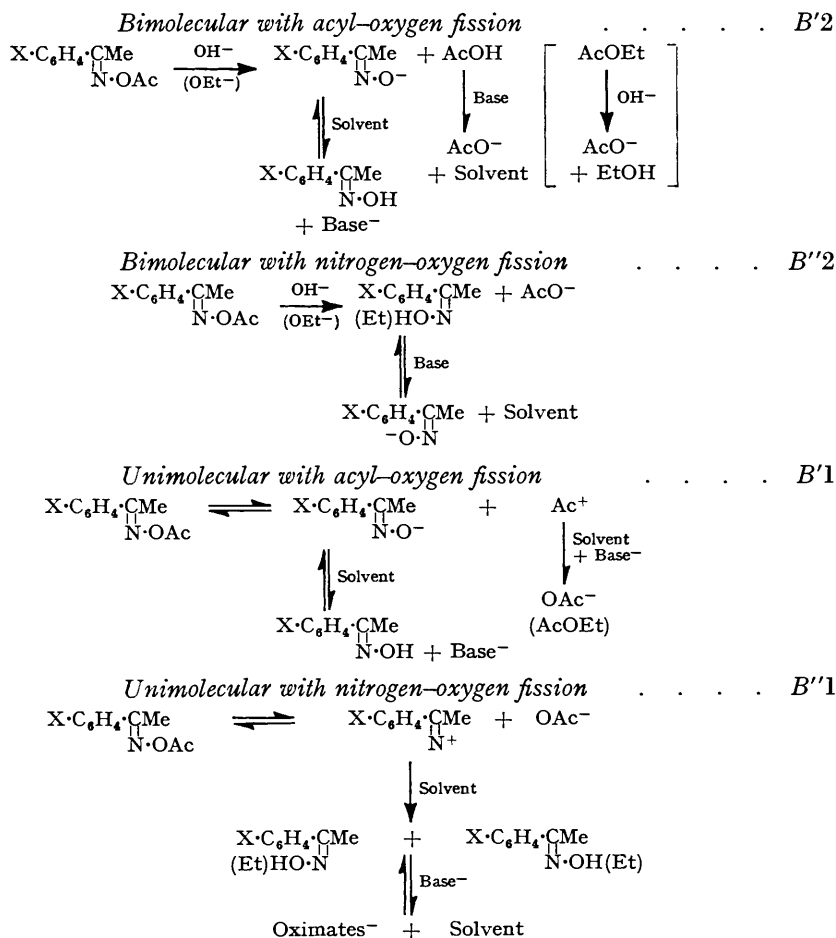**828.** *The Isomerism of the Oximes. Part XLV.* The Alkaline Hydrolysis of Acetophenone Oxime Acetates.*

By O. L. BRADY and J. MILLER.

The hydrolysis of nuclear-substituted acetophenone oxime acetates by OH^- in ethanol–water is kinetically bimolecular but fission of the transition state is facilitated by two different mechanisms. The point of fission is between the acetyl group and the hydroxyimino-oxygen atom.

The effect of varying nuclear substituents is very small and the order of the rate constants, p-OMe $> p$-Me $>$ H $> p$-Br $> m$-$NO_2 > p$-NO_2, is the reverse of that usual in alkaline hydrolysis of esters. The reactions are complicated by an important reversible reactant–product interaction and an unimportant concurrent attack by OEt^-. A general method of correcting for the former has been evolved which could be applied to other similar reactions such as the alkaline hydrolysis of phenol esters.

A STUDY has been made of the hydrolysis of nuclear-substituted acetophenone oxime acetates by OH^- in ethanol–water mixtures. The possibility of a neutral hydrolysis was first eliminated by showing that no hydrolysis occurred in solvent alone. If the hydrolysis is analogous to the usual ester hydrolysis (Day and Ingold, *Trans. Faraday Soc.*, 1941, **37**, 686) the possible reaction mechanisms are :

Bimolecular with acyl–oxygen fission . . . . . B′2

Bimolecular with nitrogen–oxygen fission . . . . B″2

Unimolecular with acyl–oxygen fission . . . . B′1

Unimolecular with nitrogen–oxygen fission . . . . B″1

* Part XLIV, *J.*, 1953, 3612.

