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Mass Spectral Fragmentation Pattern of 3-(2′-Hydroxyethyl)quinolin-2(1*H*)-one and its Derivatives

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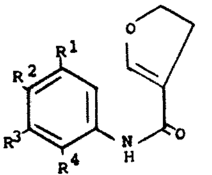
Fragmentation patterns resulting from electron impact ionization of 3-(2′-hydroxyethyl)quinolin-2(1*H*)-one, three of its monosubstituted derivatives and four of its disubstituted derivatives were studied. The molecular ion of quinolinone-2-ethanol undergoes initial fragmentation with the loss of OH$^{\cdot}$, H_2O, CO, $^{\cdot}$CHO, CH_2O, $CH_2OH^{\cdot}$, $CH_2{=}CHOH$ and HCNO species. The $[M - CHO]^+$ ion is tentatively suggested to have been formed by the expulsion of H$^{\cdot}$ from the $[M - CO]^{+\cdot}$ ion and the $[M - CHO]^+$ peak may be considered as diagnostic of a 2-quinolone-3-ethanol.

INTRODUCTION

In connection with our syntheses of furo-, thieno-, selenolo-, tellurolo- and 1*H*-pyrrolo[2,3-*b*]quinolines, we needed several 3-(2′-hydroxyethyl)quinolin-2(1*H*)-ones as starting materials. Two convenient one-step procedures for the transformation of *N*-aryl-4,5-dihydrofuran-3-carboxamides (**1**) into the 3-(2′-hydroxyethyl)quinolin-2(1*H*)-ones (**2**) were developed in our laboratory.[1] One method involves the reaction of **1** with a Lewis acid and other consists of a photochemical rearrangement.



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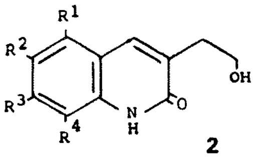

2

	R¹	R²	R³	R⁴
a	H	H	H	H
b	H	Br	H	H
c	Cl	H	H	H
d	H	H	Cl	H
e	H	Cl	H	H
f	H	Cl	Cl	H
g	H	H	Cl	CH_3
h	Cl	H	H	OCH_3
i	H	OCH_3	OCH_3	H

RESULTS AND DISCUSSION

Having obtained the quinolinonylethanols in several varieties,[2] it was of interest to study their mass spectra. Mass spectra of compounds **2a**–**i** are reported in Tables 1–8. Structures written for the fragment ions are nominal only, but are intended to relate the fragmentation processes to the structure of the intact molecule.

Table 1. Mass spectrum of 2a: principal fragments, *m/z* (relative intensity (%) in parentheses)

190 (8), 189 (60), 188 (18), 187 (17), 186 (15), 185 (6), 174 (8), 173 (5), 172 (30), 171 (52), 170 (48), 169 (10), 161 (5), 160 (31), 159 (100), 158 (70), 155 (8), 154 (8), 146 (9), 145 (18), 144 (4), 143 (12), 142 (12), 141 (26), 140 (13), 132 (10), 131 (24), 130 (74), 129 (16), 128 (28), 127 (14), 126 (8), 125 (9), 119 (6), 117 (14), 116 (10), 115 (25), 114 (6), 113 (16), 112 (8), 111 (16), 105 (12), 103 (22), 102 (24), 101 (10), 99 (20), 97 (25), 96 (9), 95 (16), 92 (8), 91 (16), 90 (6), 89 (17), 85 (48), 83 (34), 82 (12), 77 (26), 76 (7), 75 (12), 73 (17), 71 (60), 70 (28), 69 (48), 57 (52), 55 (50), 51 (12), 43 (56), 42 (20), 41 (36), 39 (20)

