The packing of the molecules is illustrated in a projection along [010] in Fig. 5. Only intermolecular distances less than 3·60 Å between the heavy atoms are indicated. A few very short distances are listed in Table 7. Very small intermolecular C–O distances have also been reported by other authors (Davies & Blum, 1955; Chu, Jeffrey & Sakurai, 1962; Romers, 1964) and their existence is less exceptional than might be expected on the basis of spherical van der Waals radii of atoms.

All calculations mentioned in this paper were carried out on the X1 computer of the University of Leiden with programs devised by:

1. Elisabeth W. M. Rutten, Fourier and structure factor-least-squares programs (Rutten, 1963).
2. Mr W. M. H. Rutten, correlation and scaling of different zones and also 'best least-squares' planes.
3. Dr H. Geise, reduction of reflexion intensities to structure factors (Geise, 1964).
4. The second author (B.H.), computation of intramolecular bond distances and bond angles and intermolecular bond distances.

A part of this investigation dealing with the −120 °C data was performed under the guidance of Prof. J. Waser (now at Caltech, Pasadena, Calif.) at Rice University, Houston, Texas, during the tenure of a postdoctoral fellowship by one of us (C.R.) in 1956–1957. The first author wishes to express his gratitude for the instruction and hospitality received during that time.

We also wish to thank Prof. L. J. Oosterhoff for his continuous interest in this research. We are indebted to Mr A. Kreuger, who collected the −120 °C data, and to Miss H. van Oijen, who made the diffraction photographs at −180 °C. Finally we thank Dr H. Geise for his assistance with several computations.

References

Altona, C. (1964). *Acta Cryst.* **17**, 1282.

Berghuis, J., Haanappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. & Veenendaal, A. L. (1955). *Acta Cryst.* **8**, 478.

Chu, S. S. C., Jeffrey, G. A. & Sakurai, T. (1962). *Acta Cryst.* **15**, 661.

Coppens, P. & Schmidt, G. M. J. (1965). *Acta Cryst.* **18**, 654.

Davies, D. R. & Blum, J. J. (1955). *Acta Cryst.* **8**, 129.

Fischmann, E., MacGillavry, C. H. & Romers, C. (1961*a*). *Acta Cryst.* **14**, 753.

Fischmann, E., MacGillavry, C. H. & Romers, C. (1961*b*). *Acta Cryst.* **14**, 759.

Geise, H. J. (1964). Thesis, Leiden.

Hanson, A. W. (1953). *Acta Cryst.* **6**, 32.

Hauptman, H. & Karle, J. (1953). *Solution of the Phase Problem.* A.C.A. Monograph, no. 3.

Hirshfeld, F. L. (1964). *Israel J. Chem.* **2**, 87.

Kehrmann, F. & Decker, H. (1921). *Ber. dtsch chem. Ges.* **54**, 2439.

McWeeny, R. (1951). *Acta Cryst.* **4**, 513.

Mulder, J. J. C. & Lugt, W. T. A. M. van der, unpublished.

Oijen, H. van & Romers, C. (1966). *Acta Cryst.* **20**, 169.

Pople, J. A. (1953). *Trans. Faraday Soc.* **49**, 1375.

Rollett, J. S. & Sparks, R. A. (1960). *Acta Cryst.* **13**, 273.

Romers, C. (1964). *Acta Cryst.* **17**, 1287.

Rutten, E. W. M. (1963). *Acta Cryst.* **16**, A171.

Schomaker, V., Waser, J., Marsh, R. E. & Bergman, G. (1959). *Acta Cryst.* **12**, 600.

Vroom, R. (1966). To be published.

Wilson, A. J. C. (1949). *Acta Cryst.* **2**, 318.

Woolfson, M. M. (1961). *Direct Methods in Crystallography.* Oxford: Clarendon Press.

Acta Cryst. (1966). **20**, 169

Isomerism of Benzoquinone Monoximes (Nitrosophenols). XIV. The Crystal Structure of α-5-(2′-Chloroethoxy)-*o*-quinone-2-oxime at −180 °C*

By Johanna W. L. van Oijen and C. Romers
Organic Chemistry Department, University of Leiden, The Netherlands

(*Received* 14 *May* 1965)

Crystals of α-5-(2′-chloroethoxy)-*o*-quinone-2-oxime are monoclinic with space group $P2_1/c$ and $Z=4$. The lattice constants at −180 °C are $a=4{\cdot}01$, $b=10{\cdot}87$, $c=20{\cdot}08$ Å, $\beta=103°\ 36'$. The structure was refined by the least-squares method; the data used were integrated reflexions recorded at −180 °C with a Weissenberg goniometer and measured with an automatic recording microdensitometer. The final R value is 6·77 %.

The molecule has an oxime structure with CO and NOH groups in the *anti* configuration. Intermolecular hydrogen bridges connect CO and NOH groups of different molecules by means of a screw axis operation. The $Cl–C_2H_4–O$ group has the *gauche* conformation.

Introduction

In an earlier paper of this series Romers & Umans (1960) published an investigation of 5-alkoxy-*o*-quinone-2-oximes occurring in two modifications α and β that were discovered by Henrich & Eisenach (Henrich, 1904; Henrich & Eisenach, 1904). Romers & Umans reported that in the β modification an intramolecular hydrogen bridge exists between the CO(H) and NO(H) groups in the *syn* configuration and that intermolecular hydrogen bonds connect CO and NOH groups of dif-

---

* Part XIII: Romers & Hesper, 1966.

ferent molecules in the crystalline α modification. Bartindale, Crowder & Morley (1959) derived the same *anti* configuration of the α modifications from the crystal structure determination of α-5-methoxy-*o*-quinone-2-oxime. The preliminary work of Romers & Umans had been performed with two-dimensional methods that made more accurate structure refinements desirable. An accurate structure determination of the β modification of 5-n-propoxy-*o*-quinone-2-oxime was published in this journal (Romers, 1964) and this research has now been complemented with a refinement of the structure of α-5-(2′-chloroethoxy)-*o*-quinone-2-oxime (abbreviated α-CEQ).

Experimental

The compound α-CEQ, $C_8H_8NO_3Cl$, m.p. 152–153 °C, was prepared as described by Hodgson & Clay (1932). The orange crystals are monoclinic and consist of heavily twinned shafts elongated along the *a* axis. The cell dimensions (Table 1) were determined at 20 °C and at − 180 °C by means of zero-layer Weissenberg photographs rotated about [100] and [010] and superposed with aluminum powder lines for calibration purposes. It follows from the observed density (1·52 g.cm^{-3}) and the absent reflexions, $h0l$ for l = odd and $0k0$ for k = odd, that four molecules have to be located in a unit cell with space group symmetry $P2_1/c$. The cell dimensions of the corresponding isomorphous bromo compound are also given in Table 1.

A very small needle (0·07 × 0·14 × 1·0 mm) was selected for the recording of integrated equi-inclination Weissenberg photographs rotating about [100] at − 180°C with nickel-filtered Cu $K\alpha$ radiation (λ = 1·5418 Å). The cooling technique developed in this laboratory by Altona (1964) was used with a split camera loaded with four films, exposure time 40–50 hours. Additional shorter exposures were made with a whole camera in order to obtain the small angle reflexions. In this way the layers h = 0, 1, 2 and 3 of the reciprocal lattice could be obtained. Crystals cut for rotation about [010] proved to be heavily twinned and their diffraction photographs were only used for unit-cell measurements. Difficulties were met in indexing the diffraction pattern with small c^* spacings and errors due to false indexing were discovered during the refinement process.

The reflexion intensities were measured with a double beam servo null balance instrument, the micro-densitometer III† that permits the continuous recording of the optical density of photographic patterns. From previous knowledge of the density/intensity curve of the film material in use it was possible to adjust a linearizer unit for intensity-linear recording. In this way the intensities of nearly all reflexions could be properly measured. A few of the strongest reflexions were measured by setting the instrument on density-recording. Special care had to be taken to correct the measured intensities for two effects that have a strong influence on the observed diffraction patterns. In the first place the crystal used consisted of two slightly misaligned individuals. Their reflexions overlap perfectly on the zero layer, but are separated on some parts of the upper level films and coincide partly or completely on other parts. This effect is largest on the level h = 3. In the second place the integration was insufficient to compensate the extension of spots with small ξ-values (Buerger, 1949) on upper level photographs. A careful measurement of the intensities and the lengths of symmetry-related reflexions occurring simultaneously on extended and contracted parts of the films revealed the necessary correction factor for this effect.

Finally, the intensities were reduced to structure factor moduli in the usual way with a program developed by Geise (1964). Since the cross-section of the crystal in use was very small it was not necessary to correct for absorption. From the total number of 1217 measured reflexions ultimately 1183 were used during the refinement.

The refinement

Initial atomic parameters (see Fig. 2 for the numbering of the atoms) were derived from the model found by Romers & Umans by means of the isomorphous replacement technique. The refinement of the structure was carried out by the method of least squares with a program devised by Mrs E. W. M. Rutten (Geise, Romers & Rutten, 1966). This program uses 3 × 3 blocks for positional parameters, 1 × 1 blocks for individual isotropic temperature factors and 6 × 6 matrices for anisotropic thermal parameters. An $(n+1) \times (n+1)$ matrix, containing n scale factors and one overall temperature factor, is optional and was used in this refinement in order to obtain the four scale values of the levels h = 0, 1, 2 and 3.

An outline of the procedures applied is given in Table 2. The weight factor used was $w=(a+F_{\text{lim}})/(a+F_o+bF_o^2)$ with $F_{\text{lim}}=2{\cdot}5$, $a=5$ and $b=0{\cdot}01$. The scattering curves were those proposed by Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for oxygen, nitrogen and carbon, McWeeny

Table 1. *Unit-cell dimensions* (Å) *of some* α-5-(2′-R-*ethoxy*)-*o*-*quinone*-2-*oximes**

R	*a*	*b*	*c*	*β*	*T*
Bromo	4·34	10·8	20·9	105°	20 °C
Chloro	4·12 ± 0·02	10·83 ± 0·02	20·35 ± 0·04	104°48′ ± 10′	20
Chloro	4·01 ± 0·01	10·87 ± 0·03	20·08 ± 0·05	103°36′ ± 10′	− 180

* The *b* and *c* axes in Table 1 in the earlier paper of Romers & Umans (1960) are accidentally interchanged.