In mechanisms $B'2$ and $B'1$ the products are the original oxime and acetoxyl ion from the main attack by OH^-, and the original oxime and ethyl acetate (subsequently hydrolysed) from the subsidiary attack by OEt^-. In mechanism $B''2$ the isomeric oxime and OAc^- are formed by OH^- attack, and the oxime O-ether and OAc^- by OEt^- attack; whilst in $B''1$ a mixture of isomeric oximes and OAc^- are produced by OH^- attack, and a mixture of isomeric oxime O-ethers and OAc^- by OEt^- attack. In the acetophenone series, however, geometrical isomerides are not known, so the failure to obtain an isomeric oxime or O-ether is not significant.
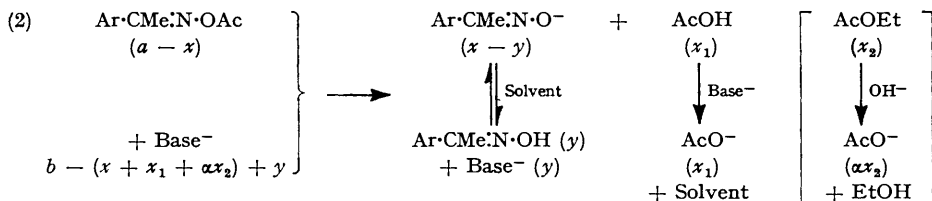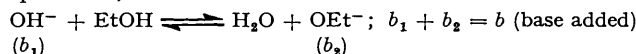
Of the possible mechanisms, those involving nitrogen–oxygen fission are eliminated because oxime O-ethers are not formed and even with OMe^- in pure methanol the original oxime was the sole product; further, large substituent effects would be expected for such fission whereas, in fact, they are very small.

The unimolecular mechanism is eliminated by : (1) the absence of reaction in solvent alone; (2) the absence of a mass-law effect—OAc^- has no effect at all, and oximate ions cause acceleration instead of deceleration; (3) after allowance is made for reversible reactant–product interaction, the second-order rate equation is obeyed; and (4) the activation energies and frequency factors have values typical of bimolecular reactions involving attack by an anion on a neutral molecule, but not of unimolecular reactions.

The mechanism, therefore, is bimolecular, with acyl–oxygen fission ($B'2$).

Calculation of Results.—When the reaction is followed by means of the reduction in the alkaline titre the concurrent attack by OEt^- shows only in the different behaviour of acetic acid and ethyl acetate with OH^-. The stages of the reaction are :

(1) Preliminary equilibrium,

$$OH^- + EtOH \rightleftharpoons H_2O + OEt^-; \quad b_1 + b_2 = b \text{ (base added)}$$
$$(b_1) \qquad\qquad\qquad (b_2)$$

(2)
$$\begin{array}{l}
Ar \cdot CMe \colon N \cdot OAc \\
(a - x) \\
\\
+ \, Base^- \\
b - (x + x_1 + \alpha x_2) + y
\end{array}
\Bigg\} \longrightarrow
\begin{array}{c}
Ar \cdot CMe \colon N \cdot O^- \;+\; AcOH \\
(x - y) \qquad\qquad (x_1) \\
\Big\updownarrow \text{Solvent} \qquad \Big\downarrow Base^- \\
Ar \cdot CMe \colon N \cdot OH \; (y) \qquad AcO^- \\
+ \, Base^- \; (y) \qquad\qquad (x_1) \\
\qquad\qquad + \text{Solvent}
\end{array}
\quad
\begin{bmatrix}
AcOEt \\
(x_2) \\
\Big\downarrow OH^- \\
AcO^- \\
(\alpha x_2) \\
+ \, EtOH
\end{bmatrix}$$

The factor α is introduced since the hydrolysis of the small amounts of EtOAc by OH^- is a slow reaction. Note that $x_1 + x_2 = x$. Alkali is consumed by hydrolysis of oxime acetate (x), neutralisation of acetic acid (x_1), and hydrolysis of ethyl acetate (αx_2). This totals $x + x_1 + \alpha x_2$, and as OEt^- tends to zero this tends to $2x$. Alkali is formed by hydrolysis of oximate ion (y).

