Crystal and Molecular Structure of Octachlorostyrene, C_8Cl_8, a Great Lakes Pollutant

JOHN C. HUFFMAN, Molecular Structure Center,
Department of Chemistry, Indiana University, Bloomington, Indiana 47405
and
RAY KAMINSKY and RONALD A. HITES,
School of Public and Environmental Affairs and Department of Chemistry,
Indiana University, Bloomington, Indiana 47405

Introduction

Octachlorostyrene (OCS, C_8Cl_8) has been found in the environment with increasing frequency in recent years. Although clearly anthropogenic, it was of no commercial importance, and the reasons for its presence were unknown. It has now been determined (5) that most of the OCS in the Great Lakes results from the waste products of electrolytic chlorine production. This study was undertaken to confirm the identity of OCS and to provide detailed structural information for this pollutant.

Experimental

Nearly all crystals of OCS examined suffered from splitting or twinning to some extent. After examination of numerous samples, we finally utilized a crystal which, athough not split, was of questionable quality due to a large mosiac spread along one axis[1]. The crystal, with a maximum dimension of 0.14 mm, was affixed to a glass fiber using silicone grease and transferred to a Picker four-circle goniostat where it was cooled to -163 °C using a gas flow cooling system (2). The diffractometer and data reduction techniques used in this study have been described in detail elsewhere (3).

A systematic search of a limited hemisphere of reciprocal space located a set of diffraction maxima which were consistent with the monoclinic space group $P2_1/a$. Cell dimensions, as determined by a least squares fit of 34 reflections centered automatically, are: a = 16.706(7), b = 10.124(5), c = 7.498(2) Å, β = 86.12(2)° V = 1265.3(3) Å^3, and D_{calc} = 1.993 gm/cm^3 for Z = 4. Continuous theta-two theta scan data were collected for $6 \leq 2\Theta \leq 40°$ for all data with indicies +h, +k, ±l. Less than 5% of the data were observed in the range $40 < 2\Theta \leq 45°$, indicative of the poor quality of the crystal. A number total of 1578 reflections were collected and reduced to 1180 unique intensities for the structural analysis. Of these, 881 were considered observed on the basis of $I \geq \sigma(I)$, and were used in the final refinements.

The structure was solved by a combination of direct methods, an interactive Patterson interpreter (1), and Fourier techniques. All atoms were refined anisotropically, and the final cycles included an isotropic extinction parameter as well as the positional parameters and an overall scale factor. The goodness of fit for the final cycle was 1.853 and the residuals[2] were R(F) = 0.069 and Rw(F) = 0.047. A final difference Fourier was featureless, the largest peak being 0.45 e/Å^3. Final coordinates are listed in Table 1. Anisotropic thermal parameters, observed and calculated structure factors, and other details are available[3].

Discussion

An ORTEP (4) drawing of the molecule is shown in Figure 1, and bonded distances and angles are listed in Table 2. In general, all distances and angles are within the normally expected values. Two distinct planes in the molecule, one defined by C(1)-C(6), Cl(9)-Cl(13) and the other by C(6)-C(8), Cl(14)-Cl(16) are planar within ±0.08Å, with

TABLE 1: *Fractional Coordinates for C_8Cl_8. Numbers in parenthesis represent the estimated errors in the least significant digits.*

Atom	x	y	z
C(1)	1.0099(6)	.2801(13)	.2047(13)
C(2)	.9786(7)	.4027(16)	.2103(15)
C(3)	1.0199(10)	.5108(15)	.2672(16)
C(4)	1.0986(8)	.4913(13)	.3196(14)
C(5)	1.1321(6)	.3682(11)	.3068(13)
C(6)	1.0888(7)	.2586(11)	.2536(14)
C(7)	1.1246(7)	.1171(15)	.2677(16)
C(8)	1.1590(7)	.0680(14)	.1344(18)
Cl(9)	.9567(2)	.1417(4)	.1439(4)
Cl(10)	.8812(2)	.4244(5)	.1455(4)
Cl(11)	.9794(3)	.6650(4)	.2749(5)
Cl(12)	1.1530(2)	.6241(3)	.3873(4)
Cl(13)	1.2287(2)	.3450(3)	.3685(4)
Cl(14)	1.1106(2)	.0449(3)	.4743(4)
Cl(15)	1.1719(2)	.1405(3)	-.0717(3)
C(16)	1.2025(2)	-.0913(3)	.1446(5)

C(7) lying 0.17° below the C(1)-C(6) plane. The two planes intersect with a dihedral angle of 94.6°, due primarily to the steric interactions of the large chlorine atoms, as shown in the space filling model drawing (6) of Figure 2.

Acknowledgments

The authors wish to thank the Bloomington Academic Computing Services for a generous gift of computer time. We also thank Prof. Manuel Ballester of the Instituto de Quimica Organica Aplicada, Barcelona, Spain, for the OCS sample.

Notes

1. The width of omega scans varied from 0.5 to 1.8°.
2. Residuals are defined as $R(F) = \Sigma|F_o-F_c|/\Sigma|F_o|$ and $Rw(F) = \Sigma w|F_o-F_c|/\Sigma w|F_o|$.
3. Complete crystallographic details are available in microfiche form from the Chemistry Library, Indiana University, Bloomington, Indiana 47405. Request Molecular Structure Center Report 82037.

