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The Crystal Structure of Aflatoxin B₂

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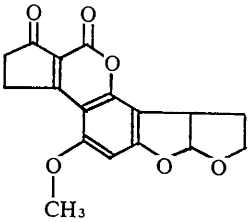
Aflatoxin B_2 crystallizes as orthorhombic bipyramids in the space group $P2_12_12_1$ with four molecules in the unit cell with dimensions $a = 6{\cdot}927$, $b = 10{\cdot}146$, $c = 20{\cdot}171$ Å. The structure, solved by Patterson methods, consists of strings of coplanar molecules in the direction of the b axis. The molecules within a string are interconnected by an interaction between the methyl group and a carbonyl oxygen atom, which may be called a hydrogen bond; the $CH_3 \cdots O$ distance is 2·930 Å. The strings are stacked at an interplanar distance of 3·463 Å in the direction of the a axis. The libration tensor of the molecule is practically diagonal in the reference system defined by the principal axes of the inertia tensor.

Introduction

Aflatoxin B_2 is one of the carcinogenic metabolites of the mould *Aspergillus flavus* Link ex Fries (Hartley, Nesbitt & O'Kelly, 1963), which, in 1960 caused the death of 100,000 young turkeys as a result of liver damage (turkey X-disease). An excellent review of aflatoxins, including for instance carcinogenic action, metabolism and biochemical effects on DNA, RNA and proteins, has been presented by Schoental (1967).

According to van Dorp, van der Zijden, Beerthuis, Sparreboom, Ord, de Jong & Keuning (1963), aflatoxin B_2 has the following structural formula:



This formula has been confirmed by van Soest & Peerdeman (1964).

The present paper describes in detail the crystal and molecular structure of aflatoxin B_2.

Experimental

Aflatoxin B_2 (kindly supplied by Professor D. A. van Dorp of the Unilever Research Laboratory, Vlaardingen) was recrystallized from a chloroform–ethanol mixture. The crystals obtained were pale-yellow, well-formed orthorhombic bipyramids.

The unit-cell dimensions were first determined from Weissenberg and rotation photographs and refined, later on, by a least-squares fit of 60 θ-values ($\theta > 68°$) measured with a General Electric diffractometer (Furnas, 1957) using Cu radiation, $\lambda(K\alpha_1) = 1{\cdot}54051$ Å.

The space group was uniquely determined from the systematically absent reflexions.

Crystal data

Aflatoxin B_2, $C_{17}H_{14}O_6$ ($M = 314{\cdot}3$).
Orthorhombic $a = 6{\cdot}927 \pm 0{\cdot}002$, $b = 10{\cdot}146 \pm 0{\cdot}002$, $c = 20{\cdot}171 \pm 0{\cdot}002$ Å. $U = 1417{\cdot}7$ Å³, $D_m = 1{\cdot}47$ g.cm^{-3}, $Z = 4$, $D_x = 1{\cdot}472$ g.cm^{-3}. Space group $P2_12_12_1$, $\mu = 11{\cdot}0$ cm^{-1} for Cu $K\alpha$ radiation.

Two sets of intensity values were measured with a General Electric diffractometer, using nickel-filtered Cu radiation and a scintillation counter with pulse height discrimination. By measuring the apparent absorption factor for a nickel filter as a function of the intensity of the primary beam, it was found that the counting apparatus was linear up to only 1000 counts.sec^{-1}. Seven attenuating filters of increasing thickness were used to keep the counting rate below this value. These intensities were measured by the θ–2θ scanning technique at a speed of 2°.min^{-1}. The scan range was adjusted to account for the α_1–α_2 splitting. Background was counted for 15 sec at each end of the scan. 1106 of the 1895 theoretically possible independent reflexions were measured, using a crystal with a largest dimension of 0·2 mm. For a second set of data (1725 reflexions measured), a different crystal was used with a maximum dimension of 0·6 mm. The intensities were corrected for Lorentz and polarization factors as well as for absorption – using a program written by Duisenberg (1966) by which absorption corrections for a crystal of arbitrary shape can be calculated – and placed on an approximately absolute scale by Wilson's method.

Table 1. *Observed and calculated structure factors* (× 10) *and phase angles*

Asterisks indicate unobserved reflexions. *E*'s denote extinction.

