Two Modifications of Cobalt Dipotassium Tetrachloride

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Abstract. High-temperature K_2CoCl_4: orthorhombic, $a = 26 \cdot 838$ (3), $b = 12 \cdot 406$ (1), $c = 7 \cdot 262$ (1) Å, space group $Pna2_1$, $Z = 12$, adopts a slightly modified β-K_2SO_4 structure. Low-temperature K_2CoCl_4: monoclinic, $a = 6 \cdot 801$ (1), $b = 9 \cdot 569$ (1), $c = 12 \cdot 757$ (1) Å, $\beta = 107 \cdot 0$ (1)°, space group $P2_1/c$, $Z = 4$, adopts a strongly distorted β-K_2SO_4 structure.

Introduction. The structure determination of K_2CoCl_4 is part of an investigation of structural relationships and magnetic properties of ternary halides $A_xB_yX_z$ with A an alkali metal, B a first-row transition element or an alkaline-earth metal and X = Cl, Br or I.

The phase diagram of the system $KCl-CoCl_2$ was constructed by Seifert (1961). Apart from K_2CoCl_4 (m.p. 436 °C) it shows another compound, $KCoCl_3$, but not the phase transition we observe for K_2CoCl_4. By melting stoichiometric amounts of KCl and $CoCl_2$ in an evacuated quartz tube and annealing the mixture at 400 °C for one week, the orthorhombic modification of K_2CoCl_4 is obtained. After a few months this phase changes at room temperature into the monoclinic modification. The former is again obtained after annealing the latter at 400 °C. The two phases are easily recognized by their X-ray powder diagrams. We were unable to measure the transition temperature by means of DTA, but visual inspection of samples after heating suggests a phase transition between 350 and 360 °C.

Lamotte & Vermeire (1975) report that powder diffractograms of K_2CoCl_4 and $(NH_4)_2ZnCl_4$ can be indexed on the basis of an orthorhombic cell with axes $a = 8 \cdot 90$, $b = 12 \cdot 39$ and $c = 7 \cdot 25$ (Å) for K_2CoCl_4 and $a = 9 \cdot 20$, $b = 12 \cdot 56$ and $c = 7 \cdot 17$ (Å) for $(NH_4)_2ZnCl_4$. In both cases our diagrams show powder lines between $d = 3 \cdot 47$ and $3 \cdot 51$ Å, which cannot be indexed with these cell parameters. On tripling the a parameter these reflexions can be indexed as 202, 231 and 112. Klug & Sears (1945) also report a tripled a parameter for K_2ZnCl_4, based on single-crystal measurements.

Dry KCl was obtained by heating *in vacuo* at 400 °C, and dry $CoCl_2$ by heating $CoCl_2 \cdot 6H_2O$ in a stream of dry HCl gas at 400 °C. Low- and high-temperature K_2CoCl_4 were obtained by melting a stoichiometric mixture of KCl and $CoCl_2$ in an evacuated sealed quartz tube and slowly lowering the temperature over the course of a few days to room temperature with a temperature gradient of about 50 °C, with the higher temperature at the top of the melt. All manipulations of

K_2CoCl_4 were carried out in a glovebox filled with dry N_2 or under dry paraffin oil. The crystallized blue material was broken into pieces and crystals were selected. A series of Weissenberg photographs showed some crystals with orthorhombic and some with monoclinic symmetry. One suitable crystal of each phase was collected and measured by means of an Enraf–Nonius three-circle single-crystal diffractometer. Intensities were recorded by the θ–2θ scan method for all reflexions with θ between 3 and 30°.

Monochromatic (graphite) Mo $K\alpha$ radiation was used for measuring the intensities. Background intensities were determined at $\theta \pm \frac{1}{2}\Delta°$, with $\Delta = 0 \cdot 7 + 1 \cdot 3$ tan $\theta°$. The mean counting time was 25 s for each background and 50 s for the scan. Standard deviations were calculated from counting statistics. Absorption corrections were applied with a computer program written by de Graaff (1973), and new standard deviations (σ_F) were calculated taking into account the inaccuracy of the absorption correction and attenuation factors of the filters used. Non-equivalent significant reflexions were reduced to F values and Wilson plots were calculated, yielding approximate values for the scale factors and overall isotropic temperature parameters, B. Table 1 gives the crystal and diffraction data.

