The Crystal Structure of *trans*-Dichloridodiaquoethylene-diaminechromium(III) Chloride, *trans*-[CrCl₂(H₂O)₂en]Cl

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The structure of one of the three possible isomers of [CrCl₂(H₂O)₂en]Cl has been determined from visually estimated X-ray data by Patterson and Fourier methods and refined by the least-squares technique to a final R-value of 0.117 for 866 observed reflexions.

The crystals are orthorhombic, belonging to space group $Pmc2_1$, with $a = 8.811$ Å, $b = 7.539$ Å, and $c = 14.256$ Å. There are four formula units in the cell. Every chromium atom is octahedrally surrounded by two chlorine atoms in *trans* positions, the two nitrogen atoms of the ethylenediamine molecule, and two water oxygen atoms. The crystals are composed of [CrCl₂(H₂O)₂en]⁺ and Cl⁻ ions.

Average bond distances and angles are: Cr—Cl 2.32 Å, Cr—N 2.08 Å, Cr—O 2.05 Å, Cl—Cr—Cl 179.1°, N—Cr—N 83.1° and O—Cr—O 96.7°.

House and Garner[1] have used diperoxidoamines of chromium(IV) in the synthesis of amine complexes of chromium(III). In particular, they have devised a simpler and more efficient method to synthesize Weinmann's blue, [CrCl₂(H₂O)₂en]Cl.[2,3] In connection with this work the question has arisen which of the three theoretically possible geometric configurations is adopted by the [CrCl₂(H₂O)₂en]⁺ ion.

The kinetics of aquation of the [CrCl₂(H₂O)₂en]⁺ ion in acid solution has been investigated by these authors[4] in the hope that the kinetic data and identification of aquation products might have been of help in characterizing the geometric configuration. Since the "wet" chemical methods have failed to give a conclusive answer, one of the present authors (R. S.) has been asked by Dr. House to perform the crystal structure analysis of Weinmann's blue.

Table 1. Observed lines in the powder photograph of trans-[CrCl$_2$(H$_2$O)$_2$en]Cl. λ(CuKα_1)=1.54051 Å.

h k l	$10^5 \times \sin^2\theta_{obs}$	$10^5 \times \sin^2\theta_{calc}$	I_{obs}	d_{obs}
0 0 2	1176	1168	w	7.103
0 1 1	1341	1336	st	6.651
1 1 0	1810	1808	w	5.725
1 0 2	1936	1932	vst	5.536
1 1 1	2107	2100	vst	5.307
0 1 2	2216	2212	m	5.174
1 1 2	2974	2976	vw	4.466
2 0 0	3062	3057	w	4.402
0 1 3	3673	3671	vst	4.019
2 1 0	4096	4100	vvw	3.806
2 0 2	4224	4224	m	3.748
0 2 1	4464	4467	vst	3.636
0 0 4	4676	4671	st	3.562
1 0 4	·5446	5435	vvw	3.301
0 1 4	5716	5715	w	3.222
1 1 4	6480	6479	vw	3.026
2 1 3	6728	6728	w	2.970
0 2 3	6794	6803	w	2.955
2 2 1	7527	7524	w	2.808
1 2 3	7570	7567	w	2.800
2 0 4	7726	7728	w	2.771
3 1 0	7945	7921	vvw	2.733
3 0 2	8047	8045	w	2.715
3 1 1	8214	8213	w	2.688
0 1 5	8342	8342	vvw	2.667
2 1 4	8776	8771	m	2.600
1 1 5	9116	9107	vw	2.551
2 2 3	9864	9859	vw	2.452
0 3 2	10550	10562	m	2.370
0 2 5	11467	11474	m	2.274
0 1 6	11579	11554	w	2.266
4 0 0	12227	12226	m	2.203
3 1 4	12599	12592	vvw	2.171
4 1 1	13570	13562	vw	2.092
3 2 3	13660	13680	vvw	2.082
0 3 4	14075	14065	vvw	2.054
4 1 2	14443	14438	vvw	2.027
2 2 5	14553	14530	vvw	2.021
0 2 6	14686	14685	vvw	2.010
1 3 4	14841	14829	vvw	2.000
3 1 5	15244	15220	vvw	1.9728
0 1 7	15347	15349	m	1.9662
4 1 3	15911	15897	m	1.9310
4 2 1	16711	16693	m	1.8842
4 0 4	16913	16897	vw	1.8729
1 2 7	19233	19245	vvw	1.7563
1 0 8	19471	19449	vvw	1.7456
0 1 8	19734	19728	vw	1.7339
1 3 6	20654	20668	vvw	1.6948
0 4 4	21388	21372	vvw	1.6655
4 0 6	22743	22736	vw	1.6151
4 2 5	23700	23700	vvw	1.5822
4 1 6	23780	23780	vvw	1.5795
3 2 7	25380	25358	vvw	1.5289
4 3 4	26306	26291	vvw	1.5018
4 1 7	27561	27575	vw	1.4672
6 0 2	28670	28677	vvw	1.4385
0 0 10	29198	29194	vw	1.4255
4 1 8	31943	31954	vvw	1.3628

EXPERIMENTAL

Preparation and analysis of the crystals. Crystals of Weinmann's blue, suitable for X-ray diffraction experiments, were produced by the method proposed by House and Garner.[1] Our crystals showed exactly the same X-ray powder pattern as those kindly analyzed and sent to us by Dr. House.

X-Ray methods. Accurate cell dimensions were obtained from X-ray powder photographs taken in a Guinier focusing camera with $CuK\alpha$ radiation, using KCl ($a = 6.2929$ Å at 20°C)[5] as an internal standard.

For the single crystal work multiple film (5 films) equi-inclination Weissenberg photographs were taken of crystals of approximate dimensions $0.1 \times 0.1 \times 0.2$ mm with rotation about [100] (layer lines $0-8$) and [001] (layer line 0), using $CuK\alpha$ radiation. Three crystals were used.

The relative intensities of the reflexions were estimated visually by comparison with a standard scale (obtained by the rotating sector method) and were corrected using Lorentz' and polarization factors. Absorption and extinction effects were not considered. The $|F_o|$-values were brought to an absolute scale by comparison with the finally calculated structure factors.

Computing methods. The computational work, including least-squares refinement of the cell dimensions, Lorentz' and polarization corrections, Fourier summations, structure factor least-squares refinements and calculation of interatomic distances and angles, was performed on the electronic computer SAAB D21 using a set of crystallographic programmes written by Abrahamsson, Aleby, Larsson, Lindqvist and Wengelin.[6-11]

The atomic scattering factors used in the calculation of the structure factors were taken from Volume III of the *International Tables for X-ray Crystallography*, 1962.

UNIT CELL AND SPACE GROUP

The unit cell parameters were obtained from the measured $\sin^2\theta$ values by a least-squares procedure using 59 observed lines. $[CrCl_2(H_2O)_2en]Cl$ was then found to be orthorhombic with

$a = 8.811 \pm 0.003$ Å, $b = 7.539 \pm 0.006$ Å, $c = 14.256 \pm 0.006$ Å, $V = 947.0$ Å³.

(The errors given are 3σ).

Calculated and observed $\sin^2\theta$ values are given in Table 1.

The systematically absent reflexions were

$h0l$ with $l = 2n+1$

and $h00$ with $h = 2n+1$

The first condition is characteristic of the space groups $Pmc2_1$ (No. 26), $P2cm$ (No. 28), and $Pmcm$ (No. 51). The second condition does not apply to anyone of the ordinary space groups. It is taken to be "accidental" but it has, of course, a significant structural meaning (see below).

