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The Crystal Structure of *N*-Chloroacetylchlorothiazide, $C_9Cl_2H_7N_3O_5S_2$

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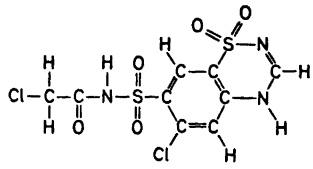
The crystal structure of *N*-chloroacetylchlorothiazide (6-chloro-7-*N*-chloroacetylsulfamoyl-1,2,4-benzothiadiazine 1,1-dioxide), has



been investigated. This compound which is an inhibitor of the enzyme carbonic anhydrase crystallizes in the space group $P2_12_12_1$ with $a=17.577$, $b=10.153$, $c=7.867$ Å and $Z=4$. A projection of the structure was solved by the symbolic addition method and the complete structure was then determined by means of three-dimensional Patterson and electron density calculations. A least squares refinement, based on 1123 independent reflexions yielded a final R value of 0.093.

The ring systems of the different molecules are packed almost parallel to one another, the molecules being connected through fairly strong N–H···O hydrogen bonds of distances 2.78 and 2.89 Å.

As indicated in the chemical formula there is a double C–N bond with a bond distance of 1.24 Å in the sulfonamide ring. This fact, together with the positions of the hydrogen bond contacts, permits the location of the amide hydrogen belonging to this ring.

Aromatic and heterocyclic sulfonamides constitute an important group of inhibitors of the enzyme carbonic anhydrase.[1,2] Usually the inhibition is reversible. By attachment of a reactive chloroacetyl group to the inhibitor molecule, however, compounds have been prepared which can irreversibly inactivate the enzyme.[3,4] One example is *N*-chloroacetylchlorothiazide (6-chloro-7-*N*-chloroacetylsulfamoyl-1,2,4-benzothiadiazine 1,1-dioxide) which in its reaction with human carbonic anhydrase B inactivates the enzyme under

modification of a histidine residue in the active site region of the enzyme. The modification is believed to take place by an initial reversible binding at the ordinary inhibitor site followed by a reaction of the chloroacetyl group with a histidine side chain in the close vicinity. A knowledge of the three-dimensional structure of the inhibitor molecule would then be expected to yield information about the topography of the active site region of the enzyme. It was therefore considered important to carry out an X-ray structure investigation of *N*-chloroacetylchlorothiazide.

EXPERIMENTAL

A sample of crystalline *N*-chloroacetylchlorothiazide was kindly provided by Dr. Georg Fölsch. Most of the crystals were very small, but some of them were large enough for data collection with Weissenberg techniques. The crystal selected for the investigation had the dimensions $0.05 \times 0.07 \times 0.24$ mm^3 and was mounted along the needle axis (c axis). Weissenberg films corresponding to $hk0 - hk6$ were taken using Cu$K\alpha$ radiation, six films

Table 1. Guinier powder diffraction data, Cu$K\alpha_1$ radiation. Cell parameters: $a = 17.577(2)$ Å, $b = 10.153(2)$ Å, and $c = 7.867(1)$ Å.

h k l	$\sin^2\theta_{obs}$	$\sin^2\theta_{calc}$	Intensity
0 1 1	1535	1534	s
3 0 0	1728	1728	m
2 1 1	2300	2302	s
3 1 1	3261	3262	m
1 2 1	3455	3453	s
4 1 0	3645	3648	m
1 0 2	4027	4027	w
0 1 2	4405	4410	m
1 1 2	4599	4602	s
1 3 0	5367	5372	vvw
5 0 1	5763	5759	vvw
3 1 2	6139	6139	s
1 2 2	6330	6329	vs
2 2 2	6902	6905	s
6 0 1	7874	7872	w
5 2 1	8057	8061	vvw
6 1 1	8439	8447	vw
6 2 0	9213	9215	w
1 1 3	9399	9396	w
2 1 3	9961	9972	m
7 0 1	10366	10368	vvw
3 4 0	10938	10937	w
1 2 3	11110	11122	w
0 4 2	13042	13044	vw
1 4 2	13233	13236	vw
0 3 3	13811	13808	vw
1 3 3	13997	14000	m
8 2 0	14593	14592	w
1 0 4	15537	15531	w
1 1 4	16104	16107	vw
9 1 1	17082	17088	vw
8 3 1	18431	18428	w

being recorded for each layer line. The photographs indicated an orthorhombic cell with the systematic absences $h00$: $h=2n+1$, $0k0$: $k=2n+1$ and $00l$: $l=2n+1$, uniquely establishing the space group to be $P2_12_12_1$.

The unit cell dimensions were determined from a Guinier powder photograph, the resulting parameters and observed and calculated $\sin^2\theta$ values being given in Table 1.

The 1320 independent reflexions occurring in $hk0-hk6$ were estimated visually, 1123 of them having intensities strong enough to be measured. The unobserved reflexions were included in the data set as $I_{\text{unobs}}=0.5I_{\text{obs}}$ (min). After Lp-correction of the data, approximate scale factors between the layer lines were calculated using $h0l$ reflexions recorded from another crystal. The absolute scale of the structure factors and an overall temperature factor ($B=3.65$ Å^2) were calculated according to Wilson [5] using the program GAASA 1.[6]

The linear absorption coefficient for *N*-chloroacetylchlorothiazide is 70.7 cm^{-1} for Cu$K\alpha$ radiation [7] and a correction was made for absorption first after an approximate structure had been determined.

STRUCTURE DETERMINATION AND REFINEMENT

A three-dimensional Patterson summation was calculated using the $hk0-hk6$ reflexions. Since this, however, showed appreciable overlap, it was decided to use the symbolic addition procedure,[8,9] as applied in the program system GAASA.[6,10] As space group $P2_12_12_1$ has centric projections, it was, after an unsuccessful treatment of the three-dimensional data, considered most efficient to continue the calculations using the $hk0$ reflexions only.

The 202 $|F_{hk0}|$ values on absolute scale, including unobserved reflexions, were converted to normalised structure factor magnitudes, $|E|$. For 121 reflexions with $|E|>0.5$, all $\sum_2$ pairs with $|E|$ values greater than 1.0 were tabulated. Four of the strongest normalized structure factors (*cf.* Table 2)

Table 2. Initial set for the symbolic addition procedure.

h	k	l	$\|E\|$	Symbol
4	1	0	2.761	a
5	12	0	2.523	b
1	11	0	2.240	c
1	5	0	2.180	d

were assigned the symbols $a-d$, and after four cycles of symbolic addition, a was solved as $-bc$ and the signs of 54 $|E|$ values were expressed in terms of b, c, and d. By choosing b and d negative, thereby specifying the origin in the xy projection, two alternative $\sum_2$ summations were performed, with c either positive or negative. In the first case 96 signs were determined with a probability [10] greater than 0.95, while 92 signs could be determined when c was negative. An E-map based on the 96 E_{hk0} magnitudes obtained for $a=-$, $b=-$, $c=+$, and $d=-$ gave a fairly well resolved picture of the structure, as is shown in Fig. 1. Fifteen of the independent atomic positions indicated in this figure could be identified, and a preliminary refinement based on these using all F_{hk0} reflexions gave an R value of 0.22 ($R=\sum||F_o|-|F_c||/\sum|F_o|$.)

Once the x and y coordinates of S_1, S_2, and Cl_1 were known it was possible to determine the z coordinates for these three atoms from the three-dimensional Patterson function. The complete set of atomic coordinates in the structure was then obtained through successive electron density calculations.

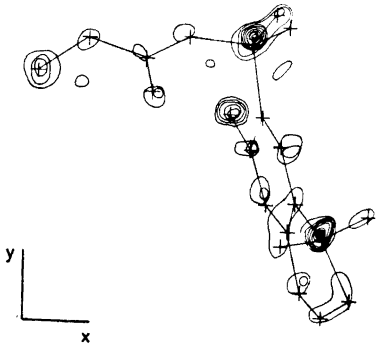


Fig. 1. E-map based on the $hk0$-reflexions. The final position of the chloroacetyl-chlorothiazide molecule is indicated.