H	F-O	F-C	ARG
H	K= 0	L= 0	
2	542	542	180
4	481	480	0
6	411	421	0
8	31	32	0
10	266	273	180
12	396	387	0
14	286	283	180
16	629	626	0
18	332	335	180
20	122	122	180
22	19	24	180
24	51	46	0
H	K= 1	L= 0	
1	701	706	90
2	140	140	0
3	140	142	270
4	379	372	180
5	655	649	270
6	83	86	0
7	493	468	270
8	28	30	180
9	144	140	270
10	16	15	180
11	37	28	270
12	314	321	0
13	132	141	270
14	190	192	180
15	208	215	270
16	434	447	0
17	232	248	90
18	11*	22	0
19	11*	6	90
20	48	43	180
21	7*	8	270
22	146	145	0
23	8*	5	90
24	57	59	0
H	K= 2	L= 0	
0	587	583	180
1	668	683	270
2	327	330	0
3	170	178	270
4	97	90	0
5	16	4	270
6	35	24	0
7	396	395	90
8	248	257	0
9	433	429	90
10	8*	18	0
11	8*	11	90
12	154	153	180
13	231	240	270
14	13	14	0
15	96	94	270
16	10*	18	180
17	145	147	90
18	101	100	0
19	75	74	270
20	11*	24	0
21	50	49	270
22	60	66	0
23	38	37	90
24	7*	10	0
25	57	55	270
H	K= 3	L= 0	
1	508	499	90
2	153	155	180
3	94	87	90
4	16	13	180
5	43	31	270
6	65	66	180
7	366	364	90
8	13	6	180
9	7*	31	270
10	274	272	180
11	280	287	270
12	143	138	180
13	137	143	90
14	13	34	0
15	87	85	270
16	10*	8	180
17	11*	19	90
18	43	37	180
19	11*	16	90
20	10*	1	180
21	73	70	270
22	7*	11	180
23	7*	16	270
24	6*	17	180
25	52	48	90
H	K= 4	L= 0	
0	259	282	0
1	14	23	90
2	31	24	0
3	69	64	90
4	537	504	0
5	116	112	270
6	179	169	0
7	204	212	270
8	149	147	0
9	228	215	270
10	178	175	0
11	46	40	90
12	195	201	0
13	46	39	90
14	46	37	180
15	79	71	270
16	154	170	0
17	141	158	270
18	123	133	180
19	11*	28	90
20	8*	3	0
21	123	123	270
22	7*	12	180
23	35	38	270
24	25	23	0
H	K= 5	L= 0	
1	211	199	90
2	431	401	0
3	485	472	270
4	270	272	0
5	144	142	90
6	73	67	0
7	86	81	270
8	50	45	0
9	208	199	270
10	201	195	0
11	48	50	270
12	10*	13	180
13	60	66	90
14	48	36	0
15	234	248	270
16	124	125	0
17	11*	29	90
18	71	77	0
19	73	74	270
20	40	40	0
21	16	32	270
22	33	38	180
H	K= 6	L= 0	
0	168	178	0
1	85	73	270
2	31	29	180
3	64	57	90
4	241	238	180
5	97	90	90
6	190	189	0
7	281	292	90
8	113	108	180
9	64	68	270
10	10*	3	180
11	150	155	270
12	22	10	0
13	57	61	90
14	100	107	180
15	230	238	270
16	10*	12	180
17	67	64	270
18	49	47	0
19	33	26	270
20	111	114	180
H	K= 7	L= 0	
1	25	33	270
2	117	114	180
3	45	50	90
4	67	59	180
5	50	48	90
6	99	93	0
7	471	460	270
8	122	119	180
9	536	545	90
10	38	35	0
11	49	44	90
12	102	99	0
13	67	65	270
14	77	84	180
15	35	36	270
16	90	87	180
17	20	18	90
18	36	33	0
19	29	30	90
20	39	39	180
H	K= 8	L= 0	
0	115	114	0
1	11*	10	90
2	127	132	180
3	156	161	270
4	11*	25	0
5	43	41	90
6	62	61	180
7	267	276	270
8	483	490	0
9	22	20	90
10	101	101	180
11	10*	19	270
12	43	42	180
13	105	98	270
14	77	71	180
15	50	37	90
16	78	77	0
17	29	28	90
18	6*	19	180
19	150	141	270
20	23	21	0
H	K= 9	L= 0	
1	191	194	90
2	115	131	180
3	105	110	90
4	94	98	0
5	229	244	270
6	11*	15	0
7	198	203	90
8	101	99	0
9	157	154	270
10	55	46	0
11	7*	12	270
12	8*	6	0
13	8*	16	90
14	66	66	0
15	50	49	270
16	40	38	0
17	13	16	270
18	109	100	180
H	K=10	L= 0	
0	21	21	180
1	178	185	90
2	34	35	0
3	34	35	270
4	100	101	180
5	20	17	270
6	10*	13	180
7	57	55	90
8	67	65	180
9	85	78	270
10	7*	9	180
11	7*	15	270
12	74	70	180
13	51	46	270
14	83	78	0
15	35	37	270
H	K=11	L= 0	
1	89	86	270
2	36	36	0
3	19	17	90
4	40	41	0
5	8*	4	90
6	47	43	180
7	52	47	270
8	48	50	0
9	107	98	90
10	77	73	0
11	14	27	90
12	84	81	180
H	K=12	L= 0	
0	48	53	0
1	62	65	270
2	62	60	180
3	7*	2	90
4	60	58	0
5	30	31	90
6	52	43	180
7	94	89	270
8	32	23	0
9	78	71	270
H	K= 0	L= 1	
1	122	120	270
2	537	547	270
3	215	227	90
4	85	82	90
5	39	44	90
6	543	548	90
7	295	297	270
8	38	35	90
9	199	196	270
10	157	151	270
11	48	49	270
12	50	42	90
13	58	58	270
14	44	41	90
15	53	55	270
16	47	50	270
17	74	75	270
18	199	200	270
19	11*	14	90
20	123	118	270
21	50	46	90
22	59	55	90
23	7*	26	270
24	7*	15	90
25	58	55	270
H	K= 1	L= 1	
0	450	460	90
1	753E	877	177
2	726E	767	66
3	350	354	213
4	702	725	64
5	494	489	259
6	665	673	290
7	285	278	26
8	149	144	120
9	254	257	178
10	110	113	307
11	88	88	80
12	124	119	91
13	69	70	39
14	208	208	112
15	168	161	206
16	72	63	94
17	192	193	218
18	185	189	298
19	153	149	359
20	36	31	166
21	34	34	270
22	52	52	260
23	47	41	353
24	49	49	177
25	124	115	179
H	K= 2	L= 1	
0	214	227	0
1	413	417	45
2	215	273	87
3	92	83	39
4	179	176	265
5	349	359	26
6	308	312	180
7	738	697	168
8	461	462	54
9	609E	657	352
10	171	167	168
11	59	57	339
12	195	187	121
13	120	121	204
14	220	220	83
15	132	132	260
16	51	49	27
17	69	69	315
18	62	64	99
19	70	69	215
20	67	61	69
21	20	36	262
22	8*	17	293
23	35	34	151
24	106	100	78
25	71	62	319
H	K= 3	L= 1	
0	369	362	270
1	55	54	13
2	251	259	260
3	204	208	172
4	47	33	50
5	63	57	228
6	208	197	116
7	591E	644	199
8	572E	624	292
9	283	277	128
10	298	287	47
11	137	133	13
12	35	32	73
13	147	149	166
14	71	67	331
15	95	99	127
16	43	41	7
17	169	173	34
18	164	166	128
19	158	156	175
20	8*	13	19
21	31	32	90
22	77	76	97
23	22	19	264
24	33	30	270
H	K= 4	L= 1	
0	90	83	180
1	206	203	245
2	207	203	240
3	328	321	192
4	62	56	234
5	256	252	189
6	26	20	71
7	125	116	215
8	126	117	244
9	38	39	204
10	120	110	201
11	121	122	185
12	63	62	124
13	79	79	307
14	87	81	185
15	82	79	273
16	150	158	241
17	107	109	180
18	22	29	326
19	28	23	173
20	88	89	56
21	72	74	317
22	17	26	327
23	41	39	283
24	93	84	293
H	K= 5	L= 1	
0	723	816	90
1	344	336	241
2	302	291	271
3	39	41	183
4	52	36	248
5	93	87	36
6	205	198	109
7	38	36	314
8	221	215	105
9	116	107	308
10	71	71	172
11	25	24	122
12	178	188	100
13	59	60	219
14	36	27	298
15	94	92	125
16	238	250	77
17	50	50	306
18	42	40	13
19	37	49	356
20	8*	6	223
21	8*	10	139
22	15	21	127
23	42	38	190
H	K= 6	L= 1	
0	154	145	180
1	160	144	181
2	86	80	132
3	139	121	166
4	164	158	82
5	71	62	21
6	278	281	93
7	96	96	318
8	75	75	117
9	50	48	328
10	206	209	263
11	162	167	96
12	215	226	84
13	75	78	1
14	42	43	253
15	77	76	67
16	100	105	98
17	8*	21	206
18	47	46	78
19	44	43	268
20	8*	15	350
21	61	62	320
22	42	38	254
H	K= 7	L= 1	
0	139	126	270
1	116	112	125
2	278	279	94
3	112	108	242
4	256	262	268
5	140	137	66
6	156	162	131
7	215	217	176
8	222	219	266
9	333	342	150
10	255	260	46
11	225	227	356
12	181	181	275
13	149	152	51
14	165	164	93
15	30	29	235
16	32	29	351
17	7*	12	270
18	39	39	47
19	46	44	326
20	28	30	256
21	42	33	141
H	K= 8	L= 1	
0	189	186	180
1	200	195	185
2	30	21	140
3	88	83	329
4	150	151	259
5	248	259	17
6	247	253	141
7	269	274	190
8	45	37	331
9	62	62	265
10	164	163	272
11	66	57	152
12	35	34	298
13	96	93	172
14	64	66	33
15	28	28	301
16	26	21	44
17	36	33	252
18	27	29	257
19	36	35	193
H	K= 9	L= 1	
0	163	172	270
1	130	132	33
2	35	34	116
3	64	64	71
4	82	77	77
5	123	127	24
6	118	118	104
7	22	19	158
8	64	62	74
9	11*	5	211
10	35	31	74
11	71	24	0
12	67	69	105
13	69	69	202
14	25	25	9
15	98	95	348
16	87	78	214
17	74	65	151
18	89	77	109
H	K=10	L= 1	
0	176	125	0
1	235	249	6
2	71	74	178
3	32	36	164
4	49	46	219
5	115	111	170
6	49	48	337
7	21	21	52
8	20	22	19
9	65	59	2
10	43	37	130
11	27	27	234
12	45	39	266
13	32	28	42
14	67	59	117
15	69	65	198
16	52	50	310
H	K=11	L= 1	
0	28	23	90
1	34	36	205
2	40	37	283
3	30	30	170
4	41	44	305
5	26	30	94
6	86	81	80
7	37	38	293
8	75	72	265
9	43	40	124
10	60	53	70
11	58	54	9
12	41	39	121
H	K=12	L= 1	
0	24	29	180
1	22	23	162
2	36	33	112
3	24	25	196
4	7*	6	322
5	66	64	0
6	67	62	141
7	43	47	208
8	31	27	225
9	22	19	186
H	K= 0	L= 2	
0	1693E	2335	0
1	1525E	2082	180
2	744	777	180
3	99	56	180
4	78	56	0
5	17	5	0
6	197	191	0
7	152	152	180
8	116	122	0
9	75	75	180
10	50	38	180
11	225	227	0
12	266	258	0
13	205	203	180
14	138	134	180
15	203	198	0
16	329	392	0
17	476	485	180
18	215	205	180
19	55	60	180
20	62	55	180
21	8*	5	0
22	7*	7	180
23	34	31	0
H	K= 1	L= 2	
0	596	578	90
1	585	594	50
2	276	273	323
3	239	243	41
4	135	139	262
5	345	352	357
6	199	187	1
7	95	89	326
8	164	159	77
9	74	69	20
10	108	104	146
11	121	112	358
12	127	121	347
13	189	188	205
14	155	149	174
15	254	248	324
