49. *Thermochromism and Related Effects in Bixanthenylidenes and Bianthronylidenes. Part I. Crystal Structure Analyses.*

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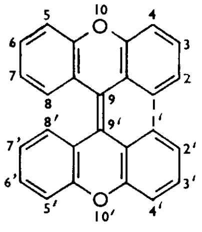
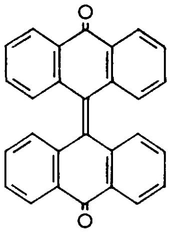
Two polymorphic forms of 9,9′-bixanthenylene, β (yellow) and α (blue-green), have been subjected to *X*-ray analysis and shown to contain molecules in the same " doubly bent " conformation.

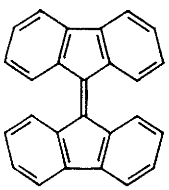
9,9′-Bixanthenylidene (I) belongs to a class of organic compounds sometimes called " thermochromic ethylenes " because, in the crystalline state and in solution, it changes colour with rise in temperature. We defer the detailed discussion of this and related phenomena to the following paper where the reasons for undertaking the crystal structure analyses of these two polymorphs will be made clear. Briefly, a widely held view is that thermochromic ethylenes can exist in at least two, and in some cases three, different molecular conformations. One of these conformations is regarded as the normal state of the molecule and in this it has either no colour absorption or, at most, only a pale colour. The other two conformations are associated with thermal excitation and irradiation of the material, respectively, and then the molecules exhibit strong absorption in the visible spectrum. To the normal form is ascribed the " doubly bent " conformation established by *X*-ray structure analysis for the closely related molecules bianthronyl[1] (II) and 9,9′-bifluorenylidene[2] (IIIa), whereas the other conformations are said to have each half of the molecule planar (instead of bent) but to be twisted to a greater or lesser extent about the central double bond. It appeared that an *X*-ray crystal structure analysis of the pale yellow and of the deep blue-green crystals of 9,9′-bixanthenylidene would throw some light on the general problem of thermochromism.



(I)



(II)



(IIIa)

When we commenced these *X*-ray analyses we held the view that because of steric interaction it would be impossible for hydrogen atoms in the 1,1′- or 8,8′-positions to pass each other in order for the twisted conformation to be attained. Scale drawings indicate the extent of the conflict (following paper). However, we have recently been informed of the careful experimental work of Theilacker and his collaborators[3] on the optical activity of certain derivatives of bixanthenylidene and bianthronylidene. The results are discussed in more detail in Part II, but the most important observations concern the two optically active enantiomorphs of 9,9′-bithioxanthenylidene-4-carboxylic acid, the sulphur analogue of the corresponding bixanthenylidenecarboxylic acid. That these optically active isomers can be prepared is not itself surprising since neither the doubly bent nor the twisted conformation would be superimposable on its mirror image. What is surprising is that these acids, which have large specific rotations of the order of 280°, racemise

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[1] Harnik and Schmidt, *J.*, 1954, 3295.
[2] Nyburg, *Acta Cryst.*, 1954, **7**, 779.
[3] Theilacker, personal communication.

spontaneously in dioxan. This can only be explained if the outer benzene rings " flip over " to occupy the opposite side of the mean molecular plane.

Had we known of these experimental details we should have had less confidence in assuming that the molecules in the two crystalline forms of bixanthenylidene both had doubly bent conformations. In the event, however, this assumption proved correct.

EXPERIMENTAL

9,9′-Bixanthenylidene was generously provided as a gift in powder form by Imperial Chemical Industries Limited, Dyestuffs Division. Harnik *et al.*[4] reported on the unit-cell dimensions and possible space groups for two crystalline forms of bixanthenylidene, but did not give details as to how the crystals were obtained. We have prepared two forms and the unit-cell dimensions and systematically absent *X*-ray reflections agree with those given by Harnik *et al.*

Good crystals of the β-form (yellow) were obtained by slowly cooling *m*-xylene solutions. Unlike Harnik *et al.*, we never obtained blue crystals concurrently with yellow crystals from solution.

The α-crystals (deep blue-green) were obtained by subliming the β-form in a 250—130° gradient at about 10^{-3} mm. No extensive study of the best experimental conditions was carried out, and those blue-green crystals that we did obtain were always accompanied by a certain amount of the yellow β-form. The outcome of sublimation was not highly reproducible, repeated trials under ostensibly the same conditions not always giving the required blue-green form.* It was, however, possible from successful sublimations to pick out clean single crystals suitable for *X*-ray analysis which showed no diffraction effects due to β-contamination. Measurement of the unit-cell dimensions and observation of the systematic absences indicated that these blue-green crystals were of the polymorph examined by Harnik *et al.* Our crystals, however, unlike theirs, appeared to be stable indefinitely.