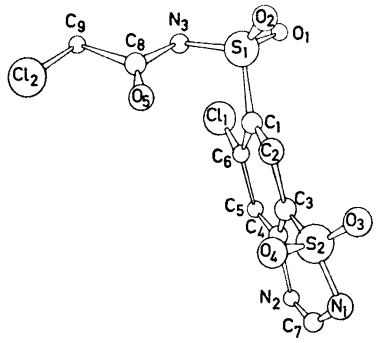


Fig. 2. Structure and numbering of the molecule.

After the absorption correction had been performed (program DATAP2 [11]), the structure was refined with isotropic temperature factors and separate scale factors for the seven layer lines using the least squares program LALS.[11] The resulting R value was 0.102.

The final refinement included anisotropic thermal parameters, only an overall scale factor being allowed to vary. Atomic scattering factors given by Doyle and Turner [12] were used, and the sulfur and chlorine values were corrected for the real part of the anomalous scattering.[13] The structure factors were assigned weight according to the formula $w=(a+F_o+bF_o^2+cF_o^3)^{-1}$ with $a=6.0$, $b=0.02$, and $c=0.0003$, and the final R value was 0.093. The corresponding atomic parameters are given in Table 3 and the observed and calculated structure factors are listed in Table 4.

A difference electron density summation based on the final parameters showed no important maxima. The highest peaks ($0.5-0.7$ $e/Å^3$) occurred in the vicinity of the sulfur and chlorine atoms and no definite peaks corresponding to the hydrogen atoms could be detected. All Fourier calculations were carried out with the program DRF,[11] and bond distances and angles were calculated with the program DISTAN.[11]

DESCRIPTION OF THE STRUCTURE

The N-chloroacetylchlorothiazide molecule is depicted in Fig. 2 and the packing of the molecules in the structure is illustrated in Fig. 3. As is apparent from the latter figure, the ring systems are mainly parallel to the yz plane and the chloroacetyl group is elongated along the direction of the x axis. Each molecule is connected by hydrogen bonds to four others, two-dimensional

Table 3. Final atomic parameters ($\times 10^4$). The anisotropic temperature factor is of the form $\exp[-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+hka^*b^*U_{12}+hla^*c^*U_{13}+klb^*c^*U_{23})]$.

Atom	*x/a*	*y/b*	*z/c*
Cl 1	7882(2)	2404(3)	−3366(1)
Cl 2	5211(2)	3326(5)	1522(6)
S 1	8168(2)	4014(2)	0195(4)
S 2	9138(2)	−0513(3)	3058(4)
O 1	8549(6)	4719(11)	−1131(17)
O 2	8308(7)	4357(12)	1882(13)
O 3	9755(5)	0135(8)	3656(13)
O 4	8566(4)	−0732(7)	4198(12)
O 5	6851(4)	2882(7)	1907(11)
N 1	9395(5)	−1926(12)	2292(17(
N 2	8870(5)	−1680(8)	−0360(13)
N 3	7249(5)	4233(8)	−0184(15)
C 1	8330(5)	2315(9)	−0058(17)
C 2	8606(6)	1669(10)	1422(16)
C 3	8773(6)	0320(10)	1306(16)
C 4	8660(6)	−0347(10)	−0240(18)
C 5	8373(6)	0311(11)	−1694(16)
C 6	8208(6)	1631(10)	−1495(16)
C 7	9218(9)	−2327(12)	0860(18)
C 8	6990(6)	3669(10)	0819(16)
C 9	5903(6)	4123(12)	0381(16)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl 1	90(2)	63(2)	58(2)	25(3)	−31(4)	14(3)
Cl 2	52(1)	147(4)	82(3)	3(4)	−5(4)	42(6)
S 1	54(1)	41(1)	55(2)	9(2)	6(3)	6(3)
S 2	63(1)	44(1)	54(2)	−5(3)	−26(3)	5(3)
O 1	63(4)	50(4)	69(7)	20(8)	24(9)	−20(8)
O 2	58(4)	47(4)	67(7)	10(7)	25(9)	−11(8)
O 3	103(7)	70(6)	116(9)	35(10)	−10(15)	−27(13)
O 4	95(6)	125(9)	52(7)	−25(10)	15(11)	−46(13)
O 5	47(3)	55(4)	56(6)	−23(7)	0(8)	−5(8)
N 1	144(11)	39(6)	64(8)	−34(15)	−70(17)	−16(13)
N 2	65(5)	61(4)	46(7)	−11(8)	−7(10)	1(8)
N 3	52(5)	50(5)	72(7)	−14(8)	21(11)	−9(10)
C 1	36(4)	41(4)	52(7)	6(7)	5(10)	7(10)
C 2	40(4)	43(5)	56(8)	−13(9)	−13(10)	−9(10)
C 3	46(5)	39(5)	47(8)	−17(9)	−13(10)	3(9)
C 4	47(5)	42(4)	75(9)	−15(9)	−19(12)	35(12)
C 5	49(5)	56(6)	32(7)	−5(9)	8(10)	−3(10)
C 6	58(5)	44(5)	48(8)	−1(11)	−19(11)	23(10)
C 7	98(9)	43(6)	64(10)	−3(15)	−19(16)	0(12)
C 8	59(5)	47(5)	33(7)	−21(9)	14(10)	−10(9)
C 9	53(5)	71(5)	34(7)	−33(12)	7(10)	−12(12)

Table 4. Observed and calculated structure factors. The columns are h, $10|F_o|$, $10|F_c|$ and the phase in radians. * indicates unobserved reflexions.

H	C	C	
2	622	632	3.14
4	1172	991	3.14
6	1133	1114	0.0
8	437	405	3.14
10	263	293	0.0
12	272	263	0.0
14	76	83	3.14
16	103	101	3.14
18	137	129	3.14
20	223	190	3.14

H	1	0	
1	105	78	1.57
2	273	220	-3.14
3	1187	1173	-1.57
4	1693	1566	3.14
5	44	43	-1.57
6	104	117	0.0
7	737	729	1.57
8	38	62	-3.14
9	144	126	-1.57
10	130	140	3.14
11	23	25	1.57 *
12	159	181	3.14
13	194	199	1.57
14	215	208	3.14
15	112	115	-1.57
16	103	109	3.14
17	27	2	1.57 *
18	26	61	3.14 *
19	69	64	1.57

H	2	0	
0	325	269	0.0
1	11	24	-1.57 *
2	286	285	0.0
3	622	588	1.57
4	895	888	3.14
5	616	614	1.57
6	29	25	-0.00
7	129	175	1.57
8	323	352	0.0
9	251	262	1.57
10	189	224	3.14
11	183	210	-1.57
12	242	233	0.0
13	26	60	1.57 *
14	27	14	-0.00 *
15	94	93	-1.57
16	27	21	3.14 *
17	77	69	-1.57
18	62	54	3.14
19	23	9	1.57 *
20	21	6	0.00 *
21	101	79	-1.57
22	72	67	0.0

H	3	0	
1	45	62	-1.57
2	108	99	3.14
3	198	232	-1.57
4	235	277	3.14
5	231	270	-1.57
6	18	7	-0.00 *
7	381	361	-1.57
8	711	671	0.0
9	22	44	-1.57 *
10	324	355	3.14
11	217	259	-1.57
12	266	286	0.0
13	352	312	-1.57
14	227	210	3.14
15	103	101	-1.57
16	165	148	3.14
17	162	141	-1.57
18	197	170	-0.00