As expected, the parent compound **2a** ($m/z = 189$) gives $M^{+\cdot}$, $[M - H_2O]^{+\cdot}$, $[M - CH_2O]^{+\cdot}$, $[M - CH_2OH]^+$ and $[M - CH_2{=}CHOH]^{+\cdot}$ ions which are characteristic of a *β*-arylethanol.[3] The $(M - CH_2{=}CHOH)^{+\cdot}$ ion (*m/z* 145) further decomposes with the expulsion of CO, as has been reported for quinolin-2-ones[4a] and pyrid-2-ones,[5] to form an ion at *m/z* 117. The $[M - CH_2O]^{+\cdot}$ (*m/z* 159) and $[M - CH_2OH]^{+\cdot}$ (*m/z* 158) ions lose CHO radicals and

Table 2. Mass spectrum of 2*b*: principal fragments, *m/z* (relative intensity (%) in parentheses)

270 (2), 269 (18), 268 (5), 267 (19), 266 (12), 265 (19), 264 (10), 263 (17), 252 (13), 251 (28), 250 (23), 249 (27), 248 (14), 240 (14), 239 (84), 238 (54), 237 (83), 236 (42), 224 (3), 223 (6), 221 (7), 220 (1), 219 (4), 211 (5), 210 (22), 209 (10), 208 (26), 207 (5), 206 (4), 197 (4), 195 (4), 187 (1), 173 (4), 171 (5), 169 (6), 159 (9), 158 (20), 157 (48), 156 (10), 142 (4), 141 (6), 140 (12), 139 (1), 131 (6), 130 (35), 129 (100), 128 (46), 127 (13), 118 (8), 117 (7), 116 (10), 115 (22), 114 (16), 113 (10), 112 (2), 104 (5), 103 (22), 102 (52), 101 (23), 100 (7), 99 (6), 98 (4), 89 (10), 88 (12), 87 (8), 86 (6), 85 (6), 79 (5), 78 (10), 77 (20), 76 (19), 75 (22), 74 (13), 65 (10), 64 (13), 63 (21), 62 (12), 61 (4), 57 (6), 55 (6), 52 (13), 51 (33), 50 (20), 45 (7), 44 (8), 43 (12), 41 (7), 39 (19), 32 (3), 31 (51)

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CO molecules, respectively, presumably to form a more able quinolinonium ion at *m*/*z* 130.[4b,4c]

Table 3. Mass spectrum of 2*c* + 2*d*: principal fragments, *m*/*z* (relative intensity (%) in parentheses)

225 (5), 224 (2), 223 (10), 207 (5), 206 (5), 205 (15), 204 (7), 196 (7), 195 (25), 194 (83), 193 (88), 192 (15), 180 (4), 179 (5), 178 (3), 177 (5), 176 (2), 175 (3), 169 (12), 168 (8), 167 (17), 166 (33), 165 (5), 164 (80), 157 (10), 155 (12), 151 (5), 150 (3), 149 (37), 141 (17), 137 (2), 129 (13), 128 (17), 127 (20), 126 (10), 125 (10), 124 (3), 123 (10), 119 (8), 113 (20), 112 (13), 111 (20), 101 (10), 99 (25), 98 (17), 97 (33), 96 (17), 95 (20), 85 (72), 71 (91), 57 (88), 43 (100), 41 (91), 55 (83)

Table 4. Mass spectrum of 2*e*: principal fragments, *m*/*z* (relative intensity (%) in parentheses)

226 (4), 225 (26), 224 (15), 223 (93), 222 (13), 210 (4), 209 (3), 208 (28), 207 (56), 206 (59), 205 (70), 204 (57), 203 (9), 197 (3), 196 (32), 195 (60), 194 (73), 193 (100), 192 (63), 191 (4), 190 (5), 188 (9), 182 (7), 181 (12), 180 (23), 179 (43), 178 (12), 177 (37), 176 (15), 175 (23), 170 (6), 169 (6), 168 (7), 167 (20), 166 (52), 165 (49), 164 (64), 163 (20), 162 (32), 159 (15), 158 (48), 157 (53), 156 (24), 154 (4), 153 (17), 152 (14), 151 (37), 150 (9), 149 (14), 142 (8), 141 (14), 140 (24), 139 (4), 138 (5), 137 (4), 130 (16), 129 (21), 128 (26), 127 (6), 126 (4), 123 (5), 116 (4), 115 (10), 114 (9), 113 (8), 111 (4), 103 (10), 102 (29), 101 (28), 100 (5), 99 (7), 91 (4), 89 (12), 78 (5), 77 (16), 76 (12), 75 (22), 74 (10), 65 (6), 63 (23), 62 (11), 52 (13), 51 (27), 50 (17), 43 (24), 41 (12), 39 (24), 31 (36), 30 (4), 29 (28), 28 (25), 27 (20)