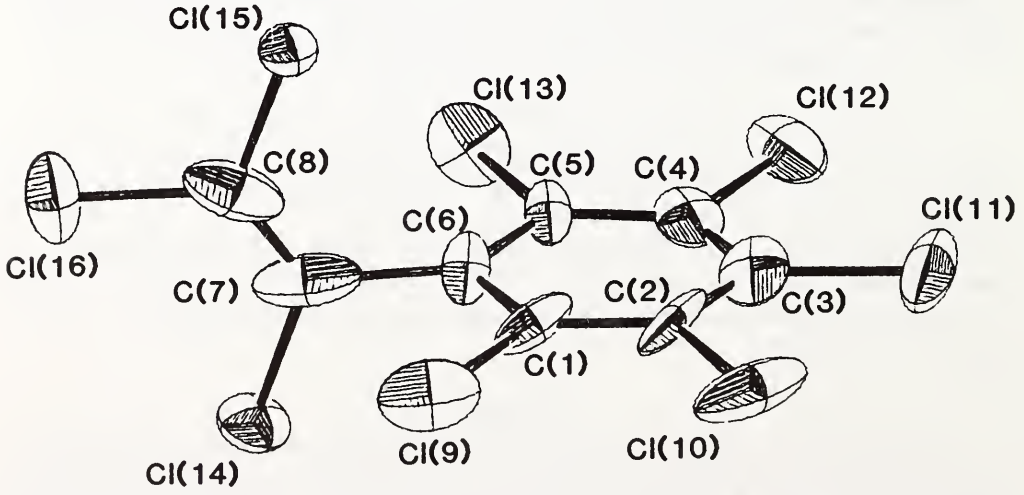

FIGURE 1. *ORTEP drawing of the C_8Cl_8 molecule showing the numbering scheme used in the tables.*

TABLE 2. *Bonded Distances (Ångstroms) and Angles (degrees) for C_8Cl_8.*

a) Distances

C(1)-C(2)	1.346(17)	C(1)-Cl(9)	1.735(13)
C(1)-C(6)	1.409(14)	C(2)-Cl(10)	1.743(12)
C(2)-C(3)	1.377(19)	C(3)-Cl(11)	1.701(15)
C(3)-C(4)	1.410(17)	C(4)-Cl(12)	1.719(13)
C(4)-C(5)	1.367(16)	C(5)-Cl(13)	1.724(10)
C(5)-C(6)	1.399(14)	C(7)-Cl(14)	1.715(13)
C(6)-C(7)	1.558(18)	C(8)-Cl(15)	1.712(13)
C(7)-C(8)	1.224(16)	C(8)-Cl(16)	1.773(15)

b) Angles

C(2)-C(1)-C(6)	120.1(12)	C(4)-C(5)-Cl(13)	119.5(10)
C(2)-C(1)-Cl(9)	123.2(10)	C(6)-C(5)-Cl(13)	118.7(8)
C(6)-C(1)-Cl(9)	116.6(10)	C(4)-C(5)-C(6)	121.7(10)
C(1)-C(2)-Cl(10)	118.4(13)	C(1)-C(6)-C(7)	122.0(11)
C(3)-C(2)-Cl(10)	118.9(12)	C(1)-C(6)-C(5)	117.6(10)
C(1)-C(2)-C(3)	122.7(12)	C(5)-C(6)-C(7)	120.1(9)
C(2)-C(3)-Cl(11)	122.2(13)	C(6)-C(7)-Cl(14)	115.0(9)
C(4)-C(3)-Cl(11)	119.7(13)	C(8)-C(7)-Cl(14)	126.3(13)
C(2)-C(3)-C(4)	118.1(12)	C(6)-C(7)-C(8)	118.7(12)
C(3)-C(4)-Cl(12)	119.6(12)	C(7)-C(8)-Cl(15)	125.9(13)
C(5)-C(4)-Cl(12)	120.7(11)	C(7)-C(8)-Cl(16)	120.4(11)
C(3)-C(4)-C(5)	119.6(12)	Cl(15)-C(8)-Cl(16)	113.7(9)

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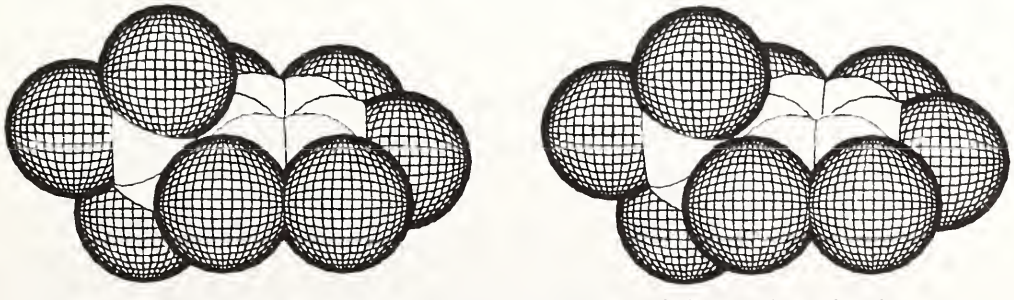

FIGURE 2. *Stereoscopic space filling model drawing of the C_8Cl_8 molecule.*