H	4	0	
0	531	465	0.0
1	691	708	-1.57
2	59	41	-3.14
3	826	749	1.57
4	45	30	-3.14
5	498	476	-1.57
6	181	189	-3.14
7	255	255	-1.57
8	80	88	0.0
9	270	287	1.57
10	25	15	3.14 *
11	327	309	-1.57
12	102	102	3.14
13	57	50	-1.57
14	147	217	-0.00
15	27	2	1.57 *
16	85	89	3.14
17	88	86	1.57
18	23	32	-0.00 *
19	184	155	1.57

H	5	0	
1	778	710	-1.57
2	103	97	-0.00
3	70	63	-1.57
4	478	498	3.14
5	69	58	1.57
6	274	288	0.0
7	124	138	-1.57
8	352	375	0.0
9	169	201	1.57
10	262	259	0.0
11	27	14	1.57 *
12	95	107	0.0
13	81	68	-1.57
14	28	1	-3.14 *
15	132	102	1.57
16	82	87	3.14
17	24	22	1.57 *
18	22	5	-0.00 *
19	72	62	-1.57
20	166	166	-0.00

H	6	0	
0	284	336	3.14
1	243	253	-1.57
2	652	611	0.0
3	86	101	-1.57
4	22	14	-3.14 *
5	566	534	1.57
6	130	143	0.0
7	24	10	1.57 *
8	432	446	0.0
9	26	47	1.57 *
10	192	189	3.14
11	28	14	-1.57 *
12	106	122	3.14
13	222	198	-1.57
14	27	3	-3.14 *
15	26	19	-1.57 *
16	24	26	-0.00 *
17	129	119	1.57

H	7	0	
1	79	85	-1.57
2	231	229	3.14
3	187	199	1.57
4	66	62	0.0
5	179	169	1.57
6	311	303	3.14
7	133	148	-1.57
8	258	235	0.0
9	27	5	-1.57 *
10	28	53	-0.00 *
11	63	72	-1.57
12	222	190	3.14
13	27	46	-1.57 *
14	26	12	0.0 *
15	166	146	1.57

H	8	0	
0	154	144	3.14
1	26	32	1.57 *
2	224	176	3.14
3	26	8	-1.57 *
4	188	195	0.0
5	234	219	1.57
6	183	167	0.0
7	134	135	1.57
8	28	64	3.14 *
9	223	190	-1.57
10	28	21	-0.00 *
11	102	89	1.57
12	91	80	3.14
13	111	107	-1.57
14	23	6	-0.00 *
15	21	8	1.57 *
16	18	21	-0.00 *
17	67	67	1.57

H	9	0	
1	218	196	1.57
2	61	44	0.0
3	177	186	1.57
4	27	35	3.14 *
5	123	132	-1.57
6	56	67	0.0
7	27	13	-1.57 *
8	27	53	3.14 *
9	205	181	-1.57
10	154	147	0.0
11	25	9	-1.57 *
12	76	72	-3.14
13	63	57	1.57
14	19	5	3.14 *
15	68	67	-1.57

H	10	0	
0	160	165	3.14
1	61	49	1.57
2	27	39	3.14 *
3	93	92	-1.57
4	93	120	0.0
5	84	73	1.57
6	177	141	0.0
7	26	5	-1.57 *
8	79	74	-3.14
9	24	51	-1.57 *
10	23	3	3.14 *
11	62	54	1.57
12	19	19	-3.14 *
13	17	25	-1.57 *
14	71	74	3.14

H	11	0	
1	218	182	1.57
2	25	46	0.0 *
3	55	44	1.57
4	76	91	0.0
5	24	29	1.57 *
6	23	47	3.14 *
7	22	9	-1.57 *
8	21	23	3.14 *
9	150	132	-1.57

H	12	0	
0	21	32	0.0 *
1	21	13	1.57 *
2	64	68	3.14
3	140	118	1.57
4	61	62	0.0
5	177	146	-1.57
6	56	53	-0.00

H	0	1	
2	1026	758	-1.57
3	445	343	1.57
4	798	689	1.57
5	1508	1297	-1.57
6	838	731	1.57
7	119	122	-1.57
8	399	382	-1.57
9	133	152	-1.57
10	410	434	1.57
11	78	89	1.57
12	27	6	-1.57 *
13	147	170	1.57
14	325	291	1.57
15	30	38	1.57 *
16	160	170	1.57
17	29	8	1.57 *
18	116	112	-1.57
19	26	24	-1.57 *
20	52	43	1.57
21	63	61	1.57

H	1	1	
0	1185	1036	1.57
1	394	325	-1.47
2	927	764	-0.31
3	215	202	0.75
4	1103	853	-0.14
5	543	450	-2.23
6	929	819	1.68
7	335	347	1.65
8	202	207	-1.96
9	519	501	0.86
10	179	203	-0.40
11	217	247	2.60
12	239	273	1.93
13	251	267	1.43
14	231	215	-0.95
15	67	70	-2.04
16	203	215	0.02
17	111	113	1.52
18	57	44	-1.43
19	26	47	0.91 *
20	95	101	-2.25
21	62	61	3.08

H	2	1	
0	1458	1326	0.0
1	1234	1099	2.42
2	205	202	-0.26
3	280	274	-1.33
4	807	652	1.39
5	685	599	1.71
6	302	274	-0.41
7	476	488	-2.41
8	433	405	-2.65
9	322	327	1.12
10	162	187	-0.70
11	279	315	2.24
12	245	265	-0.18
13	234	226	-2.85
14	158	179	2.26
15	88	82	-0.52
16	95	94	-2.48
17	71	70	-2.46
18	80	64	-2.35
19	80	88	-2.31
20	78	72	-3.12
21	59	55	-0.76

H	3	1	
0	1674	1438	1.57
1	850	779	2.82
2	587	545	-1.58
3	420	404	-1.69
4	239	217	-1.21
5	138	134	2.01
6	252	259	-3.08
7	320	299	2.50
8	497	476	-2.90
9	24	39	0.60 *
10	177	209	1.79
11	141	162	2.03
12	81	103	2.36
13	109	120	-1.94
14	30	36	-2.85 *
15	88	94	-0.46
16	30	26	-3.03 *
17	84	87	-0.67
18	27	55	-2.61 *
19	71	67	1.35
20	21	45	-2.43 *
21	45	35	0.74

H	4	1	
0	70	74	-3.14
1	364	350	2.42
2	141	133	-2.31
3	755	695	1.05
4	626	585	-2.68
5	356	374	-0.92
6	446	435	2.15
7	204	238	2.61
8	182	193	-1.91
9	73	71	0.21
10	46	51	-1.53
11	173	202	0.80
12	309	316	0.74
13	30	20	-0.69 *
14	137	146	-1.27
15	96	96	-0.61
16	66	74	-2.94
17	28	12	1.57 *
18	26	28	1.96 *
19	61	57	-1.36
20	51	59	-1.00
21	49	62	-0.32

H	5	1	
0	122	118	1.57
1	484	485	-1.00
2	234	246	-0.31
3	491	483	-2.64
4	338	360	-1.43
5	259	264	-0.90
6	144	154	-0.74
7	179	199	0.81
8	165	187	0.42
9	215	224	2.79
10	29	21	-1.44 *
11	221	205	-0.30
12	149	148	0.89
13	150	172	3.08
14	68	70	-1.83
15	295	267	-1.46
16	28	44	-0.58 *
17	91	98	-0.17
18	75	85	0.93
19	77	98	2.59

H	6	1	
0	389	413	0.0
1	159	169	-1.08
2	130	137	-1.68
3	62	49	0.78
4	49	62	-2.56
5	123	132	2.48
6	253	286	2.28
7	138	155	-1.03
8	348	361	2.29
9	257	241	2.44
10	251	251	-1.79
11	178	171	-1.81
12	258	250	0.85
13	137	142	-0.24
14	90	91	1.07 *
15	117	110	2.16
16	65	76	-2.54
17	24	16	-2.06 *
18	73	81	1.80
19	38	41	1.11