The rate constant (k_2) is obtained from the equation

$$dx/dt = k_2(a - x)[b - (x + x_1 + \alpha x_2) + y] \quad . \quad . \quad . \quad . \quad (1)$$

Titration of alkali (phenolphthalein) gives $b - (x_1 + \alpha x_2)$ since oximate ions are indistinguishable from OH^-; and as OEt^- tends to zero, so does this expression tend to $b - x$.

The values of y may be obtained by using the hydrolysis constants (K) of the oximes, thus :

$$K = [Base][Oxime]/[Oximate \; ion] = y[b - (x + x_1 + \alpha x_2) + y]/(x - y)$$

Let
$$(x + x_1 + \alpha x_2) = F; \text{ then } Kx - Ky = yb - yF + y^2,$$

i.e.,
$$y^2 - (F - b - K)y - Kx = 0$$

Then
$$2y = (F - b - K) + [(F - b - K)^2 + 4Kx]^{\frac{1}{2}}$$

For the limiting case where $OEt^- = 0$, $K = y(b - 2x + y)/(x - y)$, and thus $2y = (2x - b - K) + [(2x - b - K)^2 + 4Kx]^{\frac{1}{2}}$.

Previous estimates (Hughes, Ingold, and Shapiro, *J.*, 1936, 227) for OH^- in aqueous ethanol containing much ethanol give $[OEt]$ as small, and in view of the complexity of the corrections it is assumed in all this work that no OEt^- is present.

Equation (1) then simplifies to

$$dx/dt = k_2(a - x)(b - 2x + y)$$

and substituting for y, we have

$$dx/dt = k_2(a - x)[b - 2x + \tfrac{1}{2}\{(2x - b - K) + [(2x - b - K)^2 + 4Kx]^{\frac{1}{2}}\}]$$

whence

$$k_2 = (1/t)\int dx/\{(a - x)[(b - x - n) + (x^2 - bx + n^2)^{\frac{1}{2}}]\} \quad . \quad . \quad . \quad (2)$$

where $n = (b + K)/2$.

This integral was evaluated graphically and gives the rate constant corrected for oxime–base interaction. Without this correction we have the standard form

$$k_2 = (1/t)(b - a) \ln [a(b - x)/b(a - x)] \quad . \quad . \quad . \quad . \quad (3)$$

For all runs calculations were carried out in accordance with equations (3) and (2). The value of k_2 according to equation (3) should diminish throughout the reaction, and the divergence should be larger for those compounds in which the oxime formed is more strongly acid; the value derived from equation (2) should give a true rate constant. The experimental results accord with this.

Discussion.—The compounds investigated were $X \cdot C_6H_4 \cdot CMe\!:\!N \cdot OAc$ where $X = p$-OMe, p-Me, H, p-Br, p-NO$_2$, and m-NO$_2$. In addition the hydrolysis of α-m-nitrobenzald-oxime acetate was studied. In all except two experiments the initial oxime acetate concentration was 0·050M and the base concentrations were between 0·065 and 0·130M. The solvent usually employed was 90% ethanol, *i.e.*, 10 vols. of water per 100 vols. of ethanol–water.

By standard conductimetric procedure the values of the hydrolysis constants in 90% ethanol at 25·0° were found to be $Ph \cdot CMe\!:\!N \cdot OH$ 0·0090, p-NO$_2 \cdot C_6H_4 \cdot CMe\!:\!N \cdot OH$ 0·0018, and p-Me$\cdot C_6H_4 \cdot CMe\!:\!N \cdot OH$ 0·0136. The value of the hydrolysis constant of acetophenone oxime in water can be obtained from Brady and Chokshi's measurements (*J.*, 1929, 947), and by assuming a linear relation between 0 and 90% ethanol and extrapolating to 95% ethanol the hydrolysis constants at various concentrations of ethanol were calculated. The relation between dielectric constant and ethanol content of water–ethanol mixtures is linear. The ratio of the hydrolysis constants $X = p$-NO$_2$/H in the acetophenone oxime series in 90% ethanol is 5·0, and the ratio of the acid dissociation constants $X = p$-NO$_2$/H in the benzaldoxime series is 5·1 in water. In view of this good agreement values for other acetophenone oximes were calculated by using ratios obtained from the acid dissociation constants of the benzaldoximes in water (Brady and Goldstein, *J.*, 1926, 1919; Brady and Chokshi, *loc. cit.*).

