QUINOLINE RING FORMATION BY CYCLOADDITION OF
N-ARYLKETENIMINES WITH ENOL ETHERS UNDER HIGH PRESSURE

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Abstract - The reaction of N-aryl substituted ketenimines with enol ethers under
800 MPa gave quinoline derivatives, which formed by cyclization between the
C=C-N=C aza-diene moieties of N-aryl substituted ketenimines and the C=C
double bond moieties of enol ethers. When cyclic enol ethers such as 2,3-
dihydrofurans or 3,4-dihydro-2H-pyrans were used as dienophiles, quinoline
derivatives that had substituents with hydroxyalkyl or oxoalkyl side chains on the
C-3 positions were synthesized in one step reaction.

High-pressure sometimes accelerates organic reactions which are difficult or slow at atmospheric
pressure to give desired products in high yields. In the previous papers, we reported that enol ethers
reacted with isocyanates to give azetidin-3-one derivatives. The [2+2] cycloaddition of phenyl
isocyanate with 2,3-dihydrofuran proceeded in a sealed tube, but the reaction accelerated under high
pressure, such as 800 MPa. Isothiocyanates also reacted with 2,3-dihydrofuran under high pressure,
and 2-oxa-7-azabicyclo[3.2.0]heptane-6-thiones formed. Although the reaction of benzylideneanilines
with 2,3-dihydrofuran under high pressure gave azetidine derivatives which are the [2+2] cycloadducts,
the reactions in the presence of Lewis acids or lanthanide catalysts afforded quinoline derivatives,
which were formed [4+2] cycloaddition between the C=C-N=C aza-diene moieties of benzylideneanilines and the C=C double bond moieties of enol ethers.

It had been reported that ketenimines became starting materials of various types of heterocycles by intermolecular \(^8\) and intramolecular cycloaddition. \(^9\) We reported a convenient synthetic method of ketenimines from thioamides with haloiminium salts, such as 2-chloro-1,3-dimethylimidazolium chloride or 2-chloro-1-methylpyridinium iodide, in the preceding paper. \(^10\) \(N\)-Aryl substituted ketenimines were synthesized in high yields with this method. Because the \(N\)-aryl substituted ketenimines are regarded as analogues of both isocyanates and isothiocyanates and have the C=C-N=C moieties like benzylideneanilines, it is attractive what kinds of cycloaddition will occur in the reaction of the \(N\)-aryl substituted ketenimines with enol ethers. In this paper, we describe the cyclization of \(N\)-aryl substituted ketenimines with enol ethers under high pressure.

RESULTS AND DISCUSSION

We have already shown that the cycloaddition of isocyanates with 2,3-dihydrofuran (2a) accelerated under high pressure and the \(\beta\)-lactam derivatives were obtained in good yield. \(^3\) These reaction conditions were applied to the ketenimine reaction. \(N\)-Tolylidiphenylketenimine (1b) was dissolved in 2,3-dihydrofuran (2a) and the reaction mixture was heated 100 °C under 800 MPa for 20 h. After cooling the mixture, a product (3b) was isolated as a main product. The molecular formula of the product showed C\(_{29}\)H\(_{29}\)NO\(_2\), which meant that one molecular of 1b reacted with two molecules of 2a. Furthermore, when 3b was refluxed in methanol in the presence of \(p\)-toluenesulfonic acid, one molecular of 2a was eliminated and an alcoholic derivatives (4a) showing a molecular formula as C\(_{25}\)H\(_{23}\)NO was obtained. The structure of 3b was determined as a quinoline derivative, 2-diphenylmethyl-6-methyl-3-[2-(2-tetrahydrofuranyloxy)ethyl]quinoline, from the spectral data. This cyclization occurred only under high-pressure conditions and 3b was not obtained with the reaction in a sealed tube. It was reported that the quinoline derivatives which had a 2-hydroxyethyl group on the 3-position of the ring showed antibacterial activity. \(^11\) Therefore, the present method is convenient for synthesis of functional
group substituted quinoline derivatives.

