In all the examples which follow, 'sharpened' $|F|^2$ values were used in the calculation of the Q-functions. $Q(X_0Z_0)$ was calculated using the relative coordinates of the eleven atoms in the plane of the purine residue in deoxyadenosine. The relative coordinates were those obtained from the determination of the molecular orientation (Watson, Sutor & Tollin, 1965). The space group is $P2_1$. The map obtained is shown in Fig. 1(*a*). The expected positions of peaks due to atoms separated by $b/2$ are marked in the map. The largest remaining peak is that which determines the position of the origin. The map of $Q(X_0Z_0)$ obtained using the relative coordinates for the atoms of the sugar residue in deoxyadenosine is shown in Fig. 1(*b*). In this case only eight out of a total of twenty heavy atoms in the molecules were used. The coordinates used were the final coordinates from the refined structure with an arbitrary change of origin to $x=0{\cdot}2$, $z=0{\cdot}3$. In this case there are no atoms separated by half in their fractional y coordinates.

The function $Q(Y_0)$ was calculated from relative coordinates for pyrimidine obtained by taking the final published coordinates (Wheatley, 1960) and giving them an arbitrary shift of origin to $x=-0{\cdot}15$, $y=-0{\cdot}3$. The space group is $Pna2_1$. $Q(Y_0)$ was used to define the position of the molecule relative to the a-glide plane. Only the fifty largest 'sharpened' $|F|^2$ values were used. The resulting map is shown in Fig. 2 with the origin shifted to $y=-\frac{1}{4}$ to allow for the fact that the a-glide occurs at $y=\frac{1}{4}$. The dotted vertical line represented the correct answer of $y=-0{\cdot}3$. The results show that even with this small amount of data the origin position is well defined. It can also be seen that the determination of the y coordinate is independent of the fact that at this stage the x coordinate of the arbitrary origin is not known. In all these examples the error in determining the origin position was less than 0·05 Å.

These Q-functions have also been used to determine the structure of 4-acetyl-2′-fluorobiphenyl (Tollin, Young & Sutherland, 1965).

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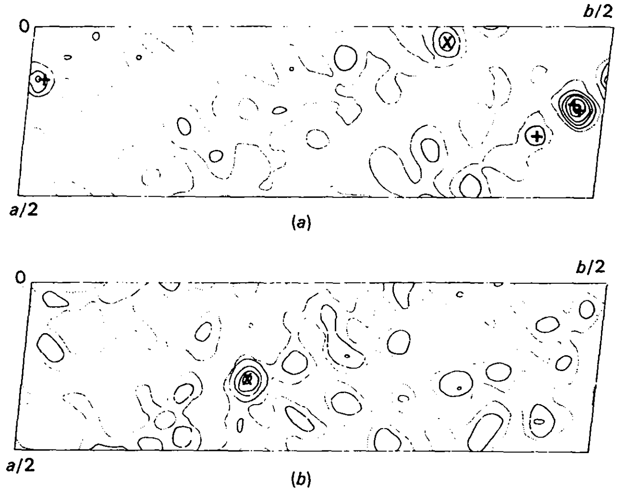

Fig. 1. $Q(X_0Z_0)$ for deoxyadenosine (*a*) using purine relative coordinates (*b*) using sugar relative coordinates. + indicates false peaks. × indicates expected positions.

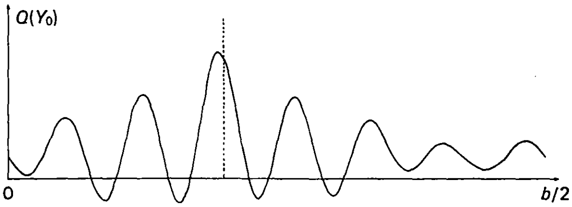

Fig. 2. $Q(Y_0)$ for pyrimidine. Dotted line indicates expected peak position.

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The crystal structure of bis(*N*-2-hydroxyethylsalicylaldiminato)copper (II).