(*Yellow*) β-*Bixanthenylidene*.—(*a*) On the basis of crystallographic axes allocated as below, the crystals grown from *m*-xylene solution were acicular along the *z*-axis, the only predominant forms being {110} and {001}. The measured unit-cell dimensions were in accord with those found by Harnik *et al.*, and since these workers cite a greater accuracy than can be claimed for our measurements we cite theirs.

The usual multiple-film technique was used for recording *X*-ray reflections on a (Leeds) Weissenberg goniometer. Unfiltered Cu-K_α radiation was generated from a demountable *X*-ray tube of the type described by Rymer and Hambling.[5] In measuring intensity values corrections for spot extension were made by Phillips's graphical method.[6]

The crystal data for this form are as follows. Formula $C_{26}H_{16}O_2$, $M = 360{\cdot}4$. Crystal system monoclinic, point group $2/m$; cell dimensions (*y*-axis unique), $a = 14{\cdot}87 \pm 0{\cdot}15$, $b = 12{\cdot}90 \pm 0{\cdot}13$, $c = 9{\cdot}41 \pm 0{\cdot}09$ Å, $\beta = 95°15' \pm 30'$; unit-cell volume 1797 (±54) Å^3, density measured by flotation in ether–carbon tetrachloride, $1{\cdot}32_6$ at 20°C. For $Z = 4$, calculated density is $1{\cdot}30_9$. $F(000) = 336$, systematic absences were *hkl* absent for $h + k$ odd, *h*0*l* absent for *h* odd (and *l* odd), 0*k*0 absent for *k* odd. Whence the space group is either *C*2/*c*, *C*2/*n*, *Cc*, or *Cn*.

Crystals for *X*-ray analysis were cut as nearly cubic in shape as possible and were about 0·5 mm. in maximum dimension. For this size, errors in intensity values due to absorption are estimated to be less than 3% for any one reflection. The crystals were dipped in liquid air before the *X*-ray examination.

To distinguish between the centric and the acentric space groups, the zero-moment test[7] was applied to the *hk*0 intensity values. The distribution found was hypercentric, indicating, as found by Harnik *et al.*,[8] either *C*2/*c* or *C*2/*n*. The centres of symmetry additional to those

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* Harnik *et al.*[4] remark that they obtained the blue form " only once."

[4] Harnik, Herbstein, Schmidt, and Hirshfield, *J.*, 1954, 3288.

[5] Rymer and Hambling, *J. Sci. Instr.*, 1952, **29**, 192.

[6] Phillips, *Acta Cryst.*, 1954, **7**, 746; 1956, **9**, 819.

[7] Howells, Phillips, and Rogers, *Acta Cryst.*, 1950, **3**, 210.

[8] Harnik, Herbstein, and Schmidt, *Nature*, 1951, **168**, 158.

required by either of these space groups were later traced to the fact that the molecules lie almost normal to the *z*-axis; in this projection each half of the molecule has virtually an additional centre of symmetry at the centre of the inner heterocyclic ring.

The Leeds Weissenberg goniometer used limited the range of accessible Bragg angle to $\sin^{-1} \frac{1}{2}\sqrt{3}$ Of the approximately 1400 reflections lying within this angle, some 951 were estimated for intensity. A Wilson plot of all the intensity data gave a mean temperature factor of 5·8 Å^2.

(*b*) *Structure analysis.* Since we believed that the molecules in both crystal modifications of bixanthenylidene had the doubly bent conformation or a closely related conformation and that elementary stereochemical considerations would enable us to assign this conformation with some certainty, the most suitable method of structure analysis seemed to be by way of the Fourier (molecular) transform obtained optically.[9] Because the molecules are not planar we were not applying the method at its simplest, but in fact we did not encounter any undue difficulties.

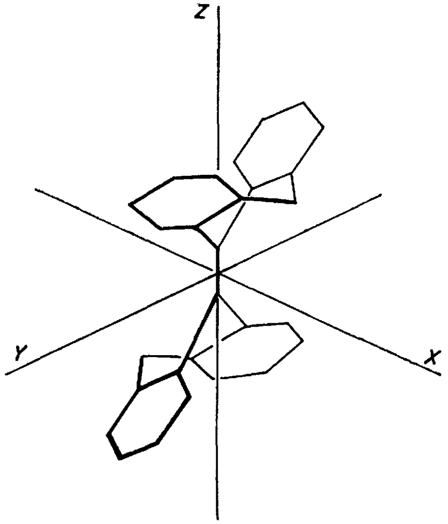

FIG. 1. Molecular axes assigned to 9,9′-bixanthenylidene.