16	329	343	45
17	227	231	146
18	43	42	262
19	64	65	198
20	39	39	132
21	87	83	349
22	100	95	36
23	30	27	185
24	56	54	54
25	48	47	183
H	K= 2	L= 2	
0	449	449	180
1	338	333	353
2	155	148	73
3	45	43	130
4	106	95	183
5	80	79	247
6	113	107	115
7	126	120	55
8	139	136	318
9	177	171	138
10	352	351	275
11	138	122	215
12	210	218	262
13	177	173	295
14	76	73	37
15	78	81	310
16	136	139	81
17	142	140	47
18	79	78	333
19	42	41	335
20	41	38	85
21	58	58	347
22	59	60	62
23	50	48	74
24	41	35	277
H	K= 3	L= 2	
0	304	296	90
1	223	221	94
2	146	138	270
3	55	55	344
4	136	130	326
5	49	44	263
6	171	161	57
7	192	185	102
8	115	115	316
9	196	188	164
10	150	140	189
11	92	87	294
12	272	276	110
13	143	139	57
14	60	59	116
15	45	49	259
16	136	139	116
17	22	15	120
18	98	91	137
19	31	28	7
20	20	23	188
21	50	49	277
22	32	28	102
23	7*	7	293
24	33	30	78
H	K= 4	L= 2	
0	252	248	0
1	117	108	184
2	25	26	349
3	106	109	37
4	256	250	342
5	221	209	198
6	55	55	333
7	199	188	225
8	72	65	66
9	173	164	241
10	179	175	74
11	101	93	202
12	93	92	21
13	157	154	198
14	63	62	231
15	72	73	300
16	129	133	333
17	150	159	214
18	64	72	144
19	58	62	54
20	75	77	291
21	29	29	291
22	31	30	51
H	K= 5	L= 2	
0	65	59	90
1	376	381	20
2	325	321	311
3	266	274	280
4	221	221	54
5	30	27	99
6	57	52	292
7	97	104	313
8	60	52	275
9	192	193	314
10	128	123	39
11	35	37	307
12	50	51	117
13	132	129	38
14	122	129	257
15	139	145	316
16	108	114	71
17	71	76	72
18	20	23	326
19	27	29	194
20	43	37	75
21	58	59	191
22	53	56	108
H	K= 6	L= 2	
0	138	129	0
1	145	136	219
2	89	81	79
3	36	42	191
4	109	107	164
5	179	179	1
6	107	108	17
7	139	140	100
8	222	230	253
9	85	82	319
10	125	129	251
11	101	102	305
12	47	42	180
13	71	73	114
14	190	191	239
15	124	125	294
16	30	31	104
17	20	30	318
18	54	53	24
19	65	63	187
20	38	40	162
21	47	47	2
22	43	43	10
H	K= 7	L= 2	
0	21	13	90
1	26	26	252
2	90	89	136
3	77	81	358
4	62	61	166
5	88	88	349
6	103	100	276
7	281	286	261
8	432	445	105
9	321	328	77
10	156	158	261
11	28	27	65
12	100	99	289
13	111	116	184
14	38	42	195
15	34	32	174
16	46	44	142
17	41	39	40
18	42	40	19
19	35	37	129
H	K= 8	L= 2	
0	96	93	0
1	93	92	203
2	105	107	224
3	150	159	289
4	34	24	72
5	13	17	40
6	150	155	247
7	235	237	318
8	276	275	20
9	276	277	174
10	87	85	193
11	68	69	161
12	21	29	248
13	43	39	218
14	77	70	119
15	69	65	32
16	57	52	0
17	34	36	133
18	53	48	264
19	97	92	284
H	K= 9	L= 2	
0	118	118	90
1	104	101	97
2	138	147	217
3	151	158	24
4	180	186	275
5	136	137	285
6	126	176	108
7	153	155	58
8	141	139	280
9	82	81	284
10	45	41	75
11	8*	10	68
12	31	35	94
13	43	46	8
14	44	41	349
15	38	35	260
16	34	34	41
17	50	48	200
H	K=10	L= 2	
0	28	22	180
1	73	70	88
2	117	122	273
3	77	76	215
4	69	69	186
5	8*	27	322
6	20	18	67
7	41	41	117
8	69	67	249
9	46	43	309
10	18	17	51
11	7*	0	0
12	42	39	221
13	80	76	338
14	58	56	344
15	28	25	219
H	K=11	L= 2	
0	45	42	270
1	49	47	296
2	59	58	77
3	26	30	90
4	25	27	37
5	25	26	149
6	36	35	183
7	56	50	337
8	74	70	74
9	71	63	75
10	58	55	323
11	48	46	174
12	72	71	217
H	K=12	L= 2	
0	14	18	0
1	42	44	229
2	51	54	138
3	51	50	356
4	40	37	15
5	25	26	139
6	67	64	242
7	79	72	299
H	K= 0	L= 3	
1	756	769	270
2	190	180	270
3	19	11	90
4	157	149	270
5	9*	13	90
6	9*	15	90
7	304	304	270
8	17	3	90
9	125	116	270
10	27	20	270
11	63	64	90
12	10*	22	90
13	29	28	90
14	44	37	90
15	82	79	270
16	48	49	270
17	156	153	270
18	74	73	270
19	45	28	90
20	42	35	270
21	61	60	90
22	8*	16	90
23	68	67	270
24	26	26	90
H	K= 2	L= 3	
0	36	36	180
1	171	171	76
2	236	239	171
3	73	76	226
4	113	102	185
5	151	157	86
6	303	311	187
7	294	292	121
8	521	540	359
9	216	219	333
10	159	147	209
11	57	56	42
12	39	37	214
13	28	28	341
14	71	75	11
15	163	165	273
16	58	57	7
17	56	51	262
18	55	54	159
19	75	78	249
20	19	18	102
21	73	68	267
22	32	31	190
23	28	25	115
H	K= 3	L= 3	
0	9*	3	90
1	126	144	80
2	88	85	145
3	183	193	97
4	77	73	32
5	183	177	57
6	132	125	145
7	200	189	263
8	342	336	340
9	439	420	91
10	272	263	18
11	40	28	7
12	52	52	180
13	72	72	183
14	52	46	349
15	94	94	144
16	75	74	355
17	77	77	80
18	178	179	177
19	61	59	226
20	29	31	301
21	36	37	96
22	15	14	169
23	26	26	261
H	K= 4	L= 3	
0	254	241	180
1	104	101	269
2	187	193	190
3	26	26	301
4	63	74	190
5	49	54	301
6	51	43	352
7	159	154	270
8	65	59	259
9	78	79	263
10	93	91	208
11	61	56	247
12	28	24	351
13	155	156	280
14	75	73	192
15	150	150	284
16	129	133	198
17	33	34	74
18	8*	13	25
19	39	43	74
20	44	45	33
21	49	52	282
22	22	20	162
H	K= 5	L= 3	
0	225	218	90
1	413	418	274
2	72	64	341
3	26	27	356
4	90	87	13
5	73	76	77
6	28	18	128
7	31	30	227
8	33	35	115
9	124	124	272
10	87	82	177
11	27	29	74
12	96	99	150
13	69	70	269
14	38	33	191
15	95	93	97
16	67	66	67
17	106	113	270
18	27	24	271
19	27	26	232
20	49	51	208
21	26	31	147
22	28	26	204
H	K= 6	L= 3	
0	179	173	180
1	99	90	114
2	32	26	196
3	94	90	103
4	79	73	343
5	70	61	78
6	39	37	73
7	128	127	280
8	32	35	130
9	144	145	290
10	102	98	236
11	224	229	83
12	49	48	74
13	69	69	306
14	65	65	209
15	92	91	77
16	52	52	185
17	8*	7	281
18	17	18	299
19	30	28	264
20	7*	24	15
21	35	33	288
H	K= 7	L= 3	
0	30	21	270
1	132	125	96
2	86	85	94
3	200	201	269
4	65	64	313
5	176	178	81
6	203	203	149
7	110	106	240
8	44	45	261
9	248	249	106
10	343	352	9
11	104	98	309
12	59	63	257
13	194	193	87
14	59	61	159
15	34	33	246
16	28	32	291
17	16	19	162
18	22	20	12
19	21	22	254
H	K= 8	L= 3	
0	222	224	180
1	38	38	173
2	46	44	31
3	72	74	291
4	30	27	297
5	163	163	62
6	341	353	171
7	131	127	252
8	37	41	342
9	144	147	284
10	74	68	228
11	63	60	85
12	20	21	224
13	21	29	102
14	48	51	2
15	24	26	294
16	15	15	58
17	50	49	266
18	19	19	147
H	K= 9	L= 3	
0	10*	13	270
1	108	113	79
2	46	47	134
3	59	54	111
4	47	49	35
5	57	60	49
6	76	71	159
7	47	48	280
8	8*	3	28
9	31	31	245
10	8*	5	0
11	8*	11	304
12	37	36	149
13	47	47	239
14	47	48	357
15	27	25	305
16	105	103	188
H	K=10	L= 3	
0	256	266	0
1	65	67	12
2	103	105	184
3	7*	2	270
4	92	49	194
5	33	36	83
6	60	60	351
7	35	38	79
8	25	24	348
9	32	32	50
10	68	64	174
11	15	15	285
12	25	23	191
13	53	49	81
H	K=11	L= 3	
0	24	20	90
1	28	28	265
2	8*	8	83
3	8*	3	314
4	16	16	3
5	41	41	103
6	32	30	90
7	70	69	261
8	23	21	204
9	55	53	101
10	48	46	30
H	K=12	L= 3	
0	49	58	180
1	7*	6	126
2	24	24	152
3	22	20	249
H	K= 0	L= 4	
0	303	310	180
1	1184E	1250	180
2	60	60	180
3	90	79	0
4	22	21	0
5	93	90	0
6	45	48	180
7	9*	14	180
8	10*	0	0
9	19	27	180
10	12	14	180
11	165	158	0
12	76	73	180
13	152	154	180
14	33	36	180
15	222	223	0
16	30	25	180
17	419	441	180
18	52	51	0
19	8*	6	0
20	74	76	0
21	25	28	0
22	53	52	0
H	K= 1	L= 4	
0	549	537	90
1	249	245	354
2	94	80	297
3	59	63	5
4	35	24	236
5	193	185	2
6	44	38	185
7	46	41	135
8	88	87	105
9	57	50	179
10	71	71	180
11	92	85	7
12	45	40	205
13	117	114	159
14	22	14	336
15	240	243	12
16	241	242	94
17	162	160	179
18	78	74	280
19	41	41	157
20	25	26	12
21	72	72	3
22	34	36	129
H	K= 2	L= 4	
0	85	82	0
1	210	206	1
2	69	63	79
3	60	61	234
4	72	71	240
5	31	33	284
6	99	94	97
7	102	94	337
8	72	75	209
9	68	64	218
10	244	229	265
11	57	57	101
12	108	108	283
13	112	112	49
14	53	55	68
15	74	76	29
16	147	145	85
17	71	71	354
18	48	46	291
19	26	21	338
20	8*	7	113
21	56	53	336
22	48	48	90
H	K= 3	L= 4	
0	190	185	90
1	61	64	212
2	71	71	242
3	33	35	277
4	53	50	169
5	19	15	151
6	155	155	113
7	40	37	176
8	67	58	140
9	193	185	168
10	30	20	350
11	66	67	67
12	166	169	85
13	89	69	9
14	11*	11	317
15	11*	11	276
16	82	83	77
17	20	17	321
18	33	33	54
19	54	54	340
20	23	20	288
21	33	25	334
22	24	26	73
H	K= 4	L= 4	
0	66	64	180
1	178	171	217
2	96	93	134
3	82	79	334
4	71	70	194
5	108	102	179
6	34	33	180
7	91	88	151
8	68	63	90
9	63	59	84
10	119	123	91
11	22	22	150
12	22	20	0
13	88	91	182
14	29	30	346
15	95	90	5
16	32	34	280
17	120	122	173
18	62	66	77
19	38	37	11
20	54	57	277
21	28	28	53
H	K= 5	L= 4	
0	166	152	90
1	226	216	6
2	203	195	247
3	82	80	105
4	156	155	108
5	54	55	104
6	50	45	214
7	63	59	50
8	83	76	242
