indicate a hexagonal unit cell of dimensions $a = 7.6519$ Å, $c = 3.3584$ Å, containing four formula units.

The Cr—As system. Arc-melting of a mixture of CrAs and Cr_4As_3 has yielded a new high-temperature phase, which could be indexed according to orthorhombic symmetry. The comparison of powder photographs and intensity calculations strongly suggested that this new phase – occurring together with CrAs – is isotypic with γ in the V—As system of the β-Yb_5Sb_3 structure type. The cell parameters found were $a = 9.263$ Å, $b = 7.446$ Å, $c = 6.393$ Å and $V = 440.9$ Å^3. In arc-melted alloys no intermediate phase more chromium-rich than Cr_3As was found. The Cr_3As (A15) phase as reported by Yuzuri [12] and Hollan *et al.*[13] was thus not obtained. Arc-melted specimens did not contain the high-temperature polymorph of Cr_3As (Fe_2P type). [14–16] This might be due to insufficiently rapid quenching. One specimen heat-treated in an induction furnace at 1450 °C showed, however, weak lines of the hexagonal phase together with the tetragonal form (Cu_2Sb type).

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On the Crystal Conformation of 1,3,9,11-Tetraoxacyclohexadecane at Room Temperature

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16-Membered rings have been studied by Dale and co-workers.[1,2] A marked tendency for the saturated ring skeleton to assume the diamond lattice is observed. Semiquantitative calculations of the conformational energies [3] show that the "square" conformation has the lowest enthalpy. This conformation was found in the crystals of 1,1,9,9-tetramethylcyclohexadecane.[4] Ring substitution by hetero atoms (oxygen) may reduce the *gauche* interactions at corner positions of the "square" conformation,[5] which indeed is the preferred crystal conformation of 1,5,9,13-tetraoxacyclohexadecane.[6] The compact "rectangular" conformation has only 5.03 kJ/mol higher enthalpy than the "square".[3] The symmetry number is 4 for both, but since only the "rectangular" form is a *d,l*-pair, it has an entropy term (at 300 K) of 1.7 kJ/mol in its favour, and the free-energy difference is only 3.4 kJ/mol.

The crystals of $C_{12}O_4H_{24}$ belong to the triclinic system with dimensions $a = 5.799(1)$ Å, $b = 7.731(1)$ Å, $c = 7.815(1)$ Å, $\alpha = 70.21(1)°$, $\beta = 82.46(1)°$, $\gamma = 88.19(1)°$ for Dirichlets reduced cell, and one molecule in the unit cell. 527 observed reflections were measured on an automatic four-circle diffractometer at room temperature (Mo$K\alpha$-radiation). Statistical tests strongly indicated the space group $P\bar{1}$. The structure was solved by direct methods [7] and refined by full-matrix least squares technique

Table 1. Final fractional coordinates (multiplied by 10^4) and thermal parameters for carbon and oxygen atoms.

Atom	X	Y	Z	B
O1	1194(43)	5805(29)	2081(29)	4.0(.6)
O2	7586(16)	6774(12)	6604(11)	4.4(.2)
O3	7636(23)	3908(18)	8940(16)	4.6(.3)
O4	4350(31)	3729(20)	2488(23)	4.3(.5)
C1	2131(23)	4551(16)	1652(17)	5.3(.3)
C2	2529(26)	7468(18)	1190(18)	5.8(.3)
C3	1787(29)	8887(21)	2262(24)	3.9(.4)
C4	2366(15)	8103(11)	4199(11)	4.0(.2)
C5	4909(33)	7804(20)	4447(22)	3.6(.4)
C6	5158(16)	6965(13)	6458(12)	5.4(.2)
C7	7899(25)	5940(19)	8379(17)	2.5(.3)
C8	9152(31)	2956(22)	8008(21)	4.7(.4)
C9	8001(22)	1312(15)	8205(16)	3.3(.3)
C10	5789(28)	1430(18)	7277(18)	4.0(.3)
C11	6172(27)	2521(17)	5194(19)	3.4(.3)
C12	3916(16)	2871(11)	4401(12)	3.4(.2)

and the minimum residual method.[8]* Hydrogen positions were calculated, but not refined. Final fractional coordinates and thermal parameters for carbon and oxygen atoms are given in Table 1. The R-value arrived at was 11.8 % (weighted value $R_w = 6.8$ %) for 527 observed reflections. Weights in least squares were obtained from the standard deviations in intensities, $\sigma(I)$, taken as

$$\sigma(I) = [C_T + (0.02C_N)^2]^{\frac{1}{2}}$$

where C_T is the total number of counts and C_N the net count.

The structure (shown in Fig. 1) is disordered, with equal amounts of *d*- and *l*-forms (ordered rings with half weight, related by the centre of symmetry in the unit cell. Averaged bond distances and angles of the title compound, (I), may be compared with those of 1,5,9,13-tetraoxacyclohexadecane,[6] (II):

	(I)	(II)
C–O	1.410 Å	1.415 Å
C–C	1.516 Å	1.507 Å
C–C–O	108.1°	108.3°
C–CO–C	112.6°	113.3°
C–C–C	113.3°	112.9°

The average O–C–O angle is 115.0°. Dihedral angles are given in Fig. 2.

A list of observed structure factors is available from the author.

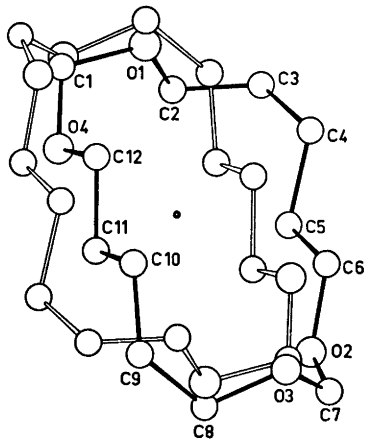

Fig. 1. Schematic drawing showing the statistical disorder in the crystal. The two (ordered) rings (with half weight) related by the centre of symmetry, are situated nearly at a common site in the unit cell.

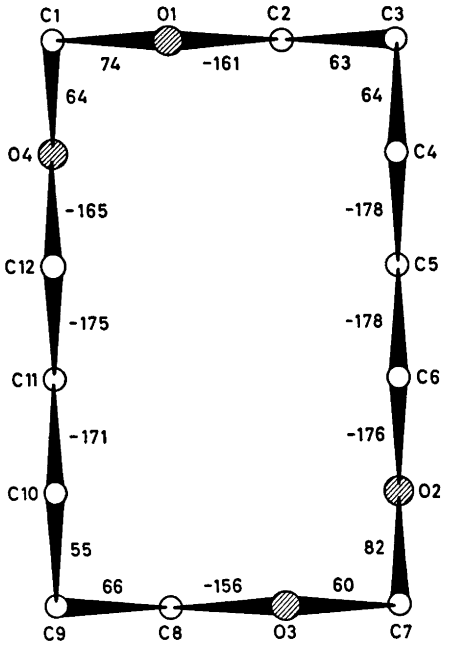

Fig. 2. Schematic drawing showing the dihedral angles.

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* All programs used (except those for phase determination) are included in this reference.