---

† Joint development of Nederlandse Instrumentenfabriek 'Nonius' N.V. and Technische Physische Dienst T.N.O./T.H., Delft, Holland.

(1951) for hydrogen and Dawson (1960) for chlorine. The hydrogen atoms, except H(8), were located in a difference Fourier synthesis after cycle 8. They were introduced at calculated positions with $B = 1{\cdot}6$ Å^{-2} and were not refined until cycle 14. A second difference Fourier synthesis (Fig. 1) with heavy atom contributions subtracted was calculated after cycle 13. It showed very clearly all hydrogen atoms with heights of approximately 0·6 e.Å^{-3}. The bridge hydrogen atom H(8) was much lower with a peak value of 0·3 e.Å^{-3}. During the final cycles 14 and 15 the heavy atoms were not refined and the hydrogen atoms were refined with isotropic B's. The B's of H(1), H(2), H(3), H(5) and H(6) became negative and the B's of H(4) and H(7) were very small (0·17 and 0·05 Å^{-2}, respectively). On the other hand the B of H(8) became 5·8 Å^{-2} in accordance with its low peak value in the difference Fourier synthesis. Since the program does not allow for negative B values the structure factors were calculated with zero B's instead of negative values. The final R value, defined in the usual way, was 6·77. The mean shifts were one tenth of the corresponding standard deviations and the maximum shift amounted to one fourth of its standard deviation. A list of calculated and observed structure factors, (based upon cycle 12, for two molecules per unit cell) is given in Table 3. The positional parameters and their standard deviations are listed in Table 4. The anisotropic thermal parameters are to be found in Table 5. The mean positional standard deviations $\sigma(r)$ are 0·0055, 0·0046, 0·0039 and 0·0013 Å for carbon, nitrogen, oxygen and chlorine atoms respectively. The standard deviation in the cell edges is approximately 0·002 Å per measured length of 1·5 Å. The standard deviations for the C–C, C–N, C–O, N–O and C–Cl bonds, including errors in cell edges, are 0·0080, 0·0074, 0·0070, 0·0064 and 0·0060 Å respectively. The standard deviation in the bond angles is about 45′.

Table 2. *Survey of the applied refinement procedures*

Cycle	R (%)	Procedure	Description
1	24·9	Structure factor least squares.	Model derived by Romers & Umans; isotropic B's.
2	20·7	SFLS	*idem*
3	16·3	SFLS	*idem*
4	15·5	SFLS	*idem*
4*a*		SF output	Correction of erroneously indexed reflexions.
5	13·4	SFLS	Isotropic B's.
6	12·6	SFLS	*idem*
6*a*		SF output	Correction of errors in the reduction of reflexion intensities.
7	10·6	SFLS	Isotropic B's
8	10·5	SFLS	*idem*
8*a*		Difference Fourier	7 hydrogen atoms located, but introduced at calculated positions.
9	10·0	SFLS	Isotropic B's; hydrogen atoms kept at fixed positions.
10	9·8	SFLS	Anisotropic U's for heavy atoms; hydrogen atoms kept at fixed positions.
11	8·3	SFLS	*idem*
11*a*		SF output	Correction of additional intensity errors; introduction of H(8), the bridge atom.
12	7·4	SFLS	Same as Cycle 11.
13	7·04	SFLS	Same as Cycle 11.
13*a*		Difference Fourier	Contributions of heavy atoms subtracted, (see Fig. 1).
14	6·93	SFLS	Heavy atoms not refined; hydrogen atoms refined with isotropic B's .
15	6·77	SFLS	*idem*

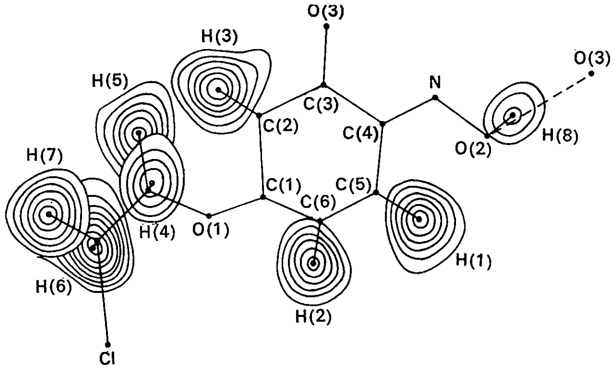

Fig. 1. Composite difference Fourier synthesis of α-CEQ projected along [100]. The contour lines, starting with 0·1 e.Å^{-3}, are drawn at intervals of 0·1 e.Å^{-3}.

Description of the structure

The mean values of small (1·351 Å) and large (1·456 Å) C–C distances in the six-membered ring as well as the values for the carbonyl and C–N bonds (1·253 and 1·306 Å, respectively) indicate a predominant oxime-like structure (Fig. 2). Table 6 shows that no significant differences† exist between the corresponding bond distances and bond angles of α-CEQ and the β modification of 5-n-propoxy-*o*-quinone-2-oxime (Romers, 1964). The main difference, however, is the *anti*-configuration of the CO and NOH groups in the former compound, in contrast with the *syn*-configuration of the same groups in β-5-n-propoxy-*o*-quinone-2-oxime. The formation of a strong intramolecular hydrogen bond coupled with internal proton dissociation (Ubbelohde & Gallagher, 1955) is impossible in α-CEQ and no experimental evidence of an n-π* transition, due to the tautomeric nitroso structure, has been found for this and other α-forms (Umans, 1959).

A second minor difference is the conformation of the R–C_2H_4–O group. This group has the *gauche* conformation in both compounds. The dihedral angle

---

† The reported value C(4)–C(5) = 1·458 Å in Table 6 of the paper of Romers (1964) should read 1·442 Å. The angle C(4)–C(5)–C(6) = 120°71′ in Fig. 1 of the same paper should read 120°11′.

Table 3. *Calculated and observed structure factors*

n	k	l	F_c	F_o
0	+ 0	+ 2	- 10.5	- 9.9
		+ 4	- 7.3	- 6.5
		+ 6	+ 9.0	+ 9.4
		+ 8	+ 13.7	+ 13.4
		+10	+ 39.5	+ 42.4
		+12	+ 7.4	+ 6.7
		+14	+ 3.5	+ 4.0
		+16	+ 7.1	+ 7.4
		+18	+ 8.7	+ 8.5
		+20	- 6.7	- 6.9
		+22	+ 6.2	+ 6.1
	+ 1	+ 2	- 0.7	- 0.8
		+ 3	- 34.8	- 32.1
		+ 4	+ 27.3	+ 26.8
		+ 5	- 28.3	- 29.2
		+ 6	- 25.7	- 25.9
		+ 7	- 8.7	- 8.2
		+ 8	- 17.7	- 17.9
		+ 9	+ 3.1	+ 3.2
		+10	- 4.7	- 4.7
		+12	- 14.4	- 15.4
		+13	- 19.9	- 20.9
		+14	- 5.8	- 6.2
		+16	- 8.2	- 8.1
		+17	- 7.9	- 7.3
		+19	- 6.0	- 6.5
		+20	- 11.6	- 11.5
		+21	+ 3.9	+ 3.7
		+22	- 3.9	- 4.2
		+23	+ 2.6	+ 2.8
		+24	- 1.4	- 8.9
	+ 2	+ 0	- 9.1	- 7.7
		+ 1	- 5.6	- 4.3
		+ 2	- 15.8	- 16.0
		+ 3	+ 4.8	+ 4.0
		+ 4	+ 6.0	+ 5.9
		+ 5	- 13.7	- 13.9
		+ 6	- 13.2	- 14.1
		+ 7	+ 23.6	+ 25.2
		+ 8	- 5.7	- 6.9
		+ 9	+ 5.5	+ 5.2
		+10	- 10.3	- 10.2
		+11	+ 11.2	+ 12.2
		+12	+ 1.4	+ 1.9
		+13	+ 20.2	+ 20.8
		+15	+ 8.1	+ 8.2
		+16	- 5.3	- 5.3
		+17	+ 7.9	+ 9.8
		+19	+ 3.4	+ 2.3
		+21	+ 10.7	+ 10.8
		+22	+ 5.6	+ 5.9
		+23	+ 8.5	+ 8.3
		+24	- 4.5	- 4.4
	+ 3	+ 1	+ 28.9	+ 28.5
		+ 3	+ 19.2	+ 19.8
		+ 4	- 7.1	- 6.2
		+ 5	+ 32.4	+ 33.0
		+ 6	+ 13.2	+ 13.6
		+ 7	+ 15.1	+ 15.5
		+ 8	- 13.1	- 14.0
		+ 9	+ 5.0	+ 5.1
		+11	+ 8.4	+ 8.2
		+12	- 9.8	- 10.0
		+13	+ 11.3	+ 11.3
		+14	- 5.8	- 5.6
		+15	+ 12.4	+ 12.3
		+16	+ 2.9	+ 1.6
		+17	+ 3.5	+ 2.3
		+19	+ 3.3	+ 3.3
		+20	- 7.5	- 7.5
		+21	- 3.7	- 3.8
		+24	- 5.1	- 4.3
	+ 4	+ 0	- 33.0	- 35.2
		+ 1	- 6.9	- 5.7
		+ 2	- 22.4	- 22.9
		+ 3	+ 8.4	+ 7.9
		+ 4	- 9.3	- 8.9
		+ 5	- 35.5	- 38.7
		+ 6	- 13.5	- 13.7
		+ 7	+ 11.8	+ 13.6
		+ 8	- 2.8	- 2.2
		+ 9	+ 2.4	+ 2.3
		+10	- 6.9	- 7.4
		+11	- 6.9	- 6.5
		+12	- 22.4	- 22.4
		+13	- 7.9	- 8.2
		+14	- 1.5	- 2.3
		+15	- 23.7	- 24.4
		+16	- 3.7	- 3.4
		+17	+ 10.0	+ 9.9
		+18	- 4.8	- 5.0
		+19	- 1.6	- 1.6
		+21	- 9.6	- 9.4
		+22	- 2.6	- 2.5
		+23	- 7.7	- 7.7
	5	+ 1	+ 5.8	+ 6.7
		+ 2	+ 9.1	+ 9.5
		+ 3	- 4.5	- 4.4
		+ 4	- 18.0	- 19.6
		+ 8	+ 26.3	+ 28.4
		+ 9	- 9.6	- 10.0
		+10	+ 14.6	+ 15.3
		+11	+ 6.5	+ 6.9
		+12	+ 8.4	+ 8.8
		+13	+ 4.2	+ 4.6
		+15	- 6.4	- 6.5
		+17	+ 6.8	+ 6.5
		+18	+ 26.1	+ 26.8
		+19	- 4.6	- 4.5
		+20	+ 4.0	+ 4.0
		+22	+ 4.4	+ 3.6
	+ 6	+ 0	+ 6.7	+ 6.5
		+ 1	- 10.4	- 11.0
		+ 2	+ 26.6	+ 29.2
		+ 3	- 1.1	- 1.8
		+ 4	+ 1.5	+ 1.8
		+ 6	+ 12.9	+ 13.4
		+ 8	+ 8.1	+ 8.5
		+ 9	- 6.1	- 6.6
		+10	+ 11.1	+ 11.4
		+11	- 3.8	- 3.7
		+12	+ 12.4	+ 12.8
		+13	- 14.4	- 15.2
		+15	- 4.6	- 4.8
		+16	+ 5.5	+ 6.0
		+17	+ 5.7	+ 6.8
		+19	- 8.2	- 8.4
		+20	+ 3.6	+ 4.0
	+ 7	+ 1	- 14.2	- 15.0
		+ 3	- 14.3	- 16.6
		+ 4	- 8.1	- 7.9
		+ 5	- 13.5	- 14.4
		+ 7	- 13.9	- 14.8
		+ 9	+ 2.5	+ 2.3
		+10	- 5.5	- 5.6
		+11	- 13.3	- 14.1
		+13	- 7.9	- 7.9
		+15	- 6.8	- 6.8
		+16	- 4.0	- 4.0
		+17	- 3.1	- 3.7
		+18	- 5.4	- 6.4
		+20	- 3.8	- 4.1
	+ 8	+ 0	- 8.8	- 9.1
		+ 2	+ 12.5	+ 13.7
		+ 3	+ 9.1	+ 10.0
		+ 4	+ 1.1	+ 1.6
		+ 5	+ 4.1	+ 3.8
		+ 6	+ 7.0	+ 7.6