According to the reflexion conditions for orthorhombic K_2CoCl_4, another space group (*Pnam*) is possible, which the Patterson synthesis proved to be incorrect.

Table 1. *Crystal and diffraction data for*
K_2CoCl_4

	Orthorhombic	Monoclinic
a	26·838 (3) Å	6·801 (1) Å
b	12·406 (1)	9·569 (1)
c	7·262 (1)	12·757 (1)
β		107·0 (1)°
Z	12	4
Space group	$Pna2_1$	$P2_1/c$
Measured reflexions	7502	3447
Independent reflexions	4017	2447
Significant reflexions	2141	1655
Reflexion conditions	$0kl: k + l = 2n$	$h0l: l = 2n$
	$h0l: h = 2n$	$0k0: k = 2n$
Absorption coefficient (μ)	44·1 cm⁻¹	44·8 cm⁻¹
Transmission factor	0·35–0·68	0·54–0·68
Crystal dimensions	0·2 × 0·2 × 0·1 mm	0·15 × 0·14 × 0·1 mm

Table 2. *Positional parameters* $(\times 10^4)$ *and anisotropic thermal parameters* $(\times 10^3)$ *for orthorhombic* K_2CoCl_4

	x	y	z	U_{11}	U_{22}	U_{33}	$2U_{21}$	$2U_{23}$	$2U_{31}$
K(1)	442 (1)	808 (3)	7879 (7)	36 (2)	78 (3)	92 (3)	−16 (3)	−10 (6)	−6 (4)
K(2)	3801 (1)	836 (4)	7662 (7)	41 (2)	98 (3)	74 (3)	2 (4)	−8 (5)	21 (4)
K(3)	7110 (1)	822 (3)	7246 (7)	33 (2)	63 (2)	71 (2)	−5 (3)	−8 (5)	−11 (3)
K(4)	3322 (1)	6883 (2)	7249 (5)	38 (1)	33 (2)	57 (2)	3 (2)	0 (3)	11 (3)
K(5)	6663 (1)	6872 (2)	7428 (5)	38 (1)	35 (2)	60 (2)	−4 (2)	4 (3)	−8 (3)
K(6)	9991 (1)	6884 (2)	7954 (5)	37 (1)	30 (2)	36 (1)	−3 (2)	10 (3)	6 (3)
Co(1)	720 (1)	4199 (2)	7500*	23 (1)	30 (1)	30 (1)	3 (1)	−2 (2)	0 (1)
Co(2)	4051 (1)	4178 (2)	7800 (3)	25 (1)	26 (1)	34 (1)	1 (1)	2 (2)	2 (1)
Co(3)	7402 (1)	4181 (2)	7351 (1)	28 (1)	27 (1)	27 (1)	−4 (1)	−4 (2)	1 (1)
Cl(1)	9917 (1)	4445 (2)	6752 (5)	29 (1)	49 (2)	47 (2)	11 (2)	−24 (3)	−10 (3)
Cl(2)	1132 (1)	5755 (2)	8166 (5)	34 (1)	31 (2)	78 (2)	−18 (2)	−10 (4)	13 (3)
Cl(3)	839 (1)	3017 (2)	9836 (5)	61 (2)	52 (2)	43 (2)	44 (2)	37 (3)	10 (3)
Cl(4)	1105 (1)	3549 (3)	4941 (5)	38 (1)	75 (2)	35 (2)	18 (3)	−37 (3)	4 (3)
Cl(5)	3228 (1)	4385 (2)	8170 (7)	23 (1)	58 (2)	94 (3)	−1 (2)	33 (4)	−4 (3)
Cl(6)	4449 (1)	5778 (3)	8139 (8)	43 (2)	31 (2)	157 (4)	−28 (3)	−6 (5)	−2 (5)
Cl(7)	4341 (1)	3074 (4)	18 (7)	65 (2)	115 (3)	56 (3)	83 (4)	89 (5)	11 (4)
Cl(8)	4299 (1)	3475 (3)	5147 (5)	104 (3)	101 (3)	42 (2)	112 (5)	−47 (5)	−5 (4)
Cl(9)	6589 (1)	4351 (2)	8077 (5)	27 (1)	60 (2)	84 (3)	−3 (2)	40 (4)	21 (3)
Cl(10)	7798 (1)	5752 (3)	6690 (7)	42 (2)	38 (2)	61 (2)	−20 (3)	9 (4)	−8 (3)
Cl(11)	7778 (1)	3606 (3)	21 (5)	56 (2)	54 (2)	37 (2)	15 (3)	14 (3)	−25 (3)
Cl(12)	7555 (1)	2935 (2)	5178 (7)	68 (2)	46 (2)	38 (2)	8 (3)	−26 (3)	5 (3)

