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The Crystal Structure of Sodium Sulfanilate Dihydrate

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The crystal structure of sodium sulfanilate dihydrate ($H_2NC_6H_4SO_3Na.2H_2O$) has been determined. The compound crystallizes in the orthorhombic space group *Pbca* with $a=23{\cdot}895$ (5), $b=10{\cdot}101$ (2) and $c=7{\cdot}944$ (2) Å. The sodium atom has a distorted octahedral coordination of oxygen atoms; the amino group of the sulfanilate ion is considerably distorted from a planar conformation, partly owing to hydrogen bonding in the crystal.

Introduction

Though the crystal structure of sulfanilic acid monohydrate was solved several years ago (Rae & Maslen, 1962), the structure of a sulfanilate salt has not yet been reported. In the framework of a study of the electronic structure and chemical bonding of sulfur-containing compounds it seemed interesting to study sodium sulfanilate, because of the many different types of bonds present in this compound including anticipated hydrogen bonds. Of interest also is the N atom of the amino group which has an sp^3 conformation in the zwitterion-like sulfanilic acid molecule, but which was expected to be approximately planar in the non-zwitterion sulfanilate salt.

Experimental

Sodium sulfanilate was prepared by slowly evaporating a solution of equimolecular quantities of sulfanilic acid and sodium bicarbonate. After several days brown-colored transparent crystals with prismatic habit were formed, which could grow to rather considerable sizes. A crystal of about 0·6 × 0·3 × 0·25 mm was chosen for the data collection. Precession and Weissenberg photographs indicated the orthorhombic space group *Pbca*. The cell parameters at 295 K are $a=23{\cdot}895$ (5), $b=10{\cdot}101$ (2) and $c=7{\cdot}944$ (2) Å. 5838 reflections were measured on a Picker diffractometer up to $\sin\theta/\lambda=0{\cdot}70$ Å^{-1} using Nb-filtered Mo $K\alpha$ radiation and applying a step-scan mode (Blessing, Coppens & Becker, 1974). 2797 of these reflections were symmetry-independent. Fluctuations in incident-beam intensity and counter response were corrected by measuring three different standard reflections after every 40 reflections and rescaling the measurements with respect to these standard reflections (maximum variation of standards was 1 %). After corrections for counter linearity (Chipman, 1969), absorption and Lorentz and polarization effects, the equivalent reflections were weighted averaged, resulting in 2604 reflections with larger than zero intensity. The weight of an individual reflection is derived from its statistical error plus a small proportionality factor. The weight of an averaged reflection is taken as the sum of the weights of the individual reflections.

Structure determination

A Patterson synthesis showed the position of the sulfur atom, but no other atoms could be found. Therefore, the direct-method program *MULTAN* (Main, Germain & Woolfson, 1970) was applied to 280 reflections using 1200 phase relationships. The first *E* map showed the positions of the sulfur, sodium and two oxygen atoms. A Fourier synthesis based on these positions showed the remaining non-hydrogen atoms. Two ad-

Table 1. *Positional and thermal parameters of the atoms*

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S	0·40242 (1)	0·03926 (3)	0·05184 (5)	0·0247 (2)	0·0222 (2)	0·0231 (2)	−0·0022 (1)	0·0008 (1)	−0·0014 (1)
Na	0·48008 (3)	0·33527 (6)	−0·09250 (8)	0·0326 (3)	0·0290 (3)	0·0277 (3)	0·0006 (2)	0·0005 (3)	−0·0009 (3)
O(1)	0·39669 (4)	−0·09204 (11)	−0·02562 (14)	0·0298 (5)	0·0250 (5)	0·0383 (6)	−0·0004 (4)	0·0040 (5)	−0·0097 (5)
O(2)	0·42723 (5)	0·13479 (11)	−0·06282 (15)	0·0336 (6)	0·0333 (6)	0·0354 (6)	−0·0068 (5)	0·0075 (5)	0·0055 (5)
O(3)	0·43039 (5)	0·03030 (12)	0·21380 (15)	0·0349 (6)	0·0399 (7)	0·0300 (6)	0·0001 (5)	−0·0096 (5)	−0·0006 (5)
C(1)	0·33336 (6)	0·09061 (14)	0·09044 (18)	0·0271 (7)	0·0198 (6)	0·0198 (7)	−0·0008 (5)	0·0019 (6)	−0·0019 (6)
C(2)	0·30077 (7)	0·01406 (15)	0·19787 (20)	0·0348 (8)	0·0214 (7)	0·0268 (8)	0·0050 (6)	0·0052 (7)	0·0047 (6)
C(3)	0·24468 (7)	0·03945 (15)	0·21413 (21)	0·0349 (8)	0·0241 (7)	0·0312 (8)	−0·0003 (7)	0·0107 (7)	0·0037 (7)
C(4)	0·21981 (6)	0·14377 (14)	0·12656 (20)	0·0276 (7)	0·0249 (7)	0·0245 (6)	0·0016 (6)	0·0019 (6)	−0·0056 (6)
C(5)	0·25332 (7)	0·22274 (15)	0·02504 (19)	0·0362 (8)	0·0246 (8)	0·0247 (7)	0·0061 (6)	−0·0004 (7)	0·0039 (7)
C(6)	0·30944 (7)	0·19608 (15)	0·00559 (20)	0·0345 (8)	0·0225 (7)	0·0239 (7)	−0·0017 (6)	0·0047 (6)	0·0036 (6)
N	0·16260 (6)	0·16432 (16)	0·14005 (22)	0·0301 (7)	0·0358 (8)	0·0413 (9)	0·0040 (6)	0·0016 (7)	0·0007 (7)
O(4)	0·47165 (5)	−0·27346 (13)	−0·17712 (17)	0·0307 (6)	0·0288 (6)	0·0395 (7)	0·0023 (5)	0·0034 (6)	−0·0030 (6)
O(5)	0·44166 (6)	0·48855 (14)	0·10304 (17)	0·0389 (7)	0·0451 (8)	0·0298 (7)	0·0114 (6)	0·0060 (6)	0·0021 (6)