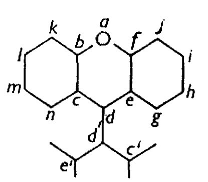

FIG. 2. Lettering scheme.

The initial conformation chosen for the molecule in both crystal modifications was assigned on the basis of considerations arising out of the *X*-ray crystal structure analysis[1] of bianthronylidene (II). We could find no previous studies which would guide us as to the extent to which the replacement of the C=O group in bianthronylidene by an oxygen atom in bixanthenylidene would change the stereochemistry. Our guess was that the heterocyclic ring would have a boat conformation, just as the corresponding ring in bianthronylidene has and, again as in bianthronylidene, that the 1,1′- and 8,8′-carbon atoms would be in as close conflic as their non-bonded atomic radii would permit. In the event, this guess proved quite accurate.

The considerations leading to a trial structure were straight forward. In *C*2/*c* or *C*2/*n* with $Z = 4$, the *y*-projected cell can be divided into four sub-cells each containing one projected molecule in the same orientation. The problem with a molecule of assumed stereochemistry thus resolves into the assigning of three parameters, the two angles needed to specify the orientation of the central double bond with respect to the crystallographic co-ordinate frame plus the angle of rotation of the molecule around this central double bond. Molecular axes *X*, *Y*, and *Z* were assigned as in Fig. 1, which is self-explanatory. Elementary packing considerations suggested that the crystallographic *y*-axis lay near the *YZ* molecular plane. Accordingly

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[9] Lipson and Taylor, " Fourier Transforms and *X*-Ray Diffraction." Macmillan, London, 1958.

masks were punched which gave, to scale, the atomic positions of the molecule as they would be observed in projection along various lines lying in the YZ molecular plane. Four such masks were made representing projections viewed 0°, 30°, 60°, and 90° from the Z-molecular axis and the agreement of the optical transform with the weighted reciprocal lattice for the 30° mask was found on rotation in its own plane to be sufficiently encouraging for us to attempt its refinement. By slight angular variations from this projection a very satisfactory fit was ultimately attained. The agreement was further improved by use of the variable structure factor graph.[10]

At this stage it was possible to distinguish between $C2/c$ and $C2/n$ which, of course, give identical y-projections. Calculation of a few $F(hkl)$ values based on atomic co-ordinates now established by the optical transform showed the true space group to be unambiguously $C2/c$.

From this point calculations of structure factors and of electron density were carried out on a Deuce computer with programmes devised by Dr. J. S. Rollett [11] and atomic scattering factors for carbon and oxygen given by Berghuis *et al.*[12] Computing times were about 75 min. for 1400 $F(hkl)$ values and 90 min. for 7750 electron density points based on these $F(hkl)$ values.

Initially 300 $F(hkl)$ values in the range $0 < \sin\theta < 0{\cdot}5$ were computed. There were no calculated $F(hkl)$ values giving outstandingly bad agreement with observed values, and the residual R was 0·44. Five cycles based on the full 1400 $F(hkl)$ values brought R down to $0{\cdot}20_6$, this calculated with the omission of a few low-order reflections clearly subject to extinction and with non-observed reflections taken as half the minimum observable. At the last cycle, there were 24 sign changes, almost all for non-observed reflections. The observed and calculated structure factors are listed in Table 1.

In order to avoid confusion with the various chemical atomic numbering schemes, the atoms have been assigned letters as in Fig. 2. Atoms related to these by the centre of symmetry at the midpoint of dd' are given corresponding primed letters.

The fractional atomic co-ordinates found for the β-bixanthenylidene molecule centred on the origin were as set out in Table 2. In addition to fractional atomic co-ordinates, co-ordinates in Å are also given for orthogonal axes. These are chosen such that X lies along x, Y along y (thus Z subtends $\beta - \pi/2$ with z).*

Stereochemical implications of these atomic co-ordinates are discussed below.

Blue-green α-9,9′-*Bixanthenylidene*.—(*a*) The blue-green crystals picked out from the sublimate were not usually sufficiently well-formed for their normal habit to be obvious; some were tabular. Within an hour of preparation, the colour of the crystals faded slightly from the initial deep blue-green to a somewhat paler shade but did not change subsequently. The X-ray photographs were free from any sign of powder either as β-contaminant or from decomposition: they were also free from signs of disordering. The largest crystal examined by X-rays was 0·5 × 0·5 × 0·25 mm. Intensity data were collected as for β-bixanthenylidene. Cell dimensions agreed with those determined by Harnik *et al.* and these are given below.