* This value was kept fixed during the refinements.

Table 3. *Positional parameters* $(\times 10^4)$ *and anisotropic thermal parameters* $(\times 10^4)$ *for monoclinic* K_2CoCl_4

	x	y	z	U_{11}	U_{22}	U_{33}	$2U_{21}$	$2U_{23}$	$2U_{31}$
K(1)	2247 (3)	8555 (1)	4935 (1)	475 (9)	356 (7)	379 (7)	126 (13)	−99 (11)	−82 (13)
K(2)	4036 (3)	961 (1)	1646 (1)	548 (10)	364 (6)	418 (7)	−25 (13)	−143 (11)	394 (14)
Co	1169 (1)	3537 (1)	3197 (1)	297 (4)	278 (3)	199 (3)	−27 (7)	−2 (6)	127 (6)
Cl(1)	2131 (3)	3997 (1)	1660 (1)	389 (9)	381 (7)	235 (6)	27 (13)	106 (10)	210 (12)
Cl(2)	2252 (3)	6884 (1)	7210 (1)	303 (8)	402 (7)	413 (8)	−74 (13)	−164 (12)	152 (14)
Cl(3)	3011 (3)	1626 (1)	3998 (2)	478 (10)	436 (8)	408 (8)	205 (15)	328 (14)	196 (15)
Cl(4)	2061 (3)	5403 (1)	4324 (1)	731 (13)	375 (8)	327 (7)	−285 (16)	−239 (12)	395 (16)

Table 4. *Distances and angles in orthorhombic* K_2CoCl_4

Cobalt–chlorine distances (Å)

Co(1)–Cl(1)	2·243 (4)	Co(2)–Cl(5)	2·240 (3)	Co(3)–Cl(9)	2·254 (4)
Co(1)–Cl(2)	2·277 (4)	Co(2)–Cl(6)	2·267 (4)	Co(3)–Cl(10)	2·271 (4)
Co(1)–Cl(3)	2·265 (4)	Co(2)–Cl(7)	2·253 (5)	Co(3)–Cl(11)	2·299 (4)
Co(1)–Cl(4)	2·274 (4)	Co(2)–Cl(8)	2·217 (5)	Co(3)–Cl(12)	2·247 (4)

Cobalt–chlorine angles (°)