Table 1 (*cont.*)

	x	y	z	U
H(1)	0·3172 (6)	−0·0556 (17)	0·2629 (22)	0·030 (4)
H(2)	0·2226 (7)	−0·0130 (17)	0·2854 (24)	0·037 (5)
H(3)	0·2386 (8)	0·2911 (17)	−0·0310 (24)	0·041 (5)
H(4)	0·3309 (7)	0·2473 (16)	−0·0625 (23)	0·032 (5)
H(5)	0·1523 (8)	0·2370 (20)	0·0934 (27)	0·044 (6)
H(6)	0·1487 (8)	0·1434 (20)	0·2424 (28)	0·051 (6)
H(7)	0·4488 (9)	−0·2296 (21)	−0·1387 (29)	0·048 (7)
H(8)	0·4534 (9)	−0·3431 (22)	−0·2004 (28)	0·059 (7)
H(9)	0·4099 (10)	0·5083 (23)	0·0807 (33)	0·063 (8)
H(10)	0·4365 (10)	0·4446 (28)	0·1923 (36)	0·081 (9)

ditional strong Fourier peaks were identified as hydrate molecules. After a least-squares refinement of the positional and anisotropic thermal parameters, a difference Fourier synthesis yielded all 10 hydrogen atoms. The scattering factors for all non-hydrogen atoms were taken from *International Tables for X-ray Crystallography* (1974), while those for the hydrogen atoms were as given by Stewart, Davidson & Simpson (1965). For the S and Na atoms, the anomalous scattering factors from Cromer & Liberman (1970) were applied. Subsequent least-squares cycles resulted in a final R value

Table 2. *Observed and calculated structure factors* (×4)

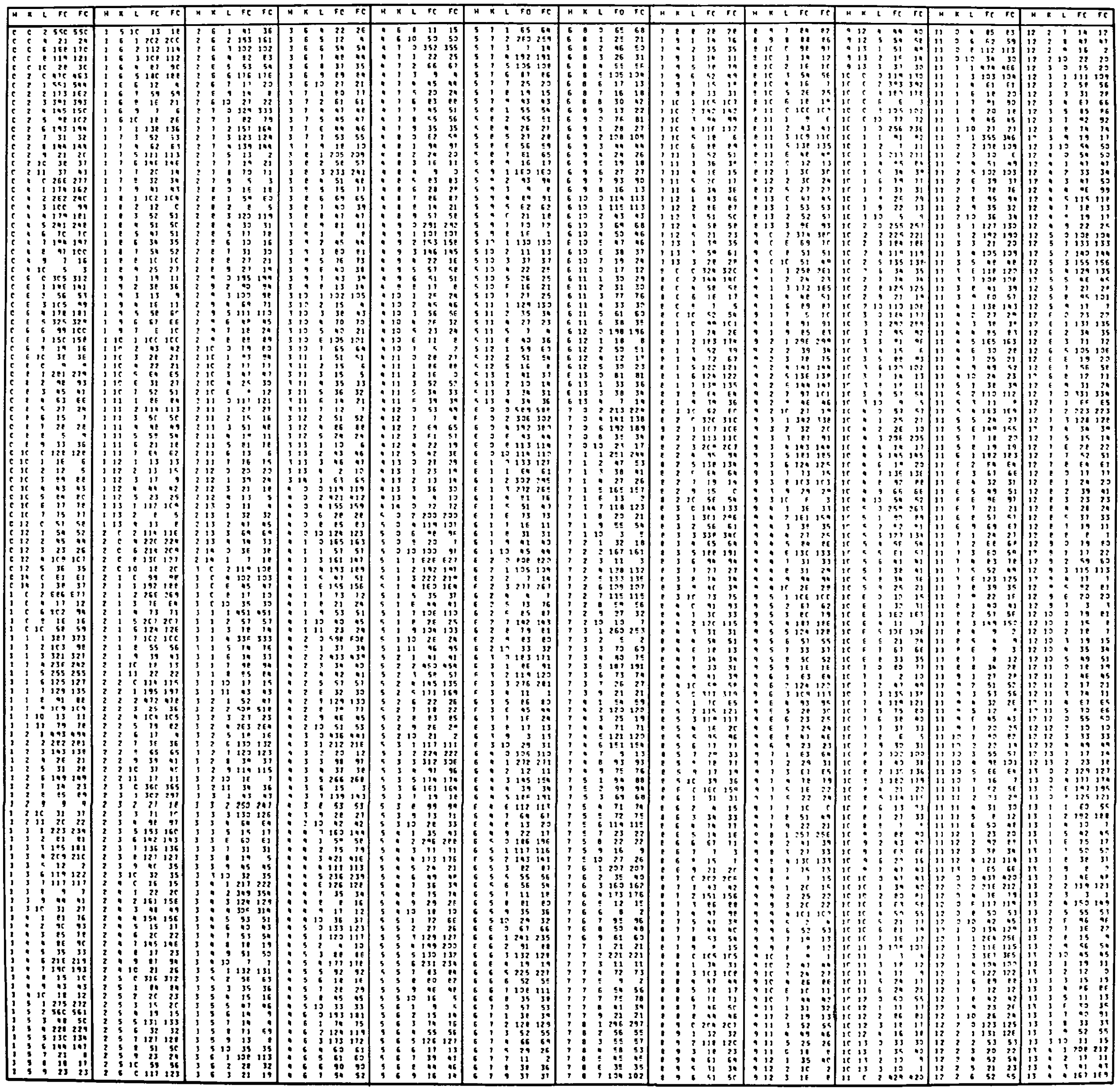

Table 2 (*cont.*)