A formation mechanism of 3b can be explained as shown in Scheme 1. Under high-pressure reaction conditions, 2,3-dihydrofuran attacked to the $sp$ carbon of a ketenimine group. Then, [4+2] cycloaddition completed between the C=C-N=C moiety of ketenimine (1b) and the C=C moiety of dihydrofuran (2a) to form a furo[3,2-$c$]quinoline ring intermediate. Next, the tetrahydrofuran ring opened and the quinoline compound (4a) which had the terminal hydroxyl group was formed. In the next stage, the formed hydroxyl group reacted with dihydrofuran which existed in large excess amount as a solvent. As a result, 3b was obtained as a final product. The same acetal formation of the terminal hydroxyl group with 2,3-dihydrofuran present in excess was reported in the cycloaddition of 1,2,4-triazines with 2,3-dihydrofuran.12

![Scheme 1](image)

Various kinds of cyclic enol ethers, such as 2,3-dihydrofurans and 3,4-dihydro-2$H$-pyrans, were treated with ketenimines under high pressure. The results are shown in Table 1. As reported in the preceding paper,10 C-monosubstituted ketenimines were too unstable to be purely isolated. Therefore, the reaction of N-tolylphenylketenimine (1d) was carried out without isolating the ketenimine and the yield of a
quinoline (3e) was calculated in the basis of the starting material, N-toly -2-phenylacetythioamide (Run 5). In the case of reaction of 3,4-dihydro-2H-pyran (2c), a hydroxy compound (3f) was exclusively isolated (Run 6). When the reaction was carried out in 2-alkoxy-3,4-dihydro-2H-pyran s (2d, e), the isolated products were 3-(3-oxopropyl)quinoline derivatives (Runs 7-9). These products (3g, h) were given by way of hemiacetal intermediates which were formed by the 3,4-dihydroquinoline ring aromatization. Although it was expected that the reaction of ketenimines with furan also gave the same aldehydes, no identifiable product was isolated after the reaction.

Table 1. Reaction of ketenimine (1) with cyclic enol ether (2)\textsuperscript{a}

<table>
<thead>
<tr>
<th>Run</th>
<th>1</th>
<th>R\textsuperscript{1}</th>
<th>R\textsuperscript{2}</th>
<th>2</th>
<th>R\textsuperscript{3}</th>
<th>R\textsuperscript{4}</th>
<th>n</th>
<th>Quinoline 3</th>
<th>R\textsuperscript{5}</th>
<th>Yield (%) of 3</th>
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<tbody>
<tr>
<td>1</td>
<td>la</td>
<td>H</td>
<td>Ph</td>
<td>2a</td>
<td>H</td>
<td>H</td>
<td>1</td>
<td>3a</td>
<td>O(2-C\textsubscript{4}H\textsubscript{7}O)</td>
<td>56</td>
</tr>
<tr>
<td>2</td>
<td>lb</td>
<td>Me</td>
<td>Ph</td>
<td>2a</td>
<td>H</td>
<td>H</td>
<td>1</td>
<td>3b</td>
<td>O(2-C\textsubscript{4}H\textsubscript{7}O)</td>
<td>86</td>
</tr>
<tr>
<td>3</td>
<td>le</td>
<td>OMe</td>
<td>Ph</td>
<td>2a</td>
<td>H</td>
<td>H</td>
<td>1</td>
<td>3c</td>
<td>O(2-C\textsubscript{4}H\textsubscript{7}O)</td>
<td>88</td>
</tr>
<tr>
<td>4</td>
<td>lb</td>
<td>Me</td>
<td>Ph</td>
<td>2b</td>
<td>Me</td>
<td>H</td>
<td>1</td>
<td>3d</td>
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<tr>
<td>5\textsuperscript{b}</td>
<td>ld</td>
<td>Me</td>
<td>H</td>
<td>2a</td>
<td>H</td>
<td>H</td>
<td>1</td>
<td>3e</td>
<td>O(2-C\textsubscript{4}H\textsubscript{7}O)</td>
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<tr>
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<td>Ph</td>
<td>2c</td>
<td>H</td>
<td>H</td>
<td>2</td>
<td>3f</td>
<td>CH\textsubscript{2}OH</td>
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<td>2</td>
<td>3h</td>
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<td>36</td>
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</table>

\textsuperscript{a} Reaction conditions: 1, 0.5 mmol; 2, 1 mL; 800 MPa; 100 °C; 20 h. \textsuperscript{b} 1 was not isolated. The yield was calculated based on N-(p-tolyl)phenylacetamide, the starting material of 1.