h	k	l	F_c	F_o
+0	+ 8	+ 3	- 3.0	- 3.7
		+ 9	+ 8.4	+ 8.3
		+10	- 3.0	- 3.6
		+11	+ 5.8	+ 6.7
		+12	+ 8.4	+ 7.9
		+13	+ 10.6	+ 11.1
		+14	+ 2.9	+ 2.8
		+15	+ 6.6	+ 7.3
		+17	+ 6.6	+ 6.9
		+19	+ 5.7	+ 5.8
	+ 9	+ 1	- 11.9	- 11.7
		+ 3	+ 26.1	+ 27.9
		+ 4	+ 2.2	+ 1.6
		+ 5	+ 9.4	+ 9.6
		+ 6	- 3.8	- 3.8
		+ 7	+ 8.5	+ 8.2
		+ 8	- 6.9	- 6.9
		+10	- 7.1	- 7.4
		+12	- 2.6	- 1.6
		+13	+ 12.1	+ 11.4
		+14	- 6.8	- 7.2
		+18	- 5.3	- 4.9
	+10	+ 0	- 24.8	- 24.9
		+ 1	+ 9.9	+ 9.5
		+ 2	- 7.9	- 8.1
		+ 3	- 11.7	- 11.4
		+ 4	- 4.4	- 5.1
		+ 6	- 2.9	- 5.6
		+ 7	+ 5.2	+ 4.9
		+ 8	- 11.0	- 10.9
		+10	- 12.9	- 12.7
		+12	- 3.4	- 3.7
		+14	- 3.6	- 3.4
		+16	- 1.7	- 2.1
		+17	- 4.6	- 4.4
	+11	+ 1	+ 7.2	+ 7.3
		+ 4	+ 6.2	+ 6.1
		+ 5	+ 6.0	+ 6.0
		+ 7	+ 3.5	+ 3.8
		+ 8	+ 3.4	+ 3.6
		+ 9	+ 2.3	+ 2.3
		+10	+ 5.0	+ 4.8
		+11	+ 3.0	+ 2.9
		+12	+ 3.4	+ 3.5
		+15	+ 3.6	+ 2.9
	+12	+ 2	+ 4.0	+ 3.5
		+ 3	- 2.6	- 2.4
		+ 4	+ 2.5	+ 2.3
		+ 7	- 7.3	- 8.1
		+ 8	+ 6.7	+ 6.5
		+10	+ 2.8	+ 2.6
		+11	- 4.9	- 4.6
	+13	+ 1	- 4.4	- 4.9
		+ 2	- 3.2	- 3.1
		+ 3	- 6.0	- 5.3
		+ 4	+ 1.9	+ 1.5
		+ 5	- 9.3	- 8.7
		+ 6	+ 2.3	+ 1.6
		+ 7	- 4.9	- 4.1
		+ 8	- 2.1	- 2.0
		+ 9	- 7.8	- 5.7
+ 1	+ 0	-24	+ 12.9	+ 14.2
		-22	+ 6.7	+ 7.4
		-20	+ 7.8	+ 8.0
		-18	+ 9.3	+ 9.6
		-16	+ 25.4	+ 27.5
		-12	+ 10.9	+ 12.1
		-10	+ 16.3	+ 16.4
		- 8	+ 27.7	+ 29.5
		- 6	+ 93.2	+ 96.5
		- 4	- 78.9	- 79.4
		- 2	- 7.8	- 6.9
		+ 0	+ 34.3	+ 35.2
		+ 2	+ 26.4	+ 29.9
		+ 4	+ 26.2	+ 32.5
		+ 6	- 16.3	- 19.2
		+ 8	- 15.4	- 16.5
		+10	+ 4.0	+ 4.9
		+12	- 4.8	- 6.1
		+14	- 11.5	- 11.3
		+16	+ 3.9	+ 2.5
		+18	- 8.3	- 8.0
		+20	- 13.7	- 12.8
	+ 1	-25	- 8.7	- 8.9
		-23	- 6.1	- 6.3
		-22	+ 3.1	+ 3.2
		-21	- 3.9	- 3.6
		-19	- 9.4	- 9.6
		-15	- 3.8	- 3.5
		-13	- 16.6	- 16.8
		-12	- 7.1	- 7.3
		-11	- 20.9	- 22.4
		-10	- 23.1	- 22.8
		- 9	- 5.1	- 4.9
		- 8	+ 5.4	+ 5.1
		- 6	- 2.0	- 2.1
		- 5	+ 8.3	+ 9.2
		- 4	- 22.4	- 21.7
		- 3	- 41.3	- 37.0
		- 2	- 5.9	- 6.8
		- 1	- 5.2	- 4.4
		+ 0	- 55.8	- 57.6
		+ 2	- 11.8	- 12.8
		+ 3	+ 6.6	+ 6.6
		+ 4	- 7.5	- 8.5
		+ 5	- 6.8	- 6.7
		+ 6	- 23.4	- 24.8
		+ 7	- 16.1	- 18.3
		+ 8	- 5.6	- 5.1
		+ 9	+ 14.2	+ 15.5
		+10	- 14.9	- 15.6
		+12	- 12.1	- 13.1
		+13	+ 10.9	+ 10.8
		+14	- 15.1	- 15.2
		+17	+ 3.2	+ 2.5
		+18	- 8.6	- 8.2
	+ 2	-23	- 6.6	- 7.0
		-22	- 4.2	- 4.8
		-21	- 4.7	- 5.3
		-19	- 5.9	- 6.2
		-18	- 4.3	- 4.5
		-17	+ 6.9	+ 6.3
		-16	- 7.2	- 8.0
		-11	+ 15.3	+ 15.6
		- 8	- 11.4	- 11.5
		- 7	+ 10.5	+ 9.7
		- 5	+ 13.5	+ 13.4
		- 4	- 5.9	- 6.7
		- 3	+ 20.8	+ 20.4
		- 2	+ 3.0	+ 4.0
		- 1	+ 16.5	+ 16.1
		+ 0	- 16.9	- 17.5
		+ 1	+ 45.9	+ 48.1
		+ 3	+ 6.4	+ 5.5
		+ 4	- 4.8	- 3.8
		+ 5	+ 26.2	+ 28.2
		+ 6	+ 9.1	+ 8.7
		+ 7	+ 19.5	+ 20.7
		+ 8	- 5.3	- 5.2
		+ 9	+ 7.6	+ 7.9
		+11	+ 11.3	+ 11.4
		+13	+ 12.0	+ 11.8
		+15	+ 14.1	+ 14.0
		+16	+ 5.8	+ 5.1
		+17	+ 8.8	+ 8.9
		+18	- 4.7	- 5.2
		+19	+ 5.6	+ 6.1
		+20	+ 5.1	+ 4.6

