Novel Syntheses with Carbon Suboxide; III. 
Cyclocondensation with N-Benzylidene- or N-
Propylidene-2-hydroxyanilines to form the Seven-
Membered Ring System of 5-Benzyl(propyl)idene-4-
oxido-2-oxo-2,5-dihydro-1,5-benzoxazepinium Betaines

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We previously showed that carbon suboxide (2) is a very good reagent for the synthesis of several or eight membered heterocyclic rings with potential pharmacological activity. Here, we report the synthesis of the previously unknown, potentially thymoleptic and/or neuroleptic 5-benzyl(propyl)idene-4-oxido-2-oxo-2,5-dihydro-1,5-benzoxazepinium betaines 3 from the reaction of carbon suboxide (2) with N-benzylidene-2-hydroxyanilines 1a-d or N-propylidene-2-hydroxyaniline (1e).

All reactions were carried out using dilute diethyl ether solutions of 1 and an equimolar amount of 2. The yields are reasonably good (Table). All the products 3a-e exist in the mesoionic structure shown and all analytical and spectroscopic data are in agreement with the proposed structures.

5-Benzyl(propyl)idene-4-oxido-2-oxo-2,5-dihydro-1,5-benzoxazepinium Betaines 3a-e; General Procedure:
To a stirred solution of 1 (24 mmol) in dry diethyl ether (200 ml), carbon suboxide (2; 1.6 g, 24 mmol) is added in 2 h at 0°C. When the addition is complete, the mixture is vigorously stirred at 0°C for 24 h and then allowed to warm and left at room temperature for 54 h. The diethyl ether is evaporated under reduced pressure and the residue crystallized from ethanol to give 3.

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Table. 5-Benzyl(propyl)idene-4-oxido-2-oxo-2,5-dihydro-1,5-benzoxazepinium Betaines 3a-e prepared

<table>
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<tbody>
<tr>
<td>3a</td>
<td>70</td>
<td>128-130</td>
<td>C₁₈H₁₇NO₃ (265.3)</td>
<td>1780-1770 (C=O); (C=CH): 7.66 (s, 1 H, CH=N); 8.0-8.6 (m, 10 H, Ar and CH=C=O)</td>
<td>265 (M⁺), 196 (M-69)</td>
<td>120 (M-145)²</td>
</tr>
<tr>
<td>3b</td>
<td>66</td>
<td>196-198</td>
<td>C₁₈H₁₇ClNO₃ (299.7)</td>
<td>1780-1730 (C=O); (DMSO-d₆): 7.5-6.8 (m, 10 H, Ar, CH=N, and CH=C=O)</td>
<td>265 (M⁺), 250 (M-69)</td>
<td>120 (M-179)²</td>
</tr>
<tr>
<td>3c</td>
<td>84</td>
<td>153-155</td>
<td>C₁₈H₁₆NO₃ (279.3)</td>
<td>1780-1770 (C=O); (CDCl₃): 7.41 (s, 1 H, CH=N); 7.3-6.7 (m, 10 H, Ar and CH=C=O)</td>
<td>231 (3 H, 3 H, Ar-CH₃)</td>
<td>279 (M⁺), 210 (M-69), 120 (M-159)²</td>
</tr>
<tr>
<td>3d</td>
<td>76</td>
<td>144-145</td>
<td>C₁₈H₁₆NO₃ (295.3)</td>
<td>1780-1770 (C=O); (CDCl₃): 7.4-6.5 (m, 10 H, Ar, CH=N, and CH=C=O)</td>
<td>3.73 (3 H, Ar-CH₃)</td>
<td>295 (M⁺), 226 (M-69), 120 (M-175)²</td>
</tr>
<tr>
<td>3e</td>
<td>61</td>
<td>166-168</td>
<td>C₁₈H₁₆NO₃ (217.2)</td>
<td>1780-1770 (C=O); (CDCl₃): 7.4-6.9 (m, 6 H, Ar, CH=N, and CH=C=O)</td>
<td>3.04 (8 H, 2 H, CH₂=CH₂, 1.13 (t, 3 H, CH₃-CH₂)</td>
<td>217 (M⁺), 118 (M-69), 120 (M-97)²</td>
</tr>
</tbody>
</table>

¹ Satisfactory microanalyses obtained: C ±0.14, H ±0.09, N ±0.10.

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