The presence of the 2-oxo group instigates several fragmentation pathways. One is the formation of the $[M-OH]^+$ ion ($m/z = 172$), which collapses further with the elimination of $CH_2{=}CHOH/CH_2O$ species to form subsequent ions with *m*/*z* 140, 142, 128, 115, etc. The loss of a water molecule from the molecular ion can be visualized to occur in two ways, to give either (i) 3-vinyl-2-oxoquinoline ion formed by β-hydrogen elimination[3] or (ii) 2,3-dihydrofuro[2,3-*b*]quinoline ion. The vinylic ion is suggested to fragment further via the ejection of acetylene in addition to CO, as expected. The $[M-H_2O]^{+\bullet}$ ion (*m*/*z* 171) eliminates an H atom and further collapses by the loss of CO/C_2H_2.

Table 5. Mass spectrum of 2*f*: principal fragments, *m*/*z* (relative intensity (%) in parentheses)

261 (3), 260 (3), 259 (17), 258 (5), 257 (26), 256 (3), 243 (5), 242 (12), 241 (27), 240 (25), 239 (39), 238 (23), 231 (11), 230 (16), 229 (65), 228 (37), 227 (100), 226 (22), 222 (2), 221 (0.5), 215 (3), 214 (5), 213 (7), 211 (8), 210 (2), 209 (5), 205 (2), 204 (3), 203 (3), 202 (5), 201 (5), 200 (23), 199 (9), 198 (33), 197 (2), 196 (5), 195 (3), 194 (6), 193 (11), 192 (16), 191 (9), 187 (5), 186 (2), 185 (7), 176 (4), 175 (3), 174 (8), 173 (2), 166 (4), 165 (7), 164 (15), 163 (15), 162 (13), 157 (7), 156 (17), 149 (5), 148 (4), 140 (5), 138 (5), 137 (6), 136 (12), 135 (7), 129 (6), 128 (34), 127 (20), 126 (14), 123 (7), 115 (3), 114 (6), 113 (9), 111 (5), 102 (12), 101 (16), 100 (11), 99 (16), 98 (6), 97 (5), 87 (10), 77 (8), 76 (7), 75 (17), 74 (12), 73 (6), 63 (9), 62 (7), 52 (6), 51 (14), 50 (10), 43 (5), 41 (3), 39 (10), 32 (2), 31 (55)

The high abundance of the $M-CHO^\bullet$ peak ($m/z = 160$ (31%)) in the spectrum of **2a** is of interest. The $[M-CHO]^+$ ion may be formed by the loss of an H radical from the $[M-CO]^{+\bullet}$ ion. This M − 29 peak may be considered as diagnostic of a 3-(2′-hydroxyethyl) quinolin-2-one, as such an M − 29 ion is not observed for any β-aryl-substituted ethanols.[3]

Table 6. Mass spectrum of 2*g*: principal fragments, *m*/*z* (relative intensity (%) in parentheses)

