Acta Cryst. (1969). B25, 2257

The Crystal Structure of Saccharin, *o*-Sulfobenzoimide, $C_6H_4CO.NH.SO_2$, an Artificial Sweetening

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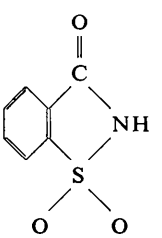
(*Received* 22 *November* 1966 *and in revised form* 27 *January* 1969)

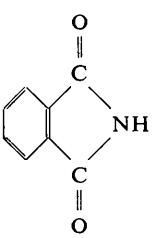
The crystal structure of saccharin, *o*-sulfobenzoimide, an artificial sweetening, was determined by the use of three-dimensional integrated intensity data collected on a computer-controlled diffractometer operated by an IBM 1620 in a closed-loop manner. The crystals are monoclinic with $a=9{\cdot}55_2\pm3$, $b=6{\cdot}91_9\pm3$, $c=11{\cdot}80_3\pm4$ Å, $\beta=103{\cdot}9°$ and the space group is $P2_1/c$. The hydrogen atoms were also located and included in the refinement. The crystal structure is molecular, with centrosymmetric dimer $(C_6H_4CONHSO_2)_2$ molecules; these dimers are formed by N–H---O hydrogen bonds between the imide nitrogen atoms and the keto oxygen atoms, both of the five-membered rings. The six-sided ring formed by the hydrogen bonds around the center of symmetry is completely planar. The location of the hydrogen atom rules out the lactim structure for the molecule. The mode of contact between aromatic rings is normal. The most remarkable feature of the molecular configuration is the narrow C–S–N angle of 92·2° in the five-membered ring. The angle relieves strain from the ring and makes it possible for the whole molecule to become quite planar. Other bond angles, as well as bond distances, are normal.

Introduction

The study is part of a series of structural studies currently undertaken by the author on *ortho*-substituted benzoic acids and their derivatives; the object of the present study is to obtain information about the size and shape of the five-membered ring formed by fusion of a carboxyamide and a sulfonate group and its influence on the overall shape of the benzene ring. It is also of significance to compare the molecular configuration with that of phthalimide, II; the crystal structure of the latter compound was studied by Post & Amendola (1965).



I. *o*-Sulfobenzoimide



II. Phthalimide

Experimental

Crystals of *o*-sulfobenzoimide were obtained from an ethyl alcohol solution of the substance. One of the crystals thus obtained was ground into a sphere of about 0·3 mm in diameter and mounted on a General Electric Goniostat; the *b* axis (the unique axis) of the specimen was set almost parallel to the φ axis of the Goniostat. The unit-cell dimensions of the crystal were obtained on the apparatus by the use of filtered Mo $K\alpha$ radiation; they are: $a=9{\cdot}55_2\pm3$, $b=6{\cdot}91_9\pm3$, $c=11{\cdot}80_3\pm4$ Å, $\beta=103{\cdot}9°$; the space group is $P2_1/c$. The axial ratios obtained from these values are $a:b:c=1{\cdot}380:1:1{\cdot}705$; the ratios are to be compared with the morphological data listed in *Chemische Krystallographie* (Groth, 1912); 2·7867:1:1·7187 and $\beta=103°51{\cdot}5'$. There are four formula units of $C_6H_4CO.NH.SO_2$. Full three-dimensional intensity data within the range of $\sin\theta/\lambda\leq 0{\cdot}85$ were recorded by the CCXD, a computer-controlled X-ray diffractometer operated in a closed-loop manner by an IBM 1620 (Cole, Okaya & Chambers, 1963). For each reflection, the crystal setting and general function of the equipment were first tested by step-scanning around the ω axis of the diffractometer; the integrated intensity data were then recorded by making $(\theta$–$2\theta)$ step scanning. The range of the $(\theta$–$2\theta)$ scanning was chosen in such a way that the first three and the last three of the twenty-four steps represent the background counts at the 2θ value of the reflection. At the ω step scanning stage, the maximum and minimum counts for each reflection were recorded and if the difference between the two counts did not exceed the statistical fluctuation or the basic noise of the counting system, the reflection was treated as a non-observed one. In the $\sin\theta/\lambda$ range studied, about 2300 reflections were recorded as observed. The integrated intensity data were calculated from the data on the $(\theta$–$2\theta)$ step scanning stage as a time-shared program on the 1620. The observed F values derived from the integrated intensity data were then used in the structure analysis of the compound. Owing to the small size and

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the low linear absorption coefficient of the specimen, no absorption correction was applied. The general concept of the experiment-controlling program used in the present study has been presented elsewhere (Okaya, 1966); copies of the actual computer program for the IBM 1620 written in the SPS (symbolic programming system) language may be obtained from the author (Okaya, 1964).

