VII)

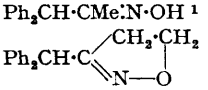
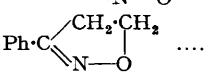
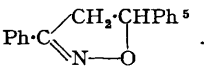


(VIII)



(VIII*A*)

methiodides of the two oximes (IV; R′ = $CHPh_2$, R = Me and Et) behaved similarly, and mixtures were obtained in which either the αβ-unsaturated oxime (VI; R′ = $CHPh_2$) or the *iso*oxazoline (VIII; R′ = $CHPh_2$) predominated. The methiodide of the morpholino-base (IV; R′ = $CHPh_2$, $NR_2$ = morpholino) yielded only the *iso*oxazoline.

| Compound | $\lambda_{max.}$, mμ (ε) | | $\lambda_{min.}$, mμ (ε) |
|---|---|---|---|
| $Ph_2CH\cdot CMe:N\cdot OH$ [1] | <210 * (>20,050) | 260 (550) | 245 (380) |
|  | <210 * (>18,900) | 260 (340) | 250 (270) |
|  | <212 * (>11,500) | 263 (12,800) | 227 (2,800) |
|  | <210 * (>18,700) | 262 (14,200) | 230 (3,850) |
| $Ph\cdot CMe:N\cdot OH$ [2] | — | 245 (10,900) | — |
| $Ph\cdot C(:N\cdot OH)\cdot CH:CHPh$ [3] | <210 * (>16,500) | 221 (12,850)<br>290 (19,500) | 215 (12,400)<br>239 (9,900) |
| $Ph_2C:CH\cdot CMe:N\cdot OH$ [4] | — | 232 (13,700)<br>288 (17,800) | 220 (12,900)<br>255 (8,000) |
| $Ph_2CH\cdot C(:N\cdot OH)\cdot CH:CH_2$ | <210 * (>21,700) | — | — |
| $CH_2Ph\cdot C(:N\cdot OH)\cdot CPh:CH_2$ | <210 * (>22,700) | — | — |
| $CMe(:N\cdot OH)\cdot CPh:CH_2$ | <210 * (>14,100) | — | — |
| $CPh(:N\cdot OH)\cdot CH:CH_2$ | 214 (12,100) | 240 (8,940) (inflexion) | |
| Compound, m. p. 102°, from $NR_2\cdot CH_2\cdot CHPh\cdot CO\cdot CH_2Ph$ | <210 * (>15,200) | — | — |

* Max. beyond the lowest accurate range of instrument used.

[1] M. p. 161—162° (Stoermer, *Ber.*, 1906, **39**, 2303). [2] M. p. 59° (Derick and Bornmann, *J. Amer. Chem. Soc.*, 1913, **35**, 1287). [3] M. p. 115—116° (von Auwers and Müller, *J. pr. Chem.*, 1933, **137**, 71). [4] M. p. 92·5—94° (Wilson and Kyi, *J.*, 1952, 1325). [5] M. p. 74° (von Auwers and Müller, *loc. cit.*).

The *iso*oxazolines obtained in these experiments contained no active hydrogen (Zerewitinoff) and have been assigned $\Delta^2$-structures (VII and VIII); $\Delta^4$-structures (*e.g.*, VIII*A*) of the type extensively employed, but without adequate proof, by Barnes and his co-workers (*J. Amer. Chem. Soc.*, 1945, **67**, 132, 134, 138; 1947, **69**, 3129, 3132, 3135;

cf. Blatt, *ibid.*, 1949, **71**, 1861) are improbable. The *iso*oxazolines are insoluble in acids and in alkalis, whereas the isomeric αβ-unsaturated oximes are usually soluble in alkalis, and have one active hydrogen atom (:N·OH group). The unsaturated oxime (VI; R′ = $CHPh_2$) is anomalous and does not dissolve in alkalis; however, it has one active hydrogen atom, and furthermore, 1 : 1-diphenylacetoxime, $Ph_2CH{\cdot}CMe{:}N{\cdot}OH$, which is closely related, is also insoluble in alkalis.

The ultra-violet absorption characteristics (Table) of the αβ-unsaturated oximes and *iso*oxazolines are consistent with their structures. The *iso*oxazoline (VIII; R′ = $CHPh_2$) and 1 : 1-diphenylacetoxime are similar in structure and have similar light absorption curves. The main absorption band of 3-phenyl*iso*oxazoline (VIII; R′ = Ph) at 263 mμ can be attributed to the Ph·C:N chromophore; similar bands are present in the curves of 3 : 5-diphenyl*iso*oxazoline and of acetophenone oxime which also contain this chromophore. In the αβ-unsaturated oxime series the introduction of a cross-conjugated phenyl group does not notably alter the absorption. However, an additional phenyl group in extended conjugation introduces an intense band near 290 mμ.