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There is considerable current interest in the crystal structures of *N*-substituted salicylaldiminato complexes of transition metals (see Table 5, Frasson, Panattoni & Sacconi, 1964; Fox, Lingafelter, Orioli & Sacconi, 1963; Wei, Stogsdill & Lingafelter, 1964; Cheeseman, Hall & Waters, 1965). Attention has been mainly focused on the dimensions and stereochemistry of the molecule, as in all but the simplest such molecules there appears to be little molecular interaction. The substituents in compounds studied to date have been alkyl or phenyl groups, and it is of interest to consider the influence on such structures of groups with hydrogen-bonding potential. In this connection studies of the crystal structure of bis(*N*-2-hydroxyethylsalicylaldiminato)copper(II) were begun both at Providence and Auckland, the results from one of these being announced at the Rome Congress (Boyko, 1963). It transpired that the two investigations were similar in scope and achievement, and a joint publication has been decided upon.

Experimental

The compound was prepared by warming stoichiometric quantities of bissalicylaldehydatocopper(II) and ethanolamine in 50% aqueous methanol, and recrystallized from ethanol as dark green needles, m.p. 176°. Chemical analysis verified the resulting composition. The constants for the monoclinic unit cell were obtained from NaCl-calibrated Weissenberg photographs around **b** and **c** as $a=18{\cdot}66\pm0{\cdot}04$, $b=4{\cdot}71\pm0{\cdot}01$, $c=19{\cdot}99\pm0{\cdot}04$ Å, $\beta=97{\cdot}8\pm0{\cdot}1°$. The density was measured by a gradient density procedure as 1·48 g.cm^{-3}, as compared with the calculated density of

Table 1. *Atom coordinates and temperature factors*

Atom in molecule *A*	*x*	*y*	*z*	*B*	Atom in molecule *B*	*x*	*y*	*z*	*B*
Cu	0	0	0	4·5 Å^2	Cu	$\frac{1}{2}$	$\frac{1}{2}$	0	4·0 Å^2
O(1)	−0·0049	−0·1437	0·0863	3·8	O(1)	0·5732	0·5245	0·0761	3·4
N	−0·0885	0·2259	0·0066	4·3	N	0·4513	0·2052	0·0483	4·5
C(1)	−0·1301	0·2196	0·0532	3·8	C(1)	0·4766	0·1061	0·1086	3·0
C(2)	−0·1187	0·2596	0·1094	3·7	C(2)	0·4577	0·8014	−0·1517	3·9
C(3)	−0·0588	−0·1357	−0·1261	4·7	C(3)	0·4130	0·5886	−0·1358	3·3
C(4)	−0·0491	−0·3195	0·1841	5·2	C(4)	0·3478	0·5001	−0·1791	4·5
C(5)	−0·1058	−0·3223	−0·2250	4·9	C(5)	0·3299	0·6501	−0·2436	4·5
C(6)	−0·1675	−0·1508	0·2121	5·4	C(6)	0·3750	0·8516	−0·2626	4·5
C(7)	−0·1723	0·0281	0·1572	4·4	C(7)	0·4397	0·9318	−0·2173	4·1
C(8)	−0·1117	0·4511	−0·0472	4·3	C(8)	0·3854	0·0479	0·0743	3·7
C(9)	−0·1597	0·2882	−0·1104	3·1	C(9)	0·3154	0·2460	0·0110	3·7
O(2)	−0·2320	0·2293	−0·0930	3·8	O(2)	0·2970	0·2767	0·0801	4·2

Estimated standard errors

	x	*y*	*z*	*B*(Å^2)
Cu	—	—	—	0·2 Å^2
O	0·015 Å	0·020 Å	0·014 Å	0·4
N	0·018	0·024	0·017	0·4
C	0·021	0·031	0·020	0·55