The crystal data for this form are as follows. Crystal system monoclinic, crystal point group probably $2/m$; cell dimensions (y-axis unique), $a = 15{\cdot}19 \pm 0{\cdot}15$, $b = 6{\cdot}26 \pm 0{\cdot}06$, $c = 18{\cdot}93 \pm 0{\cdot}19$ Å, $\beta = 93° 9' \pm 30'$. Unit cell volume 1798 (±54) Å^3 (note close correspondence with that of β-bixanthenylidene); density measured by flotation in ether–carbon tetrachloride, $1{\cdot}34_2$; for $Z = 4$, calculated density $1{\cdot}30_9$. $F(000) = 336$. Systematic absences were hkl absent for $k + l$ odd, $h0l$ for h odd, $0k0$ for k odd. Space group $A2/a$, $A2/n$, Aa, or An.

Crystals were dipped in liquid air before X-ray examination. The zero-moment test gave a centric distribution indicating $A2/a$ or $A2/n$. Of the 1400 accessible $F(hkl)$ values, 1095 were measured. These gave an average temperature factor of 4·0 Å^2.

(*b*) *Structure analysis*. The same molecular conformation was used as a trial structure as that used for β-bixanthenylidene. Also, just as for β-bixanthenylidene, the unit cell in y-projection can be divided into four subcells in each of which the molecules have the same orientation. In this case, however, the b repeat is short so that the molecules cannot lie with their mean planes close to the crystallographic xy-plane. Thus the molecular Z-axis lies close

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* These axes are not to be confused with molecular axes of Fig. 1.

[10] Morley and Taylor, *J. Sci. Inst.*, 1957, **34**, 54.

[11] Rollett, " Computing Methods and the Phase Problem in X-ray Analysis," ed. R. Pepinsky *et al.*, Pergamon, Oxford, 1961.

[12] Berghuis, Haanappel, Potters, Loopstra, MacGillavry, and Veenendaal, *Acta Cryst.*, 1955, **8**, 478.

to the crystallographic y-axis. After examination of some fifteen masks it was possible to get a reasonably good fit of the optical transform with the weighted lattice.

When the approximate atomic co-ordinates had been established and refined with the variable structure factor graph, a few general $F(hkl)$ values were calculated. These showed that the space group was $A2/n$ not $A2/a$. With the origin at $\bar{1}$ on the n glide there are eight molecular halves located at $(0,0,0;\ 0,\frac{1}{2},\frac{1}{2})$ added in turn to x,y,z; $\bar{x},\bar{y},\bar{z}$; $\frac{1}{2}-x,\ \frac{1}{2}+y,\ \bar{z}$; $\frac{1}{2}+x,\ \frac{1}{2}-y,\ z$.

After five cycles for all the 1400 data, R fell to $0{\cdot}20_8$. The observed and the calculated structure factors are given in Table 3, and from the final electron density map the atomic co-ordinates set out in Table 4 were obtained. The orthogonal axes chosen were as for β-bixanthenylidene, namely, X along x and Y along y.

Accuracy of the Analyses.—Our aim in these X-ray analyses was to establish the conformation of the molecule in the two polymorphic crystalline forms. Although we feel that were

TABLE 1.

Observed and calculated structure factors for β-9,9′-bixanthenylidene.

(Values five times absolute.)