H	K	L	FO	FC
13	4	5	170	173
13	4	6	69	69
13	4	7	139	142
13	4	8	52	54
13	4	9	36	30
13	5	1	44	45
13	5	2	65	66
13	5	3	21	19
13	5	4	37	39
13	5	5	18	19
13	5	6	35	40
13	5	8	20	8
13	5	9	8	14
13	6	1	296	298
13	6	2	135	137
13	6	3	91	91
13	6	4	80	83
13	6	5	16	10
13	6	6	61	60
13	6	7	44	44
13	6	8	101	101
13	6	9	62	58
13	7	1	32	29
13	7	2	16	11
13	7	3	110	114
13	7	5	7	5
13	7	7	23	16
13	7	8	10	6
13	8	1	42	43
13	8	2	71	69
13	8	3	110	111
13	8	4	16	4
13	8	5	40	41
13	8	6	25	20
13	8	7	28	24
13	9	1	34	40
13	9	2	57	58
13	9	3	28	27
13	9	4	4	8
13	9	5	19	16
13	9	7	6	3
13	10	1	127	125
13	10	2	24	24
13	10	3	52	54
13	10	4	42	39
13	10	5	18	8
13	10	6	91	92
13	11	1	30	32
13	11	3	18	18
13	11	5	50	48
13	12	1	17	13
13	12	2	86	81
13	12	3	11	5
13	12	4	102	96
13	13	1	25	26
14	0	0	280	282
14	0	2	242	242
14	0	4	139	138
14	0	6	62	60
14	0	8	47	46
14	0	10	36	34
14	1	1	25	29
14	1	2	104	105
14	1	3	308	313
14	1	4	52	53
14	1	5	148	151
14	1	6	161	165
14	1	7	18	29
14	1	8	46	50
14	1	9	6	12
14	2	0	64	61
14	2	1	39	41
14	2	2	152	149
14	2	3	205	205
14	2	4	14	12
14	2	5	171	171
14	2	6	16	16
14	2	7	66	66
14	2	8	45	42
14	2	10	64	64
14	3	0	129	132
14	3	1	10	20
14	3	2	90	93
14	3	3	103	105
14	3	4	7	12
14	3	5	31	31
14	3	6	42	44
14	3	7	36	32
14	3	8	47	47
14	3	9	45	43
14	4	0	132	133
14	4	1	69	67
14	4	2	42	40
14	4	3	103	106
14	4	4	18	7
14	4	5	65	66
14	4	6	15	2
14	4	7	45	41
14	4	8	34	38
14	4	9	12	3
14	5	0	30	13
14	5	1	21	17
14	5	2	111	111
14	5	3	109	110
14	5	4	50	47
14	5	5	35	38
14	5	6	23	21
14	5	7	3	7
14	5	9	22	17
14	6	0	161	175
14	6	2	30	31
14	6	3	95	97
14	6	4	21	22
14	6	5	69	71
14	6	6	15	7
14	6	7	30	30
14	7	0	82	83
14	7	1	45	50
14	7	2	120	120
14	7	3	36	34
14	7	5	30	29
14	7	6	63	65
14	7	7	42	40
14	7	9	64	59
14	8	0	140	142
14	8	1	29	29
14	8	2	51	59
14	8	3	45	43
14	8	5	34	31
14	8	6	28	28
14	8	7	26	29
14	9	0	31	36
14	9	1	7	0
14	9	2	122	122
14	9	3	48	50
14	9	4	64	68
14	9	5	13	12
14	9	7	20	17
14	10	1	20	21
14	10	2	14	21
14	10	3	49	52
14	10	5	64	65
14	10	6	29	27
14	11	0	42	39
14	11	2	10	3
14	11	5	40	40
14	12	0	38	35
14	12	1	13	21
14	12	2	20	17
14	12	3	12	20
15	0	2	150	152
15	0	4	67	68
15	0	6	20	20
15	0	8	12	6
15	0	10	19	15
15	1	1	214	215
15	1	2	42	46
15	1	3	251	256
15	1	4	16	16
15	1	5	111	114
15	1	6	104	106
15	1	7	24	28
15	1	8	31	31
15	1	9	24	21
15	2	2	45	46
15	2	3	28	28
15	2	4	88	88
15	2	5	6	1
15	2	6	10	69
15	2	7	28	28
15	2	8	47	45
15	2	9	34	34
15	3	1	296	296
15	3	2	68	71
15	3	3	83	82
15	3	4	277	280
15	3	5	135	138
15	3	6	73	73
15	3	8	40	42
15	3	9	12	10
15	4	1	153	152
15	4	2	25	28
15	4	3	110	109
15	4	4	82	83
15	4	5	29	32
15	4	6	15	22
15	4	8	29	30
15	4	9	18	7
15	5	1	67	70
15	5	2	131	138
15	5	3	68	69
15	5	4	148	149
15	5	5	94	96
15	5	6	38	34
15	5	7	21	16
15	5	8	85	82
15	5	9	47	47
15	6	1	49	51
15	6	2	71	73
15	6	3	24	22
15	6	4	84	82
15	6	5	38	38
15	6	6	20	10
15	6	7	32	29
15	6	8	56	52
15	7	1	136	138
15	7	2	64	73
15	7	3	27	25
15	7	4	128	133
15	7	5	14	15
15	7	6	50	48
15	7	7	37	30
15	7	8	79	73
15	8	1	31	33
15	8	3	51	51
15	8	4	15	11
15	8	5	18	9
15	8	6	14	12
15	8	7	39	33
15	9	1	22	20
15	9	3	17	16
15	9	4	98	98
15	9	5	43	38
15	9	6	42	43
15	9	7	43	39
15	10	1	49	49
15	10	2	10	8
15	10	3	23	19
15	10	4	14	3
15	10	5	52	48
15	10	6	30	22
15	11	1	24	25
15	11	2	32	24
15	11	3	49	52
15	11	4	56	54
15	12	1	14	5
15	12	2	26	24
15	12	3	33	36
16	0	0	238	234
16	0	2	144	143
16	0	4	144	141
16	0	6	33	37
16	0	8	11	10
16	1	0	207	204
16	1	1	142	139
16	1	2	71	69
16	1	3	84	85
16	1	4	38	40
16	1	5	59	60
16	1	6	43	39
16	1	7	24	23
16	1	9	9	12
16	2	0	34	34
16	2	1	106	108
16	2	2	339	336
16	2	3	245	251
16	2	4	143	141
16	2	5	55	55
16	2	6	4	3
16	2	7	5	11
16	2	8	39	36
16	2	9	18	18
16	3	0	88	96
16	3	1	21	19
16	3	2	28	30
16	3	3	152	151
16	3	4	54	53
16	3	6	42	41
16	3	7	11	22
16	3	8	17	4
16	3	9	13	19
16	4	0	133	134
16	4	1	19	20
16	4	2	48	43
16	4	3	135	138
16	4	4	86	84
16	4	5	133	133
16	4	7	124	129
16	4	8	131	98
16	4	9	20	27
16	5	0	189	191
16	5	1	137	130
16	5	2	154	158
16	5	3	39	37
16	5	4	94	85
16	5	5	29	24
16	5	6	59	61
16	5	7	56	52
16	5	8	27	24
16	5	9	52	60
16	6	0	29	30
16	6	1	84	85
16	6	2	10	10
16	6	3	123	122
16	6	4	52	54
16	6	5	93	93
16	6	6	21	21
16	6	7	29	23
16	6	8	11	15
16	7	0	87	89
16	7	1	58	58
16	7	2	81	83
16	7	3	32	31
16	7	4	49	51
16	7	6	21	24
16	7	7	28	32
16	7	8	13	10
16	8	0	56	52
16	8	1	66	68
16	8	2	52	53
16	8	3	173	175
16	8	4	20	22
16	8	5	44	44
16	8	6	54	53
16	8	7	52	57
16	9	0	24	24
16	9	1	11	14
16	9	2	61	65
16	9	3	30	33
16	9	4	25	31
16	9	5	8	11
16	9	6	35	34
16	10	0	114	112
16	10	1	48	45
16	10	2	90	81
16	10	3	61	63
16	10	4	12	18
16	10	5	41	38
16	11	0	9	15
16	11	1	20	16
16	11	2	35	33
16	11	3	51	55
16	11	4	45	42
16	12	0	9	6
16	12	2	35	33
17	0	2	298	302
17	0	4	282	283
17	0	6	84	88
17	0	8	16	3