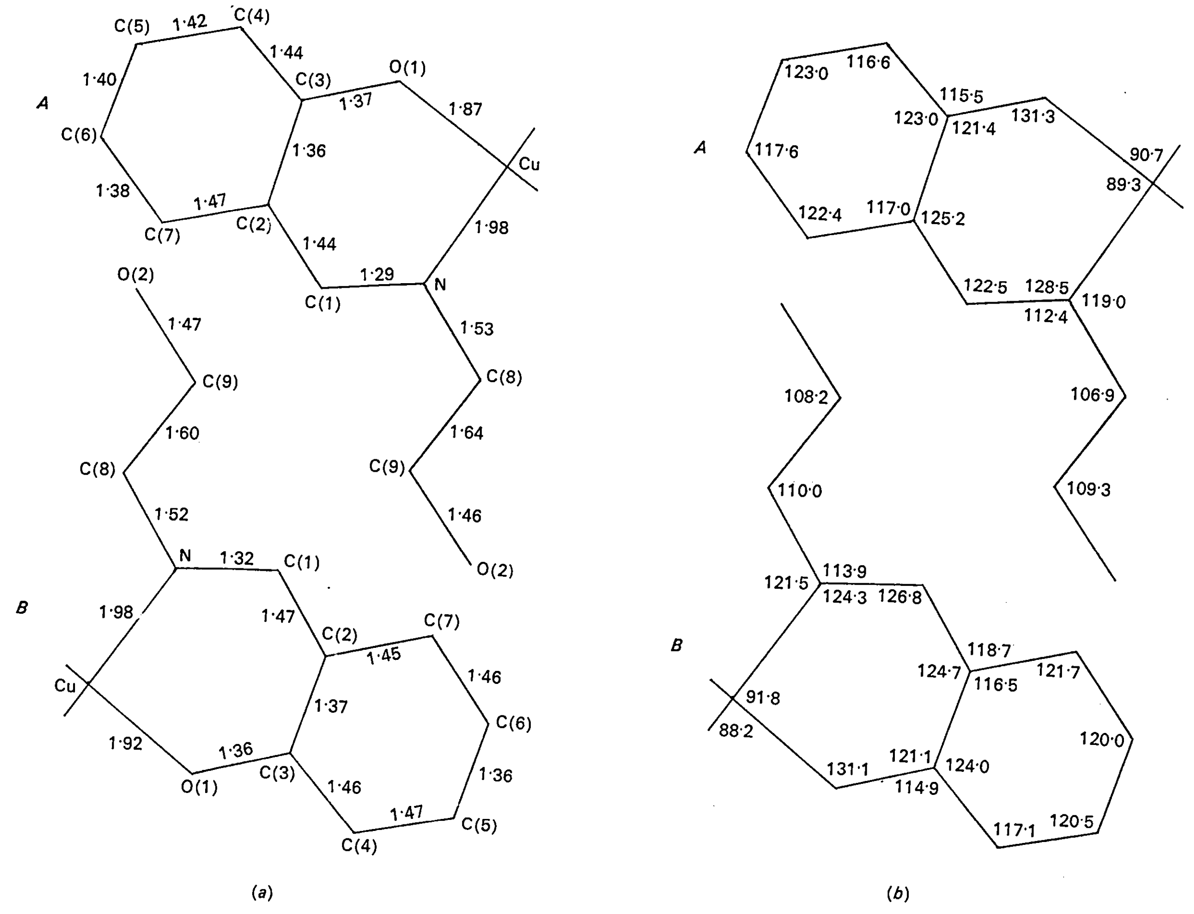

Fig. 1. (*a*) Bond lengths (Å). (*b*) Bond angles (°).

1·495 g.cm^{-3} for 4 molecules per unit cell. Systematic absences indicated the space group $P2_1/c$.

Intensities were measured visually from Weissenberg photographs of the *h0l*, *h1l* and *h2l* layers. The approximate dimensions of the crystal employed were 0·1 × 0·4 × 2 mm. No correction for absorption was made. The shape of the crystals precluded satisfactory photography other than about **b**, and the interlayer scaling constants were regarded as additional parameters during refinement. A comparison of the 600 observed structure factors from the two independent determinations showed no serious discrepancies and only one set (Providence) was employed in the refinement.

Structure determination

The Patterson function showed dominant maxima at positions $(\frac{1}{2},\frac{1}{2},0)$. $(\frac{1}{2},0,\frac{1}{2})$ and $(0,\frac{1}{2},\frac{1}{2})$, whence it was deduced that the copper atoms either occupy general positions at ($\sim$ 0·25, $\sim$ 0·00, $\sim$ 0·25) or the two independent sets of special positions at (0,0,0) and $(\frac{1}{2},\frac{1}{2},0)$. Heavy atom phased Fourier syntheses could only be interpreted on the latter assumption, *i.e.* that two independent centric molecules exist in the structure. Location of the light atoms and subsequent refinement followed conventionally, although the details of procedure in the two investigations differed somewhat. The final refinement was by a least-squares procedure in which atoms were permitted individual isotropic temperature factors. The atomic form factors employed were from Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) and the weighting factors used were $w=1$ for $F_o \le 30$, $w=30/F_o$ for $F_o > 30$. Further refinement was attempted with anisotropic temperature factors, but little improvement resulted and the isotropic refinement was preferred for its smaller number of parameters. The overall reliability index (observed structure factors only) is 0·090; individual values for separate layers are 0·091, 0·087 and 0·095 for *h0l*, *h1l*, and *h2l* respectively. Atom coordinates and temperature factors are listed in Table 1, together with the estimated standard errors calculated from the least-squares residuals.