Several of the Mannich base oximes and methiodides have been examined by Dr. P. B. Marshall; the pharmacological activities were similar to those already reported for the parent Mannich bases (Marshall, Ahmad, and Weston, *Brit. J. Pharmacol. Chemotherap.*, 1952, **7**, 85), but the oximes often had enhanced spasmolytic activity, and some spasmogenic effects were observed.

## Experimental

Ultra-violet absorption measurements were made on solutions in ethanol, with a Unicam SP500 spectrophotometer.

The Mannich bases were prepared by Wilson and Kyi's method (*J.*, 1952, 1321); some revision of the m. p.s is necessary. 4-Dimethylamino-3-phenylbutan-2-one hydrochloride had m. p. 137—140° (slow heating) or m. p. 149—151° (rapid heating), and on long storage spontaneously changed into an unidentified substance, m. p. 168—170°. 4-Dimethylamino-3-phenylbutan-2-one picrate had m. p. 114—115° after repeated recrystallisation; the methiodide had m. p. 190—191°, resolidified and remelted at 249—252°; the second m. p. is probably that of impure trimethylammonium iodide (lit., m. p. 263°). 3-Phenyl-4-piperidinobutan-2-one hydrochloride had m. p. 168°, resolidified and remelted at 223°, the picrate m. p. 121—123°, and the methiodide m. p. 175—176°. The hydrochloride (2 g.), when heated at 200° for 15 minutes, cooled, triturated with acetone, and recrystallised from ethanol, gave piperidinium chloride (0·8 g., 97%), m. p. 245—246° (lit., m. p. 244—245°). The primary m. p.s of many of these Mannich base salts are decomposition temperatures, not true m. p.s.

*4-Dimethylamino-3-phenylbutan-2-one Oxime Hydrochloride.*—4-Dimethylamino-3-phenylbutan-2-one hydrochloride (23 g.), with hydroxylamine hydrochloride (14 g.) in water (100 c.c.) at 70—80° for 10 minutes, gave, on cooling, the *oxime hydrochloride* (21 g., 77%) which formed glistening flakes, m. p. 179—180°, from ethanol (Found: C, 55·6; H, 7·8; Cl, 13·7. $C_{12}H_{18}ON_2,HCl,H_2O$ requires C, 55·3; H, 8·05; Cl, 13·6%). The free *oxime* formed needles, m. p. 86—87°, from light petroleum (b. p. 60—80°)–ethyl acetate (Found: C, 69·9; H, 8·7. $C_{12}H_{18}ON_2$ requires C, 69·9; H, 8·7%), and the *picrate* had m. p. 145—146° (from ethanol–ethyl acetate) (Found: C, 49·8; H, 4·9. $C_{12}H_{18}ON_2,C_6H_3O_7N_3$ requires C, 49·7; H, 4·8%). The *methiodide* was obtained from the base and excess of methyl iodide in ethanol, as cubes, m. p. 211—212° (Found: C, 44·9; H, 5·9. $C_{12}H_{18}ON_2,CH_3I$ requires C, 44·8; H, 6·0%).

The following oximes were obtained and crystallised similarly, unless otherwise stated: *4-Diethylamino-3-phenylbutan-2-one oxime hydrochloride* (58% yield), m. p. 158—159° (from *iso*propanol) (Found: C, 62·1; H, 8·6. $C_{14}H_{22}ON_2,HCl$ requires C, 62·1; H, 8·5%) [*base*, m. p. 48·5—49° (Found: C, 72·2; H, 9·7. $C_{14}H_{22}ON_2$ requires C, 71·8; H, 9·4%); *picrate*, m. p. 150—151° (Found: C, 51·7; H, 5·4. $C_{14}H_{22}ON_2,C_6H_3O_7N_3$ requires C, 51·8; H, 5·4%); *methiodide*, m. p. 191—192° (Found: C, 48·1; H, 6·65. $C_{14}H_{22}ON_2,CH_3I$ requires C, 47·9; H, 6·65%)].