The density of the crystals, determined by weighing a sample in air and benzene, is 1.71 g/cm³. The density calculated for a unit cell containing four formula units is 1.73 g/cm³.
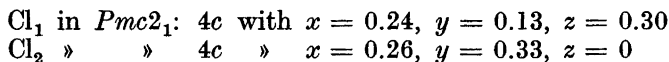
STRUCTURE DETERMINATION

Atomic positions. The Patterson function $P(uvw)$ showed the following large maxima:

u	v	w	peak height (arbitrary units)	u	v	w	peak height (arbitrary units)
0	0	0	3826	$\frac{1}{2}$	0.20	0.30	855
0.26	0	0	1455	0.26	0.29	$\frac{1}{2}$	628
0	0.27	$\frac{1}{2}$	1394	0.24	$\frac{1}{2}$	0.20	615
$\frac{1}{2}$	0	0	1333	$\frac{1}{2}$	0.24	$\frac{1}{2}$	595
$\frac{1}{2}$	$\frac{1}{2}$	0.20	1052	0.24	0.21	0.30	404

The above-mentioned fact that $h00$ reflexions are observed only for $h=2n$ means that if the x value of the position of one atom is x_1, there will be an atom of the same kind (but not necessarily symmetry-related) at, or very near, $x_1+\frac{1}{2}$. This is consistent with the fact that $P(uvw)$ has many large maxima at $u=0$ and $\frac{1}{2}$. Since all the remaining heavy peaks of $P(uvw)$ have $u\approx\frac{1}{4}$ the space group has at least approximate mirror planes perpendicular to the a axis at $x=0$ and $\frac{1}{2}$. This makes it probable that the space group is either $Pmc2_1$ or $Pmcm$ with the chromium and chlorine atoms in planes at $x=0$, $\frac{1}{4}$, $\frac{1}{2}$, and $\frac{3}{4}$. From this and space considerations one can conclude that the chromium atoms are situated in the mirror planes, the four chromium atoms in the cell thus occupying two two-fold positions. Comparison between calculated and observed interatomic vectors, with due account taken of possible interatomic distances, indicates that the only possible space group is $Pmc2_1$ with the following chromium positions:

Cr$_1$ in $Pmc2_1$: $2b$ with $x=\frac{1}{2}$, $y=0.13$, $z=0.30$
Cr$_2$ » » $2a$ » $x=0$, $y=0.33$, $z=0$
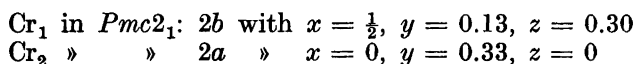
The structure was then solved in the usual, rather straightforward, manner. It was quite obvious that the highest Patterson peak (at 0.26, 0, 0) with the length 2.3 Å, which is a normal Cr—Cl bond length, could be interpreted as overlapping Cr—Cl vectors. The coordinated chlorine atoms would consequently occupy two four-fold positions with the same y and z coordinates as the chromium atoms and the x values 0.24 and 0.26. Probable coordinates for the two chlorine atoms of the asymmetric unit would thus be:

Cl$_1$ in $Pmc2_1$: $4c$ with $x=0.24$, $y=0.13$, $z=0.30$
Cl$_2$ » » $4c$ » $x=0.26$, $y=0.33$, $z=0$

A Fourier summation of the observed structure factors was then undertaken with the signs of the Fourier coefficients being determined by the contribution from the atoms Cr$_1$, Cr$_2$, Cl$_1$, and Cl$_2$. This showed large peaks at all the above positions with relative magnitudes expected for the chromium and chlorine atoms. There were also minor peaks in the mirror planes about the chromium positions. These were taken to be the oxygen and nitrogen sites. Possible positions of the remaining chlorine atoms were found both at $x=0$ and $\frac{1}{2}$ and $x=\frac{1}{4}$ and $\frac{3}{4}$. Calculation of $F(h00)$ values and comparison with observed structure factors made it obvious that the chloride ions occupy the position Cl$_3$ in $Pmc2_1$: $4c$ with $x=0.25$, $y=0.61$, $z=0.24$.

As the nitrogen atoms form tetrahedral bonds in the ethylenediamine molecule and as they lie in the mirror plane containing the chromium and

oxygen atoms, the carbon atoms must be out of this plane. There are two possible solutions to this problem. One is that the correct space group is the monoclinic one Pc. The other is to assume that the four carbon atoms of the unit cell statistically occupy two four-fold positions of $Pmc2_1$. The contribution from the carbon atoms to the structure factors is so small that it is impos-

Table 2a. Atomic coordinates (expressed in fractions of the cell edges) and isotropic thermal parameters with their standard deviations for *trans*-[CrCl$_2$(H$_2$O)$_2$en]Cl.

The temperature factor $=\exp[-B(\sin^2\theta)/\lambda^2]$. Space group $Pmc2_1$. 4 molecules in the unit cell. All atoms occupy the twofold positions $2a$ and $2b$ except Cl and C which occupy the position $4c$.

Atom	x	y	z	B Å^2	$\sigma(x)$ $\times 10^4$	$\sigma(y)$ $\times 10^4$	$\sigma(z)$ $\times 10^4$	$\sigma(B)$ Å^2
Cr$_1$	$\frac{1}{2}$	0.1311	0.3172			6	4	
Cr$_2$	0	0.3315	0.0134			6	4	
Cl$_1$	0.2372	0.1296	0.3167		6	7	4	
Cl$_2$	0.2637	0.6707	0.5145		5	7	4	
Cl$_3$	0.2547	0.6094	0.2395		7	7	6	
O$_1$	$\frac{1}{2}$	0.2895	0.2027	4.20		27	16	0.39
O$_2$	$\frac{1}{2}$	0.8945	0.2483	4.31		25	16	0.39
O$_3$	0	0.5540	0.1009	4.23		27	15	0.40
O$_4$	0	0.5318	0.3910	3.67		25	14	0.36
N$_1$	$\frac{1}{2}$	0.0001	0.4457	3.45		32	17	0.39
N$_2$	$\frac{1}{2}$	0.3578	0.4051	3.93		30	18	0.46
N$_3$	0	0.1786	0.1346	4.05		32	19	0.46
N$_4$	0	0.9085	0.4466	3.56		29	17	0.42
C$_1$	0.5415	0.8867	0.0199	4.99	47	47	34	0.96
C$_2$	0.5406	0.2984	0.5098	6.59	55	63	45	1.18
C$_3$	0.0368	0.9894	0.1213	7.84	70	84	43	1.47
C$_4$	0.0312	0.9419	0.0112	4.39	43	43	30	0.73

Table 2b. Anisotropic temperature parameters U_{ij} (with their standard deviations in parentheses). The expression used is

$$\exp-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2klb^*c^*U_{23}+2lhc^*a^*U_{31}+2hka^*b^*U_{12}).$$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{31}	U_{12}
Cr$_1$	0.055 (0.003)	0.043 (0.002)	0.034 (0.002)	0.005 (0.002)	0	0
Cr$_2$	0.045 (0.002)	0.047 (0.002)	0.037 (0.002)	-0.006 (0.002)	0	0
Cl$_1$	0.043 (0.002)	0.059 (0.003)	0.057 (0.003)	0.014 (0.003)	-0.005 (0.003)	-0.005 (0.002)
Cl$_2$	0.038 (0.003)	0.067 (0.003)	0.056 (0.003)	0.013 (0.003)	0.008 (0.003)	-0.001 (0.002)
Cl$_3$	0.052 (0.003)	0.063 (0.003)	0.062 (0.003)	-0.004 (0.003)	0.000 (0.003)	-0.005 (0.003)

