Table 4. *Geometry of hydrogen bonds*

X–H···O	*d*(X···O)	*d*(X–H)	*d*(H···O)	angle at H
O(1)—H(1)···O(2,I)	2·667 (2) Å	0·82 (3) Å	1·85 (3) Å	173 (3)°
O(3)—H(3)···O(2,II)	2·591 (2)	0·93 (3)	1·67 (3)	177 (3)
O(4)···H(4)···O(5)	2·424 (2)	1·25 (4)	1·18 (4)	173 (4)
N(1)–H(11)···O(4,III)	2·935 (3)	0·79 (3)	2·15 (3)	175 (3)
N(1)–H(12)···O(5,II)	3·130 (3)	0·82 (3)	2·40 (3)	149 (3)
N(1)–H(12)···O(1,IV)	3·160 (3)	0·82 (3)	2·81 (3)	108 (2)*
N(2)–H(21)···O(2)	2·979 (3)	0·88 (3)	2·10 (3)	177 (3)
N(2)–H(21)···O(3,V)	2·995 (3)	0·88 (3)	2·87 (3)	89 (3)*
N(2)–H(22)···O(3,V)	2·995 (3)	0·91 (3)	2·70 (2)	100 (3)*
N(2)–H(22)···O(5,II)	3·099 (3)	0·91 (3)	2·40 (3)	154 (3)

I: $\frac{1}{2}-x,\ \frac{1}{2}+y,\ z$
II: $x,\ \frac{1}{2}-y,\ \frac{1}{2}+z$
III: $-x,\ \frac{1}{2}+y,\ \frac{1}{2}-z$
IV: $-x,\ -y,\ -z$
V: $\frac{1}{2}-x,\ -y,\ \frac{1}{2}+z$

In view of their large *d*(H...O) and small angles at the proton the interactions marked* should probably not be considered as 'normal' hydrogen bonds.

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The Crystal Structure of 3,4-Epoxysulfolane

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(*Received* 14 *January* 1972)

The compound 3,4-epoxysulfolane, $C_4H_6OSO_2$, crystallizes in the orthorhombic system with four molecules in a unit cell of symmetry $P2_12_12_1$ and dimensions $a=8{\cdot}475\ (6)$, $b=10{\cdot}742\ (5)$, $c=5{\cdot}899\ (4)$ Å. Least-squares refinement of 1282 observed structure factors, measured on a Pailred diffractometer, gave a residual of 0·055. The molecule is folded so that the epoxide linkage is nearly normal to the mean plane of the sulfur and carbon atoms. Analysis of the thermal motion by the rigid-body method of Schomaker and Trueblood shows that the least librational oscillation is about the normal to the sulfur–carbon mean plane.

Introduction

Compounds with the formula SO_2X_2 may be sulfuryl halides, such as SO_2F_2, or symmetrically substituted sulfones, such as dimethyl sulfone, $(CH_3)_2SO_2$. The sulfur atom in these molecules is tetrahedrally coordinated, and the simplest prototypes have C_{2v} symmetry. We have previously determined the structures of dimethyl sulfone (Sands, 1963) and butadiene sulfone, $C_4H_6SO_2$ (preferred name is 2,5-dihydrothiophene-1,1-dioxide, also known as 3-sulfolene) (Sands & Day, 1967). The present paper reports the structure of 3,4-epoxysulfolane, $C_4H_6OSO_2$ (alternative names are butadiene sulfone epoxide and sulfolene-3-epoxide).

Experimental

The epoxidation of butadiene sulfone was accomplished by treatment with performic acid, as described by Sorenson (1959). The product was recrystallized from acetone and it melted in the range 123–125 °C. A preparation by another method by van Lohuizen & Backer (1949) was reported to melt at 130 °C, and Procházka & Horák (1959) observed a melting point in the range 124·5–126°. Sorenson (1959), however, observed a melting point of 159–160° and suggested the possible existence of two crystalline modifications, although he was unable to convert his product into a second crystalline form. Subsequently, Loev (1961) reported the isolation of two epoxides with melting points of 123–125° and 157–159° from the same reaction mixture; interconversion of these two crystalline forms was not possible.

Mass spectrometric examination of our product showed the presence of the masses reported by Weinberg, Stafford & Scoggins (1968), although the intensity distributions were rather different. Our spectrum also possessed a substantial peak corresponding to a mass of 135, whereas the parent peak of the compound should be at mass 134. High resolution measurements gave a mass of 135·0116 for this peak, which may be compared with 135·0120 expected for $C_4H_7SO_3$. It was subsequently found that upon lowering the injection temperature from 200° to 140 °C the 135 peak diminished and the 134 peak became stronger. A likely explanation of these spectra is that the compound polymerizes at the higher temperatures, and this may also be the source of the melting point ambiguity.

The single crystal selected for the structure determination was a clear, colorless, rectangular parallelepiped of dimensions 0·22 × 0·19 × 1·26 mm along directions that were later identified with the crystallographic *a*, *b* and *c* axes. Weissenberg and precession photographs showed orthorhombic symmetry, and systematic absences suggested space group $P2_12_12_1$, which was ultimately confirmed by the structure.

Accurate cell dimensions were obtained from measurements made with a Philips Pailred diffractometer using Mo $K\alpha$ radiation ($\lambda = 0·70930$ Å). These measure-

Table 1. *Observed and calculated structure factor magnitudes*

The columns are *h*, $10|F_o|$, and $10|F_c|$. The value of 10 *F*(000) is 2800.