*3-Phenyl-4-piperidinobutan-2-one oxime hydrochloride* (81% yield), needles, m. p. 201·5—202·5° (from ethanol) (Found: C, 63·7; H, 7·9. $C_{15}H_{22}ON_2,HCl$ requires C, 63·7; H, 8·1%) [*base*, needles, m. p. 96—96·5° (Found: C, 72·9; H, 8·8. $C_{15}H_{22}ON_2$ requires C, 73·2; H, 8·9%); *picrate*, m. p. 172—173° (Found: C, 53·1; H, 5·2. $C_{15}H_{22}ON_2,C_6H_3O_7N_3$ requires C, 53·1; H, 5·3%); *methiodide*, cubes, m. p. 181—182° (Found: C, 49·0; H, 5·9. $C_{15}H_{22}ON_2,CH_3I$ requires C, 49·5; H, 6·4%)].

*4-Morpholino-3-phenylbutan-2-one oxime hydrochloride* (95%), needles, m. p. 185—186° (Found: C, 59·3; H, 7·4. $C_{14}H_{20}O_2N_2$,HCl requires C, 59·05; H, 7·4%) [*base*, needles, m. p. 95—95·5° (Found: C, 68·2; H, 8·1. $C_{14}H_{20}O_2N_2$ requires C, 67·7; H, 8·1%); *picrate*, prisms, m. p. 166—167° (Found: C, 50·4; H, 4·7. $C_{14}H_{20}O_2N_2,C_6H_3O_7N_3$ requires C, 50·3; H, 4·8%); *methiodide*, needles, m. p. 208—209° (Found: C, 46·0; H, 5·8. $C_{14}H_{20}O_2N_2,CH_3I$ requires C, 46·15; H, 5·9%)].

*4-Dimethylamino-1 : 1-diphenylbutan-2-one oxime hydrochloride* (50% yield), m. p. 191—192° (Found: C, 67·3; H, 7·2. $C_{18}H_{22}ON_2$,HCl requires C, 67·8; H, 7·2%) [*base*, m. p. 128—129° (from methanol) (Found: C, 76·4; H, 7·8. $C_{18}H_{22}ON_2$ requires C, 76·6; H, 7·8%); *picrate*, yellow needles, m. p. 166—167° (Found: C, 56·4; H, 4·8. $C_{18}H_{22}ON_2,C_6H_3O_7N_3$ requires C, 56·4; H, 4·9%), and the *methiodide*, needles, m. p. 198—199° (Found: C, 53·9; H, 5·75. $C_{18}H_{22}ON_2,CH_3I$ requires C, 53·8; H, 5·9%)].

*4-Diethylamino-1 : 1-diphenylbutan-2-one oxime hydrochloride* (61% yield), plates, m. p. 192—193° (Found: C, 69·3; H, 8·0. $C_{20}H_{26}ON_2$,HCl requires C, 69·3; H, 7·8%) [*base*, needles, m. p. 98—98·5° (Found: C, 77·5; H, 8·4. $C_{20}H_{26}ON_2$ requires C, 77·4; H, 8·4%); *methiodide*, plates, m. p. 185—186° (from ethanol–acetone) (Found: C, 56·2; H, 6·4. $C_{20}H_{26}ON_2,CH_3I$ requires C, 55·8; H, 6·4%)].

*4-Morpholino-1 : 1-diphenylbutan-2-one oxime hydrochloride* (93% yield), flakes, m. p. 196—197° (from methanol) (Found: C, 65·7; H, 7·0. $C_{20}H_{24}O_2N_2$,HCl requires C, 66·6; H, 6·9%) [*base*, needles, m. p. 162·5—164° (from ethyl acetate) (Found: C, 73·9; H, 7·35. $C_{20}H_{24}O_2N_2$ requires C, 74·1; H, 7·4%); *methiodide*, needles, m. p. 190—192° (Found: C, 53·8; H, 5·8. $C_{20}H_{24}O_2N_2,CH_3I$ requires C, 54·1; H, 5·8%)].

