Crystal Structure of 3,6-Spirodicyclooctylidene-1,2,4,5-tetraoxa-cyclohexane ("Dimeric Cyclooctanone Peroxide")

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of "dimeric cyclooctanone rvstals peroxide", prepared by T. Ledaal (to

be published), belong to the monoclinic system. The space group is  $P2_1/c$ , and the unit cell, containing two molecules, has the following parameters:

$$a = 9.79_1$$
 Å,  $b = 7.33_4$  Å,  $c = 11.77_9$  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 Cochran-Douglas method 1 (programmed in FORTRAN IV for UNIVAC 1107 by the author). Least squares refinement gave the R-value  $R_{hol} = 8.5 \%$ , and Fig. 1 shows the final electron density map. The R-value arrived at in the 0kl-projection was  $R_{okl} = 9.3 \%$ . Fig. 2 shows the corresponding Fourier map. Both maps show considerable overlapping, 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 2 and dimeric cycloheptanone peroxide 3 are now at their final stages and will soon be published. The results given in Table 1 may at the moment be stated.

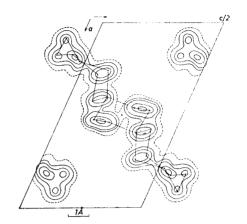


Fig. 1. Fourier projection along b-axis.

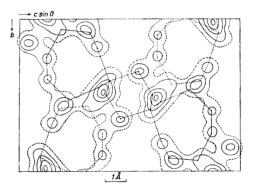


Fig. 2. Fourier projection along a-axis.

- 1. Cochran, W. and Douglas, A. S. Proc. Roy. Soc. (London) 227 (1954) 486; 243 (1958)
- 2. Groth, P. Acta Chem. Scand. 18 (1964) 1301.
- 3. Groth, P. Acta Chem. Scand. 18 (1964) 1801.

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

		Differie cyclo-	Dimeric cyclo-
		hexanone peroxide	heptanone peroxide
$\mathbf{C} - \mathbf{C}$	(mean value)	1.52, Å	1.52, Å
C-C-C	(mean value)	111.3° *	115.3° ¯
C-O	(mean value)	1.43 <sub>3</sub> Å	1.43 <sub>8</sub> Å
0-0		1.47, Å	1.47 <sub>1</sub> Å
$c_{-0-0}$	(mean value)	107.4°	107.9°
0-C-O		108.2°	107.8°

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