H,0,0		
2	159	158
4	358	364
6	31	28
10	36	37
12	64	59
14	42	38
H,1,0		
2	388	416
3	109	118
4	259	256
5	135	146
6	226	225
7	26	42
8	71	81
9	161	164
10	52	49
12	52	49
13	43	39
H,2,0		
0	447	476
1	630	711
2	103	102
3	319	341
4	21	25
5	266	263
6	58	68
7	69	70
10	54	59
11	68	67
15	30	35
H,3,0		
1	373	387
2	105	95
3	408	407
4	42	44
5	61	62
6	386	398
7	115	114
8	121	131
10	84	85
12	53	51
13	27	27
H,4,0		
0	580	594
1	313	305
2	288	285
3	141	149
4	268	260
6	61	65
7	77	84
8	29	29
10	34	33
12	51	52
14	42	40
H,5,0		
1	114	119
2	253	261
5	290	301
6	16	24
7	99	103
8	24	29
9	114	117
10	39	42
11	28	31
H,6,0		
0	179	190
1	260	251
2	309	309
3	167	172
4	23	31
5	32	40
6	103	106
7	137	133
9	35	37
11	27	32
12	32	38
15	25	15
H,7,0		
4	93	94
5	25	25
6	217	219
8	148	148
9	38	39
10	120	119
2	30	19
H,8,0		
0	97	92
1	185	200
2	29	36
3	68	71
4	169	173
5	43	47
6	48	48
7	61	64
8	123	118
9	32	22
H,9,0		
1	69	70
2	24	32
4	112	113
5	186	185
7	35	42
8	69	69
9	115	115
13	38	30
14	21	11
H,10,0		
0	302	305
1	36	39
2	31	32
3	157	157
4	146	148
7	87	86
11	31	28
H,11,0		
1	18	19
2	164	167
4	48	50
6	124	124
10	33	37
H,12,0		
1	109	113
2	28	29
3	106	110
4	46	49
5	66	62
6	30	36
8	61	61
11	34	28
H,13,0		
1	106	106
2	33	38
3	108	110
4	26	27
5	48	52
6	33	43
7	33	28
9	62	56
H,14,0		
0	166	162
1	66	63
2	35	37
3	24	23
4	66	65
5	23	26
8	26	21
10	32	25
H,15,0		
2	95	98
3	20	24
H,16,0		
1	76	76
3	55	55
4	35	39
5	36	34
7	23	25
H,17,0		
1	37	40
3	35	34
4	33	30
5	27	28
6	25	23
8	23	17
H,18,0		
0	28	31
4	49	52
H,19,0		
2	44	40
5	24	29
H,0,1		
2	196	184
3	52	62
4	181	173
5	139	138
6	175	178
7	264	262
8	182	178
9	41	44
10	68	69
11	82	77
12	61	58
H,1,1		
2	432	447
3	237	244
4	199	191
5	137	136
6	188	185
7	67	65
8	58	65
9	44	42
10	54	56
12	33	35
13	26	24
14	22	19
15	24	11
H,2,1		
1	86	91
2	334	326
3	167	157
4	260	255
5	147	149
6	119	122
7	86	89
8	144	140
9	85	87
10	30	35
11	27	29
12	61	60
H,3,1		
0	519	520
1	426	426
2	382	381
3	144	141
4	91	94
5	139	151
6	150	144
7	45	45
9	47	48
10	31	36
11	36	37
12	42	43
13	47	43
14	25	24
15	27	21
H,4,1		
1	53	58
2	274	260
3	238	234
4	103	98
5	174	170
6	222	221
7	175	174
9	29	34
10	31	38
11	65	61
12	31	35
H,5,1		
0	80	75
1	109	110
2	234	224
3	229	218
4	57	59
5	44	42
6	185	180
7	66	59
8	59	60
9	58	58
10	33	35
11	37	34
12	58	53
H,6,1		
0	227	214
1	71	69
3	80	82
4	144	142
5	109	109
6	208	202
7	97	97
8	107	105
9	92	88
10	49	44
11	44	41
12	43	42
14	27	19
H,7,1		
0	360	347
1	245	231
2	69	69
3	130	134
4	117	116
5	106	100
6	34	33
8	34	33
9	73	68
10	30	34
12	27	20
13	30	29
H,8,1		
0	207	195
1	66	70
2	79	76
3	130	124
4	83	80
5	65	65
6	102	97
7	87	85
8	57	54
9	76	74
10	36	39
11	38	40
12	31	33
H,9,1		
0	133	131
1	131	130
2	205	200
3	130	130
4	38	39
5	60	65
6	114	108
7	47	49
8	45	48
9	26	30
10	40	37
11	27	13
12	30	26
H,10,1		
0	48	48
1	72	73
2	23	24
3	102	96
4	117	113
5	78	75
6	69	64
7	101	99
8	64	65
10	39	34
11	33	30
12	31	28
H,11,1		
0	41	40
1	113	109
2	38	41
3	91	93
4	75	75
5	97	91
6	44	47
7	48	53
8	27	31
9	36	37
10	29	26
H,12,1		
0	86	82
1	92	93
2	87	84
3	64	63
4	102	102
5	44	40
6	21	23
7	32	33
8	45	46
10	26	25
H,13,1		
1	61	60
2	127	124
3	29	31
5	23	25
6	52	48
7	33	33
8	24	28
H,14,1		
0	50	53
1	65	66
2	64	62
3	57	55
4	28	32
7	60	55
8	31	28
10	30	19
H,15,1		
0	42	50
1	55	58
2	35	38
3	50	48
4	39	40
5	46	45
8	21	25
9	25	28
H,16,1		
0	30	45
3	25	29
4	41	45
6	24	22
8	28	34
H,17,1		
0	30	31
1	24	24
2	39	38
4	38	30
6	39	34
H,18,1		
2	21	25
3	41	43
H,19,1		
2	32	22
H,20,1		
0	26	28
1	22	16
H,0,2		
2	127	127
3	29	33
4	113	112
5	198	208
6	24	33
8	94	96
9	74	78
10	56	62
11	55	49
H,1,2		
1	426	437
2	234	231
3	174	168
4	122	121
5	76	78
6	195	199
7	200	198
8	97	96
9	37	38
10	36	35
11	73	71
12	55	56
13	21	12
H,2,2		
0	92	88
1	331	326
2	110	122
3	243	235
4	65	63
5	230	223
6	264	266
7	113	116
8	57	60
10	70	69
11	29	30
12	46	45
H,3,2		
0	180	165
1	119	113
2	274	266
3	168	154
4	204	200
5	196	192
6	72	75
7	176	177
8	95	96
9	30	35
10	32	37
11	69	68
12	24	21
H,4,2		
0	177	169
1	279	275
2	205	202
3	104	107
4	265	259
5	220	207
6	100	101
7	62	67
8	73	70
9	50	54
10	26	22
12	53	44
13	45	46
H,5,2		
0	490	480
1	169	167
2	115	111
3	132	130
4	245	232
5	107	107
6	55	53
7	80	82
8	51	53
9	40	39
10	22	27
11	28	30
12	58	48
13	41	40
H,6,2		