Structure determination and refinement

The crystal structure of *o*-sulfobenzoimide has been determined and refined from the three-dimensional integrated intensity data obtained from the specimen in the manner outlined in the previous paragraph. As the first step towards the structure determination, the position of the sulfur atom was studied by calculating an origin-removed sharpened three-dimensional Patterson function. The positions of the other atoms were determined from an approximate electron density function calcu-

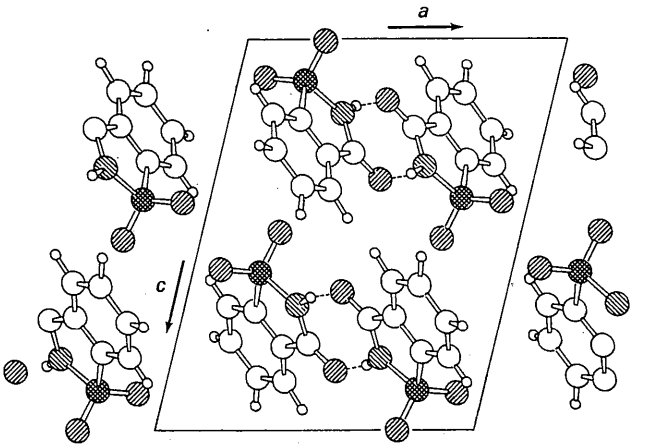

Fig. 1. View of the structure projected along the *b* axis. The N–H···O hydrogen bonds which make dimers are shown by dashed lines. The Figure has been drawn on an IBM 1627 *X–Y* plotter based on a structure drawing program on an IBM 7094 (Okaya, 1968).

Table 1(*a*). *Atomic coordinates in fractions of cell edges and their standard deviations in* 10^{-4} Å.

	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
S	0·21066	5	0·27153	6	0·34614	6
O(1)	0·08420	16	0·15665	24	0·33762	24
O(2)	0·24397	19	0·33049	23	0·24140	19
N	0·35083	18	0·15819	22	0·43090	22
C(O)	0·41360	19	0·25167	23	0·53262	22
O	0·51815	15	0·19105	19	0·60366	18
C(1)	0·22316	20	0·46355	24	0·44561	24
C(2)	0·13539	23	0·62580	29	0·43324	30
C(3)	0·16756	25	0·75874	29	0·52398	34
C(4)	0·28030	27	0·73006	29	0·62086	31
C(5)	0·36758	24	0·56664	27	0·63000	25
C(6)	0·33661	20	0·43415	23	0·53987	23
H(N)	0·387		0·432		0·411	
H(2)	0·060		0·643		0·365	
H(3)	0·114		0·877		0·513	
H(4)	0·296		0·807		0·693	
H(5)	0·444		0·539		0·705	

Table 1(*b*). *Anisotropic thermal parameters*

The β's refer to the expression:

$$\exp\{-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl)\}.$$

The estimated standard deviations are of the order of 10×10^{-5}.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
S	0·00795	0·01227	0·00446	0·00203	−0·00003	−0·00032
O(1)	0·00896	0·02119	0·00952	−0·00638	−0·00152	−0·00184
O(2)	0·01503	0·01908	0·00491	0·00490	0·00327	0·00659
N	0·00945	0·01147	0·00529	0·00612	−0·00072	−0·00253
C(O)	0·00794	0·01004	0·00405	0·00211	0·00191	0·00035
O	0·00949	0·01409	0·00503	0·00802	−0·00076	0·00028
C(1)	0·00812	0·01117	0·00473	0·00327	0·00237	0·00112
C(2)	0·00966	0·01525	0·00704	0·00990	0·00251	0·00351
C(3)	0·01263	0·01308	0·00966	0·01011	0·00860	0·00144
C(4)	0·01445	0·01296	0·00710	0·00381	0·00793	−0·00363
C(5)	0·01154	0·01248	0·00473	0·00209	0·00333	−0·00125
C(6)	0·00805	0·00962	0·00439	0·00304	0·00297	0·00126

Table 1(*c*) *Isotropic temperature factors for hydrogen atoms in* 10^{-16} cm^2

H(N)	H(2)	H(3)	H(4)	H(5)
1·7	1·9	3·9	3·5	2·6

lated by assigning the phases based on the sulfur contribution to the observed structure factors. These positions were then included in subsequent usual calculations. After several cycles of least-squares treatment of the data, depending on the contribution of the non hydrogen atoms with anisotropic thermal parameters, the positions of hydrogen atoms were studied by the usual (F_o–F_c) synthesis method. The atomic coordin-