H	7	1	
0	48	30	-1.57
1	68	74	0.52
2	115	139	2.10
3	259	266	-2.25
4	171	178	2.93
5	28	44	1.08 *
6	141	141	-1.24
7	82	60	0.48
8	219	201	0.67
9	149	144	1.91
10	179	166	-0.22
11	208	215	1.32
12	30	56	-2.45 *
13	102	120	-1.93
14	137	134	1.41
15	26	6	0.11 *
16	90	82	-1.78

H	8	1	
0	124	120	3.14
1	300	287	-0.65
2	127	125	-0.21
3	232	213	2.08
4	85	77	-1.00
5	30	49	1.11 *
6	30	55	-2.92 *
7	205	191	1.04
8	31	66	2.33 *
9	267	250	3.10
10	30	64	1.00 *
11	30	32	-1.41 *
12	91	100	-0.12
13	94	91	-0.90
14	73	56	-2.21
15	56	56	2.91

H	9	1	
0	160	166	-1.57
1	160	167	-1.90
2	124	123	2.33
3	114	130	0.56
4	162	168	-2.66
5	136	150	2.88
6	30	14	0.15 *
7	30	51	-2.22 *
8	146	156	0.54
9	111	111	2.04
10	108	102	-2.13
11	27	21	-0.09 *
12	26	45	0.61 *
13	81	87	-0.15
14	51	42	2.99

H	10	1	
0	30	12	3.14 *
1	30	60	-1.78 *
2	86	89	-2.90
3	30	8	-2.10 *
4	111	107	1.31
5	105	108	1.53
6	129	118	-1.84
7	28	44	2.33 *
8	27	32	2.09 *
9	26	11	2.20 *
10	94	82	1.34
11	67	72	-1.38
12	72	66	-1.66
13	57	62	-0.59

H	11	1	
0	48	52	1.57
1	73	65	2.76
2	94	98	-2.64
3	110	102	-0.19
4	64	68	-2.78
5	106	104	2.65
6	51	51	0.54
7	54	53	-3.03
8	61	67	-0.76
9	143	137	-0.62
10	93	89	0.60
11	73	81	2.45

H	12	1	
0	110	122	3.14
1	77	74	-2.53
2	22	45	-1.57 *
3	22	15	-2.45 *
4	21	40	0.38 *
5	66	69	0.66
6	19	25	0.09 *
7	100	104	2.28

H	0	2	
2	633	537	-3.14
3	510	394	-3.14
4	638	686	3.14

Table 4. Continued.

5	363	320	0.0
6	161	221	0.0
7	480	451	0.0
8	135	145	-0.00
9	63	68	-3.14
10	80	91	-3.14
11	338	347	0.0
12	69	74	-3.14
13	90	95	-0.00 *
14	133	141	-3.14
15	32	5	3.14 *
16	142	118	-0.00
17	90	95	0.0
18	60	54	-0.00
19	93	91	-0.00

H	1	2	
0	1277	1140	-1.57
1	748	641	0.98
2	447	322	-1.46
3	811	639	0.07
4	86	71	-1.27
5	455	326	0.96
6	518	421	-0.87
7	268	262	0.04
8	443	389	0.48
9	580	549	-2.07
10	362	361	-2.65
11	234	245	-0.43
12	350	379	-0.94
13	151	159	1.83
14	164	160	1.53
15	120	117	2.92
16	92	97	1.44
17	31	29	0.41 *
18	92	94	0.39
19	92	88	-2.04
20	68	63	2.23
21	66	77	-2.08

H	2	2	
0	271	328	0.0
1	1326	900	-3.04
2	826	685	2.18
3	500	414	2.11
4	472	428	0.75
5	508	461	-2.06
6	217	215	-0.98
7	479	412	-2.21
8	80	76	1.49
9	60	61	2.65
12	282	284	2.94
10	263	288	1.50
11	104	143	-2.63
13	262	275	1.54
14	140	130	1.50
15	320	291	-2.85
16	170	183	1.13
17	88	93	-2.06
18	28	39	3.03 *
19	82	93	1.01

H	3	2	
0	270	230	1.57
1	325	301	3.12
2	110	66	-2.83
3	184	199	1.17
4	126	130	0.37
5	372	355	0.23
6	490	428	0.95
7	153	167	-2.10
8	290	278	2.97
9	128	162	-0.13
10	60	117	2.72
11	192	212	2.74
12	152	172	-0.23
13	121	126	1.69
14	144	147	-2.06
15	101	91	1.20
16	99	95	0.59
17	61	69	-2.93
18	28	34	-0.35 *
19	72	74	0.14
20	73	82	-2.35

H	4	2	
0	649	531	3.14
1	295	257	1.09
2	417	378	0.29
3	308	313	2.95
4	295	300	1.67
5	226	223	-0.91
6	378	361	-1.61
7	328	327	-2.15
8	368	375	-0.81
9	345	365	2.39
10	266	278	-2.55
11	128	125	0.76
12	102	125	-1.89
13	144	208	2.67
14	66	75	-0.67
15	92	102	-2.27
16	10	34	2.75 *
17	77	68	-1.90
18	54	48	-1.54
19	23	23	-0.73 *
20	42	35	-1.43

H	5	2	
0	107	131	1.57
1	526	496	-2.17
2	241	234	-1.21
3	180	169	0.57
4	322	347	-0.94
5	282	287	2.58
6	170	188	-2.70
7	266	269	-0.70
8	68	81	-2.75
9	231	264	2.11
10	275	262	-2.79
11	115	132	-2.60
12	103	90	-0.06
13	122	136	-1.41
14	85	84	1.11
15	107	116	1.20
16	29	29	3.09 *
17	55	61	-1.16

H	6	2	
0	214	218	3.14
1	340	336	2.60
2	236	238	1.35
3	178	167	0.07
4	76	76	-3.04
5	55	56	-0.57
6	194	208	-2.73
7	200	215	-2.11
8	213	235	-0.93
9	233	229	-0.31
10	32	75	1.55 *
11	190	185	1.59
12	87	86	2.92
13	32	16	1.89 *
14	90	88	-0.46
15	78	77	-0.76
16	79	78	2.17

H	7	2	
1	47	51	-1.41
2	81	101	-2.76
3	89	98	-2.08
4	381	405	-0.57
5	513	490	-0.48
6	95	91	0.07
7	173	163	1.56
8	230	228	-2.98
9	103	89	2.01
10	67	60	-0.95
11	79	73	-2.10
12	110	122	2.55
13	31	31	-2.95 *
14	84	92	-0.03
15	27	47	-1.77 *
16	76	89	0.11
17	21	20	-2.85 *
18	72	84	-2.64

H	8	2	
0	168	162	0.0
1	156	155	0.06
2	183	187	1.12
3	87	83	-1.56
4	182	162	2.37
5	171	142	1.24
6	157	164	-1.50
7	176	162	-2.05
8	173	174	3.01
9	190	188	-2.64
10	32	41	1.36 *
11	152	153	1.53
12	133	133	-0.09

H	9	2	
0	188	157	1.57
1	65	63	1.27
2	101	102	2.94
3	111	111	-1.83
4	111	128	-0.10
5	102	96	-1.35
6	143	132	-2.17
7	130	141	0.65
8	31	58	-2.37 *
9	115	132	2.90
10	60	74	0.85
11	63	75	1.37
12	58	62	2.16
13	24	51	-0.21 *
14	60	65	0.17
15	64	80	-2.20

H	10	2	
0	228	203	0.0
1	31	29	-1.36 *
2	90	92	2.51
3	90	85	0.13
4	126	137	-1.69
5	30	16	-1.50 *
6	30	23	1.71 *
7	59	61	1.98
8	126	126	-3.10
9	78	76	-2.76
10	87	92	-0.02
11	47	49	2.39
12	55	57	-0.17
13	59	60	-0.49