0	41	46
1	267	254
2	338	330
3	134	126
4	124	126
5	43	48
6	107	102
7	70	67
8	57	56
10	47	48
11	32	37
12	22	23
13	39	32
H,7,2		
1	269	258
2	150	147
3	121	121
4	60	58
5	74	80
6	35	34
7	132	132
8	42	40
10	35	36
11	64	62
H,8,2		
0	50	53
1	168	162
2	144	149
3	217	210
4	92	93
5	71	75
6	64	68
7	86	81
8	48	49
9	44	46
10	35	34
11	32	23
13	31	24
H,9,2		
0	214	201
1	124	126
2	35	34
3	85	79
4	139	135
5	72	75
6	44	46
7	67	67
8	61	64
10	48	38
11	22	19
12	35	32
H,10,2		
1	120	116
2	165	156
3	75	77
4	43	44
5	93	89
6	47	51
7	31	33
8	19	27
9	24	24
10	27	25
H,11,2		
0	71	66
1	68	72
2	101	99
3	138	139
4	34	35
5	49	52
6	66	68
7	63	62
8	24	25
11	31	34
H,12,2		
0	75	70
1	51	52
2	59	60
3	73	72
4	63	61
5	63	60
6	82	79
8	22	23
9	46	41
H,13,2		
0	76	78
1	40	44
3	49	46
4	73	75
5	34	35
6	27	39
7	30	35
8	67	61
9	28	25
10	22	18
H,14,2		
0	48	52
1	40	46
2	31	34
3	35	36
4	35	40
5	56	54
9	37	41
H,15,2		
0	37	44
2	35	41
3	60	56
6	44	39
7	41	47
8	23	21
9	23	17
H,16,2		
2	62	59
3	21	22
5	23	30
6	27	38
7	30	25
H,17,2		
3	28	28
4	42	42
7	26	21
H,18,2		
1	38	29
4	27	20
5	31	31
H,19,2		
0	23	20
1	26	23
H,0,3		
2	275	260
4	118	118
6	169	161
7	73	70
8	92	94
10	123	111
H,1,3		
2	67	62
3	62	64
4	160	158
5	158	153
6	66	61
7	193	185
8	68	66
10	50	41
11	75	65
H,2,3		
0	86	62
1	371	359
2	208	197
3	121	116
4	91	86
5	255	245
6	48	50
9	79	79
10	34	33
11	58	56
13	64	56
H,3,3		
0	360	350
1	53	57
2	43	43
3	146	142
4	268	264
5	108	112
6	37	38
7	60	55
8	72	72
10	49	37
13	26	10
H,4,3		
0	84	76
1	237	221
2	172	172
3	44	45
4	151	144
5	99	89
6	165	156
8	82	81
10	69	66
11	24	28
12	43	33
13	45	28
14	28	32
H,5,3		
0	91	92
1	245	235
2	21	34
3	115	108
4	37	39
5	291	291
6	43	43
7	122	113
8	51	55
11	41	38
12	36	34
13	33	28
H,6,3		
0	26	27
1	225	222
2	76	73
3	142	145
4	69	69
5	128	123
6	72	74
7	24	26
8	32	38
9	81	80
10	36	38
11	36	33
H,7,3		
0	326	319
1	57	60
2	66	70
3	72	78
4	162	163
5	63	62
6	31	27
8	92	88
10	44	39
11	24	17
12	23	21
H,8,3		
0	46	43
1	175	170
2	215	213
5	80	80
6	110	111
7	20	22
9	41	44
10	39	39
11	50	45
13	21	15
H,9,3		
1	126	123
2	32	42
3	114	109
4	51	56
5	85	87
6	40	36
7	89	90
11	45	42
H,10,3		
1	45	48
2	88	87
3	57	65
4	68	69
5	91	91
6	84	85
7	52	49
9	50	44
10	42	36
H,11,3		
0	124	119
1	29	33
2	29	27
4	144	140
5	31	28
6	31	31
7	19	24
8	54	55
9	23	22
10	24	21
11	26	10
H,12,3		
1	65	64
2	65	65
4	24	21
5	96	93
6	29	30
9	52	39
11	23	6
H,13,3		
0	26	32
1	85	82
3	73	72
4	47	43
5	34	25
7	52	48
11	25	23
H,14,3		
2	66	65
3	47	42
4	20	27
5	22	20
6	29	31
8	31	36
H,15,3		
0	50	52
1	50	52
4	40	40
6	28	25
7	25	20
8	33	33
H,16,3		
1	43	45
3	37	38
5	32	39
H,17,3		
3	45	44
4	24	24
7	23	13
H,18,3		
2	45	41
H,19,3		
0	35	34
H,0,4		
2	112	113
3	106	104
4	113	112
5	60	69
8	24	29
9	63	60
10	49	48
13	46	36
14	22	16
H,1,4		
1	268	270
2	163	166
3	91	94
4	107	104
5	60	63
6	53	56
7	81	84
8	81	83
10	22	20
11	54	57
12	37	38
H,2,4		
0	41	43
1	166	162
2	220	217
4	35	44
5	191	197
6	137	140
7	74	76
8	33	38
9	29	28
10	25	19
11	40	38
12	25	28
13	25	27
H,3,4		
0	13	14
1	97	101
2	71	72
3	137	137
4	110	111
5	77	76
6	87	89
7	122	122
8	37	42
9	24	27
10	62	52
11	46	47
12	35	28
H,4,4		
0	165	165
1	212	218
2	89	84
3	131	134
4	153	157
5	129	126
6	70	74
7	100	98
8	40	40
9	19	21
10	26	20
13	34	28
14	24	11
H,5,4		
0	114	110
1	136	141
2	134	140
3	114	116
4	129	122
5	140	140
6	63	64
7	31	34
8	90	86
9	23	31
12	41	38
13	37	29
H,6,4		
1	116	118
2	168	170
3	77	77
4	72	72
5	53	52
6	82	83
7	85	87
8	50	45
10	38	39
H,7,4		
0	75	74
1	102	101
2	69	69
3	109	106
4	90	96
5	44	53
6	61	62
7	122	115
8	46	48
9	40	40
10	53	47
11	33	34
H,8,4		
1	89	90
2	92	90
3	48	53
4	39	42
5	88	86
6	56	59
7	77	77
8	65	60
9	52	41
10	25	24
H,9,4		
0	118	116
1	60	67
2	45	48
3	30	33
4	119	113
5	93	93
6	39	38
7	25	22
8	67	71
9	33	32
H,10,4		
0	59	65
1	68	66
2	77	79
3	95	92
4	65	63
5	42	41
6	65	63
9	31	25
H,11,4		
1	58	61
2	58	54
3	79	80
4	49	49
5	34	30
6	42	46
7	56	57
H,12,4		
0	30	33
1	79	71
2	84	80
3	54	52
4	27	20
5	66	64
6	43	43
8	20	26
9	22	24
H,13,4		
0	69	64
1	30	34
2	24	20
3	44	46
4	50	44
8	36	29
9	23	31
H,14,4		
0	83	82
1	52	56
2	30	32
3	47	50
4	30	35
5	28	26
9	25	18
H,15,4		
1	48	48
2	59	53
3	23	30
6	22	22
7	25	22
H,16,4		
1	24	28
2	41	42
3	21	23
5	24	11
6	18	21
H,17,4		
0	32	29
3	28	25
4	26	35