9	128	128	20
10	81	80	111
11	9*	17	5
12	60	62	100
13	64	61	349
14	141	140	257
15	115	116	7
16	103	110	103
17	41	44	352
18	46	49	235
19	4*	10	180
20	12	8	208
H	K= 6	L= 4	
0	76	75	180
1	79	74	171
2	82	76	101
3	27	21	311
4	33	34	90
5	151	152	347
6	75	71	75
7	49	47	297
8	177	181	281
9	9*	11	342
10	90	87	299
11	42	42	7
12	48	48	88
13	29	28	157
14	140	138	277
15	83	87	24
16	41	40	79
17	43	41	38
18	21	20	82
19	54	54	170
H	K= 7	L= 4	
0	23	20	90
1	9*	14	109
2	52	51	48
3	59	59	11
4	9*	12	338
5	53	48	11
6	83	79	276
7	78	76	145
8	395	413	89
9	60	60	326
10	165	162	262
11	29	31	331
12	85	81	243
13	83	76	169
14	26	25	283
15	8*	6	133
16	15	10	90
17	45	43	344
18	11*	6	125
H	K= 8	L= 4	
0	9*	9	0
1	99	96	171
2	87	86	274
3	39	37	343
4	38	42	86
5	9*	16	0
6	121	119	261
7	171	169	4
8	122	119	114
9	249	250	185
10	8*	14	90
11	19	20	186
12	25	30	347
13	17	19	158
14	64	53	73
15	60	56	349
16	5*	1	90
H	K= 9	L= 4	
0	95	93	90
1	71	72	209
2	76	74	273
3	123	124	353
4	161	162	266
5	20	19	61
6	119	119	94
7	85	85	351
8	116	118	260
9	29	29	15
10	47	46	117
11	22	17	80
12	20	17	122
13	46	48	355
14	24	27	212
H	K=10	L= 4	
0	8*	7	0
1	42	43	253
2	100	102	276
3	61	60	175
4	31	26	55
5	42	39	351
6	36	34	63
7	30	30	201
8	44	45	286
9	24	24	27
10	25	22	86
H	K=11	L= 4	
0	35	31	270
1	13	11	39
2	57	56	101
3	4*	3	180
4	5*	3	285
5	28	30	200
6	11*	12	239
7	51	43	0
H	K= 0	L= 5	
1	316	312	270
2	171	168	90
3	23	16	90
4	30	35	270
5	36	33	90
6	64	71	270
7	77	80	270
8	11*	8	90
9	80	83	270
10	11*	12	90
11	8*	12	270
12	8*	10	270
13	21	14	270
14	17	15	270
15	108	98	270
16	7*	11	270
17	88	86	270
18	100	98	90
19	76	78	90
20	35	34	90
21	72	70	90
H	K= 1	L= 5	
0	57	52	270
1	135	130	17
2	198	195	309
3	47	41	314
4	92	87	286
5	164	162	265
6	94	98	78
7	85	82	123
8	129	127	178
9	72	67	5
10	45	44	318
11	22	20	90
12	42	42	208
13	22	19	126
14	110	110	217
15	69	68	301
16	41	40	212
17	96	93	293
18	106	107	35
19	67	65	168
20	40	41	146
21	34	32	227
H	K= 2	L= 5	
0	65	62	0
1	70	67	124
2	91	84	216
3	47	49	262
4	47	47	94
5	26	24	134
6	82	84	189
7	118	117	9
8	286	289	344
9	165	163	217
10	125	120	180
11	30	28	140
12	63	65	223
13	9*	16	17
14	71	73	254
15	61	62	271
16	8*	11	114
17	18	20	171
18	30	32	220
19	26	29	310
H	K= 3	L= 5	
0	84	85	90
1	47	43	108
2	45	50	111
3	21	18	64
4	56	53	353
5	30	24	326
6	93	90	202
7	219	224	304
8	146	147	55
9	193	195	84
10	107	106	319
11	57	55	212
12	86	87	198
13	21	18	0
14	34	30	141
15	54	56	80
16	61	61	358
17	66	67	131
18	93	93	203
19	52	52	337
H	K= 4	L= 5	
0	54	50	180
1	105	102	291
2	37	36	58
3	62	59	330
4	11*	14	60
5	70	68	346
6	37	34	213
7	109	107	288
8	44	41	127
9	22	24	274
10	85	78	162
11	47	43	42
12	21	18	238
13	57	57	262
14	36	35	146
15	62	65	260
16	94	99	143
17	62	58	41
18	5*	15	99
19	27	29	55
H	K= 5	L= 5	
0	154	154	270
1	206	212	281
2	36	34	96
3	47	46	284
4	35	35	135
5	30	28	196
6	92	91	210
7	31	26	225
8	62	64	239
9	61	62	244
10	38	32	189
11	21	21	122
12	65	57	216
13	30	31	319
14	19	17	259
15	55	56	69
16	69	67	295
17	75	75	268
H	K= 6	L= 5	
0	107	104	180
1	40	39	329
2	22	16	246
3	39	39	350
4	70	70	312
5	40	35	108
6	80	78	282
7	47	45	252
8	34	32	342
9	77	79	260
10	68	66	116
11	162	161	87
12	54	54	276
13	29	30	223
14	46	45	169
15	47	46	87
16	56	55	214
17	21	23	349
H	K= 7	L= 5	
0	9*	1	90
1	57	56	90
2	53	50	245
3	91	91	284
4	64	65	81
5	146	149	104
6	146	144	188
7	116	117	305
8	71	72	106
9	159	159	50
10	209	210	348
11	128	128	220
12	52	52	131
13	80	84	114
14	50	51	242
15	39	39	280
16	26	21	286
H	K= 8	L= 5	
0	100	101	180
1	47	47	29
2	8*	10	321
3	47	47	264
4	58	56	55
5	127	128	136
6	186	192	196
7	130	126	315
8	53	49	0
9	94	92	279
10	82	84	107
11	59	61	60
12	5*	12	34
13	30	30	40
14	18	19	330
H	K= 9	L= 5	
0	58	60	90
1	77	81	111
2	26	26	209
3	29	28	139
4	55	56	350
5	51	49	133
6	61	60	241
7	40	38	287
H	K=10	L= 5	
0	162	161	0
1	80	80	179
2	53	57	180
3	5*	4	29
4	26	25	128
5	46	46	32
6	18	16	3
7	5*	13	108
H	K= 0	L= 6	
0	704	737	180
1	331	351	180
2	153	148	0
3	79	82	0
4	36	17	0
5	37	41	0
6	64	59	180
7	27	25	0
8	8*	0	0
9	9*	2	0
10	54	50	0
11	58	54	0
12	65	64	180
13	19	21	180
14	61	60	0
15	127	132	0
16	205	209	180
17	148	150	180
18	171	126	0
H	K= 1	L= 6	
0	231	230	90
1	209	209	293
2	63	61	209
3	31	24	168
4	22	11	25
5	49	49	53
6	43	43	154
7	57	62	115
8	29	29	46
9	44	41	174
10	35	30	0
11	45	42	25
12	30	32	161
13	28	23	160
14	73	70	342
15	124	125	37
16	179	181	152
17	144	146	246
18	56	53	264
H	K= 3	L= 6	
0	32	35	90
1	48	47	244
2	31	24	333
3	9*	7	310
4	29	27	56
5	9*	11	90
6	47	53	80
7	9*	40	285
8	48	49	114
9	66	62	237
10	89	85	358
11	29	27	57
12	47	48	43
13	49	48	296
14	33	31	240
15	16	20	95
16	23	25	78
H	K= 4	L= 6	
0	100	97	180
1	79	73	153
2	64	62	56
3	37	36	7
4	59	59	157
5	9*	11	90
6	41	42	167
7	50	51	104
8	31	26	68
9	47	48	52
10	61	57	159
11	28	32	288
12	57	57	212
13	31	31	148
14	23	30	350
15	53	51	18
16	85	82	187
17	66	68	136
H	K= 5	L= 6	
0	111	106	90
1	97	94	289
2	112	112	208
3	86	88	105
4	98	81	114
5	40	40	264
6	21	18	223
7	21	19	248
8	41	41	294
9	77	79	56
10	44	44	159
11	18	11	54
12	25	26	85
13	54	54	274
14	56	54	282
15	100	102	62
16	59	65	122
H	K= 6	L= 6	
0	25	31	180
1	39	41	105
2	54	51	16
3	30	25	9
4	20	22	12
5	44	48	0
6	48	46	94
7	95	97	289
8	64	63	233
9	46	45	124
10	34	33	333
11	56	56	97
12	41	42	107
13	36	35	229
14	65	66	320
H	K= 7	L= 6	
0	19	20	90
1	8*	5	20
2	33	33	15
3	19	18	347
4	8*	12	216
5	8*	10	37
6	36	34	233
7	144	146	102
8	159	163	74
9	198	205	271
10	73	74	281
11	23	22	304
12	27	28	204
H	K= 8	L= 6	
0	35	38	180
1	41	41	191
2	60	61	308
3	46	46	84
4	33	35	45
5	5*	8	312
6	45	47	281
7	118	119	56
8	154	159	166
9	56	83	184
10	33	51	21
H	K= 9	L= 6	
0	25	23	90
1	74	76	239
2	69	69	346
3	51	52	326
4	52	54	240
5	76	79	98
H	K= 0	L= 7	
1	39	37	270
2	157	163	90
3	20	22	270
4	21	15	90
5	8*	11	90
6	20	24	270
7	8*	21	90
8	19	28	270
9	7*	4	270
10	8*	16	90
11	17	21	90
12	7*	4	270
13	15	17	90
14	22	24	270
15	22	22	270
H	K= 1	L= 7	
0	30	26	270
1	81	81	357
2	87	89	270
3	21	21	97
4	64	53	244
5	29	24	147
6	59	53	103
7	63	57	174
8	41	38	138
9	74	59	357
10	26	21	278
11	17	18	169
12	8*	11	209
13	5*	8	213
14	59	52	258
15	65	55	358
H	K= 2	L= 7	
0	8*	0	0
1	65	53	196
2	59	52	243
3	21	21	322
4	43	39	88
5	19	16	270
6	8*	10	238
7	123	110	354
8	78	65	318
9	201	171	184
10	73	59	104
11	8*	14	130
12	16	21	314
13	5*	11	43
14	53	46	270
15	22	16	23
H	K= 3	L= 7	
0	30	27	90
1	8*	3	125
2	8*	10	81
3	8*	10	0
4	36	38	307
5	32	34	169
6	46	51	252
7	136	98	346
8	151	155	93
9	44	45	44
10	71	71	270
11	49	55	195
12	26	30	212
13	35	32	0
14	31	31	123
H	K= 4	L= 7	
0	7*	12	180
1	46	48	356
2	47	54	81
3	8*	6	23
4	27	20	72
5	8*	10	143
6	42	46	245
7	41	40	334
8	51	49	85
9	23	17	218
10	41	42	95
11	33	32	13
12	34	37	278
H	K= 5	L= 7	
0	172	172	270
1	29	31	309
2	18	15	98
3	18	16	186
4	18	23	98
5	24	25	195
6	24	23	245
7	23	21	0
8	33	34	278
9	5*	9	57
10	14	14	353
11	21	18	149
12	27	26	262
H	K= 6	L= 7	
0	7*	10	180
1	45	50	351
2	8*	7	15
3	43	45	351
4	5*	12	294
5	5*	17	155
6	60	59	265
7	4*	3	270
8	23	23	318
9	59	57	210
10	86	89	96
H	K= 7	L= 7	
0	4*	14	270
1	5*	14	146
2	45	46	273
3	13	22	322
4	62	68	85
5	58	60	150
6	90	92	257
7	89	97	336
H	K= 0	L= 8	
0	384	420	180
1	68	78	0
2	143	154	0
3	17	31	0
4	17	17	0
5	4*	9	180
6	29	25	180
7	5*	11	0
8	13	9	180
9	5*	1	0
10	12	14	0
H	K= 1	L= 8	
0	44	41	270
1	164	169	265
2	59	65	171
3	7*	11	180
4	8*	10	49
5	5*	9	105
6	28	29	173
7	5*	6	54
8	5*	15	298
9	13	15	222
10	37	35	356
H	K= 2	L= 8	
0	8*	11	0
1	33	38	108
2	16	19	282
3	16	18	251
4	16	6	298
5	28	27	73
6	15	19	335
7	31	34	241
8	34	41	180
9	42	44	288
10	30	36	52
H	K= 3	L= 8	
0	0	0	0
1	16	18	285
2	5*	6	0
3	5*	8	29
4	5*	6	159
5	5*	12	98
6	5*	6	225
7	4*	4	270
8	24	26	192
9	43	47	288
H	K= 4	L= 8	
0	84	88	180
1	28	27	71
2	39	38	1
3	5*	6	239
4	23	19	180
5	5*	7	292
6	25	26	174
7	30	31	67
H	K= 5	L= 8	
0	11*	10	270
1	78	84	255
2	51	52	167
3	59	61	87