Table 3. Observed and calculated structure factors for *trans*-[CrCl₂(H₂O)₂en]Cl. The columns are successively h, k, l, $100|F_o|$, $100|F_c|$, and phase angle α (expressed in fractions of one revolution).

h	k	l	$100\|F_o\|$	$100\|F_c\|$	α
0	0	2	5563	4523	0.7676
0	0	4	15335	14631	0.0897
0	0	6	5930	6630	0.0157
0	0	8	2813	2803	0.0499
0	0	10	9259	9248	0.1878
0	0	12	3083	3075	0.9448
0	0	14	4475	4188	0.3303
0	0	16	3500	2848	0.1277
0	1	0	352	258	0.5002
0	1	1	7399	6768	0.3396
0	1	2	7357	6798	0.6580
0	1	3	11048	13132	0.2921
0	1	4	7197	6642	0.3772
0	1	5	4919	4510	0.0215
0	1	6	9183	8509	0.8336
0	1	7	11033	10203	0.4169
0	1	8	7692	7277	0.5984
0	1	9	3399	3721	0.1712
0	1	10	691	1089	0.3423
0	1	11	3808	3534	0.5412
0	1	12	1205	1922	0.6601
0	1	13	2376	2497	0.4215
0	1	14	1064	1016	0.5462
0	1	15	1111	1251	0.3318
0	1	16	623	925	0.1493
0	1	17	1775	1640	0.5906
0	1	18	952	1204	0.7259
0	2	0	846	1664	0.5000
0	2	1	13871	15020	0.6117
0	2	2	3712	3405	0.5803
0	2	3	9243	7785	0.9030
0	2	4	4403	4297	0.5815
0	2	5	15541	12834	0.8115
0	2	6	6344	5882	0.5787
0	2	7	3654	4130	0.7955
0	2	8	1873	1842	0.6939
0	2	9	3969	4136	0.0272
0	2	10	4039	2225	0.6470
0	2	11	7124	6011	0.8177
0	2	12	769	1126	0.6576
0	2	13	1212	1521	0.2788
0	2	15	3684	3268	0.9522
0	2	16	1105	866	0.7727
0	3	0	3866	3848	1.0000
0	3	1	4259	3863	0.8403
0	3	2	10875	10391	0.0776
0	3	3	5155	4807	0.2742
0	3	4	9554	9675	0.9283
0	3	5	6931	7107	0.8912
0	3	6	4576	5192	0.2444
0	3	7	5060	4657	0.4453
0	3	8	5669	6230	0.0687
0	3	9	3449	3834	0.9869
0	3	10	912	1001	0.3424
0	3	11	1211	1314	0.4728
0	3	12	3529	3493	0.2197
0	3	14	2226	2302	0.1026
0	4	0	10139	10224	0.5000
0	4	1	4114	4531	0.2960
0	4	2	5241	5317	0.1156
0	4	3	3104	2970	0.2407
0	4	4	6483	6459	0.6326
0	4	5	4354	4783	0.3431
0	4	6	2936	2995	0.4473
0	4	7	1206	1267	0.2564
0	4	9	1407	1649	0.4108
0	4	10	4347	3808	0.7066
0	4	11	2243	2273	0.4241
0	4	12	3002	2873	0.3834
0	4	13	1806	1469	0.4422
0	4	14	2316	1940	0.8423
0	4	15	955	1055	0.5348
0	4	16	1463	1385	0.5489
0	5	0	3240	3629	0.5000
0	5	1	3630	3730	0.9278
0	5	2	2861	2670	0.3025
0	5	3	6061	6305	0.7961
0	5	4	2945	3399	0.6863
0	5	5	3985	3956	0.5098
0	5	6	4189	4119	0.8883
0	5	7	2362	2219	0.8669
0	5	8	2438	2533	0.7140
0	5	9	2530	2622	0.5883
0	5	10	1475	1433	0.0434
0	5	11	693	506	0.7832
0	5	12	2220	2292	0.9879
0	6	0	2505	2576	1.0000
0	6	1	3392	3442	0.0281
0	6	2	3412	3169	0.9865
0	6	3	2958	3350	0.6160
0	6	4	1420	1345	0.1497
0	6	5	1434	1691	0.2602
0	6	6	2779	3072	0.0154
0	6	7	2244	2324	0.0163
0	6	8	1317	1326	0.1717
0	6	9	1280	1590	0.6934
0	6	10	1493	1847	0.1352
0	6	11	1599	1838	0.2428
0	6	12	1013	971	0.1494
0	6	13	1398	1165	0.8602
0	7	0	1218	1192	1.0000
0	7	1	2436	2250	0.2723
0	7	2	2035	2116	0.5763
0	7	4	1866	1852	0.3391
0	7	5	3529	3191	0.3628
0	7	6	1276	1243	0.8522
0	7	7	2356	2049	0.5438
0	7	8	2806	2471	0.4374
0	7	9	912	752	0.2632
0	7	10	1318	1048	1.0000
0	8	0	1405	1319	0.7303
0	8	2	3188	2636	0.5966
0	8	3	684	721	0.6817
0	8	4	816	706	0.3035
0	8	5	1507	1368	0.8675
0	8	6	966	991	0.7371
0	8	7	805	814	0.6670
0	8	8	1962	1430	0.5902
0	8	9	627	701	0.9153
0	9	0	1121	1063	1.0000
0	9	1	837	964	0.6188
0	9	2	812	1052	0.9458
0	9	3	780	843	0.1849
0	9	4	656	867	0.1721
0	9	5	786	816	0.8294
0	9	6	894	975	0.9558
1	0	2	11027	9905	0.0052
1	0	4	2779	1937	0.5599
1	0	6	823	538	0.1958
1	0	8	4838	5130	0.0523
1	0	10	703	805	0.8893
1	0	12	2032	2100	0.2636
1	0	14	1145	1358	0.0750
1	0	16	497	570	0.3032
1	1	0	6362	5194	0.5000
1	1	1	8067	7573	0.1879
1	1	2	3092	2665	0.2227
1	1	3	1915	1718	0.2728
1	1	4	3921	3548	0.6985
1	1	5	4030	3638	0.3381
1	1	6	818	953	0.4888
1	1	7	3061	2496	0.1986
1	1	8	3619	3752	0.4847
1	1	9	1424	1529	0.6181
1	1	10	1719	1659	0.2933
1	1	11	782	767	0.4416
1	1	12	1189	1277	0.8491
1	1	14	1153	1221	0.4466
1	1	15	692	687	0.6196
1	1	16	610	655	0.3446
1	2	0	2280	2061	1.0000
1	2	1	865	743	0.7870
1	2	2	649	1086	0.4445
1	2	3	6706	5973	0.7321
1	2	4	2356	2314	0.5200
1	2	5	847	688	0.3127
1	2	6	1205	1203	0.4023
1	2	7	5395	5391	0.9301
1	2	8	864	870	0.6473
1	2	9	2661	2659	0.7333