H	11	2	
0	28	14	-1.57 *
1	89	91	0.90
2	88	78	0.35
3	87	87	-2.43
4	121	115	-2.72
5	70	79	-1.10
6	68	65	-0.18
7	109	120	1.69

H	12	2	
0	77	85	0.0
1	84	75	-1.09
2	22	11	-0.82 *
3	121	116	1.32
4	46	39	2.95
5	20	40	-1.30 *
6	57	67	-2.19

H	0	3	
2	486	504	-1.57
3	48	55	-1.57
4	36	35	1.57
5	317	222	1.57
6	794	690	-1.57
7	19	61	1.57 *
8	175	196	1.57
9	22	42	1.57 *
10	47	54	-1.57
11	106	120	-1.57
12	531	491	-1.57
13	85	95	-1.57
14	67	70	-1.57
15	27	52	1.57 *
16	167	146	-1.57
17	63	59	1.57
18	83	90	-1.57

H	1	3	
0	79	113	-1.57
1	160	216	0.29
2	617	694	-1.23
3	250	244	1.24
4	384	288	2.08
5	595	524	2.92
6	367	301	-2.32
7	216	206	-3.08
8	496	443	-0.70
9	228	226	0.23
10	155	177	0.37
11	54	58	2.36
12	122	111	-2.17
13	280	265	0.25
14	87	89	1.28
15	27	52	2.92 *
16	27	22	-1.06 *
17	115	110	3.09
18	98	105	1.86
19	62	55	0.28

H	2	3	
0	88	21	-0.00
1	562	537	3.12
2	412	366	1.93
3	192	196	2.28
4	704	689	-1.51
5	371	341	0.65
6	454	401	0.81
7	258	266	0.43
8	86	109	2.43
9	343	380	0.12
10	170	185	1.59
11	136	128	-0.71
12	71	75	-0.29
13	165	131	-1.53
14	80	83	2.64
15	134	123	-0.23
16	54	54	-1.64
17	25	48	1.87 *
18	23	13	-1.29 *
19	21	57	0.45 *
20	123	118	1.69
21	80	89	-0.43

H	3	3	
0	485	416	-1.57
1	694	616	0.24
2	246	188	0.00
3	218	202	1.02
4	204	214	1.96
5	350	340	0.26
6	120	141	1.72
7	93	108	0.25
8	83	92	-1.07
9	260	314	-1.75
10	66	66	0.71
11	179	165	-0.08
12	27	19	-0.10 *
13	95	124	1.69
14	232	203	-2.06
15	27	35	-1.36 *
16	165	176	1.86
17	71	90	2.67
18	22	30	-1.44 *
19	133	129	-3.05

H	4	3	
0	97	92	3.14
1	472	434	2.97
2	141	123	-2.28
3	159	172	2.38
4	128	116	2.17
5	256	271	-3.00
6	161	163	-1.32
7	76	87	-2.44
8	214	262	1.63
9	174	192	0.06
10	62	68	-1.78
11	94	104	2.80
12	135	139	1.95
13	161	166	-3.06
14	27	58	-2.40 *
15	100	107	-2.57
16	62	72	-0.68
17	23	54	2.56 *
18	72	74	1.35

H	5	3	
0	151	156	1.57
1	159	157	-1.73
2	148	168	-1.85
3	107	135	2.90
4	412	425	1.58
5	210	216	0.92
6	130	143	1.25
7	114	119	-1.69
8	70	76	0.72
9	92	99	-1.80
10	87	107	0.85
11	68	73	0.28
12	189	183	-1.46
13	28	70	2.81 *
14	27	15	0.46 *
15	26	18	0.67 *
16	24	33	-0.61 *
17	63	49	0.29

H	6	3	
0	361	349	3.14
1	172	195	-0.60
2	215	262	1.48
3	278	316	2.72
4	424	437	-1.19
5	100	105	-2.59
6	129	136	1.76
7	348	327	-0.12
8	93	92	-0.59
9	56	50	2.52
10	225	212	-1.53
11	28	22	0.95 *
12	28	55	-3.06 *
13	27	20	0.57 *
14	89	90	-1.43
15	76	80	2.97

H	7	3	
0	951	810	-1.57
1	109	121	3.04
2	259	247	1.24
3	226	221	1.18
4	201	197	1.83
5	130	123	1.27
6	144	157	-1.00
7	159	131	-0.93
8	124	126	1.48
9	57	60	2.76
10	173	162	-1.92
11	27	46	0.81 *
12	24	52	-1.59 *
13	123	122	-2.92
14	23	27	0.81 *
15	80	70	0.61

H	8	3	
0	248	236	3.14
1	199	167	2.33
2	61	52	-2.01
3	122	131	2.50
4	312	303	1.10
5	104	103	-0.83
6	135	151	-1.46
7	161	167	-0.63

H	9	3	
0	112	112	-1.57
1	95	90	3.00
2	27	42	-0.62 *
3	103	116	-0.27
4	193	180	0.74
5	94	91	-0.30
6	27	18	-2.32 *
7	100	89	-0.14
8	69	66	-2.96
9	25	69	0.70 *
10	24	43	2.38 *
11	142	135	3.04

H	10	3	
0	26	9	-0.00 *
1	117	117	0.41
2	26	7	2.22 *
3	91	79	-2.17

4	179	179	1.48
5	25	30	1.11 *
6	24	41	-2.65 *
7	24	16	-1.62 *
8	154	152	-1.86
9	21	43	-2.49 *
10	62	58	1.32
11	61	60	1.54
12	51	55	-1.25

H	11	3	
0	23	28	1.57 *
1	93	95	3.02
2	71	61	2.06
3	118	110	-0.11
4	58	58	-0.56
5	21	57	-3.09 *
6	20	17	2.06 *
7	51	57	0.59
8	46	46	-2.22

H	12	3	
0	17	17	-3.14 *
1	98	100	2.89
2	17	20	2.23 *
3	66	63	-0.28
4	48	50	1.15
5	67	74	0.05

H	0	4	
1	232	306	3.14
2	179	230	3.14
3	341	349	0.0
4	124	120	3.14
5	72	70	0.0
6	319	302	3.14
7	174	169	3.14
8	312	317	3.14
9	457	433	3.14
10	67	78	-0.00
11	310	296	3.14
12	144	141	0.0
13	241	239	0.0
14	108	105	3.14
15	26	10	3.14 *
16	121	110	0.0
17	23	14	3.14 *
18	43	50	0.0
19	63	58	-3.14

H	1	4	
0	204	247	1.57
1	237	298	1.86
2	155	199	2.15
3	199	216	-2.13
4	107	101	1.56
5	155	158	-2.70
6	111	113	-0.72
7	352	299	2.78
8	303	294	-0.27
9	169	171	-0.99
10	131	135	2.30
11	86	96	2.40
12	304	260	0.79
13	177	178	0.38
14	128	114	0.26
15	116	116	-1.49
16	102	106	-1.85
17	73	73	-3.05
18	79	68	-0.28
19	68	61	0.29

H	2	4	
0	92	108	0.0
1	526	558	-0.82
2	133	155	2.79
3	168	159	1.49
4	301	280	-0.48
5	370	336	-0.89
6	284	266	-1.89
7	76	69	-1.16
8	165	177	2.38
9	291	287	2.77
10	157	179	-0.20
11	222	193	-0.91
12	128	117	-1.91
13	177	178	0.94
14	76	60	-0.78
15	97	90	-0.17
16	84	91	-1.52
17	77	83	-2.96
18	92	94	1.67
19	45	46	2.00

H	3	4	
0	217	242	1.57
1	157	190	-0.81
2	48	88	-1.68
3	237	239	0.89
4	411	401	-2.49
5	464	438	2.84
6	284	269	-0.53
7	64	43	1.77
8	23	38	-2.12 *
9	194	182	2.24
10	137	116	-3.09

Table 4. Continued.