Discussion

The bond lengths and angles in the two independent centric molecules are shown in Fig. 1. In general they are similar, although the difference in the length of the bonds C(6)–C(7), 0·079 Å, and of Cu–O, 0·053 Å, would appear significant by normal criteria. It is unlikely that this is true, and more probable that the standard errors in Table 1 are underestimates. The excessively long values for both of the bonds C(8)–C(9) would support this conclusion. No further discussion of the individual dimensions is then profitable, other than to note the general similarity to those of the chemically related molecules bis-(*N*-methylsalicylaldiminato)copper(II) (Lingafelter, Simmons, Morosin, Scheringer & Freiburg, 1961) and bis-(*N*-phenylsalicylaldiminato)-copper(II) (Wei *et al.*, 1964).

The two molecules in this study do, however, appear to differ meaningfully with respect to planarity. The mean planes (Schomaker, Waser, Marsh & Bergman, 1959) through the molecules (other than the side chains) and the mean planes through a benzene ring were calculated. For molecule *A* the approximation to overall planarity is poor; the benzene ring is effectively planar, however, and with the exception of the nitrogen (deviation 0·125 Å), the remaining atoms of the salicylaldimine group are coplanar with it. The molecule is bent, or stepped, so that the planes of the two benzene rings are separated by 0·62 Å, this situation being similar to that observed in bis-(*N*-phenylsalicylaldiminato)copper(II). On the other hand molecule *B* can, with the sole exception of the oxygen atoms (deviation 0·092 Å), be described as coplanar within the accuracy