Experimentally determined rate constants (corrected) are given in Table 1.

Since measurements at only two temperatures have been made, and in view of the very small substituent effects, it is not considered worth while to compare individual activation energies and frequency factors. The values of these for the whole series are $E = 18,000$—19,000 cals. and log $B = 10·5$—11·5, which are typical for bimolecular reactions involving attack by an anion on a neutral molecule.

The method of correction leads to true rate constants but their numerical value depends upon the excess of base, conveniently expressed by the ratio, base/(oxime acetate). The magnitude of this dependence varies in a regular manner with the compound under investigation and also shows a saturation effect with a large excess of base (cf. Table 2).

These results may be explained by regarding the transition state (I) as relatively stable and the fission as being facilitated by ions whose basicity is greater than that of OAc⁻.

$X \cdot C_6H_4 \cdot \underset{\text{N}}{\overset{\text{C} \cdot \text{Me}}{\|}}$

(I)
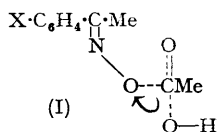
The place where the catalytic action of this base (anion) occurs is at the OH group of the forming acetic acid; conseqeuntly the acetate ion is formed before complete fission has occurred instead of as a separate stage after fission, thus accelerating the process and preventing re-formation of the reactants. This catalysis will explain why the addition of the strongly basic oximate ion increases the rate constant only just less than an equal amount of OH⁻ (Table 2), why the addition of quite large concentrations of acetate ion, or of the more weakly basic chloride ion, has no effect on

TABLE 1. *Hydrolysis of* $X \cdot C_6H_4 \cdot CMe\!:\!N \cdot OAc$ *in aqueous ethanol by sodium hydroxide.*

Initial concns. (M)		Solvent	Hydrolysis		Corr. rate constant
Oxime acetate	Base	(EtOH, %)	const. of oxime	Temp.	$10^3 k_2$ (mole^{-1} sec.$^{-1}$ l.
Acetate, X = H.					
0·050	0·065	95	0·0093	25·0°	2·20
0·050	0·065	90	0·0090	25·0	3·85
0·050	0·130	90	0·0090	25·0	5·80
0·100	0·130	90	0·0090	25·0	3·80
0·025	0·130	90	0·0090	25·0	6·90
0·050	0·105	90	0·0090	25·0	4·75
0·050	0·090	90	0·0090	25·0	4·50
0·050	0·065	80	0·0083	25·0	7·65
0·050	0·065	35	0·0053	25·0	44·4
0·050	0·065	90	0·0090	34·92	10·8
{ 0·050 0·025M-oximate	0·065 [a]	90	0·0090	25·0	4·30
{ 0·050 0·025M-oximate	0·105 [b]	90	0·0090	25·0	5·75
{ 0·050 0·050M-NaOAc	0·130	90	0·0090	25·0	5·80
{ 0·050 0·050M-LiCl	0·065	90	0·0090	25·0	3·85
Acetate, X = *p*-Me.					
0·050	0·130	90	0·0136	25·0	5·80
0·050	0·065	90	0·0136	25·0	4·00
0·050	0·065	90	0·0136	34·92	11·5
0·050	0·065	35	0·0080	25·0	48·9
Acetate, X = *p*-Br.					
0·050	0·130	90	0·0044	25·0	5·50
0·050	0·065	90	0·0044	25·0	3·00
0·050	0·065	90	0·0044	35·0	7·75
Acetate, X = *p*-OMe.					
0·050	0·130	90	0·0158	25·0	6·00
0·050	0·065	90	0·0158	25·0	4·30
0·050	0·065	90	0·0158	34·92	11·0
Acetate, X = *p*-NO₂.					
0·050	0·130	90	0·0018	25·0	4·65
0·050	0·065	90	0·0018	25·0	1·80
0·050	0·065	90	0·0018	34·92	4·95
Acetate, X = *m*-NO₂.					
0·050	0·130	90	0·0027	25·0	4·85
0·050	0·065	90	0·0027	25·0	2·10
0·050	0·065	90	0·0027	34·92	5·40
0·050	0·065	35	0·0016	25·0	22·3
m-NO₂·C₆H₄·CH:N·OAc.					
0·050	0·130	90	0·0004	25·0	4·80

[a] Slight hydrolysis of added oximate ion leads to true initial concns. 0·050, 0·068, 0·022M. [b] True initial concns. 0·050, 0·170, 0·023M.