h	k	l	F_c	F_o
–	+ 3	-25	+ 9.3	+ 5.2
		-23	+ 7.6	+ 8.0
		-21	+ 9.5	+ 10.3
		-20	+ 8.7	+ 10.0
		-19	+ 9.3	+ 9.2
		-17	+ 6.1	+ 7.0
		-16	- 5.8	- 6.3
		-15	+ 11.3	+ 12.2
		-13	+ 18.2	+ 18.9
		-12	- 11.7	- 12.0
		-11	+ 19.8	+ 19.9
		-10	+ 11.6	+ 12.1
		- 9	+ 11.5	+ 13.3
		- 8	- 8.9	- 9.0
		- 7	+ 12.6	+ 13.2
		- 5	+ 13.1	+ 14.2
		- 4	- 8.3	- 6.3
		- 3	+ 8.3	+ 7.9
		- 2	- 5.6	- 5.4
		- 1	+ 24.0	+ 25.5
		+ 0	- 7.2	- 6.9
		+ 1	- 3.8	- 3.6
		+ 2	- 14.4	- 15.1
		+ 4	+ 1.2	+ 1.2
		+ 5	+ 7.2	+ 7.3
		+ 6	- 9.5	- 8.4
		+ 7	+ 5.0	+ 6.2
		+11	- 12.4	- 11.8
		+12	- 3.0	- 2.3
		+13	- 5.4	- 5.4
		+14	- 11.4	- 11.2
		+15	- 8.1	- 7.1
		+18	- 6.9	- 6.6
		+19	- 6.0	- 6.2
		+21	- 7.9	- 8.0
	+ 4	-24	- 7.6	- 7.7
		-23	- 3.0	- 4.4
		-22	- 5.9	- 6.0
		-21	+ 18.2	+ 21.6
		-20	- 6.3	- 7.0
		-19	- 5.1	- 5.4
		-18	- 12.9	- 14.0
		-17	- 3.0	- 2.4
		-15	- 5.4	- 5.7
		-14	- 3.5	- 9.3
		-12	- 16.7	- 20.2
		-11	+ 13.2	+ 16.0
		-10	- 8.9	- 9.3
		- 9	- 15.0	- 18.5
		- 8	- 18.2	- 20.4
		- 7	- 6.3	- 7.6
		- 6	- 14.0	- 15.7
		- 5	- 13.5	- 13.9
		- 3	- 3.3	- 4.3
		- 1	- 33.8	- 36.3
		+ 0	- 8.6	- 9.3
		+ 1	+ 20.3	+ 22.5
		+ 2	- 5.8	- 5.1
		+ 3	- 12.8	- 13.8
		+ 4	- 11.8	- 12.4
		+ 5	- 20.2	- 20.7
		+ 6	- 10.5	- 10.5
		+ 7	- 15.8	- 17.0
		+ 8	+ 19.1	+ 19.1
		+ 9	- 14.5	- 14.9
		+11	+ 14.5	+ 14.2
		+13	- 9.1	- 9.5
		+14	+ 7.3	+ 7.5
		+15	- 18.0	- 17.8
		+17	- 3.3	- 2.8
		+18	+ 6.8	+ 7.6
		+20	+ 4.8	+ 9.0
	+ 5	-24	- 11.0	- 10.1
		-23	+ 3.9	+ 3.8
		-22	+ 5.5	+ 5.7
		-21	- 3.9	- 4.0
		-20	+ 2.9	+ 2.7
		-19	+ 1.8	+ 1.8
		-18	- 7.8	- 8.4
		-16	- 2.2	- 2.4
		-15	- 7.0	- 7.9
		-13	+ 4.4	+ 4.7
		-12	+ 15.2	+ 17.6
		-10	+ 16.4	+ 18.4
		- 9	- 4.0	- 4.8
		- 8	- 9.3	- 10.1
		- 7	+ 6.9	+ 3.1
		- 6	+ 14.6	+ 15.7
		- 5	- 5.1	- 5.6
		- 4	+ 8.8	+ 9.4
		- 3	- 5.1	- 5.1
		- 2	+ 6.5	+ 6.5
		+ 0	+ 25.0	+ 26.7
		+ 2	+ 41.5	+ 45.4
		+ 3	- 3.5	- 3.7
		+ 4	+ 4.1	+ 4.0
		+ 5	+ 12.5	+ 13.0
		+ 6	+ 4.1	+ 4.8
		+ 7	- 3.7	- 3.8
		+ 8	+ 8.4	+ 8.5
		+ 9	- 10.0	- 10.0
		+10	+ 14.2	+ 14.1
		+11	+ 10.2	+ 9.9
		+12	+ 34.5	+ 35.8
		+13	- 7.8	- 7.8
		+14	- 7.4	- 7.8
		+15	+ 4.2	+ 4.1
		+16	+ 5.5	+ 5.7
		+18	+ 7.0	+ 7.0
		+20	+ 8.3	+ 8.5
	+ 6	-23	- 4.2	- 3.4
		-22	+ 8.3	+ 9.3
		-21	+ 7.9	+ 8.7
		-20	+ 2.9	+ 3.2
		-19	+ 5.5	+ 6.5
		-18	+ 13.2	+ 14.8
		-17	- 6.0	- 6.6
		-16	+ 6.4	+ 6.9
		-15	- 4.1	- 5.1
		-14	+ 7.7	+ 8.6
		-13	- 5.2	- 6.0
		-12	+ 9.4	+ 10.7
		-10	+ 6.4	+ 7.1
		- 8	+ 13.5	+ 21.2
		- 7	- 3.0	- 3.8
		- 6	+ 3.7	+ 3.4
		- 5	- 14.7	- 15.9
		- 4	+ 15.3	+ 16.5
		- 3	- 3.2	- 4.0
		- 2	- 7.0	- 7.1
		- 1	- 8.3	- 8.7
		+ 0	+ 12.0	+ 11.7
		+ 1	- 7.7	- 7.5
		+ 2	+ 4.9	+ 5.3
		+ 3	- 11.9	- 12.4
		+ 4	+ 7.2	+ 7.4
		+ 5	- 4.8	- 5.4
		+ 7	- 4.0	- 4.0
		+ 8	- 9.0	- 9.1
		+13	[illegible]	- 13.5
		+14	- 3.4	- 2.4
		+15	- 3.3	- 2.9
		+16	- 5.7	[illegible]
		+17	[illegible]	- 7.0
		[illegible]	[illegible]	- 6.0
		+20	[illegible]	- 3.7
	+ 7	-22	[illegible]	- 1.5
		-21	- 6.7	[illegible]
		[illegible]	- 7.7	[illegible]

h	k	l	F_c	F_o
+1	+7	-17	[illegible]	[illegible]
		-16	[illegible]	+ 3.0
		-15	- 3.2	- 3.7
		-13	[illegible]	- 21.2
		-11	- 9.5	- 10.1
		- 9	- 10.9	- 12.4
		- 8	- 7.6	- 7.3
		- 7	- 5.5	- 5.7
		- 6	- 1.8	- 1.3
		- 5	- 12.0	- 12.9
		- 4	- 4.5	- 4.4
		- 3	- 3.2	- 3.2
		- 2	- 5.0	- 6.0
		- 1	- 9.7	- 9.0
		+ 2	- 4.6	- 4.3
		+ 3	+ 2.5	+ 2.8
		+ 4	- 7.5	- 8.0
		+ 5	- 12.2	- 13.0
		+ 7	+ 3.1	+ 2.9
		[illegible]	- 4.4	- 4.7
		+10	- 7.3	- 7.9
		+11	+ 5.8	+ 5.5
		+12	- 2.9	- 3.3
		+13	+ 3.9	+ 4.3
		+15	+ 4.4	+ 4.3
		+19	+ 9.1	+ 9.0
		-21	- 2.6	- 2.3
		-19	- 3.1	- 3.7
		-18	+ 4.0	+ 4.1
		-16	- 6.2	- 6.1
		-15	+ 1.9	+ 2.8
		-14	+ 4.3	+ 4.3
		-13	+ 5.4	+ 5.5
		-12	+ 8.1	+ 8.2
		- 8	+ 6.6	+ 7.0
		- 7	+ 12.6	+ 13.6
		- 6	- 10.9	- 11.2
		- 5	+ 3.4	+ 8.9
		- 4	+ 8.5	+ 8.9
		- 3	+ 12.1	+ 12.5
		- 1	+ 6.3	+ 6.2
		+ 0	+ 3.7	+ 3.9
		+ 1	+ 6.2	+ 5.9
		+ 2	- 6.0	- 6.2
		+ 3	+ 18.1	+ 19.5
		+ 4	+ 4.2	+ 3.7
		+ 5	+ 8.7	+ 9.4
		+ 6	+ 5.0	+ 4.4
		+ 7	+ 13.7	+ 13.5
		+ 9	+ 7.5	+ 7.3
		+10	- 4.4	- 3.6
		+11	+ 8.2	+ 7.4
		+13	+ 6.0	+ 6.1
		+15	+ 7.0	+ 7.3
		+16	- 3.4	- 3.7
		+17	+ 8.4	+ 8.5
		+18	+ 5.2	+ 5.0
	+ 9	-20	+ 2.5	+ 4.0
		-15	+ 4.3	+ 5.3
		-14	- 2.1	- 2.7
		-13	+ 10.9	+ 11.4
		-12	- 5.1	- 5.5
		-11	+ 7.2	+ 7.4
		-10	- 6.2	- 7.3
		- 9	+ 8.3	+ 8.4
		- 7	- 12.6	- 13.2
		- 6	- 4.3	- 4.5
		- 4	- 3.3	- 4.8
		- 3	+ 32.9	+ 35.2
		- 2	- 10.1	- 11.1
		- 1	- 5.9	- 6.2
		+ 0	- 11.2	- 12.0
		+ 2	- 10.0	- 10.[illegible]
		+ 3	- 5.2	- 4.5
		+ 4	- 6.1	- 6.5
		+ 6	- 4.1	- 4.9
		+ 7	+ 12.3	+ 11.1
		+ 8	- 8.6	- 8.7
		+ 9	- 6.9	- 5.8
		+10	- 4.8	- 4.9
		+12	- 9.5	- 9.8
		+14	- 5.0	- 4.9
		+16	- 4.7	- 4.9
		+17	- 9.2	- 7.6
	+10	-16	- 11.7	- 12.5
		-15	+ 3.1	+ 3.5
		-14	- 5.2	- 6.0
		-13	- 2.2	- 1.8
		-12	- 2.1	- 2.1
		-11	+ 4.4	+ 4.7
		-10	- 8.1	- 9.3
		- 9	+ 3.2	+ 3.5
		- 8	- 11.2	- 11.9
		- 7	- 11.9	- 13.0
		- 6	- 16.3	- 18.6
		- 5	+ 7.8	+ 7.9
		- 3	- 8.6	- 9.2
		- 1	+ 0.7	+ 8.0
		+ 0	- 5.1	- 5.1
		+ 1	+ 4.1	+ 4.0
		+ 2	- 9.9	- 10.3
		+ 3	- 5.5	- 5.0
		+ 4	- 3.9	- 3.9
		+ 6	+ 4.9	+ 4.7
		+ 8	+ 2.9	+ 3.2
		+ 9	- 3.7	- 4.0
		+12	+ 1.6	+ 1.8
		+13	+ 4.2	+ 5.2
	+11	-15	+ 2.7	+ 2.9
		-12	+ 2.5	+ 3.3
		-11	+ 5.6	+ 6.6
		- 7	+ 5.7	+ 6.5
		- 4	+ 6.2	+ 6.9
		- 2	+ 7.3	+ 8.1
		- 1	+ 5.2	+ 6.3
		+ 0	+ 4.7	+ 5.5
		+ 2	+ 6.0	+ 6.3
		+ 4	+ 8.5	+ 9.3
		+ 6	+ 5.4	+ 6.1
		+ 9	+ 5.4	+ 5.7
		+10	+ 10.5	+ 10.9
		+12	+ 2.1	+ 1.7
		+13	- 3.7	- 3.1
	+12	-11	- 5.5	- 5.7
		-10	+ 2.2	+ 2.2
		- 9	- 2.8	- 3.3
		- 8	+ 3.2	+ 3.5
		- 7	- 3.1	- 3.6
		- 5	- 5.4	- 5.7
		- 4	+ 5.1	+ 5.7
		- 3	- 7.1	- 7.8
		- 1	- 2.9	- 3.4
		+ 1	- 10.8	- 12.1
		+ 2	+ 5.3	+ 5.6
		+ 5	- 9.0	- 9.9
		+ 7	- 5.5	- 5.5
		+ 9	- 5.2	- 4.4
		+10	+ 1.5	+ 1.8
	+13	- 7	- 3.8	- 4.1
		- 2	+ 4.0	+ 5.4
		- 1	- 5.1	- 5.1
		+ 0	+ 4.0	+ 4.5
		+ 5	+ 3.2	+ 2.7
		+ 6	+ 2.8	+ 2.4
		+ 7	- 2.7	- 1.6
	+11	-13	+ 4.4	+ 5.1
		[illegible]	+ 9.3	+ 10.6
	+13	- 8	+ 4.1	+ 3.6
		- 5	- 4.5	- 4.6