H,18,4		
1	22	25
4	22	21
H,0,5		
2	12	3
3	86	80
4	47	47
5	110	114
6	101	98
7	128	131
8	42	42
9	29	31
10	36	36
11	47	49
H,1,5		
1	187	188
2	169	161
3	162	160
4	60	55
5	57	56
6	73	76
7	27	30
8	72	69
9	25	25
10	32	32
12	26	24
H,2,5		
0	222	219
1	19	22
2	63	64
3	37	36
4	154	149
5	75	75
6	55	50
7	74	72
8	80	77
9	54	58
10	24	25
12	37	40
H,3,5		
0	131	125
1	174	171
2	172	176
3	84	79
4	43	47
5	114	110
6	81	81
7	43	50
8	22	24
9	20	22
12	22	21
H,4,5		
0	27	38
1	21	24
2	118	117
3	141	137
4	20	21
5	55	55
6	95	92
7	116	114
11	64	56
H,5,5		
1	93	91
2	144	137
3	81	82
4	35	37
6	131	127
8	24	28
9	38	38
12	21	19
H,6,5		
0	113	118
1	63	60
2	39	36
3	49	53
4	80	83
5	25	26
6	69	68
7	80	77
8	86	76
9	32	28
11	43	35
12	29	24
H,7,5		
0	131	128
1	115	116
3	50	52
4	63	64
5	86	87
6	23	17
7	22	18
9	55	53
H,8,5		
0	119	116
1	44	42
2	43	44
3	89	87
4	41	39
5	25	26
6	48	44
7	67	67
8	38	39
9	32	37
H,9,5		
1	75	72
2	109	102
3	34	37
4	31	30
5	35	39
6	67	64
7	19	25
8	31	28
H,10,5		
1	69	71
2	30	33
3	69	68
4	65	64
5	44	47
6	33	36
7	39	47
8	32	38
H,11,5		
0	53	38
1	81	80
2	33	23
3	70	71
4	34	33
5	63	58
7	36	33
H,12,5		
0	50	57
1	48	43
2	38	37
3	21	25
4	59	61
5	33	29
6	26	25
H,13,5		
1	37	33
2	100	97
3	34	29
5	21	18
6	27	30
H,14,5		
0	42	48
1	48	49
4	25	27
6	24	26
H,15,5		
1	38	33
2	25	28
3	29	33
H,16,5		
0	32	38
3	22	18
4	41	35
H,17,5		
4	22	11
H,0,6		
3	53	54
4	134	127
5	41	41
6	22	22
7	36	38
8	53	54
H,1,6		
1	82	79
2	76	76
3	46	43
4	27	26
5	91	88
6	110	103
7	29	33
8	29	31
9	72	76
10	31	37
H,2,6		
0	94	85
1	130	128
2	14	17
3	142	138
4	25	24
5	76	74
6	35	35
7	63	68
9	23	20
11	25	23
H,3,6		
0	88	80
1	61	59
2	91	86
3	33	34
4	24	22
5	85	81
6	122	121
7	42	48
8	49	51
9	22	24
10	31	37
H,4,6		
0	171	162
1	54	51
2	57	59
3	45	45
4	129	124
5	36	36
6	30	24
8	27	35
9	21	15
H,5,6		
0	39	34
1	76	72
2	57	53
3	49	45
4	26	19
5	104	94
6	22	26
7	21	30
9	64	57
10	33	30
H,6,6		
0	103	99
1	125	124
2	37	41
3	79	79
4	42	42
5	34	38
7	49	48
11	31	23
H,7,6		
1	67	60
2	85	83
4	17	18
5	21	22
6	78	70
8	53	56
10	37	40
H,8,6		
0	106	101
1	103	93
3	46	40
4	74	70
5	23	26
8	38	32
H,9,6		
0	43	40
1	70	65
2	26	22
3	47	44
4	25	28
5	64	65
9	41	43
H,10,6		
0	106	100
1	23	30
2	39	39
3	50	50
4	65	60
5	33	34
7	22	23
H,11,6		
2	83	76
3	22	20
5	25	22
6	55	58
H,12,6		
1	34	32
3	59	59
4	31	33
5	22	30
H,13,6		
1	45	48
3	36	32
5	28	38
6	24	27
H,14,6		
0	53	55
1	21	23
4	36	34
H,15,6		
2	34	35
H,16,6		
1	23	24
H,0,7		
3	56	54
4	21	29
6	68	69
7	51	54
8	64	58
10	30	37
11	27	27
H,1,7		
1	81	83
2	110	119
3	58	74
4	39	43
6	49	50
7	38	39
H,2,7		
0	105	100
1	40	40
2	29	31
3	19	10
4	76	78
5	83	82
6	16	17
7	29	30
8	36	39
9	37	39
H,3,7		
0	131	120
1	64	61
2	54	56
3	62	65
4	60	62
5	43	40
7	29	39
H,4,7		
1	68	66
2	55	53
3	83	80
4	32	27
5	42	42
6	69	64
7	41	43
8	39	35
H,5,7		
0	19	16
1	89	84
2	56	52
3	62	67
4	25	26
5	42	47
6	51	54
7	18	12
8	23	24
H,6,7		
0	48	49
1	47	49
2	43	40
3	62	65
4	50	46
5	26	27
6	55	48
7	30	32
8	36	39
9	32	34
H,7,7		
0	138	140
1	55	56
2	20	27
3	42	39
4	45	48
5	44	44
H,8,7		
1	26	25
2	63	64
3	41	41
4	17	19
6	33	32
7	38	41
H,9,7		
1	46	50
2	66	61
3	42	47
5	25	32
6	48	50
H,10,7		
0	27	28
3	38	37
4	43	44
H,11,7		
1	41	37
2	20	26
4	42	42
5	30	31
H,12,7		
0	38	41
2	29	26
4	24	30
5	30	37
H,13,7		
2	29	36
H,0,8		
2	12	7
3	71	74
4	16	27
9	22	23
H,1,8		
1	27	27
3	29	37
4	23	30
6	33	43
7	28	33
H,2,8		
1	36	32
2	92	84
3	32	31
6	51	49
H,3,8		
0	44	40
1	32	31
2	31	28
3	65	61
4	44	46
5	28	30
7	47	49
H,4,8		
1	65	61
2	23	26
3	38	37
4	24	21
5	53	45
6	36	32
9	23	20
H,5,8		
0	78	70
1	36	35
2	30	28
4	72	69
5	14	12
6	27	24
8	48	35
H,6,8		
0	17	11
1	33	30
2	87	78
4	16	14
5	29	31
6	48	40
7	22	13
H,7,8		
1	45	36
2	32	27
3	52	44
7	49	52
H,8,8		
0	31	30
1	48	43
2	46	42
3	39	37
5	38	37
6	31	22
H,9,8		
0	38	36
3	25	21
4	43	38
5	27	16
8	31	27
H,10,8		
1	34	36
3	27	23
5	22	22
H,11,8		
2	24	17
3	32	40
5	24	15
H,13,8		
0	35	33
H,0,9		
2	22	38
4	41	37
6	43	39
8	21	20
H,1,9		
1	47	45
2	34	30
3	30	36
4	36	38
5	19	24
6	17	11
7	20	26
H,2,9		
1	51	42
3	16	6
4	23	17
5	47	55
H,3,9		
2	14	18
3	26	25
4	66	48
H,4,9		
2	40	50
H,5,9		
1	17	48
5	21	35
H,6,9		
4	17	6
H,8,9		
0	17	10
H,0,10		
4	20	29
5	16	17
H,1,10		
1	22	22
5	15	18
H,2,10		
2	18	36
6	21	17
H,3,10		
2	16	18
H,4,10		
3	21	25