11	262	264	0.61
12	168	164	0.64
13	141	133	1.26
14	127	132	2.94
15	073	62	2.95
16	58	57	-1.82
17	22	32	-2.38 *
18	86	49	1.18

H 4 4

0	311	337	3.14
1	322	333	2.68
2	158	147	0.03
3	312	292	0.31
4	279	261	-1.67
5	121	107	0.15
6	22	9	2.68 *
7	145	151	2.83
8	286	278	1.23
9	161	186	0.78
10	219	185	2.48
11	142	137	-1.79
12	85	78	-0.51
13	100	88	0.74
14	26	12	0.24 *
15	77	74	-0.37
16	23	37	2.72 *
17	65	64	-1.53

H 5 4

0	176	178	1.57
1	401	388	-0.59
2	116	121	-2.76
3	51	29	-0.89
4	49	54	-1.18
5	77	86	2.31
6	217	206	2.07
7	315	314	-2.69
8	124	137	-1.38
9	177	172	0.38
10	173	157	2.69
11	132	126	0.10
12	100	107	1.49
13	97	89	-1.29
14	72	69	-1.74
15	61	44	2.27
16	86	84	2.65
17	82	77	-2.82

H 6 4

0	288	307	3.14
1	93	91	-0.77
2	127	117	-1.23
3	322	324	-2.71
4	118	115	-1.57
5	115	129	1.98
6	253	236	-2.92
7	76	84	0.39
8	263	221	1.34
9	131	105	-1.50
10	85	95	1.34
11	92	88	1.79
12	106	106	-2.92
13	85	79	-2.98
14	94	99	-0.73
15	43	47	-1.21
16	18	24	-1.91 *
17	68	73	2.83

H 7 4

0	103	89	-1.57
1	104	99	-1.64
2	364	332	3.13
3	96	84	1.37
4	190	186	0.72
5	139	127	-3.08
6	118	115	0.55
7	27	13	2.24 *
8	142	142	2.62
9	84	77	2.55
10	107	107	-0.24
11	87	103	-0.48
12	76	63	-1.68
13	22	13	-0.54 *
14	20	47	0.23 *

H 8 4

0	99	87	3.14
1	108	97	-1.86
2	108	96	1.55
3	56	55	-2.17
4	250	235	-1.65
5	193	184	2.27
6	108	101	0.62
7	139	136	-0.67
8	26	50	1.14 *
9	95	97	-0.45
10	24	33	-2.08 *
11	72	67	2.02
12	21	20	0.93 *
13	19	38	-1.13 *
14	53	55	-2.36
15	63	73	2.89

H 9 4

0	173	161	-1.57
1	89	94	0.88
2	106	104	2.93
3	88	88	2.92
4	114	105	-0.00
5	95	88	0.61
6	25	40	1.43 *
7	108	98	2.81
8	114	103	2.13
9	60	60	-1.30
10	56	50	-1.89
11	61	51	1.44
12	90	84	-1.97

H 10 4

0	146	128	0.0
1	24	37	0.97 *
2	88	87	-2.89
3	124	116	-3.06
4	23	15	-2.86 *
5	46	43	-2.64
6	75	67	-0.33
7	78	68	0.64
8	87	93	2.65
9	67	76	-1.03
10	46	44	0.02
11	62	60	0.55

H 11 4

0	105	102	-1.57
1	104	91	2.17
2	56	49	-1.11
3	65	62	-0.12
4	90	95	2.25
5	85	76	-1.96
6	56	66	0.72
7	78	80	1.02
8	59	62	2.18

H 0 5

1	149	171	1.57
2	12	17	-1.57 *
3	93	106	-1.57
4	368	369	1.57
5	700	620	1.57
7	200	192	-1.57
8	516	471	-1.57
9	92	98	-1.57
10	153	117	1.57
11	28	42	1.57 *
12	98	98	-1.57
13	28	23	-1.57 *
14	88	89	1.57
15	27	4	1.57 *
16	157	134	1.57
17	23	6	-1.57 *
18	53	46	-1.57

H 1 5

0	156	171	1.57
1	212	275	1.92
2	94	110	-2.82
3	250	238	-1.24
4	60	62	-1.88
5	173	126	-1.25
6	81	79	1.87
7	222	226	-0.21
8	236	219	-1.70
9	304	279	-1.31
10	235	201	2.60
11	353	299	3.04
12	232	200	2.22
14	105	98	-1.31
15	77	75	-1.21
16	56	55	2.34
17	78	73	-1.98
18	73	62	-2.63

H 2 5

0	30	28	3.14
1	291	338	-2.37
2	95	108	2.95
3	166	165	-0.51
4	364	368	2.15
5	223	198	-0.85
6	196	165	-1.66
7	230	230	2.35
8	145	151	-2.46
9	184	180	-1.69
10	75	70	-2.91
11	90	81	-2.81
12	29	45	-0.52 *
13	28	44	1.61 *
14	80	74	1.18
15	117	103	-0.27
16	70	66	0.35
17	69	58	2.20

H 3 5

0	319	323	1.57
1	57	73	-2.94
2	65	64	-1.34
3	227	220	1.99
4	125	134	1.41
5	145	151	-0.68
6	119	138	-1.57
7	137	148	0.43
8	209	206	-0.91
9	87	127	-2.26
10	195	179	1.10
11	173	152	-2.31
12	108	104	0.75
13	184	170	1.46
14	55	47	-0.44
15	81	59	-1.62
16	74	53	-0.95

H 4 5

0	211	234	0.0
1	56	58	-2.60
2	235	252	1.49
3	249	249	-0.62
4	187	196	-1.23
5	161	157	-0.13
6	150	127	0.50
7	201	186	-2.29
8	88	93	-1.43
9	91	86	2.80
10	155	142	-3.69
11	71	57	-1.91
12	28	50	2.11 *
13	62	67	1.51
14	83	81	-2.65
15	91	76	2.20
16	22	30	-0.06 *
17	59	49	-1.31

H 5 5

0	231	231	1.57
1	131	143	0.67
2	67	67	1.87
3	233	234	-2.98
4	188	198	-2.43
5	190	193	0.45
6	164	163	-0.43
7	28	41	1.41 *
8	180	181	-1.94
9	182	174	3.08
10	101	115	2.83
11	172	154	0.57
12	235	211	1.66
13	99	85	-2.91
14	65	72	-1.99
15	118	98	1.63
16	87	72	-2.35
17	44	45	0.12

H 6 5

0	99	114	3.14
1	76	65	-0.59
2	110	121	0.29
3	299	264	0.96
4	189	191	-1.64
5	89	92	-0.91
6	83	67	-2.41
7	77	55	1.94
8	206	197	1.26
9	119	127	-2.52
10	90	102	-2.01
11	27	66	2.93 *
12	64	61	2.18
13	70	76	0.49
14	49	43	-1.04
15	94	105	-0.72

H 7 5

1	126	135	-0.92
2	63	58	2.20
3	63	67	1.00
4	98	107	2.47
5	83	86	-2.49
6	128	146	-1.48
7	98	107	-2.06
8	28	28	-0.18 *
9	28	29	1.26 *
10	84	94	2.77
11	87	92	-0.89
12	63	52	0.86
13	21	31	0.89 *
14	53	47	1.73

H 8 5

0	82	95	3.14
1	137	157	0.03
2	28	70	-1.99 *
3	89	84	-1.75
4	75	61	-0.45
5	28	20	-2.97 *
6	27	42	-3.12 *
7	111	98	-0.63
8	90	87	0.58
9	113	109	2.63
10	89	97	2.06
11	22	31	2.82 *
12	80	88	-2.62
13	56	58	-0.47