h	k	l	F_o	F_c
0	0	2	721	704
		4	342	308
		6	159	−176
		8	98	−92
		10	73	−72
0	2	0	10	−13
		1	171	−227
		2	733	−905
		3	330	−324
		4	7	−1
		5	27	31
		6	11	−3
		7	<32	−23
		8	122	118
		9	<32	−21
		10	44	35
0	4	0	35	46
		1	77	71
		2	61	−69
		3	134	−121
		4	147	140
		5	112	100
		6	50	−54
		7	78	46
		8	42	38
		9	<37	−14
		10	<32	−6
0	6	0	171	156
		1	66	92
		2	208	194
		3	159	−170
		4	49	51
		5	134	−134
		6	<31	−39
		7	42	−41
		8	<32	−16
		9	90	−56
0	8	0	73	−76
		1	17	3
		2	<17	−4
		3	16	−5
		4	122	−94
		5	<17	−1
		6	16	20
		7	46	54
		8	<34	11
0	10	0	<95	−83
		1	99	−97
		2	<17	13
		3	18	3
		4	59	−63
		5	<17	−14
		6	32	17
		7	<32	−3
0	12	0	121	−109
		1	94	−89
		2	<20	8
		3	<20	2
		4	<17	−8
		5	<15	−11
		6	<32	7
0	14	0	26	−22
		1	24	−22
		2	32	25
1	1	10−	<25	3
		9−	34	29
		8−	<26	22
		7−	75	97
		6−	34	−26
		5−	281	337
		4−	10	−30
		3−	684	782
		2−	391	549
		1−	183	196
		0	391	598
		1	354	−553
		2	220	−273
		3	525	−673
		4	20	−17
		5	403	−439
		6	37	29
		7	196	−189
		8	<27	−12
		9	53	−43
		10	<24	13
1	3	10−	<12	6
		9−	<27	−3
		8−	<27	−10
		7−	37	−46
		6−	39	−34
		5−	9	−6
		4−	44	−62
		3−	269	271
		2−	106	−114
		1−	196	−208
		0	106	131
		1	12	−22
		2	122	93
		3	116	151
		4	49	13
		5	171	167
		6	61	67
		7	32	29
		8	<27	16
		9	44	−26
		10	<24	7
1	5	9−	<13	10
		8−	<28	−1
		7−	<24	−24
		6−	57	64
		5−	45	−66
		4−	171	164
		3−	84	−86
		2−	159	99
		1−	147	−188
		0	134	151
		1	49	35
		2	208	188
		3	106	−95
		4	83	−85
		5	134	111
		6	17	−35
		7	45	30
		8	38	−32
		9	34	−10
1	7	9−	<24	10
		8−	<27	−2
		7−	27	34
1	7	6−	57	−66
		5−	67	72
		4−	159	−153
		3−	61	66
		2−	95	−100
		1−	53	52
		0	121	−136
		1	16	25
		2	<15	−11
		3	232	−124
		4	<15	−1
		5	64	−49
		6	50	41
		7	<27	−51
		8	13	29
		9	<24	−27
1	9	8−	12	17
		7−	13	5
		6−	21	38
		5−	48	45
		4−	51	53
		3−	106	103
		2−	147	152
		1−	39	31
		0	<20	−19
		1	22	−9
		2	61	−66
		3	39	−29
		4	57	−45
		5	18	12
		6	<17	−8
		7	<27	−23
		8	<24	6
1	11	6−	<17	−36
		5−	56	−15
		4−	67	−63
		3−	27	29
		2−	78	−62
		1−	53	−51
		0	68	−55
		1	97	−82
		2	23	25
		3	<20	10
		4	<17	7
		5	32	24
		6	<17	−3
1	13	4−	29	21
		3−	<20	3
		2−	57	40
		1−	<20	7
		0	48	−36
		1	<20	−6
		2	18	16
		3	29	27
		4	<15	−2
2	0	10−	21	26
		8−	66	−68
		6−	<7	25
		4−	13	14
		2−	758	1365
		0	106	215
		2	672	779
2	0	4	159	134
		6	45	18
		8	84	−100
		10	104	−90
2	2	10−	<10	29
		9−	<33	1
		8−	81	−71
		7−	<32	−27
		6−	31	29
		5−	183	181
		4−	196	−192
		3−	116	−138
		2−	391	470
		1−	342	−428
		0	489	−504
		1	116	−197
		2	90	−88
		3	9	5
		4	134	−114
		5	11	9
		6	116	125
		7	35	−2
		8	67	57
		9	44	−16
		10	29	32
2	4	10−	53	35
		9−	<37	−4
		8−	57	39
		7−	<37	−13
		6−	159	164
		5−	171	182
		4−	79	−90
		3−	67	77
		2−	59	65
		1−	147	140
		0	34	−48
		1	<10	19
		2	159	−118
		3	76	53
		4	208	−194
		5	50	20
		6	13	1
		7	55	−63
		8	38	−35
		9	37	−34
2	6	9−	<29	29
		8−	<32	−29
		7−	<32	28
		6−	46	−36
		5−	27	22
		4−	27	−34
		3−	39	−47
		2−	51	48
		1−	66	35
		0	220	233
		1	391	−335
		2	147	152
		3	208	−183
		4	50	49
		5	<15	6
		6	29	32
		7	46	−40
		8	32	−29
		9	<27	18
2	8	8−	<37	−14
		7−	<39	−25
		6−	57	−76
		5−	<17	−16
		4−	76	65
		3−	97	104
		2−	17	−16
		1−	134	152
		0	10	−15
		1	103	106
		2	26	−13
		3	46	36
		4	43	33
		5	29	41
		6	26	22
		7	67	−54
		8	<37	17
2	10	7−	48	54
		6−	<17	−11
		5−	49	54
		4−	49	52
		3−	38	24
		2−	116	−101
		1−	208	−202
		0	106	−102
		1	106	−97
		2	134	−112
		3	76	−73
		4	49	−41
		5	<17	15
		6	<17	−21
		7	<7	34
2	12	5−	33	−23
		4−	<17	−4
		3−	44	47
		2−	75	−57
		1−	34	−36
		0	42	46
		1	68	71
		2	29	18
		3	26	20
		4	16	18
		5	<22	20
2	14	2−	44	−32
		1−	<20	−7
		0	21	20
		1	17	16
		2	16	7
3	1	10−	<27	7
		9−	<29	2
		8−	39	−39
		7−	28	−5
		6−	50	60
		5−	39	20
		4−	147	183
		3−	181	−188
		2−	183	236
		1−	134	146
		0	171	−223
		1	76	102
		2	72	−47
		3	33	−22
		4	106	90