Table 2. *Observed and calculated structure factors*

	h00	
2	1399	1828
3	216	-244
5	216	-217
6	927	842
7	565	509
8	537	506
9	506	459
10	606	557
12	562	487
13	250	196
14	322	261
16	404	343
18	406	344
	h0 10	
0	840	901
1	371	347
2	816	795
4	1031	934
6	318	326
7	192	-221
8	409	434
9	221	-252
10	500	496
12	392	379
14	278	248
-1	223	240
-2	223	242
-3	353	268
-4	717	697
-5	218	-179
-6	800	846
-8	662	684
-9	184	142
-10	522	521
-12	600	535
-14	362	353
-16	305	247
	h1 0	
2	286	-291
4	523	518
5	335	362
6	182	-168
7	642	591
8	321	301
9	195	-218
10	140	-117
11	161	194
	h0 4	
0	400	357
1	333	240
2	85	44
3	290	-256
4	925	-856
5	633	-590
7	105	81
8	113	-91
9	138	-138
10	137	132
12	276	-291
-1	193	-151
-2	187	-138
-3	239	272
-4	328	285
-5	482	-482
-6	205	196
-8	268	-291
-9	324	-316
-10	127	-142
	h1 8	
0	776	704
1	98	-96
2	245	223
3	458	-401
5	134	159
9	262	249
11	165	154
-1	390	400
-5	445	436
-7	171	193
-8	173	202
-9	124	110
-10	197	-185
	h1 13	
1	444	460
3	367	371
7	187	231
11	299	277
-1	724	679
-3	501	512
-5	484	490
-7	460	448
-9	363	382
-11	362	353
-13	233	270
	h2 2	
0	365	444
1	547	560
2	694	730
3	204	-163
4	254	310
5	131	152
6	467	449
8	758	788
9	187	-260
12	300	292
-1	610	-548
-2	1422	1102
-3	148	107
-4	828	764
-5	256	240
-6	527	591
-8	658	633
-12	355	294
-16	401	344
	h2 7	
0	216	232
1	[illegible]	252
3	368	372
5	184	202
-2	248	206
-5	156	-147
-7	171	190
-8	200	-215
-9	307	257
	h2 13	
-4	195	-201
-9	254	-283
	h02	
0	1605	1565
2	1252	1392
3	1204	1222
4	862	1050
5	304	-303
6	603	649
7	515	420
8	1113	1097
10	1126	1123
11	290	-326
12	662	651
13	201	148
16	378	324
18	208	178
-2	873	982
-3	1282	-1397
-4	1138	1296
-5	450	-399
-6	194	295
-7	262	280
-8	706	654
-9	381	373
-10	821	767
-11	173	178
-13	248	248
-14	736	690
-16	418	325
	h0 12	
0	806	782
1	368	-321
2	337	377
3	357	387
4	1037	941
6	586	610
7	201	-151
8	288	283
10	318	372
12	522	414
-1	170	172
-2	229	249
-3	494	536
-4	250	303
-6	450	451
-8	413	491
-10	616	611
-12	272	286
-13	276	292
-14	366	360
	h1 5	
0	100	-99
1	273	361
2	228	-209
3	648	654
5	1039	973
6	297	-236
7	715	696
8	320	-326
9	394	424
11	576	503
13	386	341
15	217	194
-1	364	436
-3	497	585
-4	305	-342
-5	1158	1243
-6	309	-312
-7	1105	1204
-8	299	-319
-9	240	318
-11	342	363
-13	454	445
	h1 9	
0	425	373
1	707	646
2	263	273
3	703	697
5	522	512
7	523	479
9	566	502
11	238	203
-1	608	567
-3	603	564
-5	373	434
-6	167	-153
-7	494	498
-9	481	555
-11	412	458
-13	279	315
-15	218	267
	h1 14	
1	143	157
5	370	-357
-1	151	-179
-3	229	272
	h2 3	
1	270	-310
2	152	-149
3	414	-413
4	166	-193
5	120	-140
8	167	-211
-1	339	-283
-3	557	-510
-4	133	-158
-5	386	-317
-6	237	224
	h2 8	
0	521	502
1	275	-256
2	557	566
4	674	650
6	430	482
7	227	266
8	442	459
10	243	221
-1	189	214
-2	576	632
-4	599	617
-5	168	206
-6	344	362
-8	460	536
-10	444	496
	h2 14	
0	286	271
2	352	391
4	396	349
-1	216	-219
-2	274	291
-4	286	305
-5	203	266
-12	323	313
	h04	
0	822	772
2	661	818
3	516	450
5	604	-563
6	780	866
7	786	-753
8	900	852
9	172	70
10	931	839
11	194	156
12	470	403
16	418	430
-1	869	884
-2	571	651
-3	675	642
-5	183	-186
-6	440	541
-7	214	220
-8	462	478
-9	194	198
-10	611	554
-11	170	155
-12	344	350
-14	504	390
-16	242	229
-17	574	-577
-18	311	294
-20	188	161
	h0 14	
0	499	483
1	221	-227
2	548	548
4	238	214
7	295	278
10	335	296
12	473	396
-2	402	367
-4	606	605
-6	431	409
-8	303	270
-10	545	523
	h1 1	
2	664	776
3	665	844
4	92	-51
5	697	797
6	141	177
7	640	667
8	437	365
9	566	556
10	137	-161
11	751	654
13	622	504
15	291	242
16	153	62
17	214	207
-2	368	-397
-3	666	828
-4	502	485