A mixture of 4-dimethylamino-1 : 3-diphenylbutan-2-one hydrochloride (12 g.), hydroxylamine hydrochloride (5·6 g.) and pyridine (5·6 g.) in ethanol (50 c.c.) was boiled under reflux for 2 hours. Water was added and the pyridine was neutralised with hydrochloric acid. The *oxime hydrochloride* (7·6 g., 56%) which crystallised was washed with ether; it gave needles, m. p. 159—160·5°, from water (Found: C, 63·8; H, 7·4; N, 8·4; Cl, 10·75. $C_{18}H_{22}ON_2$,HCl,$H_2O$ requires C, 64·2; H, 7·4; N, 8·3; Cl, 10·55%). The *picrate* formed needles, m. p. 118—120° (Found: C, 57·0; H, 4·3. $C_{18}H_{22}ON_2,C_6H_3O_7N_3$ requires C, 56·4; H, 4·9%), and the *methiodide* needles, m. p. 174—175° (Found: C, 51·8; H, 5·9. $C_{18}H_{22}ON_2,CH_3I,H_2O$ requires C, 51·6; H, 6·1%).

*4-Morpholino-1 : 3-diphenylbutan-2-one Oxime.*—4-Morpholino-1 : 3-diphenylbutan-2-one hydrochloride (17·3 g.), was refluxed for 1½ hours with hydroxylamine hydrochloride (7 g.) and sodium hydroxide (6·5 g.) in 70% ethanol, to give the *oxime* (95%), m. p. 136—137°, from ethanol–chloroform (Found: C, 73·9; H, 7·2. $C_{20}H_{24}O_2N_2$ requires C, 74·1; H, 7·4%). The *methiodide*, which could not be formed at room temperature, was prepared in 63% yield by boiling the base (11 g.) with methyl iodide (10 c.c.) in chloroform (100 c.c.) for 2½ hours, and formed needles, m. p. 180—181° (Found: C, 54·1; H, 5·7. $C_{20}H_{24}O_2N_2,CH_3I$ requires C, 54·1; H, 5·8%).

*3-Dimethylamino-1-phenylpropan-1-one oxime methiodide* was obtained in 86% yield from the oxime (Mannich and Heilner, *Ber.*, 1922, **55**, 359), as plates, m. p. 191—192°, from methanol (Found: C, 43·4; H, 5·6. $C_{11}H_{16}ON_2,CH_3I$ requires C, 43·1; H, 5·7%).

*3-Morpholino-1-phenylpropan-1-one oxime*, prepared in 77% yield from the Mannich base (Harradence and Lions *J. Proc. Roy. Soc. N.S.W.*, 1939, **72**, 233; *Chem. Abs.*, 1939, **33**, 5856), crystallised as flakes, m. p. 149—150°, from ethanol (Found: C, 66·9; H, 7·8. $C_{13}H_{18}O_2N_2$ requires C, 66·7; H, 7·7%) [*methiodide*, needles, m. p. 176—178° (from methanol) (Found: C, 42·9; H, 5·8. $C_{13}H_{18}O_2N_2,CH_3I,H_2O$ requires C, 42·6; H, 5·8%)].

*Degradation of the 4-Dialkylamino-3-phenylbutan-2-one Oxime Methiodides.*—4-Dimethylamino-3-phenylbutan-2-one oxime methiodide (7 g.) was treated with 5% aqueous sodium hydroxide (100 c.c.) at 70—80° for an hour. Acidification then precipitated *3-phenylbut-3-en-2-one oxime*, prisms, m. p. 98—99° [from light petroleum (b. p. 60—80°)] (Found: C, 74·7; H, 7·1; N, 8·5; active H, 0·61. $C_{10}H_{11}ON$ requires C, 74·55; H, 6·8; H, 8·7; active H, 0·62%). The compound is very soluble in most solvents except light petroleum. It is soluble in alkalis and reprecipitated by acids.

It was obtained similarly from the diethylamino- (84% yield), piperidino- (78% yield), and morpholino-analogues (75% yield), or by working in ethanolic media.

*Degradation of 4-Dialkylamino-1 : 3-diphenylbutan-2-one Oxime Methiodides.*—The methiodides of the 4-dimethylamino- and 4-morpholino-compounds were treated with aqueous sodium hydroxide, ethanolic potassium hydroxide, or ethanolic sodium ethoxide. On acidification 1 : 3-*diphenylbut-3-en-2-one oxime* (45—61%) separated, and after recrystallisation from methanol and then from light petroleum (b. p. 60—80°) formed needles, m. p. 82—83°

(Found: C, 80·9; H, 6·1; N, 6·0; active H, 0·41. $C_{16}H_{15}ON$ requires C, 81·0; H, 6·3; N, 5·9; active H, 0·42%). The compound is soluble in alkalis and is reprecipitated by acids.