17	1	1	101	99
17	1	2	8	9
17	1	3	23	15
17	1	4	85	85
17	1	5	5	5
17	1	6	39	37
17	1	7	38	40
17	1	8	32	31
17	1	9	32	23
17	2	1	73	75
17	2	2	31	32
17	2	3	126	128
17	2	4	185	184
17	2	5	184	184
17	2	6	55	52
17	2	7	14	15
17	2	8	91	92
17	2	9	20	13
17	3	1	213	213
17	3	2	31	30
17	3	3	20	19
17	3	4	18	18
17	3	5	46	45
17	3	6	53	55
17	3	7	58	59
17	3	8	32	29
17	3	9	10	10
17	4	1	60	69
17	4	2	190	191
17	4	3	119	116
17	4	4	31	36
17	4	5	18	18
17	4	6	26	28
17	4	7	15	6
17	4	8	73	70
17	4	9	68	68
17	5	1	52	53
17	5	2	134	132
17	5	3	18	10
17	5	4	45	42
17	5	5	39	37
17	5	6	51	58
17	5	7	33	33
17	5	8	13	3
17	6	1	14	17
17	6	2	111	112
17	6	3	113	111
17	6	4	91	92
17	6	5	10	12
17	6	6	18	23
17	6	7	60	59
17	6	8	38	34
17	7	1	36	36
17	7	2	13	8
17	7	3	45	45
17	7	4	50	48
17	7	6	62	60
17	8	1	193	195
17	8	2	20	17
17	8	3	94	94
17	8	4	19	19
17	8	5	27	36
17	8	6	27	23
17	8	7	41	42
17	9	1	23	24
17	9	2	34	36
17	9	3	16	15
17	9	4	76	31
17	9	5	40	36
17	9	6	56	55
17	10	1	22	17
17	10	2	83	86
17	10	3	56	58
17	10	4	19	60
17	10	5	15	1
17	11	1	22	26
17	11	2	34	29
17	11	3	45	42
17	11	4	21	19
17	12	1	11	7
18	0	0	67	62
18	0	2	46	52
18	0	4	135	137
18	0	6	36	35
18	0	8	44	45
18	1	0	293	291
18	1	1	22	13
18	1	2	61	69
18	1	3	138	139
18	1	4	34	31
18	1	5	130	128
18	1	6	18	19
18	1	7	100	99
18	1	8	23	23
18	2	0	164	170
18	2	1	52	51
18	2	2	133	137
18	2	3	66	66
18	2	4	70	72
18	2	5	41	34
18	2	6	13	10
18	2	7	10	9
18	2	8	9	6
18	2	9	7	18
18	3	0	39	36
18	3	1	92	92
18	3	2	51	53
18	3	3	187	191
18	3	4	23	19
18	3	5	65	66
18	3	6	4	12
18	3	7	30	26
18	3	8	47	47
18	3	9	18	2
18	4	0	128	131
18	4	1	56	56
18	4	2	42	40
18	4	3	92	93
18	4	4	41	43
18	4	5	11	12
18	4	6	17	15
18	4	7	14	0
18	5	0	244	244
18	5	1	7	2
18	5	2	155	157
18	5	3	18	17
18	5	4	24	28
18	5	5	29	25
18	5	6	72	71
18	5	7	46	44
18	5	8	11	12
18	6	0	39	39
18	6	1	87	89
18	6	2	60	63
18	6	3	29	28
18	6	4	43	44
18	6	5	36	37
18	6	6	48	49
18	6	7	22	14
18	6	8	29	30
18	7	0	99	100
18	7	1	16	17
18	7	2	121	122
18	7	3	12	6
18	7	4	69	69
18	7	5	24	27
18	7	6	12	7
18	7	7	30	32
18	8	0	18	19
18	8	1	68	69
18	8	2	29	29
18	8	3	4	0
18	8	4	14	4
18	8	5	21	23
18	8	6	20	5
18	8	7	4	11
18	9	0	63	62
18	9	1	19	17
18	9	2	78	77
18	9	4	48	45
18	9	5	58	56
18	9	6	19	11
18	10	0	11	16
18	10	1	18	14
18	10	4	13	9
18	10	5	31	30
18	11	0	40	48
18	11	1	27	29
18	11	2	23	28
18	11	3	82	83
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19	0	6	168	169
19	0	8	42	43
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19	10	3	13	17
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20	0	4	175	174
20	0	6	45	49
20	0	8	36	38
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20	1	4	35	33
20	1	5	16	19
20	1	6	67	68
20	1	7	28	24
20	1	8	46	47
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20	2	7	107	110
20	2	8	87	87
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21	1	3	96	97
21	1	4	124	127
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21	1	8	14	17
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21	2	2	72	75
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21	2	5	40	40
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21	3	6	59	55
21	3	7	68	67
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21	4	3	40	43
21	4	4	13	17
21	4	5	10	9
21	4	6	47	46
21	4	7	56	52
21	4	8	11	5
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21	9	3	14	17
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22	1	6	34	30
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22	2	0	41	46
22	2	1	52	52
22	2	2	129	130
22	2	4	29	30
22	2	5	32	21
22	2	6	14	6
22	2	7	51	51
22	2	8	21	26
22	3	0	46	44
22	3	1	33	36
22	3	2	7	11
22	3	3	24	26
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22	3	6	21	31
22	3	7	86	83
22	3	8	22	24
22	4	0	34	31
22	4	1	5	1
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22	4	4	37	36
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22	6	3	12	14
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22	6	5	8	15
22	6	6	18	20
22	6	7	23	22
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22	7	1	49	53
22	7	2	126	121
22	7	3	16	18
22	7	6	27	29
22	8	0	28	30
22	8	1	45	47
22	8	2	15	20
22	8	3	71	74
22	8	4	19	30
22	8	5	63	54
22	9	0	93	93
22	9	1	42	41
22	9	2	41	44
22	9	3	19	16
22	9	4	25	20
22	10	0	5	11
22	10	1	21	26
22	10	2	25	26
22	10	3	83	85
23	0	2	83	82
23	0	4	126	121
23	0	6	64	61
23	0	8	52	51
23	1	1	4	3
23	1	2	21	17
23	1	4	53	54
23	1	5	18	10
23	1	6	8	17
23	1	7	22	19
23	1	8	43	43
23	2	1	46	49
23	2	2	65	63
23	2	3	48	46
23	2	4	160	160
23	2	5	18	11
23	2	6	14	23
23	2	7	10	5
23	3	3	53	53
23	3	4	12	4
23	3	5	16	12
23	3	6	12	5
23	3	7	48	48
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23	4	2	18	16
23	4	3	25	29
23	4	4	64	64
23	4	5	23	22
23	4	7	23	23