Table 3 (*cont.*)

h	k	l	Fc	Fo
		-3	-6.4	-6.9
		+2	-3.4	-3.6
		+4	+2.7	+1.0
+2	+0	-24	+[illegible]	+7.0
		-22	+15.6	+14.6
		-20	-3.4	-3.1
		-18	+6.7	+6.8
		-16	+6.4	+6.1
		-14	+9.3	+9.[illegible]
		-12	+11.6	+12.5
		-10	-40.8	-50.1
		-8	+14.6	+15.0
		-6	+23.8	+25.[illegible]
		+0	-35.8	-32.8
		+2	-16.9	-17.9
		+4	+8.6	+10.8
		+6	-18.9	-21.5
		+8	-11.3	-13.0
		+10	-7.3	-8.9
		+12	-19.4	-21.[illegible]
		+14	-18.1	-19.4
		-25	-3.8	-3.5
		-24	-4.3	-4.3
		-22	-5.1	-4.4
		-21	-5.4	-4.6
		-20	-5.9	-5.4
		-19	-10.7	-10.9
		-18	-8.9	-9.1
		-16	-11.6	-11.[illegible]
		-15	+6.6	+6.4
		-12	-15.[illegible]	-13.5
		-11	+3.0	+2.9
		-10	-23.8	-25.0
		-9	-24.[illegible]	-27.4
		-8	-1.2	-6.5
		-7	+15.6	+15.6
		-6	-28.5	-29.0
		-5	+9.3	+9.4
		-4	+6.4	+5.5
		-3	+10.2	+10.3
		-2	-9.7	-9.5
		+0	-21.5	-23.3
		+1	+5.6	+2.5
		+2	-5.7	-5.4
		+3	+19.5	+23.9
		+4	-14.1	-15.9
		+5	+2.3	+2.8
		+6	-6.6	-7.5
		+7	+12.8	+13.9
		+8	-3.9	-4.3
		+10	+8.4	+8.0
		+11	+7.5	+8.3
		+12	-6.6	-5.8
		+15	+13.7	+14.9
		+17	+4.0	+3.4
		-25	+5.9	+6.5
		-24	-3.7	-3.6
		-23	+10.0	+10.5
		-21	+3.5	+3.6
		-19	+5.9	+6.1
		-17	+9.8	+9.6
		-16	-3.1	-3.1
		-15	+6.5	+5.9
		-13	+16.8	+16.8
		-11	+18.7	+17.8
		-9	+13.2	+12.3
		-7	+14.8	+15.3
		-6	-2.9	-3.8
		-5	+20.5	+22.4
		-4	+6.6	+6.1
		-2	+1.4	+1.0
		-1	+24.1	+26.5
		+0	+10.7	+10.4
		+1	+10.2	+10.6
		+2	-5.5	-5.6
		+3	+7.7	+9.0
		+4	+9.1	+9.4
		+5	+9.0	+10.0
		+6	+2.7	+2.5
		+7	+8.6	+9.5
		+8	+6.0	+6.3
		+9	+1.7	+2.0
		+10	+3.5	+3.6
		+13	-4.6	-4.5
		+14	+7.9	+9.8
		+16	+2.6	+2.9
		+17	+4.0	+3.8
		-24	-8.2	-8.3
		-23	+4.4	+4.3
		-22	-3.4	-3.6
		-21	+9.5	+10.1
		-19	+7.1	+6.6
		-18	-3.4	-3.1
		-17	+3.1	+3.1
		-16	+6.1	+6.5
		-14	-12.4	-13.3
		-10	-8.5	-8.4
		-7	+4.3	+3.8
		-6	-7.1	-7.0
		-5	-19.0	-19.4
		-4	+2.5	+2.6
		-3	-4.7	-4.1
		-2	-3.2	-3.4
		-1	-6.0	-6.1
		+0	-6.3	-6.3
		+1	-9.9	-9.0
		+2	-3.4	-3.4
		+3	-12.4	-13.1
		+4	-3.1	-3.3
		+5	-20.6	-22.1
		+7	-6.3	-7.1
		+8	-5.8	-5.5
		+9	-9.5	-9.8
		+10	+7.8	+7.6
		+11	-6.8	-6.5
		+12	-5.9	-6.0
		+13	-15.3	-16.9
		+14	+6.1	+6.8
		+15	-10.7	-11.6
		+17	-6.2	-6.3
		+19	-6.9	-6.6
		-24	-6.0	-7.3
		-23	-4.9	-4.6
		-21	-2.6	-3.4
		-20	-8.0	-8.4
		-18	-6.4	-6.6
		-15	-14.4	-16.1
		-14	-3.7	-4.3
		-13	-8.6	-8.8
		-12	+2.0	+2.9
		-11	-5.8	-5.6
		-10	+4.8	+4.3
		-9	-7.1	-6.8
		-7	-15.5	-15.3
		-6	-2.6	-1.6
		-5	+10.3	+9.6
		-4	+3.8	+5.0
		-3	-20.0	-22.4
		-2	+6.7	+6.5
		-1	-16.8	-18.4
		+0	+13.4	+13.0
		+1	-6.0	-6.3
		+2	+24.0	+24.1
		+4	-4.0	-3.6
		+5	+9.0	+8.5
		+6	+3.0	+3.1
		+7	-14.3	-14.6
		+8	+12.7	+12.9
		+9	-16.4	-15.6
+2	+4	+10	+5.9	+5.4
		+11	+11.9	+11.6
		+12	+13.3	+13.4
		+14	+6.9	+7.4
		+16	+5.4	+5.9
		+18	+7.4	+7.6
	+5	-22	+7.0	+7.9
		-21	-2.1	-3.1
		-20	+7.7	+8.5
		-19	+4.3	+4.9
		-18	+13.0	+14.9
		-16	+7.2	+[illegible]
		-15	-4.5	-4.5
		-13	+2.9	+2.1
		-12	+21.2	+22.5
		-11	-5.3	-5.6
		-10	+10.7	+10.9
		-8	+15.7	+17.0
		-6	+17.7	+13.8
		-5	+4.0	+3.9
		-4	+21.2	+22.5
		-3	-4.4	-4.5
		-2	-10.6	-11.9
		-1	+6.0	+6.1
		+0	+10.9	+11.0
		+1	-10.7	-10.1
		+2	+15.6	+15.9
		+4	+16.1	+16.6
		+5	+13.6	+13.9
		+6	+18.1	+18.5
		+7	-8.8	-10.0
		+8	-24.8	-20.1
		+9	+6.1	+6.5
		+10	+7.1	+7.9
		+12	+6.0	+6.9
		+16	-3.0	-2.5
		+18	-6.1	-6.6
	+6	-23	-9.3	-10.1
		-21	-2.1	-2.3
		-20	+4.2	+4.4
		-18	+4.2	+4.6
		-16	+3.1	+2.3
		-15	-3.7	-3.9
		-14	+5.2	+6.0
		-13	-12.4	-13.8
		-11	-9.7	-9.5
		-9	-3.8	-4.1
		-8	-7.8	-7.4
		-7	-6.8	-6.6
		-6	+4.8	+4.3
		-5	-3.9	-4.4
		-4	-3.4	-3.9
		-3	-5.9	-5.9
		-2	-6.1	-5.9
		-1	-4.7	-4.9
		+0	-12.0	-11.4
		+1	-10.5	-9.1
		+2	-10.4	-10.1
		+3	+3.3	+3.5
		+4	-2.7	-2.1
		+5	-6.6	-5.8
		+6	-4.2	-3.8
		+7	-7.2	-6.5
		+8	-15.9	-13.4
		+10	-8.1	-7.5
		+12	-8.6	-9.0
		+13	+2.7	+2.9
		+14	-4.2	-3.4
		+15	+3.1	+2.5
		+16	-6.4	-6.8
		+17	-2.4	-2.3
		+18	-6.7	-6.4
	+7	-21	-5.4	-6.1
		-20	-4.1	-4.5
		-19	-8.6	-9.9
		-14	-5.6	-5.9
		-11	-5.2	-4.9
		-10	-4.9	-4.3
		-9	+5.6	+5.1
		-8	-2.9	-2.1
		-7	-3.3	-3.8
		-6	-2.5	-3.0
		-5	+9.2	+8.9
		-4	-7.6	-7.0
		-3	+4.8	+4.6
		-2	-5.4	-5.8
		+1	+13.8	+13.6
		+2	-4.6	-5.8
		+3	+9.0	+8.9
		+4	-3.1	-3.1
		+5	+8.2	+7.4
		+7	+7.0	+6.1
		+9	+8.1	+7.9
		+11	+5.8	+55
		+12	-3.7	-3.4
		+13	+14.6	+18.3
		+17	+9.2	+8.4
	+8	-20	+6.9	+7.9
		-19	+5.0	+4.8
		-18	+2.0	+1.6
		-17	+3.7	+3.6
		-16	-2.8	-3.4
		-15	+8.7	+9.4
		-13	+14.4	+16.3
		-12	-7.6	-7.8
		-11	+6.3	+5.6
		-10	+8.1	+7.9
		-9	+9.2	+9.6
		-8	+2.3	+3.1
		-7	+7.3	+7.4
		-6	-4.0	-4.5
		-5	+10.1	+10.6
		-4	-5.4	-6.0
		-3	+15.4	+16.3
		-1	+3.9	+4.4
		+0	-3.6	-4.1
		+1	+9.9	+8.8
		+2	-3.9	-3.9
		+3	+3.4	+3.1
		+4	-5.6	-4.5
		+5	+5.0	+4.5
		+9	+4.2	+3.6
		+13	-3.9	-3.4
		+14	-5.0	-5.4
	+9	-19	+7.5	+6.6
		-18	-5.9	-6.1
		-16	-4.2	-4.5
		-14	-3.5	-3.5
		-13	-5.7	-6.3
		-12	-9.0	-10.0
		-11	+7.4	+7.9
		-10	-6.6	-6.1
		-9	+16.9	+19.0
		-8	-11.8	-12.3
		-7	-21.8	-24.1
		-6	-5.2	-4.8
		-4	-4.6	-4.5
		+0	-6.1	-5.3
		+1	+4.7	+3.9
		+2	-7.3	-6.9
		+3	-14.8	-14.4
		+4	-4.5	-4.4