H 9 5

0	92	93	-1.57
1	77	83	0.85
2	118	124	1.98
3	26	39	-1.16 *
4	63	68	0.14
5	80	83	-3.14
6	66	66	-2.18
7	24	32	2.59 *
9	21	25	-3.13 *
10	85	91	-0.21
11	43	49	-0.12

H 10 5

1	67	70	0.29
2	86	93	-1.24
3	22	45	-1.23 *
4	22	37	-1.73 *
5	21	58	-2.59 *
6	20	53	-1.35 *
7	19	49	0.43 *
8	57	74	1.40

H 0 6

2	245	226	3.14
3	218	219	3.14
4	132	129	0.0
5	110	101	3.14
6	200	201	0.0
7	341	277	0.0
8	109	101	3.14
9	69	75	3.14
10	209	204	0.0
11	83	88	3.14
13	69	73	-0.00
14	53	64	3.14
15	127	107	3.14

H 1 6

1	81	95	2.93
2	191	254	0.22
3	84	101	-1.63
4	419	447	2.30
5	56	61	1.96
6	124	118	0.46
7	133	135	1.67
8	87	81	2.29
10	131	134	-2.64
11	72	67	2.34
12	115	116	-2.21
15	78	64	-0.67
16	44	36	-2.52

H 2 6

1	61	76	1.87
2	148	155	0.04
4	217	245	2.18
5	83	73	2.72
6	148	149	-2.31
7	174	142	0.49
8	185	169	-1.64
9	130	116	0.74
10	59	54	-2.00
11	132	138	-1.18
12	141	135	-0.04

H 3 6

0	90	112	-1.57
1	163	156	-2.99
2	96	97	-1.56
3	245	241	-0.56
4	149	158	2.93
5	115	124	-2.52
6	66	82	2.82
7	97	91	-0.39
8	243	249	-0.06
9	132	111	-0.06
10	145	139	2.89
11	137	120	-2.69
12	98	77	-0.50

H 4 6

0	76	89	0.0
1	176	199	-2.02
2	132	144	3.11
4	296	289	1.56
5	86	88	-0.91
7	82	81	1.84
8	59	70	-1.70
9	103	101	0.20
11	135	120	-1.51
14	130	92	1.07
16	143	117	2.07

H 5 6

0	65	65	1.57
1	197	196	-1.99
2	54	72	1.21
3	78	74	1.20
4	125	116	2.46
5	128	124	1.06
6	101	124	0.28
7	72	67	-0.80
8	105	109	-1.75
10	57	69	0.46
12	90	75	1.66
13	126	107	-3.10
14	80	65	-1.25

H 6 6

0	138	131	3.14
1	70	87	2.75
2	202	199	0.32
3	115	147	-1.76
5	171	160	1.76
8	141	133	-0.53
9	56	67	-1.45
10	121	109	-2.06

H 7 6

0	130	122	1.57
1	115	120	-2.42
2	152	132	-3.33
4	70	82	-0.36
5	127	133	0.79
6	80	92	1.57
7	55	68	-1.44
8	132	117	-0.96
10	68	61	1.48
11	54	56	2.14
12	87	88	2.33
13	38	46	-2.87

H 8 6

2	67	83	2.23
3	67	69	-0.28
4	66	77	-0.49
5	75	83	1.05
6	52	60	1.26
7	93	83	2.27
10	48	54	-1.14

H 9 6

0	49	61	1.57
4	105	98	-2.29
5	91	83	-0.54
6	87	99	-1.11

H 10 6

0	55	54	3.14
1	94	80	0.16
3	72	76	-0.40

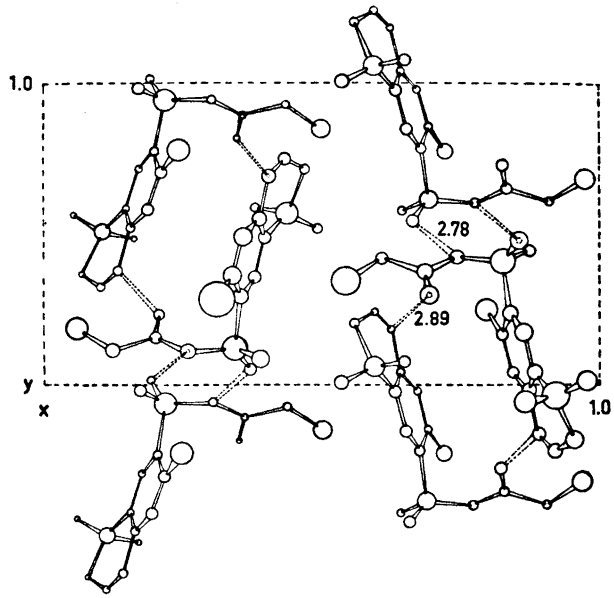


Fig. 3. Molecular packing. The structure is viewed along the *c*-axis. Hydrogen bonds are indicated by broken lines.

Table 5. Bond distances and angles with their standard deviations.

Cl_1-C_6	1.757(15) Å	N_1-C_7	1.241(16) Å
Cl_2-C_9	1.715(5)	N_2-C_4	1.405(16)
S_1-O_1	1.431(9)	N_2-C_7	1.319(17)
S_1-O_2	1.394(9)	N_3-C_8	1.385(16)
S_1-N_3	1.657(10)	C_1-C_2	1.421(17)
S_1-C_1	1.760(10)	C_1-C_6	1.344(18)
S_2-O_3	1.396(17)	C_2-C_3	1.403(14)
S_2-O_4	1.365(15)	C_3-C_4	1.406(18)
S_2-N_1	1.620(14)	C_4-C_5	1.418(17)
S_2-C_3	1.740(18)	C_5-C_6	1.379(16)
O_5-C_8	1.204(15)	C_8-C_9	1.497(12)
$N_3-S_1-C_1$	105.6(0.5)°	$S_1-C_1-C_2$	114.5(0.9)°
$N_3-S_1-O_1$	104.9(0.6)	$S_1-C_1-C_6$	125.2(1.0)
$N_3-S_1-O_2$	108.1(0.6)	$C_6-C_1-C_2$	120.3(1.0)
$C_1-S_1-O_1$	109.4(0.5)	$C_1-C_2-C_3$	117.9(1.1)
$C_1-S_1-O_2$	108.9(0.6)	$C_2-C_3-C_4$	119.8(1.1)
$O_1-S_1-O_2$	119.1(0.5)	$C_3-C_4-C_5$	121.4(1.0)
$N_1-S_2-C_3$	103.8(0.9)	$C_2-C_5-C_6$	116.2(1.1)
$N_1-S_2-O_3$	108.4(0.9)	$C_5-C_6-C_1$	124.3(1.3)
$N_1-S_2-O_4$	107.8(0.8)	$C_2-C_3-S_2$	120.0(1.1)
$C_3-S_2-O_3$	109.8(0.9)	$C_4-C_3-S_2$	120.2(0.9)
$C_3-S_2-O_4$	109.1(0.9)	$S_2-N_1-C_7$	124.4(1.0)
$O_3-S_2-O_4$	117.1(1.1)	$N_1-C_7-N_2$	127.4(1.1)
$S_1-N_3-C_8$	122.3(0.9)	$C_7-N_2-C_4$	124.0(1.2)
$N_3-C_8-C_9$	113.0(0.9)	$N_2-C_4-C_3$	119.0(1.1)
$N_3-C_8-O_5$	120.8(1.1)	$N_2-C_4-C_5$	119.6(1.1)
$C_9-C_8-O_5$	125.8(1.0)	$Cl_1-C_6-C_1$	121.5(1.0)
$Cl_2-C_9-C_8$	112.9(0.5)	$Cl_1-C_6-C_5$	114.1(1.0)

Table 6. Intermolecular distances less than 4 Å. Standard deviations are given in parentheses. The symmetry operation required to generate atom 2 from the coordinates listed in Table 3 is given before each distance.