Cl(1)–Co(1)–Cl(2)	113·8 (2)	Cl(5)–Co(2)–Cl(6)	110·5 (2)	Cl(9)—Co(3)–Cl(10)	115·0 (2)
Cl(1)–Co(1)–Cl(3)	113·9 (2)	Cl(5)–Co(2)–Cl(7)	109·0 (2)	Cl(9)—Co(3)–Cl(11)	104·8 (2)
Cl(1)–Co(1)–Cl(4)	106·7 (2)	Cl(5)–Co(2)–Cl(8)	116·5 (2)	Cl(9)—Co(3)–Cl(12)	113·9 (2)
Cl(2)–Co(1)–Cl(3)	108·7 (2)	Cl(6)–Co(2)–Cl(7)	107·0 (2)	Cl(10)–Co(3)–Cl(11)	103·9 (2)
Cl(2)–Co(1)–Cl(4)	104·7 (2)	Cl(6)–Co(2)–Cl(8)	107·3 (2)	Cl(10)–Co(3)–Cl(12)	110·9 (2)
Cl(3)–Co(1)–Cl(4)	108·6 (2)	Cl(7)–Co(2)–Cl(8)	106·2 (2)	Cl(11)–Co(3)–Cl(12)	107·4 (2)

Potassium–chlorine distances (Å) up to 4·1 Å

K(1)–Cl(9)	3·089 (4)	K(2)–Cl(1)	3·086 (4)	K(3)–Cl(5)	3·087 (4)
K(1)–Cl(3)	3·267 (5)	K(2)–Cl(2)	3·272 (6)	K(3)–Cl(11)	3·203 (6)
K(1)–Cl(6)	3·318 (8)	K(2)–Cl(4)	3·294 (6)	K(3)–Cl(10)	3·238 (7)
K(1)–Cl(8)	3·402 (6)	K(2)–Cl(11)	3·311 (5)	K(3)–Cl(12)	3·249 (5)
K(1)–Cl(6)	3·455 (8)	K(2)–Cl(10)	3·410 (5)	K(3)–Cl(4)	3·269 (5)
K(1)–Cl(7)	3·613 (6)	K(2)–Cl(7)	3·569 (7)	K(3)–Cl(2)	3·341 (5)
K(1)–Cl(8)	3·758 (6)	K(2)–Cl(8)	3·980 (6)	K(3)–Cl(10)	4·043 (7)
K(1)–Cl(6)	3·831 (8)	K(2)–Cl(2)	4·002 (6)		
		K(2)–Cl(12)	4·095 (6)		
K(4)–Cl(3)	3·180 (5)	K(5)–Cl(11)	3·153 (5)	K(6)–Cl(7)	3·153 (6)
K(4)–Cl(5)	3·180 (4)	K(5)–Cl(9)	3·169 (4)	K(6)–Cl(1)	3·156 (4)
K(4)–Cl(12)	3·182 (6)	K(5)–Cl(12)	3·181 (6)	K(6)–Cl(8)	3·173 (5)
K(4)–Cl(4)	3·234 (5)	K(5)–Cl(7)	3·216 (6)	K(6)–Cl(3)	3·179 (5)
K(4)–Cl(10)	3·280 (5)	K(5)–Cl(8)	3·281 (5)	K(6)–Cl(1)	3·222 (5)
K(4)–Cl(6)	3·382 (5)	K(5)–Cl(2)	3·315 (4)	K(6)–Cl(6)	3·248 (5)
K(4)–Cl(9)	3·403 (5)	K(5)–Cl(10)	3·390 (4)	K(6)–Cl(4)	3·319 (4)
K(4)–Cl(11)	3·421 (5)	K(5)–Cl(5)	3·476 (6)	K(6)–Cl(2)	3·371 (4)

All calculations were carried out on the Leiden University 360-65 (and later 370-158) IBM computer, with a set of computer programs written or modified by Rutten-Keulemans and de Graaff. Scattering factors and anomalous dispersion for the ions were taken from *International Tables for X-ray Crystallography* (1974). The function minimized during the least-squares refinement was $\sum w_F(|F_o| - |F_c|)^2$ using the weighting scheme $w_F = 1/\sigma_F^2$. R_w is defined by $[\sum_F w_F(|F_o| - |F_c|)^2/\sum_F w_F F_o^2]^{1/2}$ and R by $\sum_F(|F_o| - |F_c|)/\sum_F F_o$.