TABLE 1. (*Continued.*)

h	*k*	*l*	F_o	F_c
3	1	10	76	−87
		6	<10	−21
		7	134	−116
		8	<29	28
		9	<29	−39
		10	93	−58
3	3	10−	<24	−12
		9−	<32	−9
		8−	<32	−12
		7−	62	55
		6−	20	−24
		5−	342	319
		4−	44	17
		3−	106	−89
		2−	342	390
		1−	379	334
		0	232	234
		1	54	−63
		2	<10	27
		3	11	−5
		4	159	160
		5	46	−44
		6	26	−18
		7	122	−93
		8	<32	19
		9	<32	−18
3	5	9−	53	30
		8−	48	43
		7−	33	−46
		6−	45	−39
		5−	62	50
		4−	39	−54
		3−	244	−274
		2−	21	38
		1−	116	−130
		0	62	16
		1	367	−357
		2	220	−235
		3	113	100
		4	50	−32
		5	62	55
		6	62	−55
		7	<32	20
		8	<32	0
		9	56	33
3	7	9−	<29	13
		8−	<32	−4
		7−	<32	−46
		6−	48	−48
		5−	53	71
		4−	46	42
		3−	43	26
		2−	134	−123
		1−	76	62
		0	49	58
		1	183	−165
		2	26	28
		3	53	58
		4	107	91
		5	49	−52
		6	82	67
		7	38	−35
		8	<32	6
3	9	8−	<29	−5
		7−	53	33
		6−	16	13
		5−	116	121
		4−	159	153
		3−	71	62
		2−	26	−35
		1−	38	53
		0	32	42
		1	55	−55
		2	49	−49
		3	21	−12
		4	75	−85
		5	73	−70
		6	33	−28
		7	<32	−45
3	11	6−	28	−39
		5−	22	−22
		4−	81	−67
		3−	90	−90
		2−	171	−149
		1−	92	−59
		0	50	−44
		1	<20	15
		2	<20	0
		3	59	56
		4	27	26
		5	31	32
		6	53	45
3	13	4−	<15	−10
		3−	26	−18
3	13	2−	27	24
		1−	28	−16
		0	56	36
		1	<20	−1
		3	<20	5
		3	<19	−1
		4	<15	18
4	0	10−	32	−23
		8−	35	54
		6−	83	85
		4−	134	162
		2−	232	−322
		0	46	−18
		2	72	−12
		4	323	392
		6	40	56
		8	29	−16
		10	59	−44
4	2	10−	36	−36
		9−	<32	4
		8−	33	−26
		7−	67	57
		6−	60	−71
		5−	64	−109
		4−	196	220
		3−	94	−112
		2−	159	184
		1−	147	164
		0	257	295
		1	23	30
		2	122	80
		3	106	97
		4	116	108
		5	13	1
		6	49	−67
		7	76	−55
		8	<23	25
		9	9	31
4	4	10−	<32	16
		9−	<37	16
		8−	55	53
		7−	134	112
		6−	40	−13
		5−	88	82
		4−	220	203
		3−	33	−25
		2−	83	76
		1−	116	−93
		0	244	−275
		1	208	−212
		2	257	−276
		3	106	−107
		4	90	−90
		5	116	−104
		6	48	−50
		7	97	−98
		8	38	−33
		9	<37	10
4	6	9−	46	36
		8−	32	−17
		7−	44	−42
		6−	71	−59
		5−	45	46
		4−	<15	8
		3−	<17	−22
		2−	22	18
		1−	97	−83
		0	37	36
		1	<15	11
		2	66	85
		3	40	63
		4	82	64
		5	<15	6
		6	43	38
		7	<32	−3
		8	<32	4
		9	<27	2
4	8	8−	<32	−27
		7−	<37	16
		6−	46	30
		5−	68	68
		4−	<17	−19
		3−	<20	−28
		2−	<17	−8
		1−	50	−42
		0	86	87
		1	<17	6
		2	49	38
		3	56	55
		4	22	14
		5	<17	−20
		6	<17	8
		7	<34	−26
4	8	8	<32	−34
4	10	7−	18	40
		6−	17	32
		5−	44	48
		4−	76	−65
		3−	38	−25
		2−	122	−109
		1−	39	−29
		0	97	−85
		1	97	−104
		2	94	−76
		3	21	−55
		4	44	−35
		5	71	−64
		6	34	20
		7	<32	−14
4	12	5−	23	−26
		4−	<17	−2
		3−	42	−43
		2−	49	38
		1−	35	38
		0	44	29
		1	68	49
		2	10	20
		3	81	73
		4	<51	−50
		5	<15	13
4	14	1−	17	−5
		0	<20	−12
		1	<17	−8
5	1	10−	49	−29
		9−	37	31
		8−	32	38
		7−	34	32
		6−	15	11
		5−	99	110
		4−	86	67
		3−	39	44
		2−	86	60
		1−	112	−83
		0	48	−69
		1	50	95
		2	257	257