Next, the reaction of ketenimines with acyclic enol ethers was carried out under the same reaction
conditions described above. As a result, vinyl ethers behaved like acetylene compounds and quinoline derivative (6) were obtained as a main product. Vinyl acetate reacted with the ketenimine to afford 6 in spite of a low yield (Run 12). The results are listed in Table 2.

Table 2. Reaction of ketenimine (1b) with a vinyl ether or vinyl ester

<table>
<thead>
<tr>
<th>Run</th>
<th>R</th>
<th>Yield (%) of 6</th>
</tr>
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<tbody>
<tr>
<td>10</td>
<td>Et</td>
<td>83</td>
</tr>
<tr>
<td>11</td>
<td>Bu</td>
<td>59</td>
</tr>
<tr>
<td>12</td>
<td>Ac</td>
<td>26</td>
</tr>
</tbody>
</table>

* Reaction conditions: 1b, 0.5 mmol; 6, 1 mL; 800 MPa; 100 °C; 20 h.

It is concluded that the reaction of N-aryl substituted ketenimines with enol ethers under high pressure afforded the [4+2] cycloadducts and quinoline derivatives were produced. These results showed that the reactivity of ketenimines with 2,3-dihydrofuran under high pressure was different from that of isocyanates,3 isothiocyanates,4 and benzylideneaniline.5 In addition, it was found that the reaction with the cyclic enol ethers such as 2,3-dihydrofurans and 3,4-dihydro-2H-pyrans, quinolines with hydroxyalkyl or oxoalkyl side chains were synthesized in one step.

EXPERIMENTAL

Mps were determined on a Mettler FP90 microscope plate, and uncorrected. 1H and 13C NMR spectra were obtained with a Varian Gemini 300 BB (300 MHz) spectrometer with tetramethylsilane as an internal standard, and a JEOL LA-500 (125 MHz) spectrometer with chloroform as an internal standard, respectively. IR spectra were recorded on a JASCO FTIR-5300 spectrophotometer. The apparatus used for the high-pressure reaction was the same as that described previously.13
Materials. Ketenimines (1) were synthesized by treating the corresponding N-substituted thioamides with dehydrating agents as reported in our proceeding paper.10

Reaction of N-aryl substituted diphenylketenimines with enol ethers under high pressure

A homogeneous mixture of ketenimine (1, 0.5 mmol) and enol ether (1 mL, used both for reactant and for solvent) in a sealed Teflon® tube was compressed to 800 MPa, heated at 100 °C, and maintained for 20 h in a high-pressure apparatus. The resulting mixture was chromatographed on silica gel with dichloromethane as eluent. The product was recrystallized from benzene-hexane mixture.

2-Diphenylmethyl-3-[2-(2-tetrahydrofuranyloxy)ethyl]quinoline (3a).

mp 117.5-118.5 °C; 1H NMR (CDCl3) δ 1.79-1.96 (4H, m, CH2CH2), 3.07 (2H, t, J=7.1 Hz, 3-CH2), 3.60 (1H, td, J=9.9, 6.9 Hz, O-CH), 3.79-3.85 (2H, m, 3-CH2CH2-O), 3.94 (1H, td, J=9.9, 6.9 Hz, O-CH), 5.08 (1H, dd, J=3.6, 1.8 Hz, 7-H, O-CH-O), 6.04 (1H, s, 2-CH), 7.18-7.33 (10H, m, Ph2), 7.45 (1H, t, J=8.1 Hz, 7-H), 7.58 (1H, t, J=8.1 Hz, 6-H), 7.71 (1H, d, J=8.1 Hz, 5-H), 7.94 (1H, s, 4-H), 7.96 (1H, d, J=8.1 Hz, 8-H); IR (KBr) νmax 1597, 1493, 1090, 1036, 750 cm⁻¹; Anal. Calcd for C28H27NO2: C, 82.12; H, 6.65; N, 3.42. Found: C, 81.96; H, 6.60; N, 3.25.