Cl_1-O_4	$\frac{1}{2}-x,-y,z-\frac{1}{2}$	3.661(10) Å	O_4-C_5	$\frac{1}{2}-x,-y,\frac{1}{2}+z$	3.505(15) Å
Cl_1-O_2	$\frac{1}{2}-x,1-y,z+\frac{1}{2}$	3.903(10)	O_4-C_6	—»—	3.294(18)
Cl_1-N_2	$\frac{1}{2}-x,-y,z-\frac{1}{2}$	3.537(15)	O_4-C_8	—»—	3.275(15)
Cl_1-N_3	$\frac{1}{2}-x,1-y,z+\frac{1}{2}$	3.712(11)	O_4-C_9	—»—	3.687(9)
Cl_1-C_4	$\frac{1}{2}-x,-y,z-\frac{1}{2}$	3.730(12)	O_1-O_2	$\frac{1}{2}-x,1-y,z-\frac{1}{2}$	3.738(11)
Cl_1-C_7	$\frac{1}{2}-x,-y,z-\frac{1}{2}$	3.744(12)	O_1-O_5	—»—	2.967(12)
Cl_2-O_4	$\frac{1}{2}-x,-y,z-\frac{1}{2}$	3.861(9)	O_1-N_2	$x,1+y,z$	3.749(14)
Cl_2-O_1	$\frac{1}{2}-x,1-y,z+\frac{1}{2}$	3.479(9)	O_1-N_3	$\frac{1}{2}-x,1-y,z-\frac{1}{2}$	3.642(15)
Cl_2-N_2	$\frac{1}{2}-x,-y,z-\frac{1}{2}$	3.380(14)	O_1-C_7	$x,1+y,z$	3.574(13)
Cl_2-N_1	$\frac{1}{2}-x,-y,z-\frac{1}{2}$	3.684(12)	O_1-C_8	$\frac{1}{2}-x,1-y,z-\frac{1}{2}$	2.934(15)
Cl_2-C_1	$\frac{1}{2}+x,\frac{1}{2}-y,-z$	3.561(11)	O_1-C_9	$\frac{1}{2}-x,1-y,z-\frac{1}{2}$	3.137(10)
Cl_2-C_2	—»—	3.610(11)	O_2-C_8	$\frac{1}{2}-x,1-y,z+\frac{1}{2}$	3.684(15)
Cl_2-C_3	—»—	3.637(13)	O_2-C_9	—»—	3.446(9)
Cl_2-C_4	—»—	3.558(11)	O_5-C_7	$\frac{1}{2}-x,-y,z+\frac{1}{2}$	3.674(15)
Cl_2-C_5	—»—	3.517(12)	N_2-C_8	$\frac{1}{2}-x,-y,z-\frac{1}{2}$	3.752(18)
Cl_2-C_6	—»—	3.521(16)	N_1-C_2	$1-x,y+\frac{1}{2},\frac{1}{2}-z$	3.925(14)
Cl_2-C_7	$\frac{1}{2}-x,-y,z-\frac{1}{2}$	3.697(12)	N_1-C_8	$\frac{1}{2}-x,-y,z+\frac{1}{2}$	3.804(16)
S_1-O_3	$1-x,y+\frac{1}{2},\frac{1}{2}-z$	3.880(13)	N_1-C_9	$\frac{1}{2}-x,-y,z+\frac{1}{2}$	3.340(11)
O_3-O_1	$1-x,y-\frac{1}{2},\frac{1}{2}-z$	3.542(15)	C_7-C_9	$\frac{1}{2}-x,-y,z-\frac{1}{2}$	3.998(12)
O_3-O_2	$1-x,y-\frac{1}{2},\frac{1}{2}-z$	3.469(15)	Hydrogen bonds:		
O_3-N_1	$1-x,y+\frac{1}{2},\frac{1}{2}-z$	3.397(16)			
O_3-C_9	$\frac{1}{2}+x,\frac{1}{2}-y,+z$	3.810(12)	$O_5\cdots N_2$	$\frac{1}{2}-x,-y,\frac{1}{2}+z$	2.888(15)
O_4-O_5	$\frac{1}{2}-x,-y,\frac{1}{2}+z$	3.138(13)	$O_2\cdots N_3$	$\frac{1}{2}-x,1-y,\frac{1}{2}+z$	2.778(17)
O_4-N_3	—»—	3.863(12)	$N_2\cdots O_5$	$\frac{1}{2}-x,-y,z-\frac{1}{2}$	2.888(15)
O_4-C_1	—»—	3.745(14)	$N_3\cdots O_2$	$\frac{1}{2}-x,1-y,z-\frac{1}{2}$	2.778(17)

sheets thus being formed perpendicular to the x axis. These sheets are held together by van der Waals forces only.

The two independent hydrogen bonds, $N_2-H\cdots O_5$ and $N_3-H\cdots O_2$ with N – O distances of 2.89 and 2.78 Å, respectively, are fairly short compared with the corresponding distances given by Hamilton (2.97 – 3.03 Å) [14] and those found in other sulfonamides (2.85 – 3.24 Å).[15]

Bond distances and angles within the molecule are listed in Table 5, and intermolecular distances less than 4 Å are given in Table 6. The planes defined by certain groups of atoms are specified in Table 7, from which it may be seen that the two ring systems within the molecule are inclined at an angle of 174°.

Characteristic values of bond lengths in sulfonamide groups have been reported by Cotton and Stokely.[15] By comparing five recent investigations they found the S – O, S – N, and S – C distances to lie in the ranges 1.386 – 1.47, 1.61 – 1.666, and 1.74 – 1.763 Å, respectively. The corresponding distances obtained in this investigation for two different sulfonamide groups agree well with these values, although S_2-O_4 is slightly, but not significantly, shorter than the S – O distances cited. An S – N bond of about 1.65 Å has considerable double bond character,[16] involving π-bonds from the lone pair of the nitrogen atom. In the chloroacetyl group connected to N_3, the N_3-C_8 distance (1.39 Å) is therefore significantly longer than in other chloroacetyl ammides, owing to the decrease of the N_3 lone pair contribution to the C – N bond. In DL-*N*-chloroacetylalanine, for instance, the C – N bond has the usual peptide value

Table 7. Best least-squares planes. The equations are given in Cartesian coordinates.

Plane 1 (defined by C_1-C_6)

$$0.9336x+0.2193y-0.2834z=14.18$$

Atom	Deviations
C_1	0.016 Å
C_2	−0.005
C_3	−0.005
C_4	0.005
C_5	0.005
C_6	−0.016
Cl_1	0.038
S_1	0.072

Plane 2 (defined by C_3,S_2,N_1,C_7,N_2,C_4)

$$0.8983x+0.3161y-0.3051z=13.60$$

Atom	Deviations
C_3	0.040 Å
S_2	−0.072
N_1	0.064
C_7	0.000
N_2	−0.051
C_4	0.018

Plane 3 (defined by C_8,C_9,O_5,N_3)

$$-0.0140x-0.0748y-0.6639z=-3.391$$

Atom	Deviations
C_8	−0.049 Å
C_9	−0.084
O_5	0.038
N_3	0.095
S_1	0.040

Angles:		
	Plane 1 − Plane 2	6.04°
	Plane 1 − Plane 3	89.36°
	Plane 2 − Plane 3	92.66°

of 1.32 Å.[17] In this compound the Cl − C bond (1.78 Å) also differs significantly from that found in the present study, Cl_2-C_9 being only 1.715 Å. This difference is probably due to a larger transfer of negative charge from C_9 towards the weakened C_8-N_3 bond.

In the ring system the distance between N_1 and C_7 is short (1.24 Å),[16] corresponding to a strong double bond, the double bond character also leading to the proposed positions of the hydrogen bonds. All bonds not specifically mentioned have distances within the normal range.

A discussion of the interaction between *N*-chloroacetylchlorothiazide and the active site of carbonic anhydrase B is given elsewhere.[18]

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