-5	891	1016
-6	227	365
-7	506	578
-9	495	493
-10	172	163
-11	669	598
-12	153	-167
-13	810	699
-14	194	-265
-15	617	536
-17	275	211
	h1 10	
3	200	189
4	268	291
9	197	193
-1	223	-201
-2	419	423
-5	430	426
-7	175	-213
	h1 15	
1	429	391
3	294	308
5	241	259
7	378	347
9	177	202
-1	446	419
-3	548	534
-5	224	244
-9	188	202
-11	359	368
	h1 16	
-5	215	-252
	h1 17	
-1	405	379
-3	334	345
	h2 4	
0	482	535
2	390	435
4	818	859
5	602	647
6	413	413
8	506	587
9	308	-211
10	349	323
-1	364	-282
-2	876	767
-3	122	93
-4	1056	1049
-5	167	-177
-6	800	750
-7	169	-145
-8	597	571
-12	388	348
	h2 9	
1	201	221
6	213	247
8	220	294
-10	294	260
	h2 16	
0	266	235
4	273	331
-2	245	248
-6	286	277
	h2 18	
-2	226	219
-6	284	270
	h06	
0	127	220
1	243	144
2	1320	1335
3	169	-227
4	213	340
5	226	-260
6	679	636
7	231	-298
8	271	314
9	184	-224
10	325	365
12	357	369
14	218	159
15	187	297
-1	131	-87
-2	494	592
-3	292	285
-4	612	677
-6	784	767
-7	749	-722
-8	290	350
-9	599	-565
-10	377	418
-11	286	307
-12	483	485
-16	258	243
-18	269	228
-20	280	281
	h0 16	
0	300	300
2	325	338
3	306	326
-2	406	376
-4	520	475
-5	321	-343
-6	272	290
-7	224	-188
-8	249	220
-10	318	336
	h1 2	
0	[illegible]	-118
1	689	-927
2	1395	-1343
3	318	-308
4	154	-190
5	472	-482
6	118	-130
7	720	700
8	113	-136
10	161	138
12	240	-281
-1	1135	-1104
-2	1516	1581
-5	567	-631
-6	178	166
-7	242	-240
-8	252	-252
-12	214	-222
-14	147	-144
-15	180	195
	h1 6	
0	193	198
1	244	217
2	154	-152
3	263	-253
4	96	94
5	346	-309
6	416	403
9	301	286
12	159	-192
-1	160	-146
-3	316	326
-5	276	261
-6	424	-449
-7	180	167
-8	166	-115
-10	357	-391
	h1 11	
1	729	688
3	664	617
5	522	483
7	407	375
8	147	201
9	278	264
10	350	352
-1	684	634
-3	463	477
-5	461	482
-7	479	495
-8	189	270
-9	534	549
-11	504	505
-13	415	394
-15	263	250
-17	196	194
	h2 0	
2	1149	983
5	408	384
6	527	555
8	710	674
10	528	486
12	472	431
13	328	-361
14	337	297
16	214	240
	h2 5	
0	185	184
3	231	-285
4	338	-361
5	143	-167
6	306	-334
-1	219	-218
-2	417	359
-3	112	156
-4	237	-214
-5	299	318
-6	485	-448
-7	175	171
	h2 10	
0	382	373
1	401	-430
2	738	759
4	363	309
6	360	340
8	354	410
-1	159	-116
-2	616	620
-4	415	456
-6	406	452
-8	402	398
	h08	
0	952	964
1	160	132
2	660	725
3	456	-408
4	410	396
6	756	718
7	229	-214
8	534	515
10	325	318
12	238	192
-1	303	-251
-2	674	749
-3	535	559
-4	375	444
-5	231	-195
-6	364	424
-7	502	-511
-8	627	669
-9	297	-375
-10	1054	943
-12	902	865
-16	318	299
	h0 18	
0	318	336
2	306	325
-2	226	224
-3	307	-344
-5	329	-329
-8	325	384
	h0 20	
0	252	237
2	252	261
	h1 3	
0	79	-56
1	625	694
2	812	745
3	1267	1322
4	594	534
5	558	574
6	174	-182
7	372	452
9	569	604
11	562	512
13	441	386
15	297	237
-1	428	523
-2	929	-862
-3	1712	1742
-4	1062	-1037
-5	1409	1460
-7	568	618
-8	248	272
-9	434	480
-11	330	269
-13	421	366
-15	500	436
-17	292	239
	h1 7	
0	270	258
1	710	674
2	94	68
3	613	583
4	404	-421
5	677	591
6	330	-310
7	473	470
9	303	270
11	372	372
13	434	386
15	249	211
-1	760	818
-2	160	-171
-3	802	893
-5	431	484
-7	403	425
-9	491	550
-11	419	433
	h1 12	
0	146	-149
5	207	-228
8	254	270
10	263	247
-3	173	185
-4	181	-232
	h2 1	
2	226	192
3	134	-149
4	170	-189
5	117	-121
10	170	-192
-2	335	313
-3	462	-468
-4	90	-85
-6	249	-265
	h2 6	
0	850	867
1	246	-246
2	331	-325
4	467	535
5	430	430
6	714	659
7	245	239
8	441	477
10	241	272
12	255	303
-1	203	226
-3	130	160
-4	693	696
-6	914	820
-7	318	-347
-8	860	800
-10	425	455
-12	270	270
	h2 11	
-3	164	157
	h2 12	
0	246	275
2	497	582
4	357	299
6	244	271
-1	191	-196
-2	271	318
-4	230	218
-5	225	321
-6	474	568
-8	413	426
-12	257	282