		3	90	105
		4	<13	7
		5	34	−21
		6	22	22
		7	18	19
		8	89	−68
		9	<20	−9
5	3	10−	<20	3
		9−	<22	15
		8−	11	−3
		7−	76	54
		6−	59	70
		5−	106	−111
		4−	232	243
		3−	244	281
		2−	116	137
		1−	<10	14
		0	43	−88
		1	84	93
		2	67	90
		3	34	−74
		4	106	−121
		5	171	−178
		6	15	8
		7	32	−35
		8	97	−64
		9	<20	8
5	5	9−	29	−22
		8−	65	−53
		7−	134	114
		6−	<17	−19
		5−	83	−82
		4−	26	−45
		3−	31	−40
		2−	122	−119
		1−	306	−309
		0	196	−184
		1	24	−47
		2	43	75
		3	37	−43
		4	48	32
		5	<15	−1
		6	86	94
		7	<22	20
		8	<22	10
		9	<20	22
5	7	8−	26	−22
		7−	97	93
		6−	70	74
		5−	<17	−11
5	7	4−	<15	4
		3−	<17	−9
		2−	134	119
		1−	97	−100
		0	<20	−16
		1	97	88
		2	<17	−13
		3	<17	−10
		4	<15	−12
		5	<17	−4
		6	21	−23
		7	51	−45
		8	<20	0
5	9	7−	54	37
		6−	22	18
		5−	<15	−4
		4−	116	−105
		3−	26	29
		2−	49	53
		1−	43	−42
		0	26	39
		1	<20	23
		2	18	20
		3	68	−65
		4	16	24
		5	62	−48
		6	67	−55
		7	57	−37
5	11	6−	<18	1
		5−	<17	−22
		4−	75	−69
		3−	<20	9
		2−	68	−55
		1−	<20	0
		0	72	−63
		1	53	56
		2	57	−51
		3	20	14
		4	23	22
		5	16	12
5	13	3−	43	−23
		2−	<17	9
		1−	37	−26
		0	23	22
		1	21	−7
		2	<17	−12
		3	27	−26
6	0	10−	16	−9
		8−	38	37
		6−	31	37
		4−	208	228
		2−	134	185
		0	43	60
		2	208	251
		4	121	−117
		6	15	−11
		8	26	−16
6	2	10−	<27	9
		9−	<32	12
		8−	<32	15
		7−	<32	13
		6−	<15	−5
		5−	116	76
		4−	<12	−19
		3−	147	153
		2−	122	−144
		1−	68	47
		0	25	−48
		1	<15	37
		2	171	196
		3	65	−64
		4	<12	−13
		5	76	−99
		6	16	11
		7	76	48
		8	71	−48
		9	53	−31
6	4	9−	<37	8
		8−	<37	−16
		7−	38	27
		6−	112	115
		5−	64	−57
		4−	106	116
		3−	48	−63
		2−	15	8
		1−	134	−153
		0	<17	−8
		1	147	−144
		2	59	−59
		3	65	−64
		4	20	−40
		5	122	−114
		6	18	−20
6	4	7	76	60
		8	89	−51
		9	<33	−1
6	6	9−	<27	3
		8−	<32	−12
		7−	94	46
		6−	68	44
		5−	90	−94
		4−	<15	0
		3−	58	46
		2−	<17	−16
		1−	28	53
		0	34	18
		1	76	64
		2	32	21
		3	86	96
		4	86	83
		5	76	−84
		6	<17	13
		7	<32	−19
		8	<29	4
6	8	6−	40	−36
		5−	<20	−6
		4−	76	−67
		3−	26	−29
		2−	<20	−16
		1−	34	−37
		0	21	−20
		1	20	31
		2	<17	−9
		3	<20	21
		4	16	19
		5	46	−34
		6	44	−39
6	10	6−	17	−24
		5−	<20	9
		4−	44	−27
		3−	<20	11
		2−	22	22
		1−	<21	1
		0	<22	4
		1	<20	−1
		2	16	17
		3	81	−78
		4	<17	12
		5	55	−52
		6	16	−20
6	12	4−	<15	−2
		3−	<20	−13
		2−	<20	−1
		1−	<20	−1
		0	<22	−14
		1	34	35
		2	44	−41
		3	<20	0
		4	<15	0
7	1	9−	<29	32
		8−	<29	−23
		7−	67	63
		6−	29	29
		5−	121	127
		4−	116	128
		3−	28	−41
		2−	21	−17
		1−	101	−138
		0	281	303
		1	220	−289
		2	43	38
		3	134	−184
		4	−26	−19
		5	<15	16
		6	31	−34
		7	<29	16
		8	<30	1
		9	<17	−4
7	3	9−	38	1
		8−	72	57
		7−	66	−61
		6−	62	51
		5−	39	17
		4−	17	−16
		3−	62	−81
		2−	196	−177
		1−	134	152
		0	45	−16
		1	50	61
		2	59	−73
		3	16	36
		4	15	18
		5	16	23
		6	73	−77
		7	<39	6
		8	<37	−13

