De structuur van I$_2$Cl$_6$(ICl$_3$)
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SUMMARY

In the introduction some chemical and physico-chemical properties of the interhalogen compounds are reviewed. Making use of existing thermodynamical data their stability is discussed in some more detail.

The interhalogens, especially the higher ones, stimulated theoretical speculations about their structures. For the greater part the recent structure investigations on these compounds, however, are not in agreement with the theoretically proposed structures.

A contribution to the structure chemistry of the interhalogen compounds has been given by the determination of the crystal structure of ICl₃ by means of X-ray methods.

Ordinary and integrated Weissenberg photographs from the first nine levels about the needle axis of the crystals, as well as Laue-, oscillation-, and flat film photographs were made. The interpretation of the photographs caused some difficulties, which could be overcome by assuming the crystals to consist of four complexly twinned triclinic individuals. The lattice constants are:

\[ a = 5.71 \pm 0.03 \text{ Å} \]
\[ b = 10.88 \pm 0.02 \text{ Å} \]
\[ c = 5.48 \pm 0.03 \text{ Å} \]
\[ \alpha = 130° \pm 5° \]
\[ \beta = 80° \pm 5° \]
\[ \gamma = 100° \pm 5° \]

Two ICl₃ per unit cell; space group PT.

Because of the complexity of the reciprocal lattice only one - the [010] - projection was accessible for two dimensional Patterson and Fourier methods. The [010] Patterson synthesis was first tentatively interpreted in terms of flat ICl₃ molecules with valence angles of 120°. Fourier syntheses based on this structure could not be refined. A correct approximation to the structure was obtained by the application of the vector convergence method on the (101) Patterson section. It could be confirmed by a Fourier synthesis of the (101) section which, however, still showed considerable false detail. The atomic coordinates were refined by a sequence of ordinary and difference Fourier syntheses. The false detail proved to be partly due to incorrect scaling factors relating reflections of different layer lines, and partly to the finite series effect.

The latter could be reduced by the introduction of an artificial temperature factor. The final coordinates, corrected for the remaining effect of the back shift

The final value of the crystal structure is arranged in layers where the crystal may be described by two chlorine atoms at 2.68 Å from I, and two others at 2.68 Å from I, the molecular symmetry being point group symmetry.

The possibility of the presence of ICl₃⁻ and ICl₄⁻ ions orbiting in the crystal matrix is considered.
and physico-chemical properties are reviewed. Making use of stability is discussed in higher ones, stimulated by products. For the greater trend on these compounds, hypothetically proposed structures are reviewed. Motions of the interhalogen compounds, howev-

However, photographs from the first stages of the crystal structures were made. The some difficulties, which lead to a structure of four layers. The lattice constants

\[ a = 130.50^\circ \pm 10^\circ \\
 b = 80.00^\circ \pm 20^\circ \\
 c = 10.30^\circ \pm 10^\circ \]

The reciprocal lattice is only one for two-dimensional Patterson synthesis was neces-

The crystal structure consists of molecules \( \text{I}_2\text{Cl}_6 \) which are arranged in layers \((10\overline{1})\). The two types of twinning occurring in the crystal may be explained by this structure.

The molecules are planar with negligible deviations from the point group symmetry \( \text{mmm} \) (fig. 15). Each iodine atom is surround-

The molecular structure is described in terms of a mesomerism between valence patterns involving \( \text{ICl}_2^- \) and \( \text{ICl}_4^- \) ions (pag. 67). The possibility of an average structure consisting of actual \( \text{ICl}_2^- \) and \( \text{ICl}_4^- \) ions could be excluded.

The final value of the disagreement index \( R \) is 0.12.

The introduction of an artifi-

The remaining effect of termination of the series by the application of the back shift method, are:

<table>
<thead>
<tr>
<th>x</th>
<th>y</th>
<th>z</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>0.0</td>
<td>67.0</td>
</tr>
<tr>
<td>Cl_1</td>
<td>99.1</td>
<td>48.6</td>
</tr>
<tr>
<td>Cl_2</td>
<td>97.9</td>
<td>167.2</td>
</tr>
<tr>
<td>Cl_3</td>
<td>97.8</td>
<td>286.5</td>
</tr>
</tbody>
</table>

The crystal structure consists of molecules \( \text{I}_2\text{Cl}_6 \) which are arranged in layers \((10\overline{1})\). The two types of twinning occurring in the crystal may be explained by this structure.

The molecules are planar with negligible deviations from the point group symmetry \( \text{mmm} \) (fig. 15). Each iodine atom is surrounded by two chlorine atoms at a distance of 2.38 and 2.39 Å and by two others at 2.68 and 2.72 Å.

The molecular structure is described in terms of a mesomerism between valence patterns involving \( \text{ICl}_2^- \) and \( \text{ICl}_4^- \) ions (pag. 67). The possibility of an average structure consisting of actual \( \text{ICl}_2^- \) and \( \text{ICl}_4^- \) ions could be excluded.