Synthesis of Acid Dyes for Nylon

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Acid dyes for nylon have been prepared from \( m \)-aminoanilinoacrylates and gamma-acid under acidic conditions. The dyeing properties of the resulting dyes have been studied.

Among the monoazo acid dyes, the dyes obtained by acid coupling onto gamma-acid are of special interest because of their high light fastness. In such dyes, both the 2-amino and 8-hydroxy groups in the coupler are involved in six-membered rings containing strong hydrogen bonds\(^1\).

Some of the dyes which are important in the series are obtained by acid coupling of diazotized 3-benzoylaminoanilines with gamma-acid. In continuation of our studies on anilinoacrylates as synthons\(^2\) for dyes, it was considered of interest to prepare some new dyes from gamma-acid and \( m \)-aminoanilinoacrylates as diazo component and compare their properties with those of the dyes derived from \( m \)-benzoxyaminoaniline.

\( m \)-Aminoanilinoacrylates were prepared by reacting \( m \)-phenylenediamine with molar equivalent of conjugated enol ethers, as reported earlier.\(^3\).\(^4\) Benzoylaminoaniline was prepared by benzoylating \( m \)-nitroaniline with benzoyl chloride followed by reduction of the nitro group with iron and acetic acid. The dyes were prepared by the usual procedure and being water soluble could not be obtained in pure form for elemental analysis. However, the dyes were pure enough for dyeing trials. The shades and fastness data for the dyes are given in Table 1.

### Experimental Procedure

**Preparation of dyes**—The amino compound (0.01 mol) was diazotized in the usual manner. Gamma-acid (0.01 mol) was dissolved in sodium bicarbonate solution and precipitated in a fine form by acidifying with 20 ml hydrochloric acid (1:1). To the suspension of gamma-acid, the diazo solution was added slowly at 0-10°C. The coupling mixture was stirred overnight and the dye which precipitated was filtered, washed

### Table 1—Shade, Visible Absorption, Pick Up and Light Fastness of Dyes Synthesized

<table>
<thead>
<tr>
<th>Dye</th>
<th>Substituent, ( R )</th>
<th>Colour of shade (1%)</th>
<th>Pick up</th>
<th>Xeno light fastness</th>
<th>Visible absorption</th>
</tr>
</thead>
<tbody>
<tr>
<td>D1</td>
<td>( \text{NHCOPh} )</td>
<td>Brown red</td>
<td>2 +</td>
<td>3</td>
<td>( \lambda_{\text{max}} ) = 506</td>
</tr>
<tr>
<td>D2</td>
<td>( \text{NHCH} = \text{C} ) ( \text{COOEt} )</td>
<td>Maroon</td>
<td>4</td>
<td>4-5</td>
<td>522</td>
</tr>
<tr>
<td>D3</td>
<td>( \text{NHCH} = \text{C} ) ( \text{COOEt} ) ( \text{CN} )</td>
<td>Brown red</td>
<td>2</td>
<td>1-2</td>
<td>510</td>
</tr>
<tr>
<td>D4</td>
<td>( \text{NHCH} = \text{C} ) ( \text{COOEt} ) ( \text{COCH}_3 )</td>
<td>Red brown</td>
<td>4</td>
<td>3-4</td>
<td>510</td>
</tr>
</tbody>
</table>

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with saturated salt solution and dried. The samples of the dried dye were used directly for dyeing trials without further purification. The dyes derived from \( m \)-aminoanilinomethylene malonate and \( m \)-aminoanilinomethyleneacetoacetate showed better dyeing properties compared to those derived from \( m \)-benzoylaminoaniline.

References