Orthorhombic K_2CoCl_4: Interpretation of the three-dimensional Patterson synthesis proved to be a difficult task, because of the superstructure in the **a** direction. Only the Co ions could be unequivocally determined. The K ions and one Cl tetrahedron were allocated assuming that the structure resembled the β-K_2SO_4 structure. After a few cycles of least-squares refinement R_w dropped to 0·4. The remaining atoms could be found by calculating minimum functions and three-dimensional Fourier maps. With isotropic temperature factors and full-matrix refinement, R_w dropped to 0·1. R_w dropped to 0·039 and R to 0·038 after full-matrix refinement with anisotropic temperature factors. Refinement of the inverted structure results in a somewhat higher R_w (0·040). A difference Fourier synthesis revealed no significant peaks. Positional parameters and anisotropic thermal parameters are listed in Table 2.*

Monoclinic K_2CoCl_4: From a three-dimensional Patterson synthesis the positions of the Co, all the Cl and one of the K ions could be determined. These positions, isotropic temperature parameters and block-diagonal matrix refinement were used to reduce R_w to 0·32. With a Fourier synthesis the other K position could be determined and R_w dropped to 0·08 with isotropic temperature factors and block-diagonal refinement. R_w fell to 0·041 ($R = 0·051$) after full-matrix refinement with anisotropic temperature factors. A difference Fourier synthesis revealed no significant peaks. The positional parameters and the anisotropic temperature factors are listed in Table 3.*

Discussion. Tables 4 and 5 show Co–Cl distances and angles and K–Cl distances for orthorhombic and monoclinic K_2CoCl_4 respectively.

Idealized orthorhombic K_2CoCl_4 axes can be derived from β-K_2SO_4 axes (*Pnam* setting) by the following transformation:

$$\begin{pmatrix} a' \\ b' \\ c' \end{pmatrix} = \begin{pmatrix} 3 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix} \begin{pmatrix} a \\ b \\ c \end{pmatrix},$$

where a, b and c are the axes of β-K_2SO_4, and a', b' and c' are the axes of idealized orthorhombic K_2CoCl_4.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31934 (11 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 5. *Distances and angles in monoclinic K_2CoCl_4*

Cobalt–chlorine distances (Å)

| Co–Cl(1) | 2·284 (2) | Co–Cl(3) | 2·283 (2) |
| Co–Cl(2) | 2·267 (2) | Co–Cl(4) | 2·261 (2) |

Cobalt–chlorine angles (°)

Cl(1)–Co–Cl(2)	111·74 (6)	Cl(1)–Co–Cl(3)	106·34 (7)
Cl(1)–Co–Cl(4)	107·65 (6)	Cl(2)–Co–Cl(3)	110·63 (6)
Cl(2)–Co–Cl(4)	110·61 (7)	Cl(3)–Co–Cl(4)	109·73 (6)

Potassium–chlorine distances (Å) up to 3·5 Å

K(1)–Cl(1)	3·105 (2)	K(2)–Cl(2)	3·148 (2)
K(1)–Cl(4)	3·109 (2)	K(2)–Cl(4)	3·155 (2)
K(1)–Cl(3)	3·117 (2)	K(2)–Cl(1)	3·183 (2)
K(1)–Cl(1)	3·230 (2)	K(2)–Cl(2)	3·255 (2)
K(1)–Cl(3)	3·270 (2)	K(2)–Cl(4)	3·284 (3)
K(1)–Cl(2)	3·313 (2)	K(2)–Cl(3)	3·334 (2)
K(1)–Cl(2)	3·503 (2)	K(2)–Cl(1)	3·421 (2)

The CoCl$_4$ tetrahedra and K ions are more irregularly distributed than the SO$_4$ tetrahedra and K ions in β-K_2SO_4; this results in lower coordination numbers for the K ions (nine and ten in β-K_2SO_4). This effect is commonly observed in K_2SO_4-like structures. K(1), K(2) and K(3) are in irregular six coordination; K–Cl distances range from 3·089 to 3·613 Å (mean 3·304 Å). K(4), K(5) and K(6) are surrounded by Cl ions at the apices of an Archimedian antiprism. K–Cl distances range from 3·152 to 3·476 (Å) (mean 3·261 Å), and the Co–Cl distances from 2·217 to 2·299 Å (mean 2·259 Å).