Table 2. *Comparison between the observed and calculated structure factors*

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
0	0	2	463	468		1	7	146	140							2	5	209	209		3	8	107	105		4	11	66	67
	0	4	448	441		1	8	167	162	10	1	0	146	143		2	6	133	179		3	9	56	59		4	12	66	62
	0	6	28	44		1	10	322	328		1	1	33	30		2	7	30	29		3	10	26	25		4	13	74	75
	0	8	60	60		1	11	33	31		1	2	56	53		2	8	118	128		3	11	99	102		4	14	74	77
	0	10	344	349		1	12	66	63		1	3	40	38		2	9	42	45		3	12	165	164		4	15	17	17
	0	12	19	24		1	13	29	28		1	4	47	49		2	10	39	33		3	13	108	101					
	0	14	16	1		1	14	21	21		1	5	25	14		2	11	47	47						2	4	0	267	259
	0	16	43	51		1	15	13	2		1	6	74	68		2	12	60	60	4	3	0	302	294		4	1	218	209
						1	16	45	46		1	7	7	10							3	1	157	152		4	2	96	89
1	0	0	262	254							1	8	25	31	7	2	0	112	100		3	2	103	91		4	3	64	68
	0	2	166	175	2	1	0	48	42							2	1	127	124		3	3	104	94		4	4	218	207
	0	4	27	50		1	1	115	98	11	1	0	13	21		2	2	246	250		3	4	285	288		4	5	83	77
	0	6	193	183		1	2	78	81		1	1	27	29		2	4	205	206		3	5	154	148		4	6	202	198
	0	8	111	110		1	3	415	393		1	2	60	60		2	5	29	25		3	6	14	14		4	7	21	15
	0	10	378	388		1	4	391	373		1	3	8	13		2	6	69	88		3	7	41	40		4	8	68	50
	0	12	86	88		1	5	285	251		1	4	82	83		2	7	14	22		3	8	109	108		4	9	62	58
	0	14	113	123		1	6	11	0		1	5	8	8		2	9	21	23		3	9	104	100		4	10	99	102
	0	16	27	33		1	7	3	11		1	6	41	48		2	10	62	65		3	10	112	105		4	11	82	83
						1	8	276	279		1	7	54	48		2	11	10	11		3	11	65	66		4	12	59	56
2	0	0	862	882		1	9	101	101							2	12	90	86		3	12	25	19		4	13	25	31
	0	2	788	804		1	10	41	42	12	1	0	8	8							3	13	22	27		4	14	23	23
	0	4	278	262		1	11	18	12		1	1	40	39	8	2	0	86	85		3	14	35	34					
	0	6	139	133		1	12	26	22		1	2	86	83		2	1	96	100						3	4	0	229	215
	0	8	247	251		1	13	32	30		1	4	30	31		2	2	229	230	5	3	0	57	56		4	1	186	173
	0	10	328	334		1	14	74	72							2	3	7	0		3	1	131	117		4	2	251	252
	0	12	112	117						13	1	0	39	41		2	4	103	97		3	2	76	70		4	3	195	181
	0	14	58	55	3	1	0	224	204							2	5	32	28		3	3	283	295		4	4	91	83
						1	1	323	308	0	2	0	972	1065		2	6	14	28		3	4	291	293		4	5	218	215
3	0	0	348	348		1	2	157	162		2	1	162	155		2	7	7	14		3	5	25	28		4	6	176	174
	0	2	65	51		1	3	99	85		2	2	136	122		2	8	105	114		3	6	219	218		4	7	190	186
	0	4	110	114		1	4	225	211		2	3	291	265		2	9	22	29		3	7	51	47		4	8	171	171
	0	6	141	134		1	5	26	1		2	4	276	263		2	10	20	29		3	8	115	107		4	9	76	75
	0	8	280	285		1	6	71	67		2	5	117	135							3	9	11	11		4	10	112	107
	0	10	23	19		1	7	74	73		2	6	181	208	9	2	0	18	1		3	11	21	25		4	11	59	57
	0	12	28	34		1	8	200	195		2	7	252	277		2	1	44	39		3	12	59	62		4	12	42	40
	0	14	28	30		1	9	24	17		2	8	135	150		2	2	128	126		3	13	32	32		4	13	28	30
						1	10	123	124		2	9	116	120		2	3	18	17							4	14	65	65
4	0	0	43	62		1	11	22	21		2	10	116	110		2	4	30	29	6	3	0	246	251					
	0	2	637	623		1	12	140	137		2	11	35	31		2	5	51	50		3	1	33	32	4	4	0	224	216
	0	4	61	41		1	13	50	45		2	12	107	111		2	6	69	106		3	2	135	130		4	1	415	416
	0	6	144	133		1	15	20	12		2	13	24	27		2	7	11	17		3	3	56	56		4	2	218	220
	0	8	43	44							2	16	90	85		2	8	19	27		3	4	280	287		4	3	54	55
	0	10	9	18	4	1	0	127	116												3	5	63	54		4	4	41	39
	0	12	135	135		1	1	197	183	1	2	0	30	22	10	2	0	110	107		3	6	36	31		4	5	102	99
	0	14	23	36		1	2	161	142		2	1	169	150		2	1	13	18		3	7	16	11		4	6	165	163
						1	3	158	126		2	2	115	111		2	3	98	99		3	8	18	14		4	7	21	27
5	0	0	163	163		1	4	211	190		2	3	282	254		2	4	123	121		3	9	27	30		4	8	122	122