240 (3), 239 (23), 238 (16), 237 (64), 236 (19), 235 (3), 224 (6), 223 (10), 222 (27), 221 (55), 220 (58), 219 (75), 218 (55), 217 (11), 211 (5), 210 (19), 209 (70), 208 (53), 207 (100), 206 (63), 205 (6), 204 (5), 202 (4), 201 (3), 197 (5), 196 (9), 195 (5), 194 (10), 193 (13), 192 (6), 191 (8), 190 (6), 189 (5), 188 (3), 185 (3), 184 (4), 183 (6), 182 (6), 181 (7), 180 (19), 179 (11), 178 (37), 177 (10), 176 (8), 173 (7), 172 (30), 171 (15), 170 (7), 169 (8), 168 (8), 167 (7), 166 (6), 165 (6), 164 (9), 163 (5), 162 (4), 158 (7), 157 (6), 156 (12), 155 (7), 154 (12), 153 (7), 152 (7), 151 (6), 149 (6), 145 (4), 144 (12), 143 (22), 142 (22), 141 (22), 140 (15), 139 (5), 138 (4), 130 (4), 129 (8), 128 (10), 127 (11), 126 (5), 125 (6), 121 (9), 118 (4), 117 (5), 116 (9), 115 (20), 114 (6), 113 (8), 111 (6), 102 (10), 101 (10), 99 (11), 97 (13), 91 (14), 89 (16), 87 (10), 79 (9), 78 (8), 77 (26), 76 (9), 75 (16), 74 (8), 73 (27), 71 (39), 70 (16), 65 (17), 63 (19), 55 (38), 45 (39), 43 (39), 41 (38), 39 (33), 36 (5), 31 (31), 30 (7), 29 (39), 28 (32), 27 (30)

Like the parent compound **2a**, its derivatives also give characteristic $[M-OH]^+$, $[M-H_2O]^{+\bullet}$, $[M-CO]^{+\bullet}$, $[M-CHO]^+$, $[M-CH_2O]^{+\bullet}$, $[M-CH_2OH]^+$, $[M-HCNO]^{+\bullet}$ and $[M-CH_2{=}{=}CHOH]^{+\bullet}$ ions. The relative abundances of the possible first formed ions including the molecular ions are given in Table 9. In the halo derivatives **2b**–**h**, HX or X is eliminated from the molecular ion or from $[M-OH]^+$, $[M-H_2O]^{+\bullet}$, $[M-CHO]^+$, $[M-CH_2O]^{+\bullet}$ and $[M-CH_2OH]^+$ ions and other subsequent ions[6,7] (Table 10).

Table 7. Mass spectrum of 2*h*: principal fragments, *m*/*z* (relative intensity (%) in parentheses)

256 (8), 255 (47), 254 (29), 253 (79), 252 (21), 240 (7), 239 (7), 238 (38), 237 (58), 236 (64), 235 (62), 234 (55), 233 (6), 226 (18), 225 (62), 224 (62), 223 (100), 222 (58), 221 (11), 220 (10), 219 (4), 211 (6), 210 (18), 209 (20), 208 (35), 207 (13), 206 (20), 205 (10), 200 (4), 197 (6), 196 (25), 195 (17), 194 (57), 193 (21), 192 (8), 189 (5), 188 (13), 187 (22), 186 (4), 185 (10), 182 (8), 181 (18), 180 (21), 179 (43), 178 (7), 177 (5), 176 (6), 173 (5), 172 (16), 170 (6), 169 (5), 168 (5), 166 (17), 165 (8), 164 (32), 163 (6), 162 (6), 161 (4), 159 (6), 158 (8), 157 (7), 156 (7), 154 (8), 153 (21), 152 (23), 151 (52), 150 (9), 149 (4), 146 (4), 145 (11), 144 (12), 143 (6), 142 (6), 141 (6), 140 (11), 139 (7), 138 (11), 137 (13), 136 (7), 132 (5), 131 (28), 130 (22), 129 (17), 128 (38), 127 (18), 126 (14), 125 (9), 124 (7), 123 (12), 119 (5), 118 (9), 117 (38), 116 (38), 115 (24), 114 (20), 113 (11), 112 (6), 111 (7), 110 (4), 104 (9), 103 (18), 102 (39), 101 (39), 100 (17), 99 (19), 98 (6), 97 (7), 91 (10), 90 (26), 89 (37), 88 (23), 87 (22), 86 (10), 78 (14), 77 (30), 76 (30), 75 (37), 74 (22), 73 (16), 69 (13), 65 (15), 64 (18), 63 (38), 62 (28), 61 (9), 55 (10), 53 (11), 52 (22), 51 (32), 50 (21), 45 (23), 44 (17), 43 (34), 41 (9), 39 (9), 32 (35), 31 (13)