Idealized monoclinic K_2CoCl_4 axes can be derived from β-K_2SO_4 axes (*Pnam* setting) by the following transformation:

$$\begin{pmatrix} a' \\ b' \\ c' \end{pmatrix} = \begin{pmatrix} 0 & 0 & 1 \\ 1 & 0 & 0 \\ 0 & 1 & \bar{1} \end{pmatrix} \begin{pmatrix} a \\ b \\ c \end{pmatrix},$$

where a', b' and c' are the axes of idealized monoclinic K_2CoCl_4, and a, b and c are the axes of β-K_2SO_4. In β-K_2SO_4, b/c is almost $\sqrt{3}$; thus the idealized angle β in monoclinic K_2CoCl_4 is 120°. The tetrahedra are rotated more than in orthorhombic K_2CoCl_4 and are moved along with the K ions, resulting in an irregular seven coordination for K; K–Cl distances range from 3·105 to 3·503 Å (mean 3·245 Å), and the Co–Cl distances from 2·261 to 2·284 Å (mean 2·274 Å). We are not aware of other compounds with the same structure as monoclinic K_2CoCl_4. In both cases the anisotropic

Table 6. *Some compounds with β-K_2SO_4-like structures*

	Space group	a (Å)	b (Å)	c (Å)	β
K_2SO_4[a]	*Pnma*	7·456	5·776	10·08	
K_2CoCl_4[b]	*Pna2$_1$*	26·838	12·406	7·261	
K_2CoCl_4[b]	*P2$_1$/c*	6·801	9·569	12·757	107·0°
Rb_2CoCl_4[c]	*Pnma*	9·272	7·283	12·723	
Cs_2CoCl_4[d]	*Pnma*	9·737	7·792	12·972	
K_2ZnCl_4[e]	*Pma2*	26·70	12·26	7·28	
$(NH_4)_2ZnCl_4$[f]		9·20	7·17	12·56	

(a) Robinson (1958). (b) This work. (c) Amit, Horowitz & Makovsky (1972). (d) Poray-Koshits (1954). (e) Klug & Sears (1945). (f) Lamotte & Vermeire (1975).

temperature factors are unusually high; this probably indicates that there is some disorder in these compounds.

In Table 6 some other compounds with β-K$_2$SO$_4$-like structures are listed. The structure of K$_2$ZnCl$_4$ is not yet known, but the agreement between the axes of this compound and orthorhombic K$_2$CoCl$_4$ suggests that the structures may be almost the same.

References

AMIT, M., HOROWITZ, A. & MAKOVSKY, J. (1972). *Israel J. Chem.* **10**, 715–719.

GRAAFF, R. A. G. DE (1973). *Acta Cryst.* A**29**, 298–301.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 71–151. Birmingham: Kynoch Press.

KLUG, H. P. & SEARS, G. W. JR (1945). *J. Amer. Chem. Soc.* **67**, 878.

LAMOTTE, J. & VERMEIRE, M. (1975). *J. Appl. Cryst.* **8**, 689–693.

PORAY-KOSHITS, M. A. (1954). *Trud. Inst. Kristallogr. Akad. Nauk SSSR*, **10**, 117–135.

ROBINSON, M. T. (1958). *J. Phys. Chem.* **62**, 925–928.