		+5	-3.9	-4.3
		+6	-3.0	-4.8
		+7	-4.8	-4.3
		+9	-3.0	-5.5
		+11	-8.3	-9.8
		+15	+5.4	+6.1
+2	+10	-16	-5.8	-6.3
		-14	-4.3	-4.1
		-13	-5.2	-5.5
		-12	-2.1	-1.8
		-11	+8.3	+8.8
		-10	+9.5	+9.7
		-9	-4.2	-5.6
		-8	+2.2	+2.0
		-7	+3.6	+4.1
		-6	-4.7	-4.6
		-3	-7.4	-7.0
		-2	+7.8	+7.4
		+0	+10.8	+11.1
		+2	+6.0	+5.6
		+4	+2.6	+2.0
		+5	+3.5	+2.8
		+6	+6.9	+6.9
		+8	+6.1	+5.9
		+9	-3.9	-4.4
		+10	+8.2	+7.2
		+11	-2.0	-1.6
		+12	+9.3	+9.8
	+11	-14	+8.2	+8.9
		-11	-2.4	-1.5
		-10	+9.0	+9.1
		-8	+5.3	+4.9
		-7	+2.3	+2.5
		-6	+6.6	+6.5
		-5	-3.8	-3.8
		-4	+5.8	+5.6
		-3	-3.9	-4.6
		-2	+8.0	+8.5
+3	+0	-18	-[illegible]	-6.4
		-16	-21.9	-24.3
		-14	+8.6	+8.7
		-10	-30.0	-28.1
		-8	-15.4	-12.8
		-6	-13.5	-13.6
		+0	-28.4	-29.1
		+2	-9.7	-10.8
		+4	-13.1	-16.3
		+6	-14.3	-16.3
		+8	-7.5	-7.9
		+10	+9.8	+10.8
		+12	-10.0	-10.8
	+1	-24	-5.9	-6.6
		-23	+7.1	+7.2
		-22	-7.0	-6.0
		-21	+4.4	+4.3
		-18	-10.5	-11.1
		-16	-10.1	-11.4
		-15	-6.2	-5.5
		-13	+22.8	+22.7
		-12	-8.1	-6.0
		-11	+7.5	+7.3
		-10	+5.0	+4.6
		-9	+5.0	+2.7
		-8	-11.5	-9.9
		-6	-7.3	-5.3
		-5	+9.3	+5.8
		-4	+4.9	+5.2
		-3	+11.6	+8.1
		-1	+4.8	+4.7
		+0	+5.3	+5.5
		+1	+8.3	+8.9
		+2	+6.9	+6.4
		+3	+3.7	+4.0
		+4	+9.1	+9.6
		+5	+8.5	+9.9
		+6	-4.4	-4.7
		+7	+2.1	+2.9
		+8	+7.0	+7.6
		+9	+10.2	+11.0
		+10	+7.0	+7.2
		+12	+8.7	+9.5
		+14	+9.9	+10.1
	+2	-23	+8.7	+9.6
		-21	+7.9	+7.6
		-20	+2.6	+2.3
		-19	+10.2	+9.9
		-17	+6.4	+6.7
		-16	+2.1	+2.1
		-13	+7.1	+6.3
		-12	+5.3	+5.2
		-11	+4.3	+4.0
		-10	+5.4	+4.3
		-9	+2.8	+2.7
		-8	+3.5	+4.1
		-7	+10.4	+11.0
		-6	+4.7	+4.6
		-5	-2.6	-2.7
		-4	-1.1	-2.0
		-3	-2.2	-2.3
		-2	+10.2	+9.5
		-1	-3.6	-3.2
		+0	+1.0	+1.7
		+1	-3.6	-4.6
		+2	+6.2	+6.7
		+3	-8.6	-9.2
		+5	-3.0	-2.7
		+6	+2.6	+3.4
		+7	-10.6	-11.8
		+8	+5.6	+5.3
		+9	-2.5	-3.5
		+11	-4.1	-5.2
		+13	-10.5	-11.0
	+3	-23	-4.0	-3.4
		-21	-3.3	-2.9
		-20	-9.8	-10.5
		-19	-3.2	-3.4
		-17	-3.1	-3.1
		-15	-6.0	-6.7
		-13	-9.2	-8.9
		-12	-2.8	-2.9
		-11	-19.4	-17.9
		-10	+3.4	+3.4
		-9	-4.8	-5.2
		-8	-7.5	-8.2
		-7	-10.0	-12.1
		-6	-0.9	-1.5
		-5	-11.7	-13.3
		-4	-0.1	-1.5
		-3	-13.2	-13.7
		-2	+4.1	+4.1
		-1	-16.0	-17.4
		+0	+2.5	+2.0
		+1	-4.4	-4.3
		+3	-10.5	-11.4
		+4	+5.1	+5.6
		+5	-11.3	-12.5
		+6	-3.1	-3.5
		+7	-12.1	-13.1
		+8	+7.0	+7.8
		+9	-2.7	-3.1
		+10	-2.3	-3.1
		+12	+3.7	+4.3
		+13	-3.9	-4.7
		+14	+1.7	+2.3
		+15	-2.0	-1.7
	+4	-22	+5.5	+6.7
		-21	-8.9	-11.1
		-19	-3.8	-4.4
		-18	+4.4	+5.0
		-16	+4.6	+5.5
		-15	-7.0	-7.3
		-14	+4.9	+4.9
		-13	-3.9	-4.6
+3	+4	-12	+[illegible]	
		-11	+3.3	+2.9
		-10	+11.0	+14.7
		-9	-12.2	-14.5
		-8	+12.3	+14.0
		-7	-5.4	-6.7
		-6	+11.6	+12.4
		-5	+7.2	+8.2
		-4	+8.7	+10.7
		-3	+3.9	+4.6
		-2	-4.7	-5.0
		-1	+2.4	+2.0
		+0	+13.6	+14.7
		+1	-9.3	-10.4
		+2	+16.0	+16.9
		+3	-3.4	-2.9
		+4	+6.2	+7.2
		+5	+23.9	+26.6
		+6	+5.9	+9.9
		+9	+3.1	+2.4
		+12	+4.9	+6.4
		+13	+4.5	+5.6
		+15	+10.0	+11.3
	+5	-22	+4.3	+4.3
		-20	+3.6	+3.4
		-19	+3.1	+3.4
		-18	+14.4	+13.6
		-16	+2.9	+2.9
		-14	+5.8	+6.3
		-12	+5.2	+4.7
		-10	+6.1	+7.3
		-9	-4.6	-4.9
		-8	-9.9	-11.1
		-7	+3.5	+3.7
		-6	+12.6	+13.7
		-5	-4.9	-5.5
		-4	+5.1	+6.0
		-3	+6.4	+6.9
		-1	+7.3	+7.9
		+0	-6.7	-7.0
		+1	-10.4	-11.4
		+2	-25.3	-28.7
		+3	+4.2	+3.5
		+4	+11.8	+12.8
		+8	-9.7	-9.8
		+9	+2.7	+2.9
		+10	-10.8	-10.7
		+12	-9.3	-10.1
		+14	-4.7	-5.2
	+6	-21	-6.5	-7.2
		-20	-2.1	-2.1
		-19	-8.0	-9.0
		-18	-4.7	-4.7
		-16	-5.1	-6.7
		-14	-6.4	-7.0
		-13	-1.4	-2.0
		-12	-2.6	-2.6
		-11	-2.0	-2.1
		-10	-8.1	-10.7
		-9	-2.9	-2.4
		-8	-12.1	-14.3
		-7	-4.1	-4.3
		-6	-9.8	-11.8
		-4	-7.2	-8.7
		-3	+7.1	+8.7
		-2	-3.7	-3.2
		+0	-8.5	-9.5
		+1	+3.4	+3.5
		+2	-16.6	-17.7
		+3	+4.2	+4.3
		+4	-2.1	-2.1
		+5	+3.0	+2.4
		+6	-6.5	-7.5
		+7	+3.4	+4.6
		+9	+4.0	+5.0
		+10	-7.3	-7.9
		+12	-3.4	-3.1
		+13	+5.1	+4.7
	+7	-16	-5.2	-7.5
		-15	+10.3	+10.5
		-13	+3.8	+3.7
		-11	+12.6	+13.7
		-10	-3.5	-3.1
		-9	+6.0	+6.3
		-7	+8.5	+9.3
		-5	+11.6	+13.4
		-4	-3.3	-3.1
		-3	+7.4	+7.8
		-1	+6.6	+6.9
		+1	+9.8	+10.4
		+3	+7.8	+7.8
		+5	+7.3	+8.2
		+7	+10.0	+10.1
		+8	+3.4	+2.6
		+9	-2.3	-2.4
		+10	+4.4	+4.3
		+11	+6.9	+6.0
	+8	-16	+5.8	+5.0
		-15	+5.3	+4.9
		-14	-2.7	-2.6
		-13	+5.2	+4.6
		-12	-11.2	-10.7
		-11	+5.6	+5.2
		-9	+4.4	+4.1
		-7	-2.9	-3.8
		-6	-2.7	-2.0
		-5	+3.6	+3.5
		-3	-2.4	-4.3
		+2	-5.6	-5.8
		+5	-4.7	-5.2
		+6	-2.3	-2.1
		+7	-9.1	-9.2
		+9	-3.1	-2.9
		+11	-3.9	-2.1
	+9	-15	+1.6	+2.0
		-14	-4.3	-3.7
		-13	-20.7	-18.6
		-11	+4.2	+3.5
		-10	-2.1	-1.8
		-6	-3.2	-3.7
		-5	-6.0	-6.4
		-3	-15.0	-16.0
		+0	+3.7	+3.2
		+1	-2.5	-2.3
		+2	+5.4	+6.3
		+3	-4.8	-4.3
		+4	+1.3	+1.7
		+5	-4.0	-4.0
		+6	+3.1	+3.1
		+7	+3.2	+4.1
		+9	-11.5	-7.3
	+10	-10	+10.2	+11.1
		-9	-2.4	-[illegible]
		-8	+10.5	+11.4
		-7	+5.7	+6.4
		-6	+[illegible]	+9.2
		-4	+4.[illegible]	+4.4
		-2	+3.7	+4.1
		-1	+2.4	+2.4
		+0	+9.9	+9.5
		+1	-3.6	-4.0
		+2	+6.4	+5.6
		+3	-2.[illegible]	-1.4
		+4	+9.0	+7.0
		+6	+4.9	+4.4