Determination and refinement of the structure

A three-dimensional vector map, sharpened by the method of Jacobson, Wunderlich & Lipscomb (1961) was calculated. In this vector map, layers of high density are visible with maxima at the sections $u = 0{\cdot}000$, 0·132, 0·368 and 0·500 (Fig. 1). First, this means that the aflatoxin B$_2$ molecules have a plane structure and are parallel to the (100) plane and, second, that the distances from the molecule to two of the three screw axes are 0·066 x/a and 0·184 x/a. It is clear from the characteristic distribution of the peaks in these sections that the molecule contains a number of hexagonal rings. The oval peaks near the origin in the section with $u = 0{\cdot}000$ indicate that the rings are not altogether oriented parallel to the b axis. From these peaks, a rotation of about $\pm 4°$ from the axis can be deduced.

During the work on this structure, the structural formula of aflatoxin B$_1$ became known from a paper by Asao, Büchi, Abdel-Kader, Chang, Wick & Wogan (1963), from which formula we could derive a plane model containing 19 atoms. (At that stage we did not know that the carbon atom of the methoxyl group was also in the same plane.)

Three vector maps, containing the endpoints of vectors between trial molecules interrelated by screw axes, were drawn on transparent paper and used to analyse the three sections containing the intermolecular vectors. This resulted in the correct position of the model with respect to the screw axes. The four missing atoms were subsequently found by Fourier methods. Initial refinement was done with 412 structure factors ($|F| > 10$) of the first set of data and described by van Soest & Peerdeman (1964). A further refinement was carried out on Telefunken TR4 and Electrologica X-8 computers with a block-diagonal least-squares program written by Palm & Peterse (1964). The second set of data was used after omitting 56 reflexions supposed to be affected by extinction (found by comparison of the two sets of data) and replacement of the non-observed reflexions by half the minimum value observed in corresponding ranges of $\sin \theta/\lambda$.

The refinement was started at an overall temperature factor of 2·7 Å^2. During five cycles, $R = \sum||F_o| - |F_c||/\sum|F_o|$ fell to 0·18. Four further cycles with individual isotropic temperature factors reduced R to 0·142 and three more with anisotropic temperature factors to 0·081. At this point, a difference map was calculated revealing the approximate positions of the 14 hydrogen atoms. The hydrogen atoms were given isotropic temperature factors equal to those of the carbon atoms to which they are bound. During the next four cycles, R dropped to 0·051; the positions of all the atoms, the anisotropic temperature factors of the heavy atoms and the scaling factor were refined. Using these parameters, the 56 structure factors of the large crystal supposed to be affected by extinction were calculated, giving an R factor of 0·20 for these structure factors, whereas for the corresponding structure factors of the small crystal $R = 0{\cdot}07$. For 8 of these 56 reflexions, F_c is still greater than F_o and after omitting them, R decreased from 0·07 to 0·03.

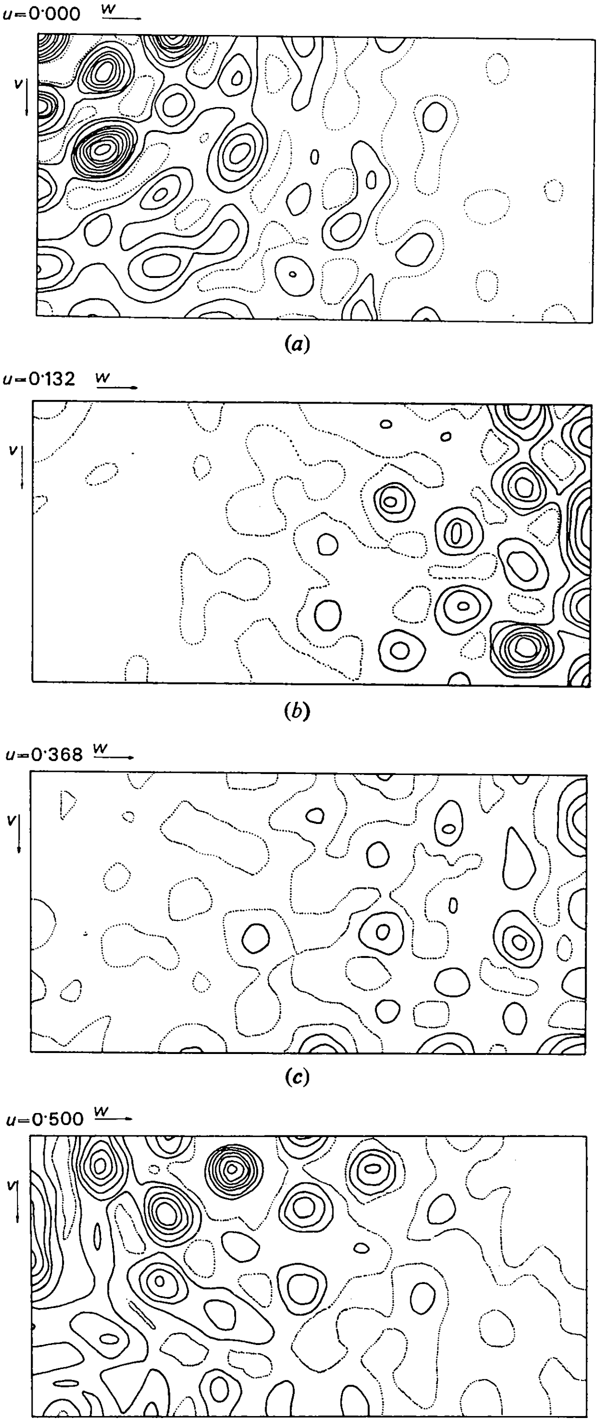


Fig. 1. Sections through the sharpened Patterson function $P(u,v,w)$ for the values u (a) 0·000; (b) 0·132; (c) 0·368 and (d) 0·500. Contours are at arbitrary intervals: solid, positive; dotted, zero.