The major fragmentation pathways observed for **2b** are the loss of HBr or Br radical from $[M - CHO]^+$, $[M - CH_2O]^{+\cdot}$ and $[M - CH_2OH]^+$ ions and also from $[M - CHO - CH_2O]^+$ and $[M - CH_2O - CO]^{+\cdot}$ species. In the spectrum of a mixture of **2c** and **2d**, the loss of Cl or HCl is predominant only from the lower daughter ions. The presence of a Cl atom at the 6-position in **2e** considerably increased the abundance of $M^{+\cdot}$, $[M - OH]^+$, $[M - (OH + X)]^{+\cdot}$, $[M - H_2O]^{+\cdot}$, $[M - CO]^{+\cdot}$ and $[M - HCNO]^{+\cdot}$ ions.

Table 8. Mass spectrum of 2*i*: principal fragments, *m/z* (relative intensity (%) in parentheses)

251 (3), 250 (11), 249 (66), 248 (8), 234 (12), 233 (5), 232 (19), 231 (43), 230 (3), 221 (4), 220 (23), 219 (100), 218 (42), 217 (5), 216 (23), 206 (7), 205 (11), 204 (63), 203 (23), 202 (8), 200 (2), 191 (3), 190 (11), 189 (6), 188 (11), 187 (4), 186 (4), 177 (3), 176 (16), 175 (15), 174 (32), 173 (9), 172 (8), 171 (4), 170 (14), 162 (5), 161 (16), 160 (35), 159 (6), 158 (20), 157 (8), 149 (6), 148 (12), 147 (6), 146 (15), 145 (9), 144 (12), 143 (5), 141 (4), 138 (5), 135 (5), 134 (8), 133 (16), 132 (35), 131 (9), 130 (14), 129 (11), 128 (13), 127 (8), 126 (5), 125 (6), 123 (4), 122 (5), 119 (5), 118 (8), 117 (13), 116 (12), 115 (15), 114 (6), 113 (8), 112 (4), 111 (8), 110 (7), 109 (7), 106 (4), 105 (8), 104 (15), 103 (13), 102 (10), 101 (8), 100 (4), 99 (8), 98 (8), 97 (16), 91 (7), 90 (8), 89 (23), 88 (10), 87 (10), 85 (14), 83 (11), 78 (11), 77 (22), 76 (8), 75 (10), 73 (10), 71 (17), 69 (18), 65 (9), 63 (14), 57 (26), 55 (26), 45 (47), 44 (24), 43 (44), 41 (25), 39 (20), 32 (17), 31 (55)

In the dichloro compound **2*f*** (*m/z* 257), loss of $2Cl^\cdot$ is noted from the molecular ions and also from $[M - CH_2O]^{+\cdot}$ and $[M - CH_2OH]^+$ ions causing further decomposition. Loss of HCl units from $[M - CH_2O]^{+\cdot}$, $[M - CHO]^+$ and $[M - CO]^{+\cdot}$ and from further derived ions seems to be the major pathways of fragmentation for **2f**.

In **2g**, the losses of HCl from the initially formed ions, viz. $[M - CO]^{+\cdot}$, $[M - CHO]^+$, $[M - CH_2O]^{+\cdot}$, $[M - CH_2OH]^+$, $[M - HCNO]^{+\cdot}$ and $[M - CH_2{=}CHOH]^{+\cdot}$ appear to be facile processes. The presence of a methyl group initiates the loss of $H^\cdot$.[6a,4a] Initial loss of CH_3 or $Cl^\cdot$ can also initiate different sets of fragmentations.