	0	2	178	163		1	5	12	3		2	4	348	328		2	6	34	46		3	10	107	102		4	9	9	13
	0	4	73	72		1	6	169	164		2	5	45	73		2	8	48	52							4	10	10	7
	0	6	90	89		1	7	100	134		2	6	16	21						7	3	0	104	101		4	11	37	38
	0	8	147	150		1	8	249	243		2	7	28	31	11	2	0	20	21		3	1	113	114		4	12	104	104
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6	0	2	415	404		1	13	49	44		2	12	83	85		2	5	30	31		3	6	17	18		4	2	119	121
	0	4	182	174		1	14	44	40		2	14	111	110		2	6	52	73		3	7	22	18		4	3	159	164
	0	6	135	125							2	15	40	39							3	8	135	132		4	5	111	103
	0	8	142	139	5	1	0	205	207		2	16	33	32	12	2	0	32	31							4	6	185	182
	0	10	9	5		1	1	228	206							2	1	12	6	8	3	0	51	52		4	7	82	86
	0	12	110	115		1	2	356	352	2	2	0	275	280		2	2	25	25		3	1	9	10		4	8	17	16
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7	0	0	390	392		1	4	138	132		2	2	419	411		2	4	72	70		3	3	95	91		4	10	161	159
	0	2	136	132		1	5	65	70		2	3	223	209							3	4	64	59		4	11	80	82
	0	4	213	206		1	6	236	235		2	4	128	110	13	2	0	11	11		3	5	97	92		4	12	23	16
	0	6	180	183		1	7	52	62		2	5	127	137		2	1	18	14		3	6	148	142					
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	0	8	135	131		1	7	58	59		2	6	111	143							3	3	17	9		4	5	51	52
	0	10	90	96		1	8	9	7		2	7	36	40	1	3	0	120	116		3	4	34	31		4	6	79	78
						1	10	155	143		2	8	239	268		3	1	401	375		3	5	45	40		4	7	6	5
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	0	6	73	76	7	1	0	131	122		2	12	13	7		3	5	204	188		3	1	10	12					
	0	8	64	59		1	1	43	39		2	14	70	70		3	6	235	224		3	2	23	25	8	4	0	106	105
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11	0	0	54	54		1	3	40	43							3	8	5	1		3	4	59	60		4	3	123	121
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	0	4	35	35		1	5	110	116		2	2	369	365		3	10	156	153							4	6	11	1
	0	6	17	13		1	6	24	24		2	3	19	22		3	11	30	36	12	3	0	8	9		4	7	47	42
						1	7	41	43		2	4	25	11		3	12	84	85		3	1	36	39		4	8	94	83
12	0	0	30	26		1	8	148	147		2	6	219	278		3	13	34	33		3	2	50	55		4	9	15	14
	0	4	66	62		1	9	19	11		2	7	54	55		3	14	50	46		3	3	34	37					
						1	10	87	81		2	8	84	99		3	15	26	22						9	4	0	35	34
13	0	2	67	67		1	11	49	46		2	9	50	53						0	4	0	264	250		4	1	44	44
						1	12	99	98		2	11	72	73	2	3	0	89	85		4	1	162	160		4	2	86	86
0	1	1	365	363							2	12	92	90		3	1	103	92		4	2	90	79		4	3	21	15
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	1	4	459	469		1	2	155	156							3	4	221	217		4	5	21	23		4	6	58	62
	1	5	26	25		1	3	26	23	5	2	0	166	158		3	5	223	208		4	6	249	246		4	7	24	21
	1	6	258	243		1	4	61	56		2	1	11	11		3	6	136	131		4	7	99	88					
	1	7	56	27		1	6	145	144		2	2	25	18		3	7	193	187		4	8	79	81	10	4	0	92	89
	1	8	355	350		1	7	16	12		2	3	19	4		3	8	124	116		4	9	87	90		4	1	54	52
	1	9	93	92		1	8	51	47		2	4	114	115		3	9	41	44		4	10	91	89		4	2	48	44
	1	10	113	111		1	9	14	16		2	5	44	66		3	10	25	25		4	11	7	5		4	3	81	85
	1	11	116	113		1	10	104	96		2	6	89	112		3	11	89	92		4	12	24	24		4	4	70	71
	1	12	160	158		1	11	6	2		2	7	123	150		3	12	22	25		4	13	68	72		4	5	57	57
	1	13	47	43							2	8	23	21		3	13	20	22										
	1	14	47	44	9	1	0	180	174		2	9	78	83		3	14	88	88	1	4	0	87	80	11	4	0	36	35
	1	15	29	24		1	1	66	67		2	10	170	168		3	15	39	37		4	1	41	23		4	1	97	103
	1	16	62	55		1	2	112	114		2	11	81	77							4	2	25	24		4	2	68	72
						1	3	11	7		2	12	60	57	3	3	0	50	33		4	3	124	107		4	3	18	19
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	1	5	14	7		1	9	36	30		2	3	9	8		3	6	224	211		4	9	13	9					
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Table 2 (*cont.*)