The 48 remaining structure factors of the small crystal were included in the last three cycles, using weights inversely proportional to mean values of $\sum(|F_o|-|F_c|)^2$ in corresponding ranges of $|F_o|$ as calculated during the refinement. After the last cycle, the shifts in the positional and thermal parameters for non-hydrogen atoms were all less than $\sigma/4$ and $\sigma/3$ respectively. Those for the hydrogen atoms were less than $\sigma/2$. The final R for 1,717 reflexions was 0·046. A list of the final structure factors [here and in the preceding cases calculated by using the atomic scattering factors taken from *International Tables for X-ray Crystallography* (1962)] is given in Table 1 and the structural parameters are given in Tables 2 to 4. The arbitrary numbering of the atoms of the molecule is given in Fig. 2.

Table 2. *Final positional parameters and e.s.d.'s* (× 10^5) *for the carbon and oxygen atoms*

	x/a	y/b	z/c
C(1)	−6577 (41)	72061 (21)	69511 (11)
C(2)	−6796 (39)	61294 (23)	64294 (11)
C(3)	−6477 (38)	48191 (21)	68027 (11)
C(4)	−6624 (37)	65511 (20)	75993 (11)
C(5)	−6542 (34)	52040 (20)	75277 (11)
C(6)	−7064 (41)	71954 (21)	82353 (11)
C(7)	−6500 (37)	43694 (20)	80971 (11)
C(8)	−6609 (38)	29701 (21)	80780 (11)
C(9)	−6717 (36)	49929 (20)	87206 (11)
C(10)	−6794 (43)	22229 (22)	86568 (12)
C(11)	−6872 (42)	42764 (24)	93011 (12)
C(12)	−7071 (41)	29161 (23)	92469 (12)
C(13)	−6638 (53)	46792 (28)	180 (12)
C(14)	−6801 (63)	33168 (31)	3665 (13)
C(15)	−6179 (56)	10448 (23)	73928 (15)
C(16)	+12162 (66)	52855 (33)	2592 (14)
C(17)	+24074 (68)	40972 (42)	4541 (17)
O(1)	−6425 (44)	83744 (17)	68374 (9)
O(2)	−7760 (37)	83462 (16)	83581 (9)
O(3)	−6908 (29)	63472 (15)	87826 (7)
O(4)	−7516 (38)	23089 (18)	98465 (9)
O(5)	−6167 (34)	24503 (15)	74679 (8)
O(6)	+10166 (47)	31964 (24)	7240 (10)

Table 3. *Final positional parameters and e.s.d.'s* (× 10^4) *and isotropic temperature factors* (Å^2) *for the hydrogen atoms*

	x/a	y/b	z/c	B
H(1)	−1936 (51)	6254 (34)	6178 (17)	2·37
H(2)	+505 (52)	6190 (33)	6168 (16)	2·37
H(3)	−1739 (50)	4254 (33)	6703 (16)	2·11
H(4)	+467 (53)	4299 (31)	6702 (15)	2·11
H(5)	−572 (61)	943 (37)	6907 (18)	3·83
H(6)	−1567 (58)	701 (36)	7615 (18)	3·83
H(7)	+351 (58)	736 (35)	7611 (18)	3·83
H(8)	−773 (56)	1297 (32)	8637 (16)	2·69
H(9)	−1701 (55)	5078 (36)	100 (19)	3·56
H(10)	−1690 (63)	3071 (41)	729 (18)	4·32
H(11)	+1539 (60)	5751 (40)	9936 (19)	4·78
H(12)	+739 (67)	5825 (37)	613 (19)	4·78
H(13)	+3035 (64)	3707 (42)	76 (21)	5·67
H(14)	+3064 (70)	4260 (44)	827 (21)	5·67

Discussion

The bond lengths and the bond angles (not corrected for thermal motion) together with their standard deviations are listed in Tables 5 and 6. The bond lengths are also shown in Fig. 2. Aflatoxin B_2 is largely a plane molecule: all heavy atoms but C(16), C(17), and O(6) lie within 0·05 Å from a plane given by the least-squares

Table 4. *Final thermal parameters and e.s.d.'s* (× 10^5) *for the carbon and oxygen atoms*

The anisotropic temperature factor is defined as:

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

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	1463 (49)	489 (19)	159 (5)	+49 (32)	−16 (16)	+24 (8)
C(2)	1271 (45)	614 (20)	126 (5)	+6 (34)	+13 (14)	+16 (8)
C(3)	1123 (42)	504 (18)	123 (5)	−16 (39)	−24 (14)	−15 (8)
C(4)	1188 (43)	465 (18)	139 (5)	+6 (29)	−6 (14)	−9 (8)
C(5)	865 (38)	437 (18)	132 (5)	+38 (27)	+12 (13)	−18 (7)
C(6)	1431 (47)	487 (19)	148 (5)	−29 (31)	−58 (16)	−6 (8)
C(7)	1043 (42)	455 (18)	135 (5)	+8 (29)	−14 (15)	+22 (7)
C(8)	1119 (42)	484 (19)	165 (5)	−7 (39)	−40 (15)	+1 (8)
C(9)	1000 (41)	483 (19)	135 (5)	+41 (29)	−5 (14)	+12 (8)
C(10)	1527 (50)	486 (20)	188 (6)	−124 (33)	−19 (18)	+50 (9)
C(11)	1348 (48)	678 (22)	140 (5)	−7 (34)	+56 (17)	+9 (9)
C(12)	1349 (47)	660 (22)	154 (5)	−99 (34)	+2 (16)	+102 (9)
C(13)	2548 (74)	865 (26)	131 (5)	+188 (49)	+134 (21)	+19 (10)
C(14)	3333 (93)	1007 (31)	143 (6)	+108 (59)	+156 (22)	+87 (11)
C(15)	2990 (80)	404 (20)	246 (7)	+13 (42)	−6 (25)	−44 (10)
C(16)	4007 (120)	1028 (33)	144 (6)	−192 (57)	−168 (24)	−12 (12)
C(17)	3626 (122)	1545 (50)	221 (9)	+16 (77)	−361 (28)	+28 (17)
O(1)	4047 (72)	497 (16)	195 (5)	−14 (36)	−24 (18)	+70 (7)
O(2)	2927 (55)	435 (14)	190 (4)	+67 (30)	−43 (15)	−50 (7)
O(3)	1794 (37)	455 (14)	127 (4)	+96 (24)	+16 (11)	−31 (5)
O(4)	2881 (56)	784 (18)	167 (4)	−179 (34)	+67 (15)	+133 (7)
O(5)	2760 (52)	393 (13)	161 (4)	+14 (28)	−52 (14)	−24 (6)
O(6)	4155 (83)	1205 (26)	178 (5)	+47 (45)	−181 (18)	+118 (10)

equation

$$-0{\cdot}99996X-0{\cdot}00552Y-0{\cdot}00764Z-0{\cdot}3139=0$$

The tetrahydrofuran ring protrudes from this plane and the equation of the plane formed by the four atoms C(13), C(14), C(16) and O(6) is

$$-0{\cdot}46563X+0{\cdot}38972Y+0{\cdot}79455Z-18{\cdot}1320=0$$

Table 5. *Bond lengths and e.s.d.'s* (Å)