2-Diphenylmethyl-6-methyl-3-[2-(2-tetrahydrofuranyloxy)ethyl]quinoline (3b).

mp 132-134 °C; 1H NMR (CDCl3) δ 1.77-1.96 (4H, m, CH2CH2), 2.50 (3H, s, 6-Me), 3.05 (2H, t, J=7.0 Hz, 3-CH2), 3.58 (1H, td, J=9.9, 7.0 Hz, O-CH), 3.79-3.85 (2H, m, 3-CH2CH2-O), 3.92 (1H, td, J=9.9, 7.0 Hz, O-CH), 5.07 (1H, dd, J=3.9, 1.8 Hz, 7-H, O-CH-O), 6.02 (1H, s, 2-CH), 7.18-7.34 (10H, m, Ph2), 7.41 (1H, dd, J=8.5, 1.9 Hz, 7-H), 7.47 (1H, br s, 5-H), 7.84 (1H, s, 4-H), 7.86 (1H, d, J=8.5 Hz, 8-H); 13C NMR (CDCl3) δ 21.5 (q, 6-Me), 23.4 (t), 32.3 (t), 32.7 (t), 54.9 (d, O-CH-O), 67.0 (t), 67.1 (t), 104.0 (d, 2-CH), 125.5 (d), 126.2 (d), 127.0 (s), 128.0 (d), 129.2 (d), 129.7 (d), 130.6 (d), 131.1 (s), 135.6 (d), 143.0 (s), 143.0 (s), 145.1 (s), 160.6 (s); IR (KBr) νmax 1597, 1493, 1453, 1090, 1034, 920, 822, 696 cm⁻¹; Anal. Calcd for C29H29NO2: C, 82.12; H, 6.65; N, 3.31. Found: C, 81.93; H, 6.94; N, 3.22.

2-Diphenylmethyl-6-methoxy-3-[2-(2-tetrahydrofuranyloxy)ethyl]quinoline (3c).

mp 131-132 °C; 1H NMR (CDCl3) δ 1.80-1.96 (4H, m, CH2CH2), 3.05 (2H, t, J=7.0 Hz, 3-CH2), 3.59
(1H, td, \(J=9.9, 7.0\) Hz, O-CH), 3.79-3.85 (2H, m, 3-CH\(_2\)CH\(_2\)-O), 3.88-3.96 (1H, m, O-CH), 3.90 (3H, s, 6-MeO), 5.07 (1H, dd, \(J=3.9, 1.8\) Hz, O-CH-O), 6.00 (1H, m, 2-CH), 6.99 (1H, d, \(J=2.8\) Hz, 5-H), 7.17-7.31 (11H, m, Ph\(_2\), 7-H), 7.84 (1H, s, 4-H), 7.86 (1H, d, \(J=9.6\), 8-H); IR (KBr) \(\nu_{\text{max}}\) 1624, 1599, 1493, 1233, 1040, 826 cm\(^{-1}\); Anal. Calcd for C\(_{29}\)H\(_{29}\)NO\(_3\): C, 79.24; H, 6.65; N, 3.19.  Found: C, 79.21; H, 6.73; N, 3.02.