1	2	10	1090	1151	0.9950
1	2	11	688	608	0.0166
1	2	13	1264	1640	0.8491
1	2	14	475	598	0.6609
1	2	16	267	203	0.6282
1	3	0	1484	1730	1.0000
1	3	1	1951	1802	0.1011
1	3	2	492	531	0.9121
1	3	3	3225	3423	0.9030
1	3	4	6635	5720	0.1421
1	3	5	1667	1793	0.3451
1	3	6	3529	3883	0.0017
1	3	7	1598	1559	0.8574
1	3	8	693	656	0.1707
1	3	9	1087	1262	0.6099
1	3	10	1936	2035	0.1739
1	3	11	609	646	0.1240
1	3	12	1057	1189	0.0262
1	3	13	765	733	0.8606
1	3	14	905	955	0.2908
1	3	16	675	762	0.1161
1	4	0	1958	2088	1.0000
1	4	1	1829	1940	0.2430
1	4	2	3309	3381	0.5030
1	4	3	3123	3083	0.2415
1	4	4	3240	2904	0.3364
1	4	5	775	917	0.3518
1	4	6	751	520	0.7860
1	4	7	642	707	0.2806
1	4	8	669	733	0.4791
1	4	9	773	853	0.3714
1	4	10	932	906	0.1408
1	4	11	862	846	0.3059
1	4	12	1235	1512	0.7948
1	4	13	1069	1215	0.4461
1	4	14	966	1122	0.5145
1	5	0	906	634	1.0000
1	5	1	2939	2773	0.7276
1	5	2	2726	2592	0.4576
1	5	3	1210	1232	0.0013
1	5	4	1279	1156	0.3121
1	5	5	1971	2239	0.8918
1	5	6	675	614	0.8752
1	5	7	773	1052	0.6314
1	5	8	497	601	0.6375
1	5	9	1273	1416	0.9516
1	5	10	493	387	0.1277
1	5	11	1386	1540	0.8540
1	5	12	630	769	0.7467
1	5	14	422	462	0.6485
1	6	1	899	744	1.0000
1	6	2	1668	1662	0.5796
1	6	3	968	1057	0.1912
1	6	4	345	392	0.1981
1	6	5	854	1070	0.5691
1	6	6	1449	1520	0.8613
1	6	7	934	1092	0.1275
1	6	8	610	695	0.5591
1	6	9	953	1563	0.1412
1	6	10	554	538	0.1621
1	6	11	513	578	0.7029
1	6	12	373	353	0.1174
1	7	0	1271	1548	0.5000
1	7	1	610	467	0.3074
1	7	2	610	649	0.2978
1	7	3	784	802	0.2714
1	7	4	984	871	0.5816
1	7	5	343	75	0.0095
1	7	6	1008	1106	0.4710
1	7	7	1299	1285	0.3672
1	7	8	535	534	0.0470
1	7	9	499	592	0.2482
1	7	10	563	664	0.6875
1	8	0	1468	1533	0.5000
1	8	1	782	734	0.7834
1	8	3	695	746	0.6498
1	8	4	801	666	0.7039
1	8	6	618	607	0.4124
1	8	7	575	503	0.0487
1	9	0	238	103	0.9999
1	9	1	473	528	0.0485
1	9	2	730	531	0.0895
1	9	3	543	521	0.7421
2	0	0	8329	7461	0.0136
2	0	2	6647	6384	0.5794
2	0	4	1455	1663	0.8150
2	0	6	1312	1414	0.3906
2	0	8	1666	1853	0.9738
2	0	10	1675	1556	0.3942
2	0	12	314	509	0.8988
2	0	14	1142	1112	0.2461
2	0	16	5715	5653	1.0000
2	1	0	3530	3376	0.5794
2	1	1	2976	2608	0.3429
2	1	2	6543	5535	0.8462
2	1	3	7105	6775	0.9477
2	1	4	1238	1149	0.3160
2	1	5	1647	1699	0.5443
2	1	6	2759	2508	0.6678
2	1	7	2058	1905	0.9078
2	1	8	3370	3139	0.1197
2	1	10	1368	1299	0.8156
2	1	11	457	617	0.9810
2	1	12	697	810	0.2886
2	1	14	434	613	0.9419
2	1	16	282	325	0.3981
2	1	17	2313	2108	1.0000
2	2	0	7212	6900	0.0020
2	2	1	3829	3591	0.9474
2	2	2	6017	5088	0.4947
2	2	3	1681	1407	0.4335
2	2	4	5604	5140	0.9355
2	2	5	1184	1015	0.6401
2	2	6	2088	1757	0.4284
2	2	7	271	116	0.2170
2	2	8	1787	1841	0.8844
2	2	9	1316	1400	0.4030
2	2	11	459	543	0.2926
2	2	12	847	1010	0.8736
2	2	14	430	488	0.3767
2	2	15	676	545	0.3293
2	3	0	1578	1483	0.5000
2	3	1	350	359	0.5695
2	3	2	2298	1899	0.8872
2	3	3	3099	2837	0.0993
2	3	4	1210	921	0.5997
2	3	5	1608	1479	0.4544
2	3	6	1436	1321	0.9378
2	3	7	1777	1592	0.8941
2	3	8	1838	1660	0.4005
2	3	9	1434	1150	0.4476
2	3	10	711	681	0.9283
2	3	11	1376	1487	0.8769
2	3	12	558	642	0.3020
2	3	13	538	618	0.4176
2	4	0	2448	2750	1.0000
2	4	1	1243	1136	0.8876
2	4	2	2247	2288	0.4805
2	4	3	1196	1225	0.5822
2	4	4	1541	1448	0.9978
2	4	5	1291	1363	0.9926
2	4	6	1220	1245	0.4439
2	4	7	1122	1047	0.7814
2	4	8	1322	1357	0.8469
2	4	9	1282	1277	0.9445
2	4	10	459	562	0.5426
2	4	11	322	516	0.4230
2	5	0	2043	2323	0.5000
2	5	1	985	1150	0.9927
2	5	2	2013	1976	0.0530
2	5	3	654	827	0.0944
2	5	4	2449	2481	0.4426
2	5	5	531	476	0.8717
2	5	6	1720	1892	0.8933
2	5	7	785	966	0.5994
2	5	8	1297	1455	0.4155
2	5	10	710	683	0.9109
2	5	12	692	799	0.4328
2	5	13	357	427	0.8659
2	6	1	947	853	1.0000
2	6	2	1704	1718	0.9538
2	6	3	636	747	0.5154
2	6	5	858	1157	0.4423
2	6	6	562	592	0.5269
2	6	7	911	1081	0.9939
2	6	8	447	401	0.0598
2	6	9	431	733	0.4519
2	6	10	407	225	0.3457
2	7	1	645	702	0.9782
2	7	2	322	520	0.3463
2	7	3	320	470	0.5174
2	7	4	317	195	0.8462
2	7	5	312	692	0.8878
2	7	7	716	969	0.3366
2	8	0	499	503	0.5000