C–C bonds			
sp^3–sp^3		–C–O bonds	
C(2)—C(3)	1·528 (3)	C(14)-O(6)	1·385 (5)
C(13)-C(14)	1·553 (4)	C(15)-O(5)	1·437 (3)
C(13)-C(16)	1·523 (5)	C(14)-O(4)	1·465 (3)
C(16)-C(17)	1·513 (6)	C(17)-O(6)	1·434 (5)
sp^2–sp^3		=C–O bonds	
C(1)—C(2)	1·521 (3)	C(9)—O(3)	1·381 (3)
C(3)—C(5)	1·516 (3)	C(8)—O(5)	1·339 (3)
C(11)-C(13)	1·504 (3)	C(12)-O(4)	1·359 (3)
sp^2–sp^2		C(6)—O(3)	1·401 (3)
C(1)—C(4)	1·467 (3)		
C(4)—C(6)	1·437 (3)	C–H bonds	
C(5)—C(7)	1·426 (3)	C(2)—H(1)	0·98 (4)
		C(2)—H(2)	1·01 (4)
C=C bonds		C(3)—H(3)	0·96 (4)
C(4)—C(5)	1·377 (3)	C(3)—H(4)	0·97 (4)
		C(10)-H(8)	0·94 (4)
C···C bonds		C(13)-H(9)	0·84 (4)
C(7)—C(8)	1·424 (3)	C(14)-H(10)	1·04 (4)
C(7)—C(9)	1·407 (3)	C(15)-H(5)	0·99 (4)
C(8)—C(10)	1·390 (3)	C(15)-H(6)	0·87 (4)
C(9)—C(11)	1·377 (3)	C(15)-H(7)	0·86 (4)
C(10)-C(12)	1·383 (3)	C(16)-H(11)	0·84 (4)
C(11)-C(12)	1·382 (3)	C(16)-H(12)	0·96 (4)
		C(17)-H(13)	0·96 (4)
C=O bonds		C(17)-H(14)	0·89 (4)
C(1)—O(1)	1·206 (3)		
C(6)—O(2)	1·197 (3)		

Table 6. *Bond angles and e.s.d.'s for the non-hydrogen atoms*

C(2)—C(1)—O(1)	125·08 (0·16)
C(4)—C(1)—O(1)	127·96 (0·20)
C(2)—C(1)—C(4)	106·94 (0·24)
C(1)—C(2)—C(3)	106·52 (0·15)
C(2)—C(3)—C(5)	104·60 (0·22)
C(1)—C(4)—C(5)	110·94 (0·17)
C(1)—C(4)—C(6)	126·17 (0·25)
C(5)—C(4)—C(6)	122·87 (0·18)
C(3)—C(5)—C(4)	110·95 (0·18)
C(3)—C(5)—C(7)	128·74 (0·23)
C(4)—C(6)—O(2)	128·90 (0·20)
C(4)—C(6)—O(3)	115·30 (0·24)
O(2)—C(6)—O(3)	115·78 (0·15)
C(5)—C(7)—C(8)	124·79 (0·16)
C(5)—C(7)—C(9)	117·15 (0·24)
C(8)—C(7)—C(9)	118·04 (0·18)
C(7)—C(8)—O(5)	114·63 (0·18)
C(7)—C(8)—C(10)	121·38 (0·18)
C(10)-C(8)—O(5)	123·96 (0·25)
C(8)—O(5)—C(15)	119·14 (0·17)
C(6)—O(3)—C(9)	122·60 (0·14)
C(7)—C(9)—O(3)	121·71 (0·18)
C(7)—C(9)—C(11)	121·65 (0·26)
O(3)—C(9)—C(11)	116·60 (0·18)
C(8)—C(10)-C(12)	116·50 (0·28)
C(9)—C(11)-C(12)	117·28 (0·19)
C(9)—C(11)-C(13)	132·24 (0·31)
C(12)-C(11)-C(13)	110·46 (0·23)
C(10)-C(12)-C(11)	125·11 (0·21)
C(10)-C(12)-O(4)	122·41 (0·31)
C(11)-C(12)-O(4)	112·46 (0·20)
C(11)-C(13)-C(14)	101·00 (0·29)
C(11)-C(13)-C(16)	115·38 (0·34)
C(14)-C(13)-C(16)	102·87 (0·38)
C(13)-C(14)-O(4)	107·32 (0·18)
C(13)-C(14)-O(6)	107·74 (0·45)
O(4)—C(14)-O(6)	110·00 (0·45)
C(13)-C(16)-C(17)	103·12 (0·48)
C(16)-C(17)-O(6)	103·86 (0·56)
C(14)-O(6)—C(17)	108·58 (0·36)
C(12)-O(4)—C(14)	107·26 (0·28)

Table 7. *Distances from the heavy atoms to the least-squares planes of the aflatoxin* B$_2$ *molecule* (Å)

$$-0{\cdot}99996X-0{\cdot}00552Y-0{\cdot}00764Z-0{\cdot}3139=0$$

C(1)	−0·007
C(2)	+0·023
C(3)	+0·003
C(4)	−0·010
C(5)	−0·006
C(6)	+0·007
C(7)	−0·013
C(8)	+0·003
C(9)	−0·011
C(10)	+0·012
C(11)	−0·005
C(12)	+0·018
C(13)	−0·035
C(14)	−0·021
C(15)	−0·004
O(1)	−0·023
O(2)	+0·047
O(3)	−0·007
O(4)	+0·043
O(5)	−0·015

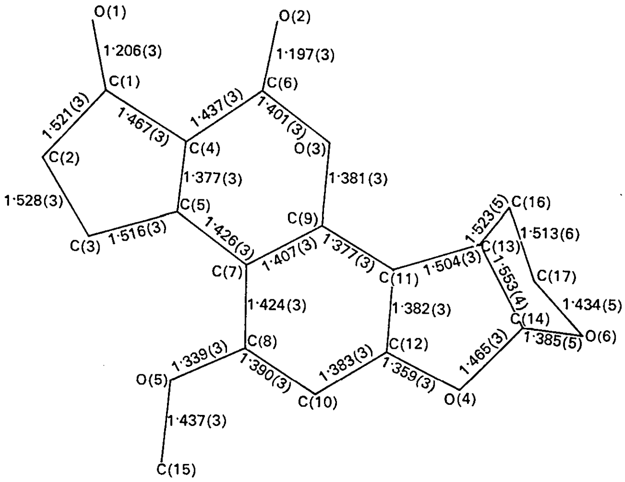


Fig. 2. Arbitrary numbering of the atoms, the bond lengths and their standard deviations of the aflatoxin B$_2$ molecule.

Table 7 (*cont.*)

$-0{\cdot}46563X+0{\cdot}38972Y+0{\cdot}79455Z-18{\cdot}1220=0$

C(13)	−0·012
C(14)	+0·013
C(16)	+0·008
O(6)	−0·009
[C(17)	−0·534]

The angle between these two planes is 117·21°. The distances from the atoms to their respective planes are listed in Table 7. The bond between the carbon atoms with the highest degree of substitution C(13) and C(14) is significantly longer than the other three sp^3–sp^3 bonds, as has been observed *e.g.* for iso-eremolactone (Oh & Maslen, 1968). The value of 1·467 Å for the sp^2–sp^2 bond in the cyclopentenone ring agrees well with 1·462 Å in cyclo-octatetraene (Bastiansen, Hedberg & Hedberg, 1957), whereas the two sp^2–sp^2 bonds in the lactone ring are significantly shorter. Although the variations in the six bond lengths of the benzene nucleus are considerable, the mean value of 1·394 Å is in agreement with the value of 1·394 Å given by Sutton (1965). Among the –C–O bonds, the short C(14)–O(6) bond of 1·385 Å deserves attention for it causes an asymmetry of the C(14)–O(4) and C(14)–O(6) bonds. The =C–O bonds present in a ring system are all longer than 1·339 Å, which is the length of the =C(8)–O(5) bond in the methoxyl group. Oh & Maslen (1968) have ascribed the extension of the two =C–O bonds (1·379 and 1·400 Å) in the γ-lactone ring of iso-eremolactone to the small interior angles that would induce a greater *p* character in the bonds. However, in the δ-lactone ring of aflatoxin B_2, the angles are normal and the same extension of the bond lengths occurs. The difference in the carbonyl bond angles O(2)–C(6)–C(4) and O(2)–C(6)–O(3) seems to be normal for γ-lactone rings [*cf.* iso-eremolactone and α-methyltetronic acid (MacDonald & Alleyne, 1963)] and for δ-lactone rings [*cf.* 4-hydroxycoumarin (Gaultier & Hauw, 1966) and 3-bromo-4-hydroxycoumarin monohydrate (Gaultier & Hauw, 1965)]. The smaller angle of the two is always at the side of the oxygen atom in the ring. The methoxyl group is coplanar with respect to the benzene nucleus of the molecule, as has been found *e.g.* for 2-*p*-methoxyphenyl-3,4-dibenzyl-1,3,4-thiadiazolidine-5-thione (Karle & Karle, 1965) and in myxin (Hanson, 1968*b*). However, in dibromo-eriostoic acid (Paton, Maslen & Watson, 1967), methyl is protruding from the plane. In aflatoxin B_2 as well as in myxin and dibromoeriostoic acid, the methyl hydrogen atoms have a staggered con-