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	5	6	161	163
	5	7	130	133
	5	8	39	40
	5	9	95	93
	5	10	24	25
	5	11	30	33
	5	12	82	79
	5	13	49	46
	5	14	18	17
1	5	0	58	53
	5	1	237	235
	5	2	46	50
	5	3	80	77
	5	4	251	137
	5	5	127	125
	5	6	146	145
	5	7	157	160
	5	8	136	135
	5	9	115	112
	5	10	55	56
	5	11	12	14
	5	12		
2	5	0	69	67
	5	1	112	108
	5	2	169	164
	5	3	168	167
	5	4	258	256
	5	5	26	26
	5	6	86	86
	5	7	88	90
	5	8	128	127
	5	9	48	45
	5	10	23	24
	5	11	24	22
	5	12	31	33
	5	13	11	11
3	5	0	111	109
	5	1	166	162
	5	2	243	233
	5	3	17	16
	5	4	268	272
	5	5	58	60
	5	6	32	30
	5	7	110	105
	5	8	139	139
	5	9	52	49
	5	10	21	23
	5	11	108	107
	5	12	90	85
	5	13	39	39
4	5	0	238	250
	5	2	127	126
	5	3	41	40
	5	4	140	139
	5	5	93	94
	5	6	107	111
	5	8	40	40
	5	9	120	121
	5	10	103	103
	5	11	15	23
	5	12	10	0
4	5	0	58	53
	5	1	122	117
	5	2	77	75
	5	3	122	120
	5	4	58	60
	5	5	5	3
	5	6	65	67
	5	7	52	55
	5	8	124	123
	5	9	55	50
	5	10	27	24
	5	11	62	60
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	5	1	45	47
	5	2	57	58
	5	3	77	76
	5	4	39	34
	5	5	32	32
	5	6	113	117
	5	8	18	22
	5	9	48	44
	5	10	81	77
7	5	0	36	33
	5	1	30	28
	5	3	53	53
	5	4	98	98
	5	6	37	36
	5	7	11	4
	5	8	83	85
	5	9	65	62
8	5	0	61	60
	5	1	86	90
	5	2	15	2
	5	3	17	23
	5	5	68	71
	5	6	61	64
	5	7	44	41
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	5	1	7	3
	5	2	13	12
	5	3	94	101
	5	4	61	63
	5	7	36	38
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	5	1	66	72
	5	2	16	17
	5	3	13	9
	5	5	19	20
11	5	0	49	58
	5	2	18	12
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	6	1	233	241
	6	2	92	97
	6	3	166	169
	6	4	62	61
	6	5	125	123
	6	6	190	189
	6	7	11	3
	6	8	55	54
	6	9	41	40
	6	10	67	66
	6	11	83	82
	6	12	57	55
	6	13	31	29
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	6	1	162	168
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	6	3	29	25
	6	4	217	214
	6	5	9	11
	6	6	132	128
	6	7	54	50
	6	8	7	6
	6	9	150	151
	6	10	8	13
	6	13	52	51
2	6	0	141	142
	6	1	170	177
	6	2	126	129
	6	3	55	55
	6	4	233	234
	6	5	85	86
	6	6	115	115
	6	7	114	112
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	6	9	18	17
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	6	12	37	34
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	6	6	62	65
	6	7	22	16
	6	8	57	59
	6	9	70	72
	6	10	31	31
	6	12	27	22
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	6	2	161	165
	6	3	48	46
	6	5	48	55
	6	6	110	111
	6	7	70	71
	6	8	98	101
	6	9	4	2
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	6	11	13	13
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	6	3	56	54
	6	4	43	44
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	6	9	38	41
	6	10	38	41