Table 3. Continued.

h	k	l	Fo	Fc	val
2	8	1	642	841	0.5927
2	8	2	492	632	0.9031
2	8	3	482	535	0.9217
2	8	4	382	283	0.5026
2	8	5	367	492	0.4797
2	8	8	348	457	0.4119
2	9	0	666	633	1.0000
3	0	2	9778	10259	0.0013
3	0	4	2239	1703	0.8463
3	0	6	2372	1978	0.2458
3	0	8	6648	5806	0.0629
3	0	10	688	507	0.9283
3	0	12	3249	2519	0.2498
3	0	14	1678	1556	0.1010
3	0	16	731	683	0.4094
3	1	0	5496	6535	0.5000
3	1	1	6673	7564	0.1772
3	1	2	2152	2368	0.2136
3	1	3	1703	1747	0.3637
3	1	4	4583	3883	0.6833
3	1	5	5126	4445	0.3376
3	1	6	1423	1527	0.4926
3	1	7	100	2446	0.2121
3	1	9	4045	3758	0.4940
3	1	10	2176	1920	0.6510
3	1	11	2470	2041	0.3230
3	1	12	990	923	0.4487
3	1	14	1481	1421	0.8462
3	1	15	1583	1458	0.4081
3	1	16	860	784	0.6231
3	2	0	558	677	0.9999
3	2	1	1589	1671	0.8704
3	2	2	2057	2040	0.5738
3	2	3	7691	6838	0.7483
3	2	4	2452	2346	0.5141
3	2	5	880	806	0.3662
3	2	6	1256	1247	0.4723
3	2	7	6070	5945	0.9372
3	2	8	1177	1255	0.6377
3	2	9	2815	2777	0.7250
3	2	10	956	928	0.8729
3	2	11	656	996	0.0346
3	2	12	659	700	0.7193
3	2	13	2439	1841	0.8777
3	2	14	733	672	0.6610
3	2	15	267	218	0.6273
3	3	0	3352	3441	1.0000
3	3	1	2566	2293	0.0744
3	3	2	1061	1113	0.8904
3	3	3	3024	2621	0.8625
3	3	4	6417	5869	0.1433
3	3	5	1742	1632	0.3159
3	3	6	5098	4261	0.0121
3	3	7	1080	1388	0.5866
3	3	8	430	782	0.2051
3	3	9	1054	1188	0.6282
3	3	10	2758	2545	0.1405
3	3	11	572	844	0.1400
3	3	12	1412	1296	0.0351
3	3	13	923	846	0.8661
3	3	14	1587	1163	0.3072
3	4	0	1684	1737	1.0000
3	4	1	2339	2262	0.2939
3	4	2	3475	3683	0.6021
3	4	3	3248	3222	0.2768
3	4	4	3359	3152	0.3465
3	4	5	1159	1303	0.3563
3	4	6	797	977	0.8515
3	4	7	766	934	0.3416
3	4	8	1070	1372	0.5160
3	4	9	1186	1261	0.3408
3	4	10	737	891	0.1421
3	4	11	723	845	0.4978
3	4	12	1685	1627	0.7862
3	4	13	1649	1395	0.4980
3	4	14	1404	1195	0.5964
3	5	0	515	508	1.0000
3	5	1	2532	2998	0.7115
3	5	2	2891	2864	0.4862
3	5	3	1015	1202	0.0176
3	5	4	764	992	0.3230
3	5	5	2467	2708	0.8302
3	5	6	324	689	0.7431
3	5	7	870	1187	0.6359
3	5	8	1044	885	0.6495
3	5	9	1423	1613	0.9499
3	5	11	1965	1722	0.8364
3	5	12	1098	1020	0.7487
3	6	0	798	710	1.0000
3	6	1	1846	1703	0.5779
3	6	2	1701	1456	0.1173
3	6	3	657	755	0.2112
3	6	4	1234	1203	0.0617
3	6	5	1867	1670	0.8451
3	6	6	1228	1266	0.1921
3	6	7	856	845	0.5130
3	6	8	1634	1698	0.1034
3	6	9	1309	1289	0.0762
3	6	10	841	667	0.0859
3	6	11	761	591	0.7085
3	6	12	617	499	0.2075
3	7	0	1557	1645	0.5000
3	7	1	794	760	0.3167
3	7	2	791	777	0.2292
3	7	3	960	1008	0.2488
3	7	4	773	886	0.6997
3	7	6	1466	1313	0.4440
3	7	7	1400	1355	0.3954
3	7	8	464	620	0.0488
3	7	9	547	623	0.3002
3	7	10	857	793	0.6769
3	8	0	1685	1629	0.5000
3	8	1	625	801	0.8167
3	8	3	935	876	0.6654
3	8	4	1043	823	0.7573
3	8	6	956	692	0.4985
3	8	7	556	542	0.9455
3	9	0	187	157	0.9999
3	9	1	614	564	0.0541
3	9	2	712	664	0.0711
3	9	3	523	613	0.7480
4	0	2	2766	3009	0.7394
4	0	4	12894	8299	0.0921
4	0	6	5204	4774	0.0128
4	0	8	1650	1640	0.0471
4	0	10	7213	6423	0.1482
4	0	12	2427	2324	0.9571
4	0	14	2906	3157	0.3984
4	0	16	2045	2168	0.1244
4	1	0	589	834	0.5000
4	1	1	3492	4451	0.3614
4	1	2	4579	4473	0.6462
4	1	3	8454	8281	0.2890
4	1	4	4348	4014	0.3835
4	1	5	2511	2595	0.0500
4	1	6	5357	5288	0.8288
4	1	7	6550	7104	0.4117
4	1	8	5178	5236	0.5289
4	1	9	2625	2717	0.1437
4	1	10	515	589	0.3393
4	1	11	3007	2683	0.5995
4	1	12	1753	1560	0.6580
4	1	13	1842	1979	0.4490
4	1	14	987	786	0.5706
4	1	15	936	1011	0.3300
4	1	16	621	683	0.0416
4	2	0	1600	1295	0.5000
4	2	1	6584	8797	0.6117
4	2	2	2510	2191	0.5870
4	2	3	4853	4602	0.9955
4	2	4	2494	2578	0.5857
4	2	5	10323	8088	0.8883
4	2	6	3549	3765	0.5732
4	2	7	2964	2823	0.7430
4	2	8	1299	1342	0.6802
4	2	9	3060	2847	0.1261
4	2	10	1736	1671	0.6465
4	2	11	4777	4484	0.8156
4	2	12	906	868	0.6940
4	2	13	1076	1123	0.2822
4	2	14	2664	2481	0.9482
4	2	15	2076	2293	1.0000
4	3	1	2148	2155	0.8344
4	3	2	5679	6551	0.0760
4	3	3	2929	3175	0.2766
4	3	4	6369	6412	0.9307
4	3	5	4138	4691	0.8904
4	3	6	3309	3636	0.2435
4	3	7	3189	3340	0.4432
4	3	8	3833	4496	0.0677
4	3	9	3533	2775	0.9880
4	3	10	530	743	0.1468
4	3	11	911	1007	0.6614
4	3	12	2985	2644	0.2154
4	3	13	1969	1763	0.1142