23	5	1	36	33
23	5	2	53	51
23	5	3	16	1
23	5	4	29	26
23	5	5	43	42
23	5	6	32	30
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23	6	3	106	106
23	6	4	81	79
23	6	5	39	38
23	6	6	1	1
23	7	1	18	22
23	7	2	25	17
23	7	3	56	57
23	7	4	19	21
23	7	5	12	9
23	8	1	100	101
23	8	2	25	19
23	8	3	71	71
23	8	5	21	0
23	9	1	34	35
23	9	2	24	25
23	9	4	20	24
23	10	1	29	30
23	10	2	41	39
24	0	0	174	176
24	0	2	118	119
24	0	4	18	19
24	0	6	27	18
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24	1	1	15	12
24	1	2	38	36
24	1	3	109	110
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24	2	2	64	65
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24	2	4	17	22
24	2	5	51	51
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24	3	7	39	36
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24	4	4	37	34
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24	4	6	15	17
24	4	7	82	84
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24	5	3	29	29
24	5	5	13	10
24	5	6	21	23
24	6	0	53	41
24	6	2	20	30
24	6	3	41	30
24	6	4	37	37
24	6	5	31	41
24	6	6	62	61
24	7	0	81	80
24	7	1	8	11
24	7	2	89	89
24	7	3	14	7
24	7	4	15	0
24	7	5	7	9
24	8	0	33	29
24	8	1	35	33
24	8	2	44	41
24	8	3	73	70
24	8	4	20	20
24	9	0	22	21
24	9	1	59	53
24	9	2	48	48
24	9	3	24	25
25	0	2	113	115
25	0	4	82	81
25	0	6	39	39
25	1	1	64	60
25	1	2	5	14
25	1	3	47	46
25	1	4	7	13
25	1	5	66	66
25	1	6	41	41
25	1	7	23	14
25	2	1	17	19
25	2	2	13	4
25	2	4	16	16
25	2	5	23	25
25	2	6	12	2
25	2	7	7	4
25	3	1	116	119
25	3	2	40	40
25	3	3	48	49
25	3	4	75	78
25	3	5	39	40
25	3	6	83	81
25	3	7	54	49
25	4	1	72	73
25	4	2	11	0
25	4	3	32	34
25	4	4	20	15
25	4	5	13	1
25	4	6	16	3
25	5	1	20	22
25	5	2	101	104
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25	6	3	43	42
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25	7	1	68	60
25	7	2	50	47
25	7	3	11	7
25	7	4	50	51
25	7	5	21	21
25	8	2	12	10
25	8	3	27	13
25	9	1	40	39
25	9	2	31	32
26	0	0	195	201
26	0	2	125	123
26	0	4	28	31
26	0	6	10	11
26	1	0	20	28
26	1	1	23	15
26	1	2	8	2
26	1	3	39	42
26	1	4	31	4
26	1	5	5	7
26	1	6	5	15
26	2	0	107	106
26	2	1	59	59
26	2	2	88	90
26	2	3	116	115
26	2	4	18	20
26	2	5	15	13
26	2	6	23	23
26	3	0	30	29
26	3	1	5	6
26	3	2	35	33
26	3	3	3	11
26	3	5	41	40
26	3	6	14	11
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26	4	2	56	53
26	4	3	32	28
26	4	5	60	58
26	4	6	13	14
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26	6	0	9	12
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26	6	3	42	45
26	6	4	51	48
26	6	5	64	68
26	7	0	107	108
26	7	1	30	28
26	7	2	9	15
26	7	3	3	8
26	7	4	18	9
26	8	0	29	30
26	8	1	32	28
26	8	2	8	10
26	8	3	61	61
26	9	0	41	38
27	0	2	22	18
27	0	4	18	15
27	0	6	50	50
27	1	1	24	23
27	1	2	8	2
27	1	3	36	38
27	1	4	32	26
27	1	6	9	10
27	2	2	13	16
27	2	3	6	2
27	2	4	27	24
27	2	6	36	40
27	3	1	93	92
27	3	2	53	56
27	3	3	45	44
27	3	4	64	64
27	3	5	18	18
27	3	6	10	4
27	4	1	57	51
27	4	2	22	30
27	4	3	21	22
27	4	4	68	67
27	4	5	4	5
27	5	1	22	17
27	5	2	48	48
27	5	3	23	28
27	5	4	73	70
27	5	5	40	35
27	6	1	72	66
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27	8	2	13	11
28	0	0	10	14
28	0	2	8	11
28	0	4	82	79
28	0	6	15	19
28	1	1	45	46
28	1	2	33	30
28	1	3	94	93
28	1	4	24	23
28	1	5	61	62
28	1	6	44	44
28	2	1	36	29
28	2	2	15	17
28	2	3	59	60
28	2	4	12	12
28	3	0	98	98
28	3	1	5	4
28	3	2	38	36
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28	3	4	18	21
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28	4	0	20	10
28	4	1	40	41
28	4	2	17	29
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28	4	5	6	4
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28	5	1	9	13
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29	0	2	73	70
29	0	4	80	83
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29	1	2	63	59
29	1	3	18	27
29	1	4	17	21
29	1	5	24	21
29	2	1	21	22
29	2	2	92	96
29	2	3	9	7
29	2	4	60	62
29	3	1	7	7
29	3	2	26	24
29	3	3	26	20
29	3	4	38	40
29	3	5	42	43
29	4	1	81	79
29	4	2	23	22
29	4	3	54	51
29	4	4	30	29
29	5	1	22	22
29	5	3	28	26
29	5	4	31	32
29	6	1	70	69
29	6	2	28	17
29	7	1	16	18
30	0	0	112	118
30	0	2	37	42
30	0	4	31	34
30	1	0	10	9
30	1	1	4	11
30	1	2	21	21
30	1	3	33	31
30	1	4	32	23
30	2	0	124	120
30	2	1	14	4
30	2	2	17	14
30	2	3	8	12
30	2	4	24	24
30	3	0	47	47
30	3	3	6	8
30	4	0	49	49
30	4	1	51	51
30	4	2	17	13
30	4	3	82	80
30	5	0	46	46
30	5	1	6	2
30	5	2	24	22
30	5	3	14	5
30	6	0	34	35
30	6	1	14	26
31	0	4	31	34
31	1	1	92	94
31	1	2	32	28
31	1	3	40	39
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32	0	2	34	32
32	1	0	21	4
32	1	1	46	41
32	1	2	9	2
32	2	0	16	23
32	2	2	34	33
32	2	3	8	12
32	3	0	50	51
32	3	1	81	77
32	3	2	20	14
32	4	0	61	67
32	4	1	16	14
33	0	2	85	81
33	1	1	22	25
33	1	2	37	31
33	2	1	77	74