Table 9. The relative abundances of the first formed fragment ions including the molecular ions of compounds 2a–i

Fragment ion	Compound (relative intensity (%))							
	2a	2b	2c + 2d	2e	2f	2g	2h	2i
M^+	60	19	10	93	26	64	79	66
$[M - H]^+$	18	12	—	13	3	19	21	8
$[M - OH]^+$	30	23	5	59	25	58	64	19
$[M - H_2O]^{+\cdot}$	52	27	15	70	39	75	62	43
$[M - H_2O - H]^{+\cdot}$	48	14	7	57	23	55	55	3
$[M - CO]^{+\cdot}$	5	84	25	60	65	70	62	4
$[M - CHO]^+$	31	54	83	73	37	53	62	23
$[M - CH_2O]^{+\cdot}$	100	83	88	100	100	100	100	100
$[M - CH_2OH]^+$	70	42	15	63	22	63	58	42
$[M - HCNO]^{+\cdot}$	9	3	4	23	5	10	18	7
$[M - CH_2{=}CHOH]^{+\cdot}$	18	6	5	43	7	13	20	11
$[M - 58]^{+\cdot}$ [a]	24	10	5	49	9	11	17	3
$[M - 59]^+$ [b]	74	26	80	64	33	37	57	11

[a] M − 58: $M - CH_2O - CO/M - CO - CH_2O$.

[b] M − 59: $M - CH_2O - CHO/M - CHO - CH_2O$.

Table 10. The relative abundances of fragment ions formed after the elimination of HX or X from molecular ions or their daughter ions of the halo derivatives 2b–h

Fragment ion	Compound (relative intensity (%))					
	2b	2c + 2d	2e	2f	2g	2h
$[M - X]^+$	—	—	9	2	4	—
$[M - HX]^{+\cdot}$	1	—	—	0.5	3	—
$[M - OH - X]^{+\cdot}$	5	—	—	2	3	—
$[M - H_2O - HX]^{+\cdot}$	6	12	6	3	6	—
$[M - CHO - X]^{+\cdot}/[M - CO - HX]^{+\cdot}$	9	—	15	11	7	5
$[M - CHO - HX]^+/[M - CH_2O - X]^+$	20	—	48	16	30	13
$[M - CH_2O - HX]^{+\cdot}$	48	10	53	9	15	22
$[M - R^5]^{+\cdot}$ [a]	100	13	21	15	22	6
$[M - R^6]^+$ [b]	46	17	26	13	22	8
$[M - 2X]^{+\cdot}$	—	—	—	5	—	—
$[M - CH_2O - 2X]^{+\cdot}$	—	—	—	7	—	—
$[M - CH_2OH - 2X]^{+\cdot}$	—	—	—	17	—	—
$[M - CH_2O - CHO - 2X]^{+\cdot}$	—	—	—	34	—	—

[a] R^5: $CHO - X - CH_2O/CH_2O - CHO - X/CHO - CH_2O - X/CH_2O - CO - HX/CH_2O - X - CHO$.

[b] R^6: $CH_2O - CHO - HX/CHO - CH_2O - HX/CH_2O - HX - CHO/CHO - HX - CH_2O$.

Table 11. ***N*-Aryl-4,5-dihydrofuran-3-carboxamides (1)**

1	Yield (%)	M.p. (°C) (solvent)	Molecular formula or m.p. (°C) reported	IR (KBr), ν (cm^{-1})
a	80	151–152 (benzene)	151–152[11]	1135, 1650, 3025
b	50	194–195 (benzene)	194–195[12]	1125, 1640, 3285
c/d	65	138–139 (benzene)	139–140[11]	1130, 1640, 3450
e	72	176–178 (benzene)	176–178[11]	1130, 1640, 3450
f	84	155–157 (benzene–$CHCl_3$)	$C_{11}H_9Cl_2NO_2$ [a] (258.1)	1125, 1635, 3350
g	81	166–168 (benzene)	167–168[12]	1135, 1650, 3240
h	82	105–109 (benzene)	$C_{12}H_{12}NO_3Cl$[a] (253.67)	1120, 1620, 3285
i	65	95–97 (benzene)	96–97[12]	1130, 1630, 3350

[a] Satisfactory microanalyses obtained: C ± 0.3, H ± 0.2%.