4	3	14	402	546	0.1190
4	4	0	6953	6950	0.5000
4	4	1	3319	3397	0.2934
4	4	2	3282	3595	0.1114
4	4	3	2155	2348	0.2788
4	4	4	3781	4391	0.6328
4	4	5	3200	3561	0.3409
4	4	6	2374	2126	0.4404
4	4	7	1194	1150	0.2693
4	4	8	430	387	0.1420
4	4	9	1336	1392	0.4033
4	4	10	3025	2823	0.7042
4	4	11	1955	1799	0.4211
4	4	12	2213	2213	0.3815
4	4	13	1122	1202	0.4380
4	4	14	1510	1445	0.8606
4	5	1	2128	2422	0.5000
4	5	3	2265	2582	0.9241
4	5	4	1880	1846	0.3898
4	5	5	4424	4522	0.7940
4	5	6	2289	2382	0.7028
4	5	7	676	865	0.5198
4	5	8	2929	2879	0.5017
4	5	9	2858	3080	0.8802
4	5	10	1988	1653	0.8729
4	5	11	1899	1976	0.5855
4	5	12	1085	1088	0.0377
4	5	13	2269	1905	1.0000
4	6	0	2955	2575	0.0223
4	6	1	2688	2434	0.9876
4	6	3	2636	2532	0.6202
4	6	4	1184	1047	0.1551
4	6	5	1052	1185	0.2901
4	6	6	2377	2353	0.0380
4	6	7	1677	1760	0.0126
4	6	8	930	1057	0.1669
4	6	9	1321	1202	0.6812
4	6	10	1625	1427	0.1323
4	6	11	1431	1381	0.2190
4	7	0	777	816	1.0000
4	7	1	1658	1728	0.2723
4	7	2	1672	1600	0.5774
4	7	3	1492	1397	0.3409
4	7	5	2540	2456	0.3815
4	7	6	1018	939	0.8476
4	7	7	555	509	0.1442
4	7	8	1568	1564	0.5619
4	7	9	1853	1907	0.4349
4	8	0	805	811	1.0000
4	8	1	903	1002	0.7334
4	8	2	2057	2013	0.5967
4	8	3	516	582	0.6823
4	8	4	699	519	0.3313
4	8	5	1079	1040	0.8966
4	8	6	534	764	0.7340
5	0	2	3125	3598	0.0655
5	0	4	863	1087	0.4457
5	0	6	462	681	0.9609
5	0	8	2311	1844	0.0478
5	0	10	470	601	0.9050
5	0	12	834	830	0.2540
5	0	14	473	693	0.0502
5	0	-8	1355	1650	0.5000
5	1	1	1535	1994	0.1876
5	1	2	730	758	0.2774
5	1	3	456	534	0.1116
5	1	4	837	1079	0.7948
5	1	5	1224	729	0.3182
5	1	6	411	334	0.3700
5	1	7	1207	957	0.1437
5	1	9	1953	1484	0.4717
5	1	10	700	615	0.5332
5	1	11	521	568	0.1172
5	1	12	622	264	0.4711
5	1	14	420	470	0.4459
5	1	15	349	351	0.6048
5	1	16	282	485	0.9999
5	2	0	314	198	0.6241
5	2	2	381	347	0.4870
5	2	3	1893	1850	0.7012
5	2	4	1089	1015	0.5134
5	2	5	335	240	0.1112
5	2	6	696	738	0.3489
5	2	7	2574	1888	0.9295
5	2	8	698	305	0.5878
5	2	9	1201	1087	0.7349
5	2	10	850	672	0.9321
5	2	11	298	158	0.8214
5	2	13	883	758	0.8044
5	2	14	527	336	0.6650
5	2	15	360	155	0.4656
5	3	0	353	238	0.5001
5	3	1	390	560	0.2008
5	3	3	1357	1766	0.9361
5	3	4	2245	2212	0.1273
5	3	5	985	861	0.3719
5	3	6	2091	1462	0.9915
5	3	7	837	790	0.8418
5	3	8	296	252	0.0683
5	3	9	521	600	0.5727
5	3	10	559	607	0.1286
5	3	12	511	612	0.0097
5	3	13	302	283	0.8957
5	3	14	365	392	0.2346
5	3	15	267	169	0.5152
5	4	0	789	758	1.0000
5	4	1	793	654	0.1928
5	4	2	1418	1428	0.5597
5	4	3	1253	1118	0.2942
5	4	4	1139	990	0.3354
5	4	5	352	105	0.2415
5	4	6	368	171	0.2626
5	4	10	353	384	0.1063
5	4	11	332	488	0.3561
5	4	12	801	803	0.8090
5	4	13	566	542	0.4218
5	5	0	413	49	0.5666
5	5	1	1096	966	0.7932
5	5	2	1267	1161	0.4313
5	5	4	594	519	0.0106
5	5	5	543	479	0.3257
5	5	6	672	695	0.7937
5	5	9	355	246	0.8780
5	5	11	521	562	0.9141
5	5	12	249	316	0.7205
5	6	0	424	372	1.0000
5	6	1	734	700	0.5343
5	6	3	473	421	0.3096
5	6	4	298	98	0.3915
5	6	5	626	519	0.0403
5	6	6	543	479	0.0841
5	6	7	346	516	0.1552
5	6	8	272	205	0.5826
5	6	9	750	803	0.0440
5	6	10	581	441	0.0010
5	6	12	298	232	0.1192
5	7	0	614	675	0.5000
5	7	3	421	345	0.5196
5	7	6	409	432	0.5059
5	7	6	526	377	0.4982
5	7	7	716	304	0.3954
5	7	8	433	276	0.0615
5	8	0	684	749	0.5000
6	0	4	1879	2248	0.9445
6	0	6	3198	2812	0.5652
6	0	8	711	667	0.7840
6	0	10	893	959	0.4258
6	0	12	966	833	0.9010
6	1	0	792	921	0.3766
6	1	1	904	1568	1.0000
6	1	2	893	1361	0.4471
6	1	3	1016	1204	0.4476
6	1	4	1946	1578	0.8776
6	1	5	3552	2656	0.9382
6	1	6	992	901	0.3440
6	1	7	939	962	0.5184
6	1	8	1249	1141	0.9145
6	1	9	1057	1077	0.8496
6	1	10	403	404	0.4242
6	1	11	2055	1655	0.4360
6	1	12	397	685	0.8116
6	2	0	266	329	1.0000
6	2	1	3326	2675	0.0498
6	2	2	1500	1435	0.9337
6	2	3	2966	2227	0.4445
6	2	4	374	434	0.4702
6	2	5	2710	2229	0.9490
6	2	6	300	300	0.1951
6	2	7	971	994	0.4148
6	2	9	876	851	0.8752
6	2	11	710	896	0.3568
6	2	13	420	575	0.8639
6	3	0	929	872	0.5000
6	3	1	331	378	0.4287
6	3	2	732	865	0.8805
6	3	3	1616	1281	0.9907
6	3	4	594	476	0.5424
6	3	5	951	1023	0.4414
6	3	7	905	611	0.9259
6	3	8	954	914	0.9338
6	3	9	812	1114	0.4441
6	3	9	736	832	0.4762