based on *F* of 4·5 % and a weighted *R* value based on *F* of 3·8 %. Table 1 gives the final positional and thermal parameters, while the observed and calculated structure factors are listed in Table 2.

Discussion of the results

The bond distances and angles are given in Tables 3 and 4. The arrangement of the atoms in the unit cell is shown in Fig. 1. The benzene ring is approximately planar. The N and S atoms are bent out of this plane by distances of respectively 0·1 and 0·2 Å. The bonds C(2)–C(3) and C(5)–C(6) are significantly shorter than the remaining four bonds in the benzene ring as can be expected by resonance in benzene with an electron-donating and an electron-receiving group substituted *para* to one another.

Table 3. *Bond distances* (Å)

S—O(1)	1·468 (1)	N—H(5)	0·86 (2)
S—O(2)	1·453 (1)	N—H(6)	0·90 (2)
S—O(3)	1·453 (1)	O(4)–H(7)	0·77 (2)
S—C(1)	1·757 (2)	O(4)–H(8)	0·85 (2)
C(1)–C(2)	1·390 (2)	O(5)–H(9)	0·80 (2)
C(2)–C(3)	1·371 (2)	O(5)–H(10)	0·85 (3)
C(3)–C(4)	1·395 (2)		
C(4)–C(5)	1·388 (2)	Na–O bonds	
C(5)–C(6)	1·376 (2)	Na–O(2)	2·398 (1)
C(1)–C(6)	1·384 (2)	Na–O(3)′	2·371 (1)
C(4)–N	1·387 (2)	Na–O(4)′	2·426 (1)
C(2)–H(1)	0·96 (2)	Na–O(4)″	2·511 (1)
C(3)–H(2)	0·94 (2)	Na–O(5)	2·377 (1)
C(5)–H(3)	0·89 (2)	Na–O(5)′	2·582 (1)
C(6)–H(4)	0·91 (2)		

The Na atom is surrounded by six oxygen atoms, four of the hydrate molecules and two of the sulfanilate anions. The O–Na–O angles are in the ranges of 77–108° and 158–166° showing a distorted octahedral conformation. The hydrate oxygens form bridges between two sodium atoms; all O–Na bonds are in the expected lone-pair directions of the oxygen atoms.

Fig. 2 shows a Newman projection of the orientation of the SO_3 group with respect to the plane of the benzene ring. It may be expected that this orientation corresponds to optimal hydrogen bonding and Na–O bonding. The SO_3 group is involved in five hydrogen

Table 4. *Bond angles* (°)

O(1)–S——O(2)	112·02 (7)	C(3)–C(2)–H(1)	119 (1)
O(1)–S——O(3)	110·96 (7)	C(2)–C(3)–H(2)	120 (1)
O(2)–S——O(3)	114·13 (7)	C(4)–C(3)–H(2)	119 (1)
C(1)–S——O(1)	104·62 (6)	C(4)–C(5)–H(3)	120 (1)
C(1)–S——O(2)	107·25 (7)	C(6)–C(5)–H(3)	119 (1)
C(1)–S——O(3)	107·21 (7)	C(5)–C(6)–H(4)	120 (1)
S——C(1)–C(2)	118·0 (1)	C(1)–C(6)–H(4)	120 (1)
S——C(1)–C(6)	122·0 (1)	C(4)–N——H(5)	112 (1)
C(2)–C(1)–C(6)	119·7 (1)	C(4)–N——H(6)	113 (1)
C(1)–C(2)–C(3)	120·1 (1)	H(5)–N——H(6)	119 (2)
C(2)–C(3)–C(4)	120·7 (1)	H(7)–O(4)–H(8)	102 (2)
C(3)–C(4)–C(5)	118·6 (1)	H(9)–O(5)–H(10)	100 (2)
C(3)–C(4)–N	119·6 (1)		
C(5)–C(4)–N	121·8 (1)		
C(4)–C(5)–C(6)	121·0 (1)		
C(5)–C(6)–C(1)	119·9 (1)		
C(1)–C(2)–H(1)	121 (1)		

bonds and two Na–O bonds, also shown in Fig. 2. Atom O(1) has three hydrogen bonds, two with an amino group and one with a hydrate molecule, while atoms O(2) and O(3) both have one hydrogen bond with a hydrate molecule and one Na–O bond. As can be seen in Table 3, the sulfur oxygen bonds S–O(2) and S–O(3) have exactly the same length, while the S–O(1) bond distance is about 0·015 Å longer; this difference may be attributed to the larger number of interactions in which O(1) participates.

The C–S bond length of 1·757 (2) Å is in good agreement with the C–S bond length of 1·77 (2) Å reported for sulfanilic acid monohydrate (Rae & Maslen, 1962).