ments gave $a=8{\cdot}475$ (6), $b=10{\cdot}742$ (5), $c=5{\cdot}899$ (4) Å, where the standard deviations in parentheses were obtained from a least-squares treatment of the observed angles. The density calculated on the basis of four molecules of $C_4H_6OSO_2$ per unit cell is 1·659 g.cm^{-3}; two measurements of the density by flotation gave 1·663 and 1·658 g.cm^{-3}.

Diffracted intensities for layers from $l=0$ to $l=10$ were measured on a Pailred diffractometer with silicon-monochromated Mo $K\alpha$ radiation and a maximum $\sin\theta/\lambda$ of 0·94. Both hkl and $\bar{h}kl$ reflections were measured, giving 3560 accessible reflections, of which 1950 were independent. Background corrections were calculated from the equation $1/B=C+Ds^2+Es^4$, where B is the background, s is $\sin\theta/\lambda$, and C, D, and E are constants obtained by least-squares fitting of measurements made on unobserved reflections.

Corrections were applied for instrumental fluctuations, crystal deterioration, peak truncation, absorption (using an adaptation of the program of Burnham, 1962), and the Lorentz-polarization factor (Jennings, 1968; Suortti & Paakkari, 1968). 1282 independent weighted average F values were obtained. A set of realistic standard deviations were obtained by adding $0{\cdot}0004\ F^2$ to the σ computed for each F on the basis of counting statistics.

Structure determination and refinement

The sulfur positions were deduced from the three-dimensional Patterson function. These atoms occupy general positions of space group $P2_12_12_1$ $(x,y,z;\ \frac{1}{2}-x,\ \bar{y},\ \frac{1}{2}+z;\ \frac{1}{2}+x,\ \frac{1}{2}-y,\ \bar{z};\ \bar{x},\ \frac{1}{2}+y,\ \frac{1}{2}-z)$ with $x=0{\cdot}134$, $y=0{\cdot}112$, $z=0{\cdot}162$. Successive Fourier and least-squares calculations yielded the positions of the oxygen and carbon atoms. Hydrogen-atom positions were obtained from a difference Fourier map. Full-matrix least-squares refinements of the scale factor and of the positional and anisotropic thermal parameters of all atoms were carried out by means of the program *CRYLSQ* of the *X-ray System* (1970) of programs. Atomic scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1962), and this source was also used for the real part of the dispersion correction for the sulfur atom, equal to 0·1. Since the $\Delta f''$ dispersion term generally affects $F(hkl)$ and $F(\bar{h}kl)$ differently, a simple correction is not strictly applicable to the averaged data, so $\Delta f''$ was taken as zero. No extinction corrections were made. The terms in the least-squares summations were weighted by $1/\sigma^2$, where σ is the adjusted standard deviation described above. The quantity being minimized in the refinements was $\sum w(|F_o|-|F_c|)^2$.

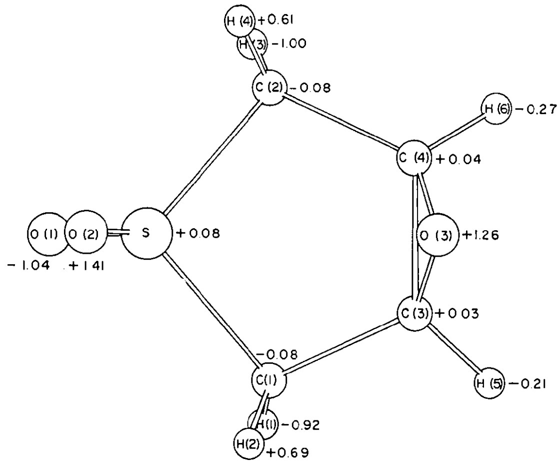

Fig. 1. The 3,4-epoxysulfolane molecule projected onto the mean plane through the sulfur and four carbon atoms.

The anisotropic temperature factors of the sulfur, oxygen and carbon atoms available at this stage were analyzed for rigid-body motion by the method of Schomaker & Trueblood (1968), using a local program. The libration, translation and screw tensors obtained from this calculation were used to compute anisotropic temperature factors for the six hydrogen atoms.

