
As part of an experimental study of the structural consequences of triaryl amine oxidation, we have initiated crystallographic investigations of tri(p-tolyl)amine and tri(p-tolyl)aminium hexafluorophosphate. The neutral amine crystallizes in space group 

**C202** 09. STRUCTURES OF ORGANIC, ORGANOMETALLIC AND COORDINATION COMPOUNDS

---

**09.2-04 THE CRYSTAL STRUCTURE OF LONCHOCILIUM FORMYLPHOSPHONATE.**


Extraction of the corrosion product formed by metallic lead and white mineral oil at 150° with morpholine gave yellow crystals identical with those produced by bubbling CO₂ gas through morpholine (Ennor, Annals, (1978), 301, 4). This compound crystallizes in space group 

**P2₁/c** with a = 10.497, b = 9.366, c = 6.365 A, α = 94.07, β = 96.56, γ = 106.67°. The structure was determined by neutron and refined to R = 0.056 for 1733 reflections.

The structure is evidently a salt (C₇H₁₀ON₂H₂)⁺ (C₅H₅ON)⁻ with pairs of ions linked by a closed cyclic hydrogen bonding system. The difference in charge between the anionic and cationic morpholine rings has a notable influence on the bond lengths and angles as shown in the diagram.

---

**09.2-05 CONFORMATIONAL ANALYSES OF α-(1-NITRO-POLYACETOXY-2-HEPTYL)-α-ALKYLAMINO-CROTONIC ACID ESTERS BY X-RAY DIFFRACTION.**

By R. Vega, M.J. Diaz, A. Lopez Castro and R. Márquez, Departamento de Optica y Sección de Física del Departamento de Investigaciones Físicas y Químicas del C.S.I.C., Universidad de Sevilla, Spain.

We report here several examples of α-(1-Nitro-polyacetoxy-2-heptyl)-α-alkylamino-crotonic acid esters for which a conformational study is being carried out.

They are by-products in the synthesis of 3-pentacoxyentyl-pyrole derivatives (Gomez-Sánchez, Mancera, Rosado + Bellenstot (1980), J.C.S. Perkin Trans. 2, 1199). In the assigned structure (1) a new chiral centre is found (marked with an asterisk) and two diastereomers could result; however, only one product was isolated and detected chromatographically. The configuration could not be established from chemical and spectroscopic considerations and an X-ray study was undertaken.

The three compounds belong to the orthorhombic space group 

**P2₁ 2₁ 2₁** with four molecules per unit cell.

Crystal data:

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>C₇H₁₀N₂O₄</td>
<td>C₂₃H₃₄N₂O₁₄</td>
<td>C₂₃H₃₄N₂O₁₄</td>
</tr>
<tr>
<td>a=11.147(1)</td>
<td>a=14.270(2)</td>
<td>a=17.376(1)</td>
</tr>
<tr>
<td>b=37.350(2)</td>
<td>b=18.743(2)</td>
<td>b=13.176(1)</td>
</tr>
<tr>
<td>c=7.705(1)</td>
<td>c=10.109(1)</td>
<td>c=9.466(1)</td>
</tr>
<tr>
<td>v=3207.9</td>
<td>V=2757.3</td>
<td>V=2161.5</td>
</tr>
</tbody>
</table>

**Dc=1.28 Mg.m⁻³ | Dc=1.31 Mg.m⁻³ | Dc=1.35 Mg.m⁻³ | Dc=1.36 Mg.m⁻³**

Intensity data were collected on an automatic Philips PW 1100 four-circle diffractometer (MoKα for I and CuKα for II and III, graphite monochromator, 0–2θ scan). The crystal structures have been solved by direct methods and refined by Fourier and least-squares calculations. The main least-squares planes in the molecule and torsion angles have been calculated. The configuration of the sugar chain will be discussed. The molecule in the three compounds has an intramolecular bond between the nitrogen and oxygen atoms of the amino and carbonyl groups respectively, showing a chelated structure. The packing is governed by normal van der Waals contacts.