Fig. 3 shows a Newman projection of the NH_2 group with respect to the plane of the benzene ring. The nitrogen atom shows a significant distortion of a planar sp^2 conformation, resulting in the inclusion of some sp^3 character in this atom. The involvement of the NH_2 group in hydrogen bonding may be responsible for some of the non-planarity of the amino group; the out-of-plane bending of the hydrogen atoms is in the direction of the hydrogen bonds (Fig. 3), and H(6) shows a larger bending than H(5) in agreement with the larger out-of-plane bending of the $N\cdots O(1)''$ hydrogen bond. A three-dimensional neutron diffraction study of β-sulfanilamide (Lum & Maslen, 1970) also shows a significant, though smaller, non-planar distortion of the ring-amino group. In β-sulfanilamide only one hydrogen atom of the ring-amino group is involved in a hydrogen bond located nearly exactly in the plane of the benzene ring. The hydrogen bonding can therefore not explain the non-planarity in this molecule and it may only be partially responsible for the non-planarity of the amino group in Na sulfanilate dihydrate. The interaction with the π-system of the benzene ring is clearly too weak to produce exact sp^2 character for the N atom of the amino group, as has been predicted by

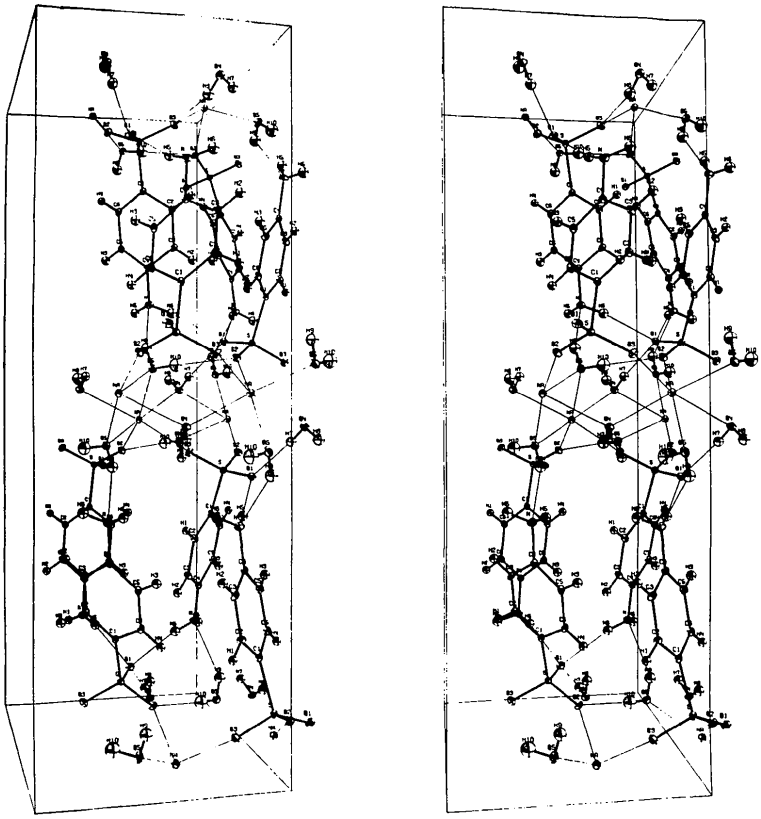

Fig. 1. Stereoscopic view of the arrangement of the atoms in the unit cell. Thermal ellipsoids enclose 10% probability.

Dewar (1969) from simple theoretical arguments. Nonplanarity has also been observed for the nitrogen atom in several amides, as discussed by Winkler & Dunitz (1971); however, no conclusions could be made about the equilibrium conformation of an undistorted amide group. The C–N bond in Na sulfanilate is significantly shorter than the value of 1·49 (3) Å reported for sulfanilic acid. Sulfanilic acid is a zwitterion compound, in which the C–N bond must be single, while in Na sulfanilate the p_π electrons of the nitrogen atom can conjugate with the π-system of the benzene ring resulting in a partial double C–N bond. A similar feature has been observed in K sulfamate (Cox, Sabine, Padmanabhan, Tu Ban, Chung & Surjadi, 1967) which has a N–S bond length of 1·666 (6) Å compared with 1·76 (2) Å in sulfamic acid (Sass, 1960). Here the double-bond character is due to a π-bond between the nitrogen p_π electrons and the sulfur d-orbitals.

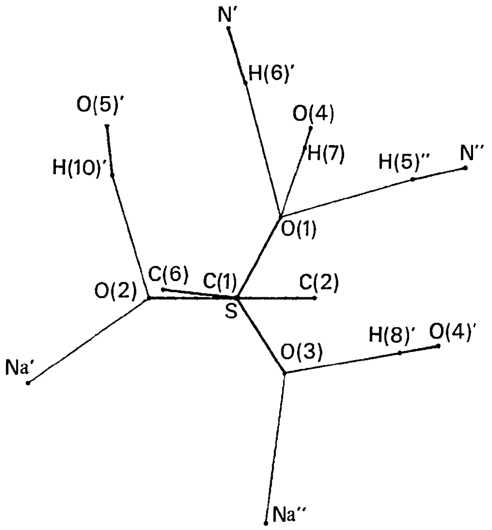

Fig. 2. Newman projection of the SO_3 group with respect to the plane of the benzene ring. The hydrogen bonds and oxygen–sodium bonds are also shown.

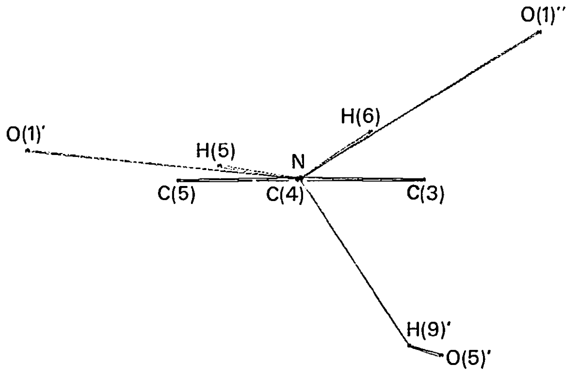

Fig. 3. Newman projection of the NH_2 group with respect to the plane of the benzene ring. The involvement of the group in the hydrogen-bond system is also shown.