TABLE 2. *Alkaline hydrolysis of* $X \cdot C_6H_4 \cdot CMe\!:\!N \cdot OAc$.

Y = initial concn. ratio, base/(oxime acetate).

X	*p*-OMe	*p*-Me	H [a]	*p*-Br	*m*-NO₂	*p*-NO₂
Rate const. ($\times 10^3$) :						
at Y = 1·3	4·30	4·00	3·85	3·00	2·10	1·80
at Y = 2·6	'6·00	5·80	5·80	5·50	4·85	4·65
ratio	1·40	1·45	1·51	1·83	2·31	2·58

[a] Also (ratios in parentheses) 4·50 (1·17) at Y = 1·8, 4·75 (1·23) at Y = 2·1, and 6·80 (1·77) at Y = 5·2; with both initial concns. doubled, 3·80 (0·99) at Y = 1·3; 4·30 (1·12) when made up as 2·14M-OH⁻ and 0·46M-oximate ion; 5·75 (1·49) at Y = 2·6 when made up as 2·14M-OH⁻ and 0·46M-oximate ion.

the rate constant (Table 1), and why the catalytic action shows a saturation effect at high base concentrations. It is in this base-catalysed fission process that the normal order of substituent effect appears, the p-NO_2-group causing the greatest and the p-OMe-group the least rise in rate constant with increasing base concentration.

The effect of varying nuclear substituents (see Table 3) is very small, as would

TABLE 3. *Ratio of rate constants for the alkaline hydrolysis of* $X \cdot C_6H_4 \cdot CMe \colon N \cdot OAc$ (0·050M) *at* 25·0°.

Initial concn. (M) of base	EtOH (%)	p-NO_2	m-NO_2	p-Br	H	p-Me	p-OMe
0·130	90	0·80	0·84	0·95	1	1·00	1·03
0·065	90	0·47	0·55	0·78	1	1·04	1·12
0·065	35	—	0·50	—	1	1·10	—

be expected in a reaction occurring at a point in a side chain separated by a considerable distance from the aromatic nucleus; but the order shown in the Table is clear and the reverse of that met in other alkaline hydrolyses of esters; nevertheless, a tendency towards the normal order is clearly occurring at higher relative base strengths whilst the abnormal order is favoured in a more aqueous solvent. This may be explained if, in addition to the base-catalysed fission of the transition state, there is an acid-catalysed fission due to water in the reaction mixture. The water molecule attaches itself through its hydrogen atom to the basic oxygen atom of the forming oximate ion and thus accelerates the fission. In this case the substituent effects would parallel the basicity of the oximes formed, *i.e.*, would be in the order shown above. Since this explanation is identical in character with that offered for the acid-catalysed hydrolysis of benzaldoxime acetates (Brady and Miller, *J.*, 1950, 1234) the similarity of the substituent effects in both order and range was expected.

The substituent effects in this hydrolysis are, therefore, the resultant of two opposing influences, a water-catalysed reaction favoured by electron-releasing substituents and a base-catalysed reaction favoured by electron-attracting substituents, the former being the more important under the conditions employed.

The effect of varying the solvent is greater than the substituent effect (see Table 4)

TABLE 4. *Ratio of rate constants for the alkaline hydrolysis of* $X \cdot C_6H_4 \cdot CMe \colon N \cdot OAc$ *at* 25·0° *and initial concns.* 0·050M-*oxime acetate* 0·065M-*base.*

X	Solvent : ethanol, %			
	95	90	80	35
p-Me	—	1	—	12·25
H	0·57	1	1·99	11·53
m-NO_2	—	1	—	10·61

and is in the opposite direction to that expected from the simple Hughes–Ingold theory of solvent action (*Trans. Faraday Soc.*, 1941, **37**, 657) owing to the specific catalysis by the water molecule already discussed. This is supported by the solvent effect's being greatest for the compound which gives the most basic oxime.