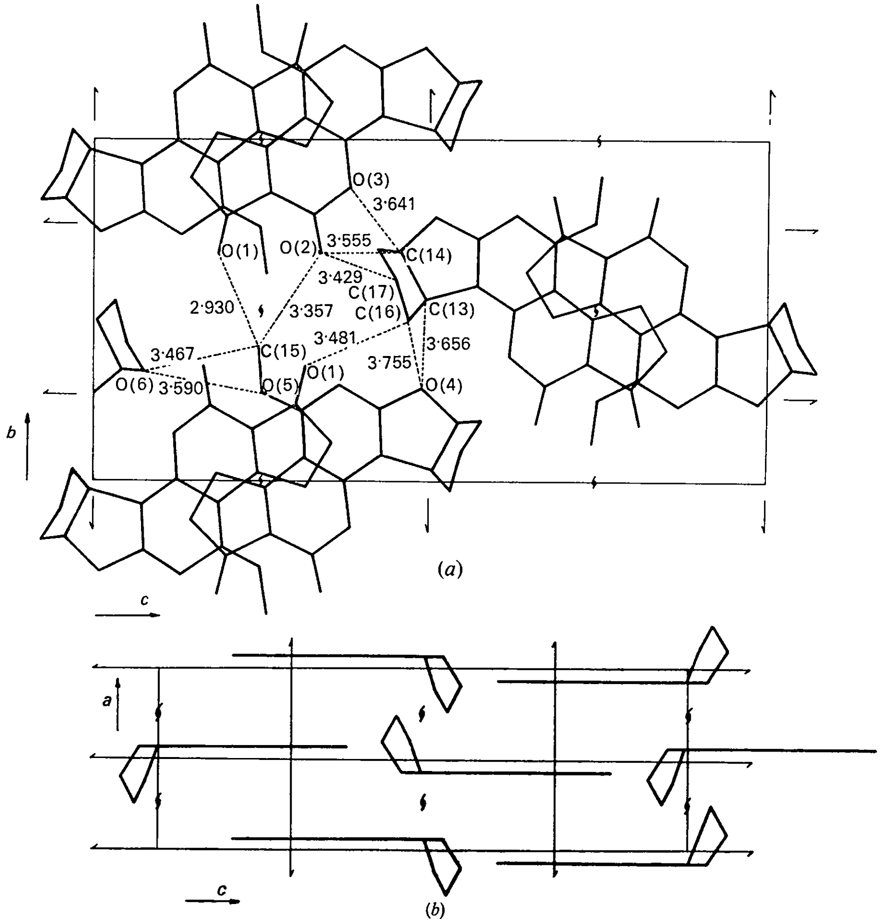


Fig. 3. Projection of the structure of aflatoxin B_2 (*a*) on the (100) plane and (*b*) on the (010) plane.

formation with respect to the C(benzene nucleus)–O bond. For this conformation, there is also little steric hindrance from the hydrogen atom of the benzene nucleus. The carbon–hydrogen bond lengths vary from 0·84 to 1·04 Å with a mean value of 0·94 Å and a standard deviation of 0·06 Å.

The crystal structure of aflatoxin B$_2$ is shown in Fig. 3(*a*) and (*b*) as a projection on the (100) and (010) plane respectively. It shows that one molecule is very close to another related to it by the translation *b*. The distance between the carbon atom C(15) of the methoxyl group of one molecule and the oxygen atom O(1) of the other molecule is 2·930 ± 0·003 Å.

Short $CH_3 \cdots O$ contacts in crystals are not unusual; a number of them have been reviewed in a paper by Sutor (1963). They mainly occur in the crystal structures of heterocyclic biologically important molecules with, in many cases, a conjugated double bond system. Aflatoxin B$_2$ clearly satisfies this classification. The shortest $CH_3 \cdots O$ contacts described in the review by Sutor are 3·00 Å. In the aflatoxin B$_2$ crystal, the distance is even 0·07 Å shorter. This short contact points to an interaction between the molecules and, because hydrogen atoms are involved, this interaction may be due to hydrogen bonding. Since the molecule is almost parallel to the (100) plane, two molecules are practically coplanar.

Thus, strings of coplanar molecules occur in the direction of the *b* axis. Within these strings, the molecules are possibly interconnected by hydrogen bonds. The strings are stacked in the direction of the *a* axis with an interplanar distance of $a/2 = 3{\cdot}463$ Å (*cf.* $C(1) \cdots C(8) = 3{\cdot}468$ and $C(5) \cdots C(5) = 3{\cdot}490$ Å).

Thermal motion

The atomic vibration ellipsoids have been analysed in terms of the rigid-body tensors of translation (**T**), libration (**L**) and screw motion (**S**) according to Schomaker & Trueblood (1968). Their program, available from the ACA library (No. 1) was adapted for use on an IBM 1800 computer. All the 23 heavy atoms were considered and the results of the analysis are given in Table 8. The r.m.s. deviation between the thermal parameters obtained by the least-squares refinement (further referred to as 'observed') and those calculated from the rigid-body tensors is 0·0043 Å^2. Although this value is not very low, the resulting modes of motion are physically reasonable. The translation tensor **T** is slightly anisotropic, the direction of the smallest vibration being almost parallel to the *a* axis (the angle between the two directions is 4°48′). A possible explanation is that the packing between the molecules is closest in the a direction, which is approximately perpendicular to the plane of the molecule. The unique origin obtained by symmetrizing **S** was used to calculate the inertia tensor **I** (principal axes and the cosines of their angles with respect to the crystallographic axes) of the aflatoxin B$_2$ molecule. There is a striking relation between **L** and **I**, which are both anisotropic. The largest principal axis of **L** has almost the same direction as the smallest principal axis of **I**. In other words, the libration is largest around an axis, the moment of inertia of which is smallest. The reverse is also valid (see Table 8): the libration is smallest around an axis of which the moment of inertia is largest. The angles between the corresponding axes of both tensors are 2°38′, 6°18′ and 5°4′, respectively. The libration tensor related to an axial system defined by the principal axes of the inertia tensor has also been calculated (Table 8) and is practically diagonal. The distance between the unique origin and the centre of gravity is small: 0·137 Å, compared for example with 0·542 Å for 7,7,8,8-tetracyanoquinodimethane (Hanson, 1968*a*). There are relatively large differences between observed and calculated U_{11}'s (r.m.s. $\Delta U_{11} = 0{\cdot}0091$ Å^2), as can be seen in Table 9. Especially, the values found for the atoms O(1) and O(5) are too large compared with the calculated values, indicating a possibly non-rigid thermal motion of these atoms perpendicular to the plane of the molecule.

Table 8. *Rigid-body thermal parameters*

$$\mathbf{T} = \begin{pmatrix} 193 & -4 & 8 \\ & 247 & 0 \\ & & 273 \end{pmatrix} \times 10^{-4}\ \text{Å}^2$$

$$\sigma(\mathbf{T}) = \begin{pmatrix} 17 & 13 & 11 \\ & 12 & 10 \\ & & 10 \end{pmatrix} \times 10^{-4}\ \text{Å}^2$$

$$\mathbf{L} = \begin{pmatrix} 37 & -5 & 5 \\ & 93 & -24 \\ & & 142 \end{pmatrix} \times 10^{-1}\ (^\circ)^2$$

$$\sigma(\mathbf{L}) = \begin{pmatrix} 4 & 5 & 5 \\ & 6 & 5 \\ & & 8 \end{pmatrix} \times 10^{-1}\ (^\circ)^2$$

Unique origin (Å)*	−0·336	4·885	15·941
Centre of gravity (Å)	−0·308	4·976	15·866

Principal axes of **I**† Eigenvalue	Direction cosines (× 10^4)		
1219 Å^2	831	−3283	9409
3268	−418	−9445	−3259
4318	9957	−122	−922

Principal axes of **T**‡ Eigenvalue	Direction cosines (× 10^4)		
0·0271 Å^2	−659	1707	−9831
0·0221	−517	9833	1742
0·0155	9965	616	−564

Principal axes of **L** Eigenvalue	Direction cosines (× 10^4)		
15·20 (°)^2	594	−3767	9243
8·34	514	−9236	−3798
3·70	9969	694	−359

Libration tensor **L** related to the principal axis of **I**

$$\mathbf{L} = \begin{pmatrix} 46 & 1 & 0 \\ & 25 & 1 \\ & & 11 \end{pmatrix} \times 10^{-4}\ \text{rad}^2$$

r.m.s. difference between observed and calculated U_{ij}: 43×10^{-4} Å^2.

* This origin symmetrizes **S**.

† Calculated for the unique origin, using atomic weights instead of mass weights.

‡ Calculated after **S** had been symmetrized.

Table 9. *Differences between observed and calculated* U_{ij} ($\times 10^4$ Å^2)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	−117	+4	+7	+10	+6	−2
C(2)	−128	+7	−23	−2	+20	+1
C(3)	−23	−7	−26	−15	−8	+4
C(4)	−8	+24	−9	+1	+1	−4
C(5)	+37	+0	+0	+19	+9	−12
C(6)	−107	+25	−11	−9	−46	+13
C(7)	+108	+6	+0	+2	−13	+12
C(8)	+1	+16	+13	+8	−28	+3
C(9)	+11	−3	+8	+15	−17	+15
C(10)	−50	−4	+6	−21	−26	−2
C(11)	−24	+30	+12	+3	+8	−17
C(12)	−80	+28	−10	−27	−19	+32
C(13)	−36	−10	−4	+58	+55	+7
C(14)	+3	−17	−12	−0	+55	−9
C(15)	−105	−22	+30	+2	+31	−3
C(16)	+85	−34	+9	−32	+26	−4
C(17)	+22	+105	+70	+32	+15	−66
O(1)	+207	−3	+5	+4	−0	+1
O(2)	−68	−20	−0	+2	−29	+33
O(3)	+52	−44	−28	+21	+3	+2
O(4)	+8	−12	−32	−51	−1	−0
O(5)	+237	−30	−31	+5	−18	−1
O(6)	−24	−37	+26	−29	−15	−4
r.m.s. ΔU_{ij}	91	30	20	22	25	18

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