Crystal Structure of 3,6-Spiro-
dicyclooctyliden-1,2,4,5-tetra-
oxa-cyclohexane ("Dimeric Cyclo-
looctanone Peroxide")

P. GROTH

Universitets Kjemiske Institutt, Blindern,
Oslo 3, Norway

Crystals of "dimeric cyclooctanone
peroxide", prepared by T. Ledal (to
be published), belong to the monoclinic
system. The space group is P2\textsubscript{1}/c, and the
unit cell, containing two molecules, has
the following parameters:

\[ a = 9.79 \text{ Å}, \quad b = 7.33 \text{ Å}, \quad c = 11.77 \text{ Å}, \]
\[ \beta = 114.2^\circ \]

Fourier map for the h0l-projection was
obtained by a combined application of
Harker-Kasper inequalities and a computer
procedure based on the Cochrane-Douglas
method\textsuperscript{1} (programmed in FORTRAN IV
for UNIVAC 1107 by the author). Least
squares refinement gave the R-value
\[ R_{\text{hkl}} = 8.5\% \], and Fig. 1 shows the final
electron density map. The R-value arrived
at in the 0kl-projection was \[ R_{\text{kl}} = 9.3\% \].
Fig. 2 shows the corresponding Fourier
map. Both maps show considerable over-
lapping, and publication of interatomic
distances and angles will be postponed
until the three-dimensional analysis, now
in progress, has been finished.

The three-dimensional refinements of
dimeric cyclohexanone peroxide\textsuperscript{2} and
dimeric cycloheptanone peroxide\textsuperscript{3} are
now at their final stages and will soon be
published. The results given in Table 1
may at the moment be stated.

Table 1. Distances and angles in dimeric cyclohexanone and cycloheptanone peroxide.

<table>
<thead>
<tr>
<th>Bond</th>
<th>Value</th>
<th>Dimeric cyclohexanone peroxide</th>
<th>Dimeric cycloheptanone peroxide</th>
</tr>
</thead>
<tbody>
<tr>
<td>C–C</td>
<td>(mean value)</td>
<td>1.52\textsubscript{4} Å</td>
<td>1.52\textsubscript{4} Å</td>
</tr>
<tr>
<td>C–C–C</td>
<td>(mean value)</td>
<td>111.3\textsuperscript{3}</td>
<td>115.3\textsuperscript{3}</td>
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<tr>
<td>C–O</td>
<td>(mean value)</td>
<td>1.43\textsubscript{3} Å</td>
<td>1.43\textsubscript{3} Å</td>
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<tr>
<td>O–O</td>
<td></td>
<td>1.47\textsubscript{4} Å</td>
<td>1.47\textsubscript{4} Å</td>
</tr>
<tr>
<td>C–O–O</td>
<td>(mean value)</td>
<td>107.4\textsuperscript{5}</td>
<td>107.9\textsuperscript{5}</td>
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<tr>
<td>O–C–O</td>
<td></td>
<td>108.2\textsuperscript{2}</td>
<td>107.8\textsuperscript{2}</td>
</tr>
</tbody>
</table>

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