between the planes R–C(8)–C(7) and C(8)–C(7)–O(1) is nearly ideal (62°) in α-CEQ, but is significantly smaller (47°) in the corresponding β-propoxy compound.

The ring atoms C(1), · · · C(6) are planar within the limits of error and define a 'least-squares' plane (Schomaker, Waser, Marsh & Bergman, 1959):

$$-0{\cdot}82287X-0{\cdot}01411Y+0{\cdot}56805Z-2{\cdot}3336=0$$

with respect to an orthogonal coordinate system XYZ with directions a, b and c^*. The atoms C(7), C(8), O(2), N and Cl (Table 7) are significantly, and O(1) and O(3) are possibly, outside this plane. In contrast with the

Table 4. *Final atomic parameters* (*fractions of cell edges*) *and their standard deviations* (Å) *for* α-5-(2′-chloroethoxy)-*o*-quinone-2-oxime

Atom	x	y	z	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
C(1)	−0·1138	−0·0564	0·1292	0·0057	0·0052	0·0055
C(2)	0·1423	−0·0540	0·1871	0·0056	0·0052	0·0053
C(3)	0·2736	0·0607	0·2171	0·0057	0·0053	0·0053
C(4)	0·1156	0·1757	0·1819	0·0057	0·0050	0·0053
C(5)	−0·1598	0·1664	0·1218	0·0057	0·0053	0·0055
C(6)	−0·2749	0·0555	0·0969	0·0060	0·0056	0·0055
C(7)	−0·0953	−0·2746	0·1218	0·0057	0·0054	0·0056
C(8)	−0·2556	−0·3741	0·0729	0·0058	0·0055	0·0056
O(1)	−0·2536	−0·1584	0·0967	0·0038	0·0035	0·0037
O(2)	0·1311	0·3795	0·1718	0·0042	0·0037	0·0040
O(3)	0·5086	0·0673	0·2706	0·0040	0·0039	0·0040
N	0·2680	0·2772	0·2074	0·0048	0·0044	0·0047
Cl	−0·1812	−0·3491	−0·0107	0·0014	0·0013	0·0013
H(1)	−0·268	0·245	0·102	0·066	0·068	0·067
H(2)	−0·475	0·044	0·055	0·064	0·064	0·066
H(3)	0·218	−0·128	0·206	0·063	0·064	0·064
H(4)	0·172	−0·274	0·128	0·072	0·073	0·077
H(5)	−0·147	−0·295	0·166	0·065	0·069	0·068
H(6)	−0·525	−0·378	0·063	0·065	0·063	0·065
H(7)	−0·166	−0·464	0·091	0·068	0·068	0·072
H(8)	0·303	0·428	0·192	0·099	0·105	0·101

Table 5. *Anisotropic thermal parameters for* α-5-(2′-*chloroethoxy*)-*o*-*quinone*-2-*oxime*

The thermal parameters are in the form: $\exp\,[-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2hka^*b^*U_{12}+2hla^*c^*U_{13}+2klb^*c^*U_{23})]$

Atom	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{13}$
C(1)	0·0141	0·0187	0·0245	0·0004	−0·0077	0·0212
C(2)	0·0148	0·0204	0·0201	−0·0028	0·0007	0·0153
C(3)	0·0190	0·0211	0·0188	0·0071	0·0000	0·0177
C(4)	0·0139	0·0197	0·0214	−0·0048	−0·0037	0·0192
C(5)	0·0162	0·0205	0·0237	0·0060	0·0028	0·0158
C(6)	0·0236	0·0242	0·0193	−0·0023	−0·0030	0·0128
C(7)	0·0147	0·0234	0·0217	0·0063	−0·0020	0·0031
C(8)	0·0154	0·0266	0·0238	0·0021	0·0035	0·0214
O(1)	0·0152	0·0168	0·0208	0·0010	−0·0053	0·0044
O(2)	0·0264	0·0166	0·0223	0·0071	0·0093	0·0037
O(3)	0·0198	0·0211	0·0236	0·0008	−0·0077	−0·0013
N	0·0214	0·0179	0·0193	0·0079	0·0004	0·0047
Cl	0·0184	0·0242	0·0180	0·0066	−0·0023	0·0098

Table 6. *Intramolecular bond distances* (Å) *and bond angles in* α-CEQ *and in* β-5-*n*-*propoxy*-*o*-*quinone*-2-*oxime*

Distance	α	β	Angle	α	β
C(1)–C(2)	1·358	1·357	C(8)–C(7)–O(1)	108°17′	107°31′
C(2)–C(3)	1·430	1·410	C(7)–O(1)–C(1)	116 52	117 51
C(3)–C(4)	1·502	1·482	O(1)–C(1)–C(2)	125 21	125 12
C(4)–C(5)	1·435	1·442	O(1)–C(1)–C(6)	112 31	112 9
C(5)–C(6)	1·345	1·344	C(2)–C(1)–C(6)	122 8	122 39
C(1)–C(6)	1·457	1·458	C(1)–C(2)–C(3)	120 30	119 37
C(1)–O(1)	1·343	1·344	C(2)–C(3)–O(3)	122 40	122 33
C(3)–O(3)	1·253	1·270	C(2)–C(3)–C(4)	117 1	118 38
C(4)–N	1·306	1·319	O(3)–C(3)–C(4)	120 20	118 49
O(2)–N	1·365	1·353	C(3)–C(4)–C(5)	119 36	119 9
O(1)–C(7)	1·448	1·451	C(4)–C(5)–C(6)	120 19	120 11
C(7)–C(8)	1·501	1·511	C(1)–C(6)–C(5)	120· 21	119 50
C(8)–Cl	1·792	—	C(3)–C(4)–N	114 18	124 19
C(8)–C(9)	—	1·523	C(5)–C(4)–N	125 53	116 32
			C(4)–N–O(2)	112 29	116 47

β-propoxy compound where the oxime group nearly coincides with the planar system the oxime group is in α-CEQ definitely outside this plane.