The transition state is highly polar and is probably as solvated as the reactants, and no salt effects would be expected, as was found experimentally.

<div align="center">EXPERIMENTAL</div>

Materials.—The acetophenones were commercial products purified by standard methods, except the *m*-nitro- and *p*-methoxy-compounds which were prepared as described in *Org. Synth.*, **10**, 74, and **5**, 19. The acetophenone oximes were prepared from the ketones in the usual way, except for the *p*-nitro-compound, and crystallised from light petroleum in the cases of the lower-melting compounds or from light petroleum–ether. The *p*-nitro-derivative was prepared by the action of hydroxylamine on *p*-nitrocinnamic acid (Posner, *Annalen*, 1912, **389**, 42).

The acetophenone oxime acetates were prepared from the oximes by the usual procedure, with pure acetic anhydride below 35°. Crystallised from light petroleum–dry ether, the following derivatives formed (generally colourless) rhombic prisms : m-*nitro*-, pale yellow, m. p. 83°;

p-*methyl*-, m. p. 106—107°; p-*bromo*-, m. p. 96°; p-*methoxy*-, m. p. 53—54°; and p-*nitro*-, yellow (from light petroleum–acetone), m. p. 124°. All gave a theoretical saponification value.

Ethanol was purified by Smith's method (*J.*, 1927, 1288) and was better than 99·9% pure. The water was boiled distilled water, kept in a carbon-dioxide-free atmosphere. The thermostat was of standard type with a toluene–mercury regulator and was constant to 0·01°.

The hydrolysis constants (K) were measured by standard conductivity procedures. During the work the specific and equivalent conductivity of sodium hydroxide in 90% ethanol was measured with the following results :

Concn. of NaOH (c) (M)...	0·130	0·065	0·0325	0·01625	0·0065	0·00325	0·0008125
Equiv. conductivity (Λ)	22·25	26·20	30·39	33·30	36·33	38·70	41·54

At infinite dilution, $\Lambda = 44\cdot35$. The Λ–$\sqrt{c}$ curve is not linear.

The reaction products were isolated by dilution of the reaction mixtures with water, neutralisation with dilute hydrochloric acid, and extraction with chloroform; the solvent was removed by a current of dry air and the residue examined by m. p.s and mixed m. p.s. Absence of *O*-ethers was ascertained by complete solubility of the product in aqueous sodium hydroxide.

TABLE 5. *m*-$NO_2\cdot C_6H_4\cdot CMe\colon N\cdot OAc$ = 0·050M; NaOH = 0·130M; 90% ethanol; 25·0°.

x	$b - x$	$a - x$	t (sec.)	$\dfrac{t(b-a)}{2\cdot303}$	$\log_{10}\left[\dfrac{a(b-x)}{b(a-x)}\right]$	$10^3 k_2$ (mole⁻¹ sec.⁻¹ l.)
0·01688	0·11321	0·03312	0	—	—	—
0·02321	0·10679	0·02679	480	16·67	0·0670	4·020
0·02766	0·10234	0·02234	960	33·34	0·1275	3·824
0·03094	0·09906	0·01906	1440	50·51	0·1823	3·645
0·03361	0·09639	0·01639	1920	66·68	0·2359	3·538
0·03590	0·09410	0·01410	2400	83·35	0·2909	3·490
0·03794	0·09206	0·01206	3000	104·2	0·3493	3·352
0·04010	0·08990	0·00990	3600	125·0	0·4247	3·397
0·04235	0·08765	0·00765	4800	166·7	0·5255	3·153
0·05000	0·08000	0·00000	Inf.	—	—	—

Corrected : $a = 0\cdot0500$; $b = 0\cdot13000$; $n = 0\cdot06635$; $n^2 = 0\cdot004402$; $b - n = 0\cdot06365$.