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	6	3	52	56
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	6	6	9	5
	6	7	92	96
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	6	4	25	23
	6	5	17	20
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	6	2	54	55
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	6	2	40	41
	6	3	19	17
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	7	2	57	56
	7	3	147	150
	7	4	104	101
	7	6	134	106
	7	7	101	109
	7	8	74	75
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	7	3	62	67
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	7	9	106	106
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	7	11	39	36
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	7	9	81	84
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	7	2	45	47
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	7	4	77	81
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	7	5	43	43
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	7	1	7	1
7	7	0	7	9
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	7	3	61	64
	7	4	78	82
	7	5	43	43
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	8	2	18	24
	8	3	61	64
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	8	6	63	69
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	8	9	12	19
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	8	1	55	58
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	0	4	161	162
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	0	8	262	256
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	0	4	160	152
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	0	8	91	88
	0	10	135	134
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	0	16	35	31
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	0	4	83	83
	0	6	163	158
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	0	6	43	45
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	1	9	151	164
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	1	11	92	86
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	1	3	191	196
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	2	4	112	114
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	2	14	60	57
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	2	8	139	142
	2	9	68	71
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	2	11	53	54
	2	12	32	31
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	2	4	204	203
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	2	5	42	45
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	2	10	32	31
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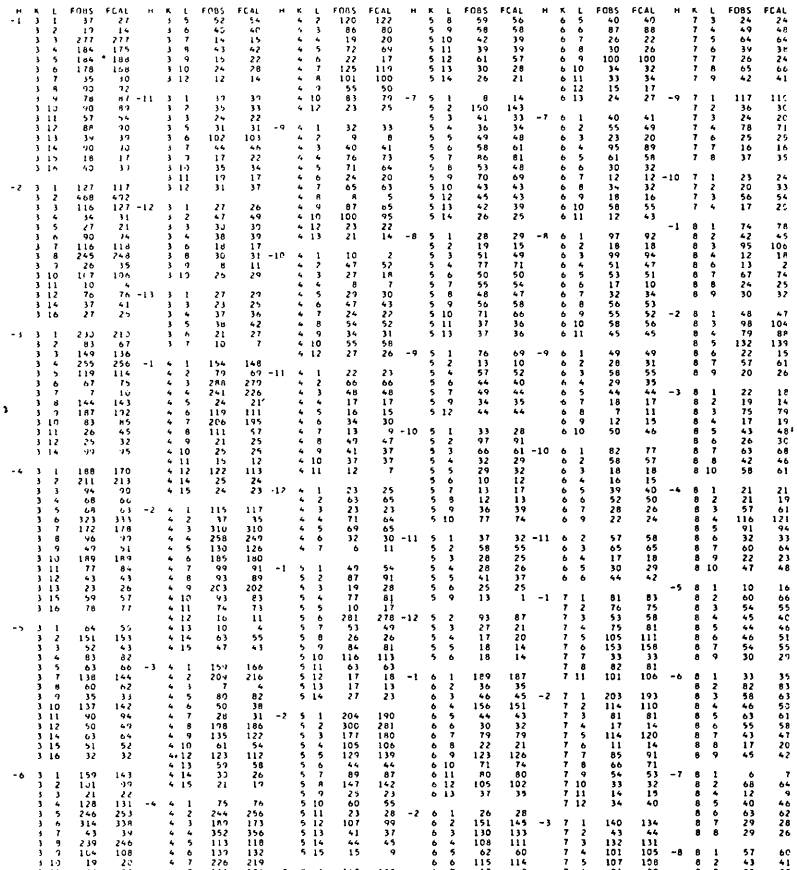
	2	9	15	14