Table 3. *Continued.*

h	k	l	Fo	Fc	val	h	k	l	Fo	Fc	val	h	k	l	Fo	Fc	val
6	3	10	320	333	0.9071	7	1	10	1111	1346	0.6662	7	6	4	1262	832	0.0087
6	3	11	740	786	0.0758	7	1	11	1112	1404	0.3416	7	6	5	1246	949	0.8713
6	3	13	336	419	0.4378	7	1	12	555	614	0.4553	7	6	6	1130	891	0.1156
6	4	0	1526	1553	1.0000	7	1	14	437	905	0.8673	7	6	7	793	624	0.4718
6	4	1	536	446	0.9920	7	2	0	550	432	0.5001	7	6	8	1378	1134	0.1177
6	4	2	1289	1240	0.4991	7	2	1	1724	1324	0.8628	7	7	0	1048	992	0.5000
6	4	3	673	651	0.5158	7	2	2	1653	1510	0.5437	7	7	1	759	669	0.3419
6	4	4	791	955	0.9450	7	2	3	4025	3749	0.7589	7	7	2	453	517	0.2389
6	4	5	695	643	0.0161	7	2	4	1356	1268	0.5461	7	7	3	787	762	0.2473
6	4	6	405	637	0.4155	7	2	5	491	346	0.4079	7	7	4	458	532	0.6699
6	4	8	565	718	0.6476	7	2	6	726	854	0.5251	8	0	0	1130	1228	0.6079
6	4	9	706	708	0.9407	7	2	7	3409	3470	0.9286	8	0	2	3070	2991	0.0463
6	4	10	298	345	0.3479	7	2	8	656	947	0.6610	8	0	4	1753	1436	0.0048
6	4	11	273	369	0.4136	7	2	9	1547	1636	0.7342	8	0	6	748	601	0.0488
6	5	0	1191	966	0.5000	7	2	10	362	542	0.7815	8	0	10	2469	2573	0.1853
6	5	1	522	564	0.0080	7	2	11	586	815	0.0358	8	0	12	1421	1072	0.9458
6	5	2	1365	1234	0.9910	7	2	12	302	579	0.7188	8	1	0	527	661	0.5100
6	5	4	1553	1193	0.4163	7	2	13	1087	1243	0.8692	8	1	1	1334	1765	0.3735
6	5	5	233	329	0.8926	7	3	0	3428	2485	1.0000	8	1	2	1507	1717	0.6417
6	5	6	1235	1175	0.9026	7	3	1	1642	1380	0.8624	8	1	3	3106	3297	0.2992
6	5	7	446	534	0.5043	7	3	2	1080	876	0.8464	8	1	4	1570	1653	0.4033
6	5	8	881	864	0.3497	7	3	3	1162	1072	0.8048	8	1	5	1090	970	0.6012
6	5	9	283	137	0.9419	7	3	4	2884	3156	0.1407	8	1	6	2114	1988	0.8179
6	5	10	315	461	0.9483	7	3	5	523	799	0.2992	8	1	7	3162	3033	0.4002
6	5	12	324	447	0.4139	7	3	6	2917	2542	0.0232	8	1	8	2479	2392	0.5277
6	6	0	645	527	1.0000	7	3	7	656	718	0.9258	8	1	9	1631	1216	0.1994
6	6	1	938	1058	0.5162	7	3	10	1643	1743	0.1797	8	1	10	1378	264	0.4426
6	6	2	506	432	0.5243	7	3	12	706	857	0.0456	8	1	11	1165	1256	0.5562
6	6	3	387	276	0.0673	7	3	13	326	541	0.8810	8	2	0	733	627	0.5000
6	6	5	748	622	0.4787	7	4	0	827	606	1.0000	8	2	1	3156	3250	0.6674
6	6	6	355	313	0.5012	7	4	1	1996	1437	0.3090	8	2	2	631	964	0.5808
6	6	7	561	639	0.9499	7	4	2	2982	2179	0.6659	8	2	3	1484	1799	0.9023
6	6	8	273	394	0.4473	7	4	3	2836	1838	0.2733	8	2	4	853	959	0.5002
6	6	10	178	184	0.3182	7	4	4	2204	1822	0.3597	8	2	5	2457	2961	0.7669
6	7	0	277	67	0.9098	7	4	5	928	963	0.3426	8	2	6	1742	1472	0.5485
6	7	1	478	389	0.9465	7	4	6	921	762	0.8195	8	2	7	1184	1188	0.7455
6	7	2	272	288	0.3491	7	4	7	639	736	0.3723	8	2	11	1533	2000	0.6185
6	7	3	325	298	0.5852	7	4	8	1066	1171	0.5334	8	3	0	703	991	1.0000
6	7	5	294	458	0.8815	7	4	9	748	974	0.3853	8	3	2	2148	2448	0.7721
6	7	7	302	607	0.3992	7	4	10	431	450	0.1332	8	3	3	892	1304	0.2450
6	8	1	278	520	0.5304	7	4	11	263	564	0.4805	8	3	4	2456	2597	0.9359
6	8	2	263	412	0.9065	7	4	12	655	1069	0.7771	8	3	5	1907	1850	0.8811
6	8	3	231	321	0.9949	7	5	1	2199	1766	0.6677	8	3	6	1977	1511	0.2376
7	0	2	4234	4809	0.0447	7	5	2	2193	1866	0.5100	8	3	8	1872	1933	0.0422
7	0	4	2137	2046	0.9398	7	5	3	647	636	0.0193	8	4	0	2798	2853	0.5000
7	0	6	1564	1796	0.2401	7	5	5	2049	1766	0.8145	8	4	1	1673	1586	0.2924
7	0	8	3266	3520	0.0752	7	5	6	608	556	0.7146	8	4	2	1674	1473	0.1018
7	0	12	1383	1736	0.2920	7	5	7	669	745	0.6423	8	4	3	1183	1223	0.2915
7	0	14	697	1094	0.1246	7	5	8	540	724	0.6506	8	4	4	1940	1630	0.3351
7	1	0	2835	3429	0.5000	7	5	9	886	1041	0.9864	8	5	1	509	1017	0.9140
7	1	1	2937	3497	0.1719	7	5	10	404	85	0.9746	8	5	2	505	771	0.3451
7	1	2	879	930	0.2230	7	6	0	491	437	1.0000	8	5	3	2384	1904	0.7934
7	1	3	924	1006	0.4142	7	6	1	1095	966	0.5431	8	5	6	879	1250	0.4497
7	1	4	1993	2010	0.8972	7	6	2	1569	1115	0.0879	9	0	0	6672	3580	1.0000
7	1	5	2493	2548	0.3351	7	6	3	671	685	0.2233	9	0	0	7341	7274	1.0000
7	1	6	984	1126	0.4415							10	0	0	609	266	0.5000
7	1	7	1156	1259	0.2969												
7	1	9	1560	2063	0.4992												

sible to decide which possibility is the correct one. The principal aim of this investigation was to establish the coordination about the chromium atoms, which is unaffected by either alternative. Since all other atoms fit the orthorhombic space group and since the structure of the ethylenediamine molecule is wellknown, the authors have chosen to describe the structure in terms of the space group $Pmc2_1$ with the carbon atoms statistically occupying two four-fold positions.

Structure refinement. At this stage of the structure determination the R-value was 0.20. The structure was then refined by the structure factor least-squares method using all 866 observed reflexions. For each reflexion the weight w was computed according to $w = [1 + (|F_o| - a)^2 b^{-2}]^{-1}$ with $a = 50$ and $b = 40$. Anisotropic temperature factors were used for all heavy atoms and isotropic ones for the light atoms. After about ten cycles the refinement converged to the parameters given in Table 2, the final R-value being 0.117.

Observed and calculated structure factors are given in Table 3. Calculated bond distances and angles and their standard deviations are given in Table 4 and some packing distances in Table 5. All bond distances were found to be within the normal range, thus supporting the correctness of the structure.

The result obtained from the refinement was further checked by calculating a three-dimensional electron density difference map. This showed only small residual maxima or minima, the largest maximum having a magnitude of about 15 % of the height of a true nitrogen peak in the $|F_o|$-synthesis.

Table 4. Bond distances and angles in the *trans*-dichloridodiaquoethylenediaminechromium(III) ion.

	Distance (Å)	e.s.d. (Å)		Distance (Å)	e.s.d. (Å)
Cr$_1$—Cl$_1$	2.32	0.01	Cr$_2$—Cl$_2$	2.32	0.00
Cr$_1$—O$_1$	2.02	0.02	Cr$_2$—O$_3$	2.09	0.02
Cr$_1$—O$_2$	2.04	0.02	Cr$_2$—O$_4$	2.03	0.02
Cr$_1$—N$_1$	2.08	0.02	Cr$_2$—N$_3$	2.08	0.03
Cr$_1$—N$_2$	2.12	0.02	Cr$_2$—N$_4$	2.04	0.02
N$_1$—C$_1$	1.41	0.05	N$_3$—C$_3$	1.48	0.07
N$_2$—C$_2$	1.60	0.07	N$_4$—C$_4$	1.48	0.04
C$_1$—C$_2$	1.58	0.06	C$_3$—C$_4$	1.71	0.07