The presence of methoxy group in **2h** and **2i** causes a variety of fragmentations,[6a,8] expelling $CH_3^{\cdot}$, $OCH_3^{\cdot}$, CH_2O and of course $CHO^{\cdot}$ either from the molecular ion or from the derived daughter ions, viz. $[M - OH]^+$, $[M - H_2O]^{+\cdot}$, $[M - CO]^{+\cdot}$, $[M - CHO]^+$, $[M - CH_2O]^{+\cdot}$, $[M - CH_2OH]^+$, $[M - HCNO]^{+\cdot}$ and $[M - CH_2{=}CHOH]^{+\cdot}$ species.

EXPERIMENTAL

The spectra were determined by the introduction of the sample into the ion source of Hitachi–Perkin-Elmer RMU-6E mass spectrometer. The energy of the electron beam was 70 eV. The compounds used in the study were of analytical purity[1,2,9–12] and all of them were prepared as reported previously[1,2] (for physical data see Tables 11 and 12).

Preparation of *N*-aryl-4,5-dihydrofuran-3-carboxamides[11,1]

A mixture of 4,5-dihydrofuran-3-carboxylic acid (0.1 mol) and purified thionyl chloride (0.2 mol) was heated on a steam-bath at 40–50 °C until the reaction was complete (3 h). Excess of thionyl chloride was distilled off and the residue was taken up in benzene (50 ml) and slowly added to a well cooled mixture of the appropriate aniline (0.1 mol) and pyridine (0.1 mol) in dry benzene (100 ml). The mixture was then allowed to stand for 30 min and then poured into ice–water. The precipitated solid was filtered, dried and recyrstallized from a suitable solvent (see Table 11).

Table 12. **3-(2′-Hydroxyethyl)-2-oxo-1,2-dihydroquinolines (2)**

2	Yield (%)	M.p. (°C) (solvent)	Molecular formula or m.p. (°C) reported	IR (KBr), ν (cm^{-1})
a	76	195–196 (methanol)	195–196[1]	3300, 1640
b	74.5	175–183 (decomp.) ($CHCl_3$–methanol)	$C_{11}H_{10}NO_2Br$[a] (268.1)	3300, 1640
c + d	65	165–170 ($CHCl_3$–methanol)	$C_{11}H_{10}NO_2Cl$[a] (223.6)	3285, 1640
e	77	209–210 (methanol)	209–210[1]	3320, 1640
f	72	204–205 ($CHCl_3$–methanol)	$C_{11}H_9Cl_2NO$[a] (258.1)	3320, 1635
g	66.4	226–228 ($CHCl_3$–methanol)	227–228[10]	3300, 1640
h	70	211–212 ($CHCl_3$)	211–212[10]	3300, 1635
i	62.5	213–215 ($CHCl_3$–methanol)	215–216[9]	3320, 1640

[a] Satisfactory microanalyses obtained: C ± 0.3, H ± 0.2%.

Preparation of 3-(2′-hydroxyethyl)quinoline-2(1*H*)-ones[1]

A solution of furan-3-carboxanilide, **1** (0.5 g), in dry methanol (300 ml) was placed in a quartz tube, purged with oxygen-free nitrogen for 15 mins and then irradiated in a Rayonet Model 208 preparative photoreactor using 253.7 nm light for 20–24 h until thin-layer chromatography (silica gel plates eluted with chloroform containing a few drops of ethyl acetate) showed the absence of a spot corresponding to **1**. The solvent was then evaporated *in vacuo* and the residue placed on a silica gel (20 g) column. The product was eluted with chloroform–ethyl acetate (1:1), the eluate was evaporated *in vacuo* and the residue recrystallized (see Table 12).

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