Table 2 (*cont.*)



ates of the hydrogen atoms thus obtained were then included in the least-squares treatment by assigning isotropic temperature factors to account for their thermal motion.

The atomic coordinates, their standard deviations and thermal parameters after five further cycles of least-squares treatment are shown in Table 1. The final conventional error index $R=\Sigma\,||F_o|-|F_c||/\Sigma\,|F_o|$ was 0·050 (or 0·059 if unobserved reflections are included with $F_{obs}=0$). Comparison between the observed and calculated structure factors is given in Table 2. The following are some of the computational details. All the analyses were performed on an IBM 7094 at the IBM Research Center. The atomic scattering factors

used in the computation were those for the neutral atoms listed in *International Tables for X-ray Crystallography* (1962). The parameter shifts at the last stage of the refinement were almost negligible compared with their standard deviations. The weighting scheme used in the least-squares treatment was: $\omega = 1{\cdot}0$ for $|F_{obs}| \leq$ $\omega = 20{\cdot}0/|F_{obs}|$ for $|F_{obs}| \geq 20{\cdot}0$ and zero weight for the non-observed reflections. Interatomic distances and bond angles were calculated from the coordinates in Table 1; they are the basis for the subsequent discussions.

Discussion of the structure

The crystal structure of *o*-sulfobenzoimide is a molecular one and consists of centrosymmetric dimer molecules $(C_6H_4.CO.NH.SO_2)_2$ formed by N–H...O hydrogen bonds of $2{\cdot}79_6$ Å around centers of symmetry. These hydrogen bonds are formed between the hydrogen atoms (or protons) on the imino nitrogen atoms and the ketone oxygen atoms of the mate molecules. A similar dimer formation is also found in the structure of phthalimide, C_6H_4 CO.NH.CO, II. The dimeric molecules thus formed pack in the structure by the usual mode of contact between aromatic rings with normal van der Waals distances. Fig. 1 shows the structure viewed down the *b* axis, illustrating the packing of the molecules in the crystal. Fig. 2 demonstrates the geometry of the six-membered ring formed around the center of symmetry by the two N–H...O hydrogen bonds; the ring is completely planar as is shown by the deviations of atoms from the least-squares plane. The N–H...O hydrogen bond is almost linear with an angle of 170° around the proton. A similar completely planar configuration is found in the centrosymmetric dimer of ε-caprolactam, $C_5H_{10}CONH$, (Tomiie, Okaya & Nitta, 1964); in this crystal the dimer is again formed by N–H...O hydrogen bonds.

Bond distances and angles in the molecule are shown in Figs. 3 and 4. The most important feature in the shape of the molecule is the sharp C(1)–S–N angle of 92·2°. The value is much smaller than the tetrahedral angle of 109·5°, and no doubt relieves strain from the five-membered ring; the effect is demonstrated in the complete planarity of the molecule as shown in Fig. 5. This is in contrast to the configuration of phthalimide, C_6H_4 CO.NH.CO, where no such sharp angle can be formed in the five-membered ring which is therefore less planar than that in the sulfoimide molecule. The O(1)–S–O(2) angle of 117·7° now becomes larger than the usual tetrahedral angle with the remaining four angles being close to the normal value. As is expected, the angles around C(1), C(6) and C(O) in the five-membered ring are still smaller than the normal angles found in conjugated systems. The angles in the benzene ring, especially those around C(2) and C(5) demonstrate that the ring takes a partial quinoid structure. This situation is also illustrated in the short C(1)–C(6) bond distance. The benzene ring in the phthalimide structure also exhibits the quinoid structure; in this molecule, the corresponding C(1)–C(6) and C(3)–C(4) bonds become shorter than the remaining C–C bonds. However, there is no clear-cut explanation for the normal C(3)–C(4) bond length in the *o*-sulfoimide structure. Apart from the nature of the benzene ring, other bond distances are normal. For example, the average of the five regular C–C distances in the benzene ring is $1{\cdot}38_8$ Å and the bond distances in the five-membered ring all have values expected in view of possible resonance formulas and the hydrogen-bond formation. The position of the hydrogen atom belonging to the five-membered ring indicates that the molecule takes the lactam form, I, and is not a lactim, III, at least not in the crystalline state. The next-neighbor N–O(1) and N–O(2) distances of 2·52 Å are unique; separations of this length can