Finally, the scale factor, the positional parameters of all fourteen atoms and the anisotropic temperature factors of the sulfur, oxygen and carbon atoms were refined. In this refinement, the hydrogen temperature factors were those given by the Schomaker–Trueblood rigid-body treatment, and these were not refined. The value of $R_1=\sum||F_o|-|F_c||/\sum|F_o|$ obtained was 0·055 for the observed data, and the weighted residual, $\sum w||F_o|-|F_c||/\sum w|F_o|$, was 0·060. The standard deviation of an observation of unit weight, $[\sum w(|F_o|-|F_c|)^2/(n-m)]^{1/2}$, where n is the number of observations included in the calculations (1282) and m is the number of variables being refined (91), was 1·40. The largest ratio of shift to standard deviation in the last least-squares cycle was 0·63 for coordinates of atoms other than hydrogen; including all parameters refined this maximum ratio was 5·33, and the average ratio was 0·39.

Results

The observed and calculated structure factor magnitudes for the observed reflections are listed in Table 1, and Table 2 gives the fractional positional coordinates of the fourteen atoms in one molecule. The intramolecular distances and angles and the intermolecular distances less than 3·0 Å are listed in Table 3. These dimensions were calculated by means of the program *BONDLA* of the *X-ray System* (1970). Table 3 also lists the intramolecular dimensions after correction for libration by the formula of Schomaker & Trueblood (1968), using our local program.

Table 4 compares the average dimensions of the 3,4-epoxysulfolane molecule with the corresponding dimensions of the butadiene sulfone molecule (Sands & Day, 1967) and the dimethyl sulfone molecule (Sands, 1963). Appropriate librational corrections were not applied in the earlier work, so the comparison is valid for only the uncorrected distances. The agree-

Table 2. *Positional parameters and standard deviations (in parentheses)*

	x	y	z
S	0·13425 (8)	0·11210 (6)	0·16219 (12)
O(1)	0·3006 (2)	0·0849 (2)	0·1420 (5)
O(2)	0·0839 (4)	0·1862 (2)	0·3526 (4)
O(3)	−0·2002 (2)	0·0930 (2)	0·0042 (5)
C(1)	0·0234 (3)	−0·0303 (3)	0·1512 (6)
C(2)	0·0615 (4)	0·1797 (3)	−0·0954 (5)
C(3)	−0·0970 (3)	−0·0118 (3)	−0·0316 (6)
C(4)	−0·0774 (3)	0·1011 (3)	−0·1634 (5)
H(1)	0·088 (5)	−0·097 (5)	0·110 (11)
H(2)	−0·025 (6)	−0·037 (5)	0·304 (10)
H(3)	0·152 (6)	0·176 (5)	−0·210 (9)
H(4)	0·043 (5)	0·265 (5)	−0·052 (9)
H(5)	−0·150 (5)	−0·090 (5)	−0·070 (11)
H(6)	−0·116 (5)	0·102 (5)	−0·312 (9)

Table 3. *Interatomic distances and angles*

Standard deviations of the last significant digit are given in parentheses.

Intramolecular distances (Å)

	Uncorrected	Corrected for libration
S—O(1)	1·445 (2)	1·456
S—O(2)	1·441 (3)	1·454
S—C(1)	1·797 (3)	1·809
S—C(2)	1·793 (3)	1·804
O(1)-O(2)	2·470 (4)	2·492
O(1)-C(1)	2·657 (4)	2·674
O(1)-C(2)	2·665 (4)	2·682
O(2)-C(1)	2·662 (4)	2·682
O(2)-C(2)	2·650 (4)	2·670
C(1)-C(2)	2·704 (4)	2·721
C(1)-C(3)	1·498 (5)	1·507
C(1)-H(1)	0·94 (5)	0·95
C(1)-H(2)	0·99 (6)	1·00
C(1)-H(5)	2·07 (5)	2·08
C(2)-C(4)	1·504 (4)	1·513
C(2)-H(3)	1·02 (5)	1·03
C(2)-H(4)	0·96 (6)	0·97
C(3)-C(4)	1·450 (4	1·460
C(3)-O(3)	1·442 (4)	1·454
C(3)-H(1)	2·00 (5)	2·01
C(3)-H(5)	0·98 (5)	0·99
C(3)-H(6)	2·06 (6)	2·07
C(4)-O(3)	1·438 (4)	1·450
C(4)-H(5)	2·22 (6)	2·23
C(4)-H(6)	0·94 (5)	0·94

Table 3 (*cont.*)

Intramolecular angles (°)

O(1)-S—O(2)	117·69 (16)	117·83
O(1)-S—C(1)	109·60 (14)	109·53
O(1)-S—C(2)	110·32 (16)	110·24
C(1)-S—C(2)	97·72 (16)	97·74
O(2)-S—C(1)	110·10 (16)	110·10
O(2)-S—C(2)	109·58 (15)	109·55
C(3)-C(1)-S	105·62 (21)	105·61
C(4)-C(2)-S	105·55 (21)	105·51
C(1)-C(3)-C(4)	114·78 (25)	114·76
C(2)-C(4)-C(3)	114·59 (25)	114·62
C(1)-C(3)-O(3)	114·31 (26)	114·25
C(2)-C(4)-O(3)	114·64 (25)	114·60
C(3)-O(3)-C(4)	60·47 (19)	60·35
O(3)-C(4)-C(3)	59·88 (19)	59·95
C(4)-C(3)-O(3)	59·65 (20)	59·70
H(1)-C(1)-H(2)	114·7 (48)	114·9
H(3)-C(2)-H(4)	109·4 (41)	109·6
H(5)-C(3)-C(1)	111·4 (33)	111·4
H(6)-C(4)-C(2)	121·3 (30)	121·3
H(5)-C(3)-C(4)	130·2 (36)	130·3
H(6)-C(4)-C(3)	118·0 (34)	118·0
H(5)-C(3)-O(3)	115·2 (27)	115·2
H(6)-C(4)-O(3)	113·0 (27)	113·0

Intermolecular distances (Å)