SEIFERT, H. J. (1961). *Z. anorg. allgem. Chem.* **307**, 137–144

Hydrogen-Bond Motifs of Carboxylic Acids: the α-Form of Monochloroacetic Acid

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Abstract. C$_2$H$_3$O$_2$Cl, monoclinic, $P2_1/c$; $a = 5.380$ (1), $b = 19.150$ (4), $c = 7.860$ (2) Å, $\beta = 109.14$ (10)°; $Z = 8$, $D_c = 1.64$ g cm^{-3}. The structure consists of centrosymmetric tetramers formed by hydrogen bonding between carboxyl groups of the two independent molecules. Corresponding bond distances and angles involving non-hydrogen atoms of the independent molecules are very similar; their conformations differ: one molecule is planar, the other has a dihedral angle of 20.3°.

Introduction. C$_2$H$_3$O$_2$Cl exists in three distinct crystalline varieties (Aumeras & Minangoy, 1948). The α-form is the stable modification at ambient conditions with a m.p. of 64°C, the β- and γ-forms are metastable with m.p.'s of 54 and 51 °C respectively. From IR and Raman spectroscopy it was concluded that the metastable β-form crystallizes in the cyclic centrosymmetric hydrogen-bonded dimer type (Sinha, Katon & Jakobsen, 1975), whereas the results for the α-form are inconsistent with a dimer structure and strongly indicate a hydrogen-bond motif with a polymeric nature (Sinha, Katon & Jakobsen, 1974). The analysis of the α-form was performed to elucidate the hydrogen-bond motif.

Prismatic crystals were obtained by sublimation *in vacuo*. A crystal $0.3 \times 0.4 \times 0.5$ mm was selected and

Table 1. *Fractional coordinates* ($\times 10^4$ *for* Cl, O *and* C; $\times 10^3$ *for* H) *and anisotropic thermal parameters* ($\times 10^4$) *of the non-hydrogen atoms*

The estimated standard deviations are given in parentheses and refer to the last decimal position of respective values. The anisotropic temperature factor is of the form $\exp[-2\pi^2(a^{*2}h^2U_{11} + \ldots + 2b^*c^*klU_{23})]$.

	x	y	z	U_{11} or B (Å^2)	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Molecule 1									
Cl	−379 (2)	6706 (1)	9904 (1)	722 (6)	560 (5)	498 (5)	−116 (4)	353 (4)	−107 (4)
O(1)	3057 (5)	6596 (1)	6272 (3)	625 (15)	415 (14)	541 (14)	−97 (11)	334 (12)	−61 (10)
O(2)	782 (5)	5806 (1)	7228 (4)	706 (16)	340 (11)	735 (17)	−110 (11)	454 (14)	−89 (11)
C(1)	1669 (6)	6384 (2)	7276 (4)	426 (15)	345 (15)	379 (15)	11 (12)	158 (13)	21 (12)
C(2)	1461 (9)	6961 (2)	8517 (5)	841 (29)	394 (18)	511 (21)	−71 (18)	373 (20)	−21 (15)
H(1)	322 (7)	713 (2)	925 (5)	3·8					
H(2)	62 (7)	730 (2)	786 (5)	3·8					
H(3)	302 (7)	631 (2)	549 (5)	3·7					
Molecule 2									
Cl	−6497 (2)	3409 (1)	6768 (1)	529 (5)	422 (4)	696 (6)	−115 (4)	274 (4)	−59 (4)
O(1)	−3047 (5)	5177 (1)	8216 (3)	596 (15)	518 (14)	589 (15)	−187 (12)	361 (12)	−190 (12)
O(2)	−2747 (5)	4434 (1)	6128 (3)	564 (14)	448 (13)	564 (14)	−96 (11)	350 (12)	−119 (11)
C(1)	−3783 (6)	4623 (2)	7186 (4)	369 (15)	396 (15)	402 (15)	−6 (12)	166 (13)	2 (12)
C(2)	−6106 (7)	4282 (2)	7501 (6)	499 (20)	396 (17)	659 (23)	−66 (15)	324 (18)	−70 (16)
H(1)	−611 (6)	430 (2)	873 (5)	3·6					
H(2)	−741 (7)	451 (2)	688 (5)	3·6					
H(3)	−157 (7)	546 (2)	806 (5)	3·9					