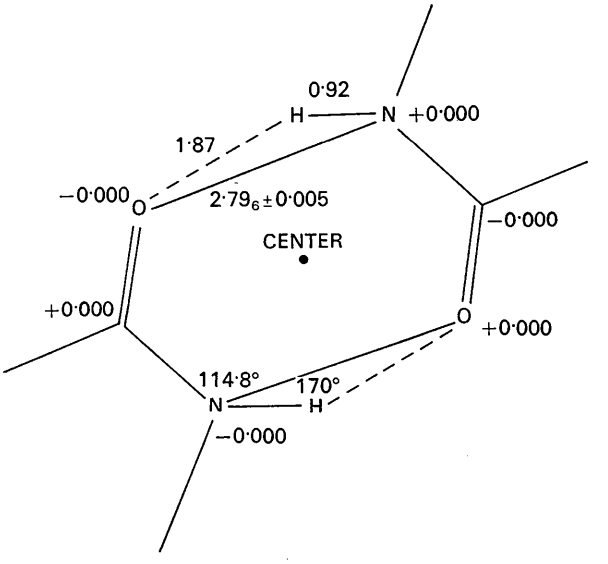


Fig. 2. The centrosymmetric dimer configuration. The six-membered ring formed around the centers of symmetry. Deviations of atoms from the least-squares plane are also shown; the equation is $0{\cdot}7363X + 0{\cdot}4883Y - 0{\cdot}4685Z + 0{\cdot}2104 = 0$, where $X = ax + cz \cos\beta$, $Y = by$ and $Z = cz \sin\beta$.

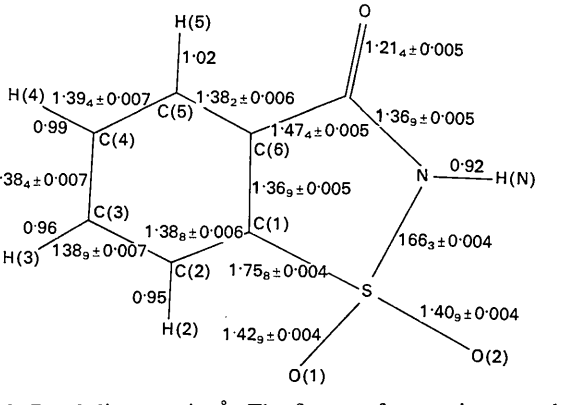


Fig. 3. Bond distances in Å. The figures after ± signs are the corresponding e.s.d.'s.

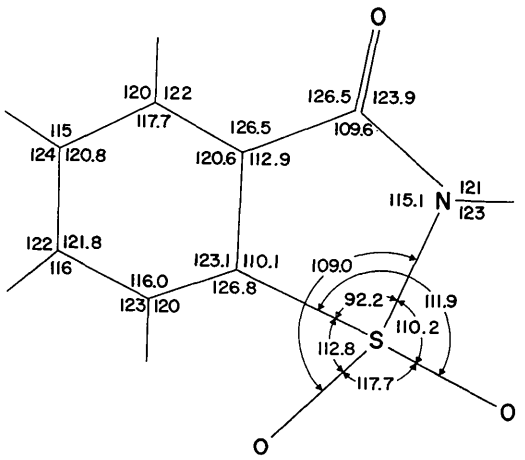


Fig. 4. Bond angles in degrees.

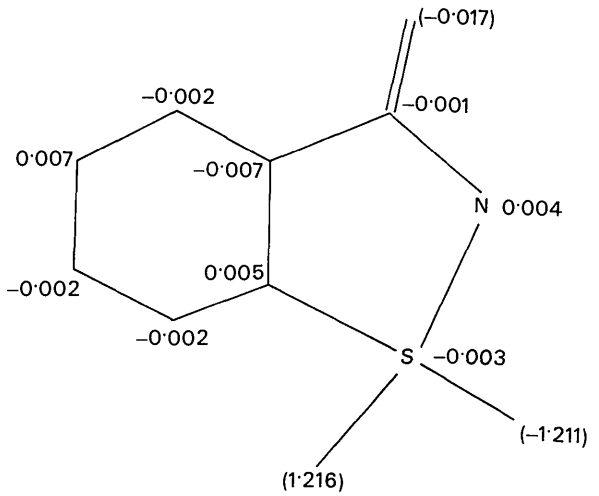
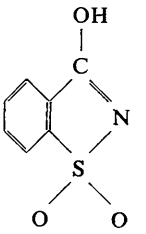


Fig. 5. Deviations of atoms from the plane of the molecule. The equation of the plane is $0{\cdot}7451X+0{\cdot}4814Y-0{\cdot}4616Z+0{\cdot}1603=0$. The atoms with deviations in parentheses were not included in the evaluation of the equation.

occur when the nitrogen atom acts as a bridge between a tetrahedral sulfur atom and the remaining part of a molecule. This N–O configuration in conjunction with the ketone oxygen atom might play some role in the physiological activities of the molecule.



III

The anisotropic thermal parameters for each atom were decoded into its vibration ellipsoid. The atoms undergo vibrations with large amplitudes in the directions perpendicular to the plane of the molecule.

The author is grateful to N. R. Stemple for his help in collecting the three-dimensional diffractometer data used in the structure analysis. He also wishes to thank Dr G. Hamor of the University of Southern California for interesting discussions on the physiological importance of derivatives of *o*-sulfobenzoimide.

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