$10^2 x$	$10^2(a - x)$	$10^2(b - n - x)$	$10^2(x^2 - bx + n^2)^{\frac{1}{2}}$	$10^2(b - 2x + y)$
1·688	3·312	4·677	4·993	9·670
2·321	2·697	4·044	4·386	8·430
2·766	2·234	3·599	3·964	7·563
3·904	1·906	3·271	3·657	6·928
3·361	1·639	3·004	3·410	6·414
3·590	1·410	2·775	3·200	5·975
3·794	1·206	2·571	3·016	5·587
4·010	0·990	2·355	2·824	5·179
4·235	0·765	2·130	2·627	4·757
5·000	0·000	1·365	2·006	3·371

$10^2 (a - x)(b - 2x + y)$	$[(a - x)(b - 2x + y)]^{-1}$	Time (sec.)	Total area	$10^3 k_2$ (mole⁻¹ sec.⁻¹ l.)
0·3203	312·2	0	—	—
0·2258	442·8	480	2,385	4·968
0·1690	591·9	960	4,680	4·876
0·1321	757·3	1440	6,884	4·780
0·1051	951·2	1920	9,160	4·770
0·0842	1187	2400	11,690	4·869
0·06738	1484	3000	14,410	4·804
0·05127	1950	3600	18,110	5·030
0·03639	2748	4800	23,360	4·868
—	—	Inf.	—	—

Average value of $k_2 = 4\cdot85 \times 10^{-3}$ mole⁻¹ sec.⁻¹ l.

Table 5 records a typical experiment.

The following are examples of the rate constants obtained in a number of other experiments :

$C_6H_5\cdot CMe\colon N\cdot OAc$ = 0·050M; NaOH = 0·0650M; 90% ethanol; 25·0°.

Uncorr. : $k_2 \times 10^3$ (mole⁻¹ sec.⁻¹ l.) = 3·275, 2·930, 2·866, 2·786, 2710, 2·677, 2·660, 2·554, 2·433, 2·283, 2·208, 1·850 from 600 to 82,200 sec.

Corr. : 4·078, 3·797, 3·852, 3·934, 4·022 from 600 to 3600 sec.

p-Me·$C_6H_4\cdot CMe\colon N\cdot OAc$ = 0·500M; NaOH = 0·1300M; 90% ethanol; 25·0°.

Uncorr.: $k_2 \times 10^3$ (mole^{-1} sec.$^{-1}$ l.) = 4·559, 4·446, 4·351, 4·288, 4·271, 4·203, 4·111, 4·004, 3·773 from 480 to 6600 sec.

Corr. : 5·508, 5·558, 5·608, 5·669, 5·793, 5·864, 5·867 from 480 to 4860 sec.

p-Br·C_6H_4·CMe:N·OAc = 0·050M; NaOH = 0·1300M; 90% ethanol; 25·0°.

Uncorr. : $k_2 \times 10^3$ (mole^{-1} sec.$^{-1}$ l.) = 4·206, 4·156, 4·077, 4·014, 3·970, 3·898, 3·868, 3·795, 3·663, 3·611 from 480 to 9000 sec.

Corr. : 5·175, 5·330, 5·409, 5·489, 5·5911, 5·675, 5·804 from 480 to 3600 sec.

p-MeO·C_6H_4·CMe:N·OAc = 0·050M; NaOH = 0·130M; 90% ethanol; 25·0°.

Uncorr. : $k_2 \times 10^3$ (mole^{-1} sec.$^{-1}$ l.) = 4·505, 4·470, 4·302, 4·274, 4·247, 4·203, 4·138, 4·063, from 480 to 4800 sec.

Corr. : 5·798, 5·899, 5·808, 5·918, 6·006, 6·072, 6·090, 6·146 from 480 to 4800 sec.

p-NO_2·C_6H_4·CMe:N·OAc = 0·050M; NaOH = 0·1300M; 90% ethanol; 25·0°.

Uncorr.; $k_2 \times 10^3$ (mole.$^{-1}$ sec.$^{-1}$ l.) = 3·934, 3·626, 3·519, 3·224, 3·127, 2·807, 2·381 from 600 to 12,690 sec.

Corr. : 4·890, 4·735, 4·738, 4·483, 4·501, 4·316 from 600 to 5400 sec.

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