	Angle (°)	e.s.d. (°)		Angle (°)	e.s.d. (°)
Cl$_1$—Cr$_1$—Cl$_1$	179.3	0.3	Cl$_2$—Cr$_2$—Cl$_2$	178.9	0.3
Cl$_1$—Cr$_1$—O$_1$	90.0	0.2	Cl$_2$—Cr$_2$—O$_3$	90.1	0.2
Cl$_1$—Cr$_1$—O$_2$	89.7	0.2	Cl$_2$—Cr$_2$—O$_4$	90.5	0.2
Cl$_1$—Cr$_1$—N$_1$	90.0	0.2	Cl$_2$—Cr$_2$—N$_3$	89.4	0.2
Cl$_1$—Cr$_1$—N$_2$	90.3	0.2	Cl$_2$—Cr$_2$—N$_4$	89.8	0.2
O$_1$—Cr$_1$—O$_2$	97.3	0.9	O$_3$—Cr$_2$—O$_4$	96.1	0.8
O$_1$—Cr$_1$—N$_1$	172.2	0.9	O$_3$—Cr$_2$—N$_4$	171.1	0.9
O$_1$—Cr$_1$—N$_2$	90.1	0.9	O$_3$—Cr$_2$—N$_3$	87.1	0.9
O$_2$—Cr$_1$—N$_1$	90.5	0.9	O$_4$—Cr$_2$—N$_4$	92.8	0.9
O$_2$—Cr$_1$—N$_2$	172.6	1.0	O$_4$—Cr$_2$—N$_3$	176.9	0.9
N$_1$—Cr$_1$—N$_2$	82.1	1.0	N$_4$—Cr$_2$—N$_3$	84.0	1.0
Cr$_1$—N$_1$—C$_1$	112.0	2.2	Cr$_2$—N$_3$—C$_3$	115.1	2.8
Cr$_1$—N$_2$—C$_2$	109.0	2.1	Cr$_2$—N$_4$—C$_4$	112.7	2.0
N$_1$—C$_1$—C$_2$	110.4	3.7	N$_3$—C$_3$—C$_4$	104.1	3.8
N$_2$—C$_2$—C$_1$	103.4	3.7	N$_4$—C$_4$—C$_3$	109.9	3.0
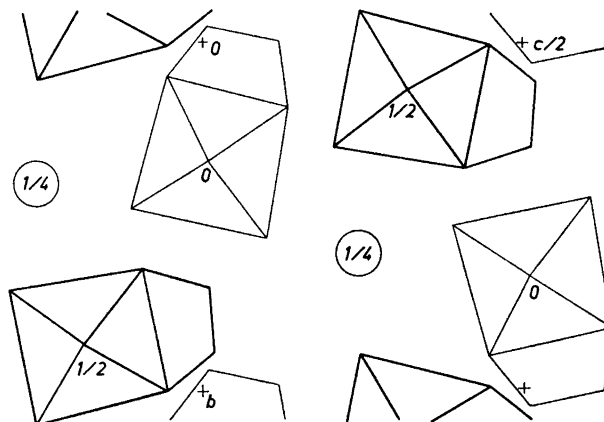
Fig. 1. The packing of the ions in *trans*-[CrCl$_2$(H$_2$O)$_2$en]Cl as viewed along the x-direction.

Table 5. Packing distances in *trans*-[CrCl₂(H₂O)₂en]Cl. (The first atom symbols refer to the coordinates given in Table 2. After the second atom symbols stand, within brackets, the unit cell translations in the *a*, *b*, and *c* directions and after that the number of the symmetry operation performed on the original coordinates of the atom as given in the *International Tables for X-ray Crystallography* Vol. I).

$Cl_1 - Cl_1$	(0	0	0)2		4.18 Å
$- Cl_2$	(0	-1	0)1		4.47
$- Cl_2$	(0	0	0)1		4.56
$- Cl_3$	(0	0	0)1		3.78
$- Cl_3$	(0	-1	0)1		4.08
$- N_3$	(0	0	0)1		3.35
$- N_4$	(0	-1	0)1		3.25
$- C_3$	(0	-1	0)1		3.46
$- C_4$	(0	1	0)4		3.36
$Cl_2 - Cl_2$	(1	0	0)2		4.16
$- Cl_3$	(0	0	0)1		3.95
$- Cl_3$	(0	1	0)4		3.84
$- O_1$	(1	1	0)3		3.41
$- N_1$	(0	1	0)1		3.39
$- N_2$	(0	0	0)1		3.51
$- C_2$	(1	0	0)2		3.29
$Cl_3 - O_1$	(0	0	0)1		3.28
$- O_2$	(0	0	0)1		3.05
$- O_3$	(0	0	0)1		3.02
$- O_4$	(0	0	0)1		3.17
$- C_2$	(1	1	-1)3		3.80
$- C_3$	(0	0	0)1		3.84
$O_3 - C_3$	(0	0	0)1		3.31
$- C_4$	(0	0	0)1		3.20

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

The crystals of Weinmann's blue, [CrCl₂(H₂O)₂en]Cl, consist of dichlorido-diaquoethylenediamine ions and chloride ions as illustrated in Fig. 1. The complex ion is shown in Fig. 2. The structure investigation has shown that
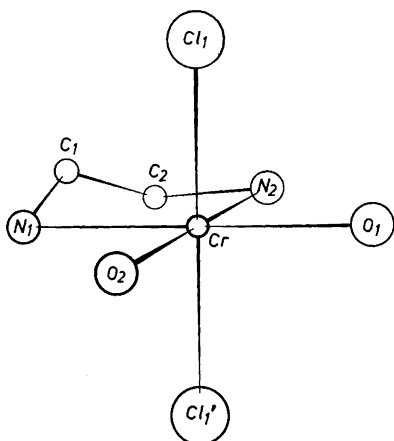
Fig. 2. The complex ion *trans*-[CrCl₂(H₂O)₂en]⁺.

the two chlorine atoms of the octahedral complex are situated in *trans* positions. The two water molecules and the two nitrogen atoms of the ethylenediamine molecule are situated at the corners of a somewhat distorted square. These atoms together with the chromium atom lie in a crystallographic mirror plane.

In the course of the structure determination it became obvious that there are two possible solutions, one being based on space group Pc and the other on space group $Pmc2_1$. They cannot be distinguished experimentally. Following the arguments given above, the authors have chosen to describe the structure in terms of space group $Pmc2_1$ instead of Pc. This means that the two carbon atoms of the ethylenediamine molecule statistically occupy fourfold positions (half-filled) in such a way that C_1 and C_2 are situated on opposite sides of the mirror planes one way or the other throughout the crystal. The same statistical manner was also chosen by Stomberg [12] in describing $[Cr(O_2)_2(H_2O)en](H_2O)$.

The Cr—Cl bond lengths are all 2.32 Å which compares well with the value 2.33 Å found by Ooi *et al.*[13] in $[CrCl_2en_2]Cl \cdot HCl \cdot 2H_2O$. The distances between the chromium and oxygen atoms lie between 2.02 and 2.09 Å, which is normal for a Cr—O_{water} bond; 2.03 Å was found in $[Cr(O_2)_2(H_2O)en](H_2O)$.[12] The Cr—N bond distances are 2.04—2.12 Å and thus not significantly different, the mean value being 2.08 Å. 2.06 Å was found in $[Cr(O_2)_2(H_2O)en](H_2O)$,[12] 2.12 Å in both $[CrCl_2en_2]Cl \cdot HCl \cdot 2H_2O$ [13] and $[Cr(O_2)_2(NH_3)_3]$ [14] (see also Ref. 15).

The errors in the positional parameters of the carbon atoms are, as expected, rather large, the standard deviations being about 0.07 Å as compared with 0.02 for nitrogen. The C—C and C—N bond lengths are, therefore, quite uncertain as well as the angles N—C—C and Cr—N—C but they do not differ significantly from their normal values.

The two independent N—Cr—N angles are not significantly different, being 82.1° and 84.0°. The value 82.6° was obtained for $[Cr(O_2)_2(H_2O)en](H_2O)$ [12] and 85.2° for $[CrCl_2en_2]Cl \cdot HCl \cdot 2H_2O$.[13]

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