2-Diphenylmethyl-3-(2-hydroxyethyl)-4,6-dimethylquinoline (3d).

mp 195-195.5 °C; \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 1.38 (1H, br t, OH), 2.53 (3H, s, 6-Me), 2.65 (3H, s, 4-Me), 3.19 (2H, t, \(J=7.4\) Hz, 3-CH\(_2\)), 3.71 (2H, br q, 3-CH\(_2\)CH\(_2\)-O), 6.08 (1H, s, 2-CH), 7.18-7.36 (10H, m, Ph\(_2\)), 7.41 (1H, dd, \(J=8.5, 1.7\) Hz, 7-H), 7.72 (1H, br s, 5-H), 7.83 (1H, d, \(J=8.5\) Hz, 8-H); IR (KBr) \(\nu_{\text{max}}\) 3403 (O-H), 1572, 1493, 1055, 831, 739, 706 cm\(^{-1}\); Anal. Calcd for C\(_{26}\)H\(_{25}\)NO: C, 84.98; H, 6.86; N, 3.81.  Found: C, 84.54; H, 6.91; N, 3.64.

6-Methyl-2-phenylmethyl-3-[2-(2-tetrahydrofuranyloxy)ethyl]quinoline (3e).

oil; \(^1\)H NMR (CDCl\(_3\)) 1.74-2.00 (4H, m, CH\(_2\)CH\(_2\)), 2.53 (3H, s, 6-Me), 2.94 (2H, t, \(J=6.9\) Hz, 3-CH\(_3\)), 3.50 (1H, td, \(J=9.9, 6.9\) Hz, O-CH), 3.79-3.90 (3H, m, 3-CH\(_2\)CH\(_2\)-O, O-CH\(_2\)), 4.43 (2H, s, 2-CH\(_2\)), 5.03 (1H, dd, \(J=6.0, 1.8\) Hz, O-CH-O), 7.18-7.26 (5H, m, Ph), 7.50 (1H, dd, \(J=9.3, 1.9\) Hz, 7-H), 7.51 (1H, br s, 5-H), 7.86 (1H, s, 4-H), 7.97 (1H, d, \(J=9.3\) Hz, 8-H); IR (KBr) \(\nu_{\text{max}}\) 1601, 1493, 1453, 1348, 1182, 1038, 918, 826, 727, 698 cm\(^{-1}\).  This compound could not be purified to be satisfying elemental analysis. Therefore, the following hydrolysis was carried out for determining the structure.

2-Diphenylmethyl-3-(3-hydroxypropyl)-6-methylquinoline (3f).

mp 138.5-139 °C; \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 1.23 (1H, br t, OH), 1.80-1.90 (2H, m, 3-CH\(_2\)CH\(_2\)CH\(_2\)O), 2.50 (3H, s, 6-Me), 2.91 (2H, t, \(J=7.8\) Hz, 3-CH\(_3\)), 3.66 (2H, br q, CH\(_2\)OH), 5.98 (1H, s, 2-CH), 7.17-7.31 (10H, m, Ph\(_2\)), 7.42 (1H, dd, \(J=8.5, 2.1\) Hz, 7-H), 7.48 (1H, br s, 5-H), 7.82 (1H, s, 4-H) and 7.86 (1H, d, \(J=8.5\) Hz, 8-H); IR (KBr) \(\nu_{\text{max}}\) 3353 (O-H), 1599, 1493, 1057, 824, 743, 706 cm\(^{-1}\); Anal. Calcd for C\(_{26}\)H\(_{25}\)NO: C, 84.98; H, 6.86; N, 3.81.  Found: C, 84.73; H, 6.86; N, 3.75.

2-Diphenylmethyl-3-(3-oxopropyl)quinoline (3g).

mp 125-126.5 °C; \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 2.62 (2H, td, \(J=7.6, 1.0\) Hz, CH\(_2\)CHO), 3.17 (2H, t, \(J=7.6\) Hz, 3-
2-Diphenylmethyl-6-methyl-3-(3-oxopropyl)quinoline (3h).

mp 156-157 °C; \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 2.50 (3H, s, 6-Me), 2.59 (2H, td, \(J=7.6, 1.1\) Hz, \(CH_2CHO\)), 3.14 (2H, t, \(J=7.6\) Hz, 3-CH\(_2\)), 5.93 (1H, s, 2-CH), 7.20-7.28 (10H, m, Ph\(_2\)), 7.43 (1H, dd, \(J=8.5, 1.9\) Hz, 7-H), 7.48 (1H, br s, 5-H), 7.78 (1H, s, 4-H), 7.86 (1H, d, \(J=8.5\) Hz, 8-H) and 9.71 (1H, t, \(J=1.1\) Hz, CHO); IR (KBr) \(\nu_{\text{max}}\) 2834, 2733 (O=C-H), 1717 (C=O), 1599, 1493, 828, 714 cm\(^{-1}\); Anal. Calcd for C\(_{26}\)H\(_{23}\)NO: C, 85.45; H, 6.34; N, 3.83. Found: C, 85.11; H, 6.30; N, 3.70.