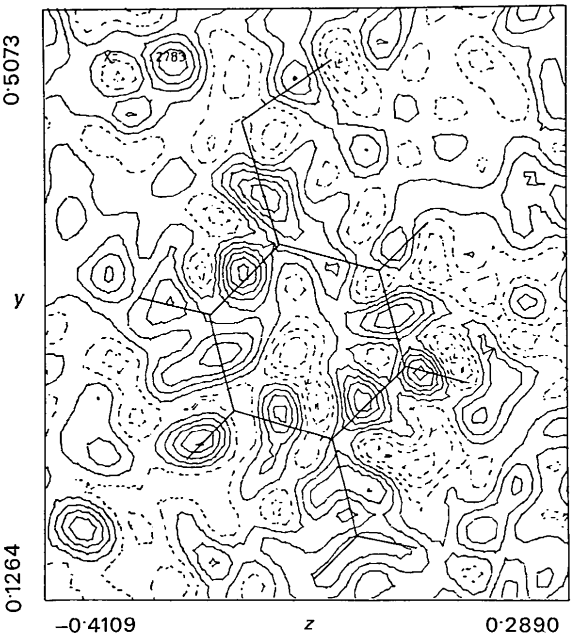

Fig. 4. Final difference Fourier in the plane of the benzene ring. The contour interval is 0·05 e Å^{-3}. Negative contours dotted. Shown are the amino group (below) and the S–O(2) bond (top).

Na sulfanilate dihydrate shows six different hydrogen bonds reported in Table 5. Three are O–H···O bonds with O···O distances in the range of 2·831 to 2·952 Å, two are N–H···O bonds with N···O distances of 3·098 and 3·130 Å and the fifth is an O–H···N bond with an O···N distance of 3·074 Å. The O–H···N bond is much more bent (O–H–N bond angle of 144°) than the other hydrogen bonds, and should therefore be relatively weak.

Finally, Fig. 4 gives a difference Fourier synthesis in the plane of the benzene ring, using the results of the final cycle of the least-squares refinement. Although a difference density synthesis based on room-temperature

Table 5. *Hydrogen bonds* (*X*···H–*Y*)

X···*Y* (Å)		*X*···H (Å)		*X*···H–*Y* (°)	
O(1)···O(4)	2·831 (2)	O(1)···H(7)	2·07 (2)	O(1)···H(7)—O(4)	172 (2)
O(1)···N'	3·098 (2)	O(1)···H(6)'	2·21 (2)	O(1)···H(6)'—N'	172 (2)
O(1)···N''	3·130 (2)	O(1)···H(5)''	2·30 (2)	O(1)···H(5)''–N''	165 (2)
O(2)···O(5)'	2·952 (2)	O(2)···H(10)'	2·11 (2)	O(2)···H(10)'–O(5)'	170 (2)
O(3)···O(4)'	2·907 (2)	O(3)···H(8')	2·09 (2)	O(3)···H(8)'—O(4)'	164 (2)
N·····O(5)''	3·074 (2)	N·····H(9)''	2·41 (2)	N·····H(9)''–O(5)''	144 (2)

X-ray data up to $\sin\theta/\lambda=0{\cdot}70$ Å^{-1} cannot be expected to show bonding features in much detail, the map clearly shows bond populations in the benzene ring and the C–S and S–O(2) bond.

The present work is being continued to study the chemical bonding in Na sulfanilate dihydrate and sulfamic acid at 78 K by a combination of X-ray and neutron diffraction.

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Affinement de la Structure du Bis(pyridyl-2 acétato)cuivre(II) Dihydraté: $[Cu(C_5H_4N\text{–}CH_2\text{–}COO)_2].2H_2O$

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Crystals of bis-(2-pyridineacetato)copper(II) dihydrate, also known as copper(II) 2-pyridyl acetate, are monoclinic, space group $P2_1/c$ with $a=12{\cdot}196$, $b=7{\cdot}259$, $c=18{\cdot}045$ Å, $\beta=114{\cdot}05°$ and $Z=4$. The structure was determined by Faure & Loiseleur [*Acta Cryst.* (1972). B**28**, 2733–2740], from Weissenberg photographs using space group *Pc*. New intensities were collected on an automatic four-circle diffractometer using Cu $K\bar{\alpha}$ radiation and anisotropic full-matrix least-squares refinement was performed considering each molecule as centrosymmetric with respect to the copper atom. The space group is $P2_1/c$; the final *R* is 0·036 for the 2358 observed reflexions. Each copper atom is surrounded by an octahedral arrangement. The base of the octahedron is constituted by the chelating nitrogen atoms (Cu–N 2·00 Å) and the chelating carboxylic oxygen atoms (Cu–O = 1·94 Å) of two symmetrical 2-pyridineacetato groups. The apices of the octahedron are occupied by two carboxylic oxygen atoms belonging to the neighbouring molecules situated at $\pm b/2$ (Cu–O = 2·67 Å). The results are compared with those of 1972.

Introduction

Au cours d'un travail de synthèse sur les relations entre la stabilité et la structure des chélates d'acides pyridinecarboxyliques (Faure, 1973), il est apparu que la technique photographique et le choix du groupe *Pc* utilisés pour la détermination de la structure cristalline du bis(pyridyl-2 acétato)cuivre(II) (Faure & Loiseleur, 1972) ne permettaient pas d'obtenir des résultats suffisamment cohérents pour les longueurs des liaisons métal–coordinat.

De meilleures mesures ont pu être obtenues d'une part grâce à l'utilisation d'un diffractomètre automatique qui nous a permis d'acquérir des intensités plus nombreuses et plus précises en particulier pour les ordres pseudo-éteints (*hkl*, $k=2n+1$ et/ou $l=2n+1$), d'autre part grâce à l'hypothèse justifiée par les résultats, de la centro-symétrie autour de son propre atome de cuivre de chaque molécule du motif asymétrique, et, partant, du choix du groupe d'espace $P2_1/c$.

Données expérimentales

Le chélate $[Cu(C_5H_4N\text{–}CH_2\text{–}COO)_2].2H_2O$ (noté $CuL_2.2H_2O$) présente les caractéristiques cristallographiques suivantes: $a=12{,}196$, $b=7{,}259$, $c=18{,}045$ Å $\beta=114{,}05°$; groupe $P2_1/c$; $V=1459$ Å^3; $M=371{,}6$; $D_m=1{,}69$, $D_c=1{,}693$ g cm^{-3}; $Z=4$; $F(000)=764$; $\mu=$