O(1)-H(1)	2·92 (6)	O(3)-H(5)	2·81 (6)
O(1)-H(2)	2·80 (6)	O(3)-H(6)	2·83 (5)
O(1)-H(3)	2·96 (5)	C(3)-H(6)	2·92 (5)
O(1)-H(4)	2·67 (5)	H(1)-H(3)	2·59 (7)
O(2)-H(1)	2·75 (5)	H(2)-H(4)	2·58 (8)
O(2)-H(3)	2·64 (5)	H(2)-H(6)	2·82 (8)
O(2)-H(5)	2·78 (6)	H(3)-H(5)	2·82 (8)
O(2)-H(6)	2·76 (5)	H(4)-H(5)	2·86 (8)
O(3)-H(2)	2·68 (5)	H(5)-H(6)	2·50 (7)
O(3)-H(4)	2·67 (5)		

ment between corresponding dimensions is generally quite good, and the epoxidation has produced only slight changes in the bond angles at the sulfur atom. The preservation of these angles upon increasing the carbon–carbon distance across the ring from the double bond value of 1·30 to 1·45 Å in the epoxide has required folding of the ring. This folding is apparent in Fig. 1, which shows the molecule projected onto the mean plane through the sulfur and four carbon atoms. The S–C(1)–C(2) plane and the C(1)–C(2)–C(3)–C(4)

Table 4. *Comparison of mean dimensions of molecules*

Distances in ångströms, angles in degrees. Standard deviations in parentheses.

	Dimethyl* sulfone uncorrected	Dimethyl† sulfone uncorrected	Butadiene‡ sulfone uncorrected	This work uncorrected	This work corrected
S–O	1·445 (16)	1·446 (3)	1·440 (4)	1·444 (2)	1·455
S–C	1·778 (17)	1·765 (5)	1·794 (5)	1·795 (2)	1·806
O–O	2·48 (3)	2·473 (6)	2·455 (8)	2·470 (4)	2·492
C–C (adjacent to sulfur)			1·479 (6)	1·502 (3)	1·510
C–C (across ring)			1·299 (8)	1·450 (4)	1·460
O–S–O	117·9 (8)	117·3 (2)	117·0 (3)	117·69 (16)	117·83
O–S–C	108·0 (8)	108·5 (1)	110·2 (2)	109·91 (11)	109·84
C–S–C	103·0 (8)	104·8 (2)	97·0 (3)	97·72 (16)	97·74

* Sands (1963).
† Langs, Silverton & Bright (1970).
‡ Sands & Day (1967).

Table 5. *Anisotropic temperature factors*

Observed values from least-squares refinement. Calculated values from rigid-body treatment of Schomaker & Trueblood (1968). The β^{ij} are the coefficients in $[\exp -(h^2\beta^{11}+2hk\beta^{12}+\cdots)]$.

Atom	ij	β^{ij}(obs)	β^{ij}(cal)	Principal axes (crystal coordinates)	$\overline{u^2}$ along principal axes (Å^2)
S	11	0.00906(8)	0.00923	-0.1097, 0.0210, 0.0494	0.0343
	12	-0.00049(5)	-0.00048	-0.0139, -0.0867, 0.0586	0.0227
	13	-0.00102(10)	-0.00086	0.0412, 0.0268, 0.1512	0.0245
	22	0.00401(4)	0.00401		
	23	0.00038(8)	0.00042		
	33	0.01424(14)	0.01423		
O(1)	11	0.00796(27)	0.00808	-0.0143, -0.0769, 0.0934	0.0416
	12	-0.00096(19)	-0.00104	0.1126, 0.0053, 0.0499	0.0258
	13	-0.00372(39)	-0.00435	-0.0324, 0.0522, 0.1324	0.0648
	22	0.00836(23)	0.00840		
	23	0.00309(39)	0.00305		
	33	0.03086(78)	0.03163		
O(2)	11	0.02407(51)	0.02130	-0.0005, -0.0839, 0.0734	0.0395
	12	0.00013(25)	-0.00040	-0.0033, 0.0403, 0.1527	0.0239
	13	0.00066(48)	-0.00084	0.1179, 0.0008, 0.0045	0.0876
	22	0.00625(19)	0.00614		
	23	-0.00188(34)	-0.00272		
	33	0.01525(58)	0.01532		
O(3)	11	0.00682(25)	0.00635	-0.0039, -0.0685, 0.1147	0.0438
	12	0.00155(18)	0.00128	-0.1136, 0.0192, 0.0300	0.0220
	13	0.00270(36)	0.00270	0.0318, 0.0601, 0.1211	0.0600
	22	0.00849(24)	0.00916		
	23	0.00208(40)	0.00210		
	33	0.02916(78)	0.02980		
C(1)	11	0.00990(36)	0.00940	-0.0637, 0.0351, 0.1275	0.0391
	12	-0.00089(21)	-0.00043	-0.0315, -0.0861, 0.0461	0.0220
	13	-0.00007(49)	0.00014	0.0941, -0.0051, 0.1018	0.0362
	22	0.00419(18)	0.00388		
	23	0.00137(41)	0.00103		
	33	0.02087(77)	0.02078		
C(2)	11	0.00938(37)	0.00887	-0.0332, 0.0741, 0.0909	0.0367
	12	-0.00048(24)	-0.00046	-0.0250, -0.0556, 0.1312	0.0207
	13	0.00077(40)	0.00027	0.1104, 0.0097, 0.0571	0.0346
	22	0.00530(22)	0.00533		
	23	0.00227(34)	0.00199		
	33	0.01525(64)	0.01477		
C(3)	11	0.00842(34)	0.00782	0.0502, -0.0587, 0.1100	0.0429
	12	-0.00143(23)	-0.00121	0.0930, 0.0569, 0.0131	0.0257
	13	0.00064(45)	-0.00014	-0.0525, 0.0446, 0.1283	0.0350
	22	0.00592(23)	0.00579		
	23	-0.00114(39)	-0.00042		
	33	0.02169(79)	0.02256		
C(4)	11	0.00740(27)	0.00759	0.0004, 0.0914, 0.0321	0.0415
	12	0.00011(24)	0.00003	0.1038, -0.0087, 0.0791	0.0256
	13	-0.00097(42)	-0.00076	-0.0561, -0.0154, 0.1465	0.0315
	22	0.00704(25)	0.00697		
	23	0.00066(47)	0.00066		
	33	0.01736(63)	0.01667		