*Degradation of* 4-*Dialkylamino*-1 : 1-*diphenylbutan*-2-*one Oxime Methiodides.—In aqueous media.* 4-Dimethylamino-1 : 1-diphenylbutan-2-one oxime methiodide (2·5 g.) was treated with 3% aqueous sodium hydroxide (150 c.c.) at 60—70° for an hour. The oil which separated solidified on cooling (m. p. 150—160°, sintered at 80°). Recrystallisation from ethanol gave 1 : 1-*diphenylbut*-3-*en*-2-*one oxime* (0·8 g., 57%), plates, m. p. 166—167° (Found: C, 81·2; H, 6·5; N, 5·9; active H, 0·44. $C_{16}H_{15}ON$ requires C, 81·0; H, 6·3; N, 5·9; active H, 0·42%), which was insoluble in alkalis. Similar degradation of the diethylamino-analogue (2·5 g.) gave an identical product (0·8 g., 62%).

*In ethanolic media.* 4-Dimethylamino-1 : 1-diphenylbutan-2-one oxime methiodide (3 g.) was treated with sodium hydroxide (4·5 g.) in 60% ethanol (150 c.c.) at 60—70° for an hour. Excess of water was added and the mixture was extracted with ether. Evaporation of the ether and recrystallisation of the crude residue (m. p. 83—160°) from ethanol afforded 3-*diphenylmethyl*-$\Delta^2$-iso*oxazoline* (1·3 g., 78%), prisms, m. p. 88—89° (Found: C, 80·8; H, 6·1; N, 6·1; active H, 0. $C_{16}H_{15}ON$ requires C, 81·0; H, 6·3; N, 5·9; active H, 0%). Similar degradation of the diethylamino-analogue (1·25 g.) gave the same product (0·35 g.; 51%).

The morpholino-analogue gave exclusively the *iso*oxazoline (65%) in either aqueous or ethanolic media. The *iso*oxazoline and the αβ-unsaturated oxime could not be interconverted by heating with acids or alkalis.

*Degradation of the* 3-*Dialkylamino*-1-*phenylpropan*-1-*one Oxime Methiodides.*—3-Dimethylamino-1-phenylpropan-1-one oxime methiodide (8 g.) with aqueous sodium hydroxide gave 1-*phenylprop*-2-*en*-1-*one oxime* (0·5 g., 14%), needles, m. p. 109—110° [from light petroleum (b. p. 60—80°)–ethyl acetate] (Found: C, 73·2; H, 6·2; active H, 0·64. $C_9H_9ON$ requires C, 73·5; H, 6·1; active H, 0·68%), and unchanged 3-dimethylamino-1-phenylpropan-1-one oxime (0·4 g., 9%). On treatment with 5% ethanolic potassium hydroxide (80 c.c.), however, it (8 g.) gave 3-phenyl*iso*oxazoline (1·6 g., 45%), flakes [from light petroleum (b. p. 40—60°)], m. p. 65·5—66·5°, not depressed on admixture with an authentic sample made by von Auwers and Müller's method from β-chloropropiophenone and hydroxylamine (*J. pr. Chem.*, 1933, **137**, 125), which contained no active hydrogen. Treatment of the morpholino-compound (8 g.) with aqueous sodium hydroxide gave both the *iso*oxazoline (1·1 g., 35%) and the vinyl ketoxime (0·3 g., 10%); the latter separated on acidification after removal of the *iso*oxazoline. The *iso*oxazoline (51%) was the only product on degradation with ethanolic potassium hydroxide.

*Abnormal Oximation of* 4-*Dialkylamino*-1 : 3-*diphenylbutan*-2-*ones.*—Sodium acetate trihydrate (48 g.) was added to an aqueous solution (200 c.c.) of 4-dimethylamino-1 : 3-diphenylbutan-2-one hydrochloride (36 g.) and hydroxylamine hydrochloride (17 g.). The mixture was heated at 90—100° for 15 minutes; an oil separated and solidified on cooling. Recrystallisation from methanol gave the *compound*, fine needles (16 g., 56%), m. p. 101—102° [Found: C, 81·0; H, 6·3; active H, 0·40%; *M* (Rast), 255. $C_{16}H_{15}ON$ requires C, 81·0; H, 6·3; one active H, 0·42%; *M*, 237]. From the aqueous reaction liquors, 4-dimethylamino-1 : 3-diphenylbutan-2-one oxime hydrochloride (3·5 g., 9%) crystallised. The corresponding diethylamino-base oxime afforded the same product (m. p. 101—102°) on similar treatment.

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