Table 7. *Distances from the molecular plane*

C(1)	−0·018 Å	C(8)	−0·343 Å
C(2)	+0·008	O(1)	−0·024
C(3)	+0·011	O(2)	−0·248
C(4)	−0·009	O(3)	+0·028
C(5)	+0·003	N	+0·077
C(6)	+0·015	Cl	+1·790
C(7)	−0·155		

It is interesting to note that the angle C(5)–C(4)–N (125° 53′) inside the quadrangle C(5), C(4), N and O(2) is significantly larger than the outside angle C(3)–C(4)–N (114° 18′). This difference can be interpreted as a steric effect of hydrogen atom H(1) and oxygen atom O(2) on the nitrogen atom thus inducing a non-linear overlap of σ-orbitals between C(4) and N (Hirshfeld, 1964). The same phenomenon was observed in β-5-n-propoxy-*o*-quinone-2-oxime (124° 19′ and 116° 32′; Romers, 1964), in *p*-methoxyindophenol *N*-oxide (124° 26′ and 114° 50′; Romers & Hesper, 1966),

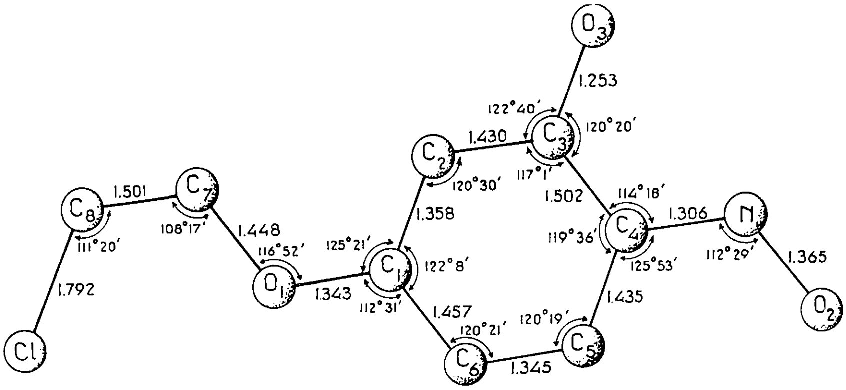

Fig. 2. Intramolecular bond distances and bond angles of α-CEQ.

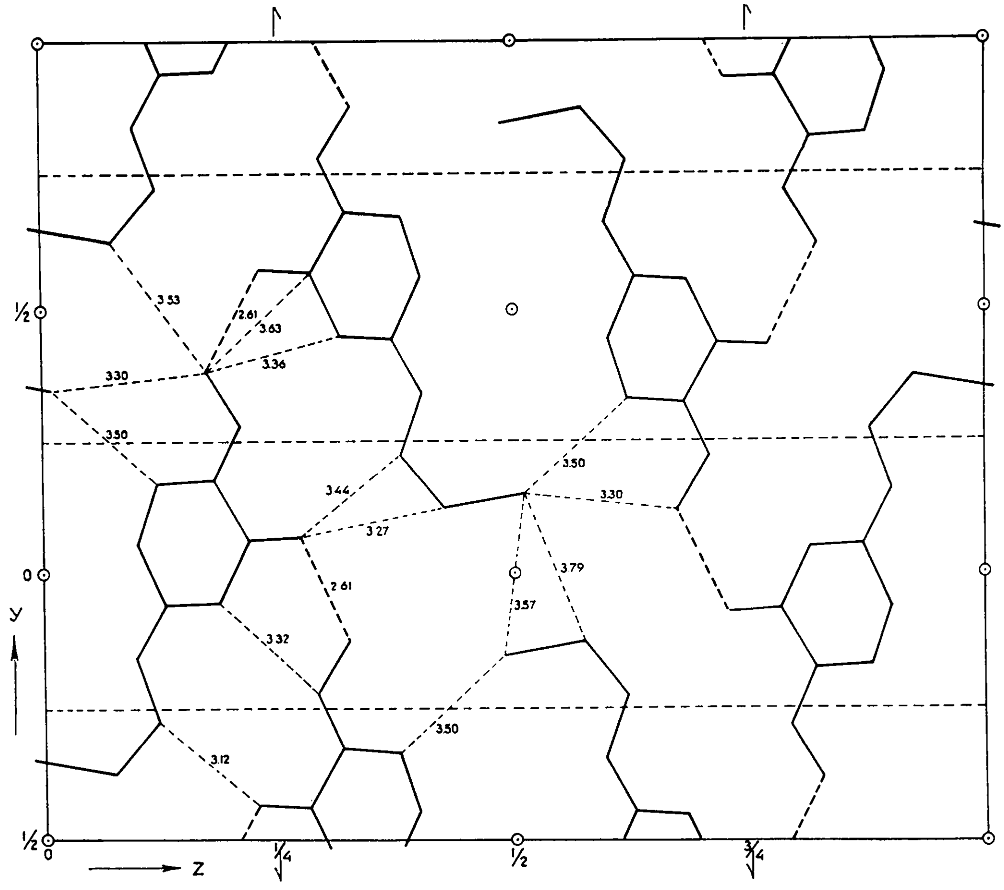

Fig. 3. The packing of α-CEQ viewed down the *a* axis.

in the two forms of 2-chloro-*p*-benzoquinone-4-oxime acetate (127°, 114° and 125°, 116°, respectively; Fischmann, MacGillavry & Romers, 1961*a*, *b*) and in *β*-*p*-nitrophenol (123·0° and 116·5°; Coppens & Schmidt, 1965). The same effect is observed for the angles C(6)–C(1)–O(1) (112° 31′) and C(2)–C(1)–O(1) (125° 20′), but can be attributed to the single- and the double-bond character of the respective bonds C(1)–C(6) and C(1)–C(2).

Table 8. *Intramolecular bond distances* C–H- *and* O–H (Å)

C(5)–H(1)	1·00	C(7)–H(5)	0·99
C(6)–H(2)	1·02	C(8)–H(6)	1·05
C(2)–H(3)	0·91	C(8)–H(7)	1·07
C(7)–H(4)	1·05	O(2)–H(8)	0·89

Table 9. *Intermolecular distances* (Å)

Bond	Symmetry relation	Distance
O(2)–O(3)	$1-x,\ \frac{1}{2}+y,\ \frac{1}{2}-z$	2·61
O(2)–C(3)	$-x,\ \frac{1}{2}+y,\ \frac{1}{2}-z$	3·63
O(2)–C(2)	$-x,\ \frac{1}{2}+y,\ \frac{1}{2}-z$	3·36
O(2)–C(8)	$x,\ 1+y,\ z$	3·53
O(2)–Cl	$-x,\ -y,\ -z$	3·30
N–C(2)	$-x,\ \frac{1}{2}+y,\ \frac{1}{2}-z$	3·50
N–C(2)	$1-x,\ \frac{1}{2}+y,\ \frac{1}{2}-z$	3·33
O(3)–C(7)	$-x,\ \frac{1}{2}+y,\ \frac{1}{2}-z$	3·44
O(3)–C(7)	$1-x,\ \frac{1}{2}+y,\ \frac{1}{2}-z$	3·12
O(3)–C(8)	$-x,\ \frac{1}{2}+y,\ \frac{1}{2}-z$	3·27
Cl–Cl	$-x,\ -1-y,\ -z$	3·57
Cl–C(5)	$-x,\ -y,\ -z$	3·50
Cl–C(8)	$1-x,\ -1-y,\ -z$	3·79

The intramolecular carbon, hydrogen and oxygen hydrogen bond lengths are given in Table 8. The variance in measured C–H distances (0·16 Å) is of the same order as their standard deviation (0·11 Å).

Fig. 3 shows the packing of the molecules viewed along [100]. Intermolecular distances smaller than 3·8 Å are indicated by dashed lines in this figure and are also listed in Table 9. Screw axes at $0, y, \frac{1}{4}$ and at $\frac{1}{2}, y, \frac{1}{4}$ take care of the packing in the direction [010]. The former gives rise to almost all short distances between the reference molecule at x, y, z and a molecule at $-x, \frac{1}{2}+y, \frac{1}{2}-z$. The latter relates the oxime group of the molecule at x, y, z with the carbonyl group of a molecule at $1-x, \frac{1}{2}+y, \frac{1}{2}-z$ by means of a hydrogen bridge of 2·61 Å (indicated by bold dashed lines in Fig. 3). In this way a double layer of molecules parallel to (0 0 1) is obtained.

The stacking of molecules in the direction [001] is obtained by the inversion operation resulting in a number of short distances between the chlorine atom of the reference molecule and the atoms O(2) and C(5) at $-x, -y, -z$, Cl at $-x, -1-y, -z$ and C(8) at $-1-x, -1-y, -z$.

We thank Professor E. Havinga for his continuous interest in this investigation and Miss G. M. la Roy for the preparation of the crystals.

References

Altona, C. (1964). *Acta Cryst.* **17**, 1282.

Bartindale, G. W. R., Crowder, M. M. & Morley, K. A. (1959). *Acta Cryst.* **12**, 111.

Berghuis, J., Haanappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. & Veenendaal, A. L. (1955). *Acta Cryst.* **8**, 478.

Buerger, M. J. (1949). *X-Ray Crystallography*, p. 229. New York: John Wiley.

Coppens, P. & Schmidt, G. M. J. (1965). *Acta Cryst.* **18**, 654.

Dawson, B. (1960). *Acta Cryst.* **13**, 403.

Fischmann, E., MacGillavry, C. H. & Romers, C. (1961*a*). *Acta Cryst.* **14**, 753.

Fischmann, E., MacGillavry, C. H. & Romers, C. (1961*b*). *Acta Cryst.* **14**, 759.

Geise, H. J. (1964). Thesis, Univ. of Leiden.

Geise, H. J., Romers, C. & Rutten, E. W. M. (1966). Submitted to *Acta Cryst.*

Henrich, F. (1904*a*). *J. prakt. Chem.* [2], **70**, 332.

Henrich, F. & Eisenach, H. (1904*b*). *J. prakt. Chem.* [2], **70**, 313.

Hirschfeld, F. L. (1964). *Israel J. Chem.* **2**, 87.

Hodgson, H. H. & Clay, H. (1932). *J. Chem. Soc.* **134**, 869.

McWeeny, R. (1951). *Acta Cryst.* **4**, 513.

Romers, C. (1964). *Acta Cryst.* **17**, 1287.

Romers, C. & Hesper, B. (1966). *Acta Cryst.* **20**, 162.

Romers, C. & Umans, A. J. H. (1960). *Proc. K. Ned. Akad. Wet. Afd. B.* **63**, 32.

Schomaker, V., Waser, J., Marsh, R. E. & Bergman, G. (1959). *Acta Cryst.* **12**, 600.

Ubbelohde, A. R. & Gallagher, K. J. (1955). *Acta Cryst.* **8**, 71.

Umans, A. J. H. (1959). Thesis, Univ. of Leiden.