H(1)	11	0.01248	H(4)	11	0.01472
	12	0.00080		12	-0.00015
	13	-0.00270		13	-0.00255
	22	0.00362		22	0.00456
	23	-0.00020		23	0.00328
	33	0.03373		33	0.02479
H(2)	11	0.01378	H(5)	11	0.01289
	12	-0.00206		12	-0.00393
	13	0.00157		13	-0.00445
	22	0.00676		22	0.00747
	23	0.00454		23	-0.00064
	33	0.02210		33	0.03681
H(3)	11	0.01011	H(6)	11	0.01180
	12	-0.00228		12	-0.00131
	13	0.00218		13	-0.00492
	22	0.00986		22	0.01107
	23	0.00357		23	0.00170
	33	0.01670		33	0.01994

plane make an angle of 167·3°, with the folding such that O(3) is moved closer to O(2). The O(3)–C(3)–C(4) plane makes an angle of 105·1° with the C(1)–C(2)–C(3)–C(4) plane, with the folding again moving O(3) closer to O(2). The O(3)–C(3)–C(4) plane thus makes an angle of 92·4° with the S–C(1)–C(2) plane, and the O(2)–O(3) distance is 3·32 Å as compared with 4·32 Å for O(1)–O(3). The weighted mean of the six angles at the sulfur atom is 109·20°, as compared with 109·17° for butadiene sulfone and 109·47° for a regular tetrahedron. The folding of the ring has lowered the molecular symmetry to C_s–m, as compared with C_{2v}–$mm2$ for butadiene sulfone.

The anisotropic temperature factors of the fourteen atoms are listed in Table 5. The β's are the coefficients in the exponent of the factor applied to the calculated structure factors, $\exp -(\sum h_i h_j \beta^{ij})$; the value of the mean square displacement, u^2, along the direction of the reciprocal axis a^{i*} is given by $\beta^{ii}/2\pi^2 a^{i*2}$. The calculated values of β^{ij} given in Table 5 are based on the rigid-body treatment of Schomaker & Trueblood (1968), where the 48 β^{ij} values given by least-squares for the eight heavy atoms were used to determine least-squares values of the six independent parameters of a molecular libration tensor, the six independent parameters of a molecular translation tensor, and the eight determinable components of a screw tensor. The agreement between the columns of β^{ij} (obs) and β^{ij} (cal) suggests that the approximation of rigid-body motion is quite reasonable, and the hydrogen temperature factors based upon this model are probably at least as good as those that might be obtained by refining them directly at the expense of increasing the number of variables included in the least-squares calculations from 91 to 127. Table 5 also gives the crystal coordinates of the directions of the principal axes of the temperature factors, and the values of $\overline{u^2}$ along these principal axes.

The components and principal axes of the librational tensor, **L**, the translational tensor, **T**, and the screw tensor, **S**, are given in Table 6. These components are in terms of a set of Cartesian axes, which in this case are parallel to the crystallographic axes. In this Cartesian system, the calculated β's of Table 5 are given by

$$\beta_{ij} = e_{imk} e_{jnl} L_{mn} r_k r_l + e_{imk} S_{mj} r_k + e_{jnl} S_{ni} r_l + T_{ij} ,$$

where the convention of summing over repeated indices is followed, e_{imk} is a component of the permutation tensor equal to 1 if i, m and k are a cyclic permutation of 1, 2, and 3, to -1 for a non-cyclic permutation, and to 0 otherwise, and r_k is the kth Cartesian component of the particular atom being considered. If the matrix **M** transforms from Cartesian axes to crystal axes, the contravariant tensor $\boldsymbol{\beta}$ in crystal components is given by the transformation

$$\boldsymbol{\beta}\ (\text{crystal}) = \bar{\mathbf{M}}^{-1}\ \boldsymbol{\beta}\ (\text{Cartesian})\ \mathbf{M}^{-1},$$

where $\bar{\mathbf{M}}$ is the transpose of **M**. In the present orthorhombic case

$$\beta^{ij}\ (\text{crystal}) = \beta^{ij}\ (\text{Cartesian})/a_i a_j$$

(no summation implied). The principal values of **L** and **T** in Table 6 should be divided by $2\pi^2$ to obtain mean square displacements, in rad^2 and Å^2 respectively. The values for **L** have been converted to (°) in Table 6.

The smallest component of the librational tensor corresponds to a mean square oscillation of 11·8 (°)^2, and the direction of the associated axis of libration is about

Table 6. *Cartesian components, principal axes and principal values of librational tensor*, **L**, *translational tensor*, **T**, *and screw tensor*, **S**

Component	Value	Principal axes (Cartesian components)	Principal value
$L(1,1)$	0·1475	0·657, −0·152, 0·739	0·1951 = 32·4 (°)^2
$L(1,2)$	0·0270	−0·577, 0·531, 0·621	0·0712 = 11·8
$L(1,3)$	0·0478	−0·486, −0·834, 0·261	0·1681 = 28·0
$L(2,2)$	0·1414		
$L(2,3)$	−0·0350		
$L(3,3)$	0·1454		
$T(1,1)$	0·5210	0·288, −0·392, 0·874	0·6601 = 0·0334 Å^2
$T(1,2)$	0·0002	−0·240, 0·854, 0·462	0·4322 = 0·0219
$T(1,3)$	0·0459	−0·927, −0·343, 0·151	0·5136 = 0·0260
$T(2,2)$	0·4768		
$T(2,3)$	−0.0823		
$T(3,3)$	0·6081		
$S(1,1)$ $-S(2,2)$	0·0147		
$S(1,2)$	0·0652		
$S(1,3)$	−0·1552		
$S(2,1)$	−0·0126		
$S(2,2)$ $-S(3,3)$	0·0774		
$S(2,3)$	0·0274		
$S(3,1)$	0·1058		
$S(3,2)$	−0·0474		

5° away from the normal to the mean plane through the sulfur and four carbon atoms. That is, the normal to the molecule shown in Fig. 1 is the axis of least rotational oscillation. The other two orthogonal principal axes of the **L** tensor are then nearly in the plane of Fig. 1, and a magnitude of 28·0 (°)^2 is associated with a direction within two degrees of being perpendicular to the C(1)–C(3) bond, while the third direction has the value of 32·4 (°)^2. No such clear-cut interpretation of the directions of the principal axes is apparent for the **T** tensor, although there does seem to be a tendency for the greatest translational freedom to be in the plane of Fig. 1, with lesser motions along the normal.

The author gratefully acknowledges the assistance of Mr John T. McPhearson who prepared the compound, of Mr Benjamin F. Blyton who ran some n.m.r. patterns and helped with the melting-point determinations, of Dr Robert W. Kiser who ran and interpreted the mass spectra, and of Miss Doris M. Thompson who made several helpful suggestions.

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