of these observations. Alternatively, consideration of the benzene rings alone would again suggest that it be described in terms of individually planar but not coplanar salicylaldimine groups, separated by 0·2 Å. The experimental accuracy is not sufficient to distinguish a planar molecule from one that is slightly stepped. These variations between *A* and *B* must presumable stem from packing effects, and would support the contention (Cheeseman, Hall & Waters, 1965) that the resistance of such molecules to deformation from overall planarity is rather less than has often been supposed.

The coordination is square planar, the closest contacts made by the copper atoms in the octahedral axial direction being in each case to atoms C(8) of adjacent molecules along [010], and of length 3·37 and 3·39 Å for molecules *A* and *B* respectively. The molecules are so oriented that the ethanolic hydroxyl groups of the independent molecules make contacts of 2·65 and 2·70 Å. The structure may then be described in terms of two-dimensional sheets of hydrogen-bonded molecules, parallel to (001). The compactness of this structure, resulting from the hydrogen bond formation, may be seen by comparing the density, 1·495 g.cm^{-3}, with the values of 1·405 for bis-(*N*-ethylsalicylaldiminato)-copper (Clark, 1964) and 1·34 for bis-(*N*-butylsalicylaldiminato)copper (calculated from Frasson *et al.*, 1964).

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Refinement of the L-alanine crystal structure.

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Shortly after we had completed the measurement of a set of three-dimensional intensities for a crystal of L-alanine, we learned that the crystal structure had recently been determined by Simpson & Marsh (1966). In order to test the constancy of the molecular parameters derived from different data sources, we have carried out a series of full-matrix least-squares refinements with our data. We present here the comparison of our results with those of Simpson & Marsh (SM).

Our intensity measurements were made with a Hilger–Watts linear diffractometer, using Mo radiation with SrO/ZrO_2 balanced filters. The intensities of 522 independent reflexions were recorded in the layers $hk0-hk6$ and converted to relative F values in the usual way. Absorption corrections were not deemed necessary.

Starting with SM's published parameters for the C, N and O atoms (hydrogen atoms were included in the structure factor calculations but not refined), our analysis leads to the results shown in Tables 1 and 2. The agreement is good as far as the chemical significance of the results is concerned; however, the differences, although small, seem statistically significant on the basis of the estimated standard deviations cited by SM. (We have not calculated the least-squares standard deviations of our parameters, but they should be of about the same order of magnitude as those of SM.) On the basis of tests using the function $R''=[\sum_i w_i(F_o-F_c)^2/\sum_i w_iF_o^2]^{\frac{1}{2}}$ (Hamilton, 1965) we find that our data reject the SM model at better than 0·005 level of significance, while the SM data reject our model at about the same level.

Table 1. *Positional parameters* (× 10^4) *and bond lengths*

	x_{DR}	x_{SM}	σ_{SM}	y_{DR}	y_{SM}	σ_{SM}	z_{DR}	z_{SM}	σ_{SM}
O(1)	7278	7287	3	843	843	1	6280	6283	3
O(2)	4499	4501	3	1856	1850	1	7604	7609	3
C(1)	5606	5606	4	1413	1418	1	6023	6016	4
N	6565	6560	3	1375	1382	1	1853	1856	3
C(2)	4764	4769	4	1611	1612	1	3559	3563	4
C(3)	2744	2746	5	919	915	2	3021	3025	5

Bond	d_{DR}	d_{SM}
C(1)–O(1)	1·239 Å	1·247 Å
C(1)–O(2)	1·257	1·256
C(1)–C(2)	1·533	1·525
C(2)–C(3)	1·523	1·525
C(2)–N	1·496	1·491

$$\frac{R''\ (\text{SM data: our model})}{R''\ (\text{SM data: SM model})}=\frac{0{\cdot}091}{0{\cdot}070}=1{\cdot}30$$

$$\frac{R''\ (\text{our data: SM model})}{R''\ (\text{our data: our model})}=\frac{0{\cdot}066}{0{\cdot}049}=1{\cdot}34\,.$$