2-Diphenylmethyl-6-methylquinoline (6).

mp 120-122 °C; \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 2.52 (3H, s, 6-Me), 5.90 (1H, s, 2-CH), 7.20-7.33 (11H, m, Ph\(_2\), 3-H), 7.52 (1H, dd, \(J=8.5, 1.9\) Hz, 7-H), 7.53 (1H, br s, 5-H), 7.96 (1H, d, \(J=8.5\) Hz, 4-H), 7.97 (1H, d, \(J=8.5\) Hz, 8-H); IR (KBr) \(\nu_{\text{max}}\) 1595, 1493, 828, 714 cm\(^{-1}\); Anal. Calcd for C\(_{23}\)H\(_{19}\)N: C, 89.28; H, 6.19; N, 4.53. Found: C, 89.01; H, 6.17; N, 4.43.

Hydrolysis of 3-[2-(2-tetrahydrofuranyloxy)ethyl]quinolines (3)

A quinoline compound (3b or 3f, 100 mg) was dissolved in methanol (15 mL) and p-toluenesulfonic acid (40 mg) was added. After the reaction mixture was refluxed for 4 h, the solvent was evaporated off. Sodium carbonate (10% in water) was added to the reaction mixture, and the product was extracted with dichloromethane. The organic layer was washed with water and dried over magnesium sulfate. The solvent was evaporated off and the product was recrystallized from benzene-hexane.

2-Diphenylmethyl-3-(2-hydroxyethyl)-6-methylquinoline (4a).

Yield 89 %; mp 171-172 °C; \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 1.40 (1H, br t, OH), 2.50 (3H, s, 6-Me), 3.07 (2H, d, \(J=6.7\) Hz, 3-CH\(_2\)), 3.81 (2H, br q, \(CH_2OH\)), 5.99 (1H, s, 2-CH), 7.18-7.30 (10H, m, Ph\(_2\)), 7.43 (1H, dd, \(J=8.7, 2.1\) Hz, 7-H), 7.49 (1H, br s, 5-H), 7.86 (1H, s, 4-H), 7.86 (1H, d, \(J=8.7\) Hz, 8-H); IR (KBr) \(\nu_{\text{max}}\)
3329 (O-H), 1599, 1493, 1038, 822, 749, 704 cm⁻¹; Anal. Calcd for C₂₅H₂₃NO: C, 84.95; H, 6.56; N, 3.96. Found: C, 84.84; H, 6.55; N, 4.05.

3-(2-Hydroxyethyl)-6-methyl-2-phenylmethylquinoline (4b).

Yield 60 %; mp 129-130 °C; ¹H NMR (CDCl₃) δ 1.65 (1H, br s, OH), 2.52 (3H, s, 6-Me), 2.95 (2H, t, J=6.6 Hz, 3-CH₂), 3.75 (2H, t, J=6.6 Hz, CH₂OH), 4.42 (2H, s, 2-CH₂), 7.17-7.26 (5H, m, Ph), 7.49-7.52 (2H, m, 5-H, 7-H), 7.86 (1H, s, 4-H), 7.97 (1H, d, J=9.1 Hz, 8-H); IR (KBr) νmax 3179 (O-H), 1599, 1495, 1341, 1076, 1047, 822, 733, 708 cm⁻¹; Anal. Calcd for C₁₉H₁₉NO: C, 82.28; H, 6.90; N, 5.05. Found: C, 81.96; H, 6.95; N, 4.95.

REFERENCES AND NOTES