TABLE 1. (*Continued.*)

h	*k*	*l*	F_o	F_c
7	5	9−	86	46
		8−	89	51
		7−	57	−33
		6−	67	44
		5−	<15	11
		4−	39	27
		3−	79	−71
		2−	44	−49
		1−	<17	14
		0	<22	15
		1	49	−76
		2	<17	9
		3	53	−56
		4	75	85
		5	71	−87
		6	37	−38
		7	<37	36
		8	<34	−2
7	7	6−	71	−65
		5−	<20	15
		4−	<17	10
		3−	95	−50
		2−	<20	8
		1−	18	−6
		0	62	70
		1	59	−63
		2	28	21
		3	55	−41
		4	<17	6
		5	94	−107
		6	<17	−12
	9	6−	18	−16
		5−	18	−24
		4−	17	22
		3−	29	−47
		2−	20	−21
		1−	67	58
		0	<22	−7
		1	29	17
		2	<20	−13
		3	<10	25
		4	29	−21
		5	<17	−14
		6	13	11
7	11	5−	16	27
		4−	<15	−10
		3−	<20	−3
		2−	<20	−6
		1−	39	38
		0	44	−34
		1	29	−33
		2	<20	−19
		3	21	−24
		4	<15	−5
7	13	1−	<17	−3
		0	44	−27
		1	26	−17
8	0	8−	16	−6
		6−	147	167
		4−	111	130
		2−	196	216
		0	275	270
		2	147	−188
		4	43	−89
		6	83	−101
		8	45	−45
8	2	9−	<29	24
		8−	<32	−9
		7−	62	64
		6−	37	25
		5−	75	76
		4−	122	−124
		3−	82	83
		2−	122	−130
		1−	50	−58
		0	53	39
		1	67	−86
		2	<15	−5
		3	75	−92
		4	57	84
		5	<17	16
		6	<17	29
		7	97	−61
		8	39	30
8	4	9−	32	9
		8−	71	31
		7−	64	−48
		6−	31	14
		5−	29	−43
		4−	<15	−12
		3−	17	15
		2−	21	23
		1−	<17	−11
8	4	9	50	59
		1	73	88
		2	65	83
		3	72	−89
		4	59	−74
		5	46	51
		6	73	90
		7	38	−28
		8	<32	−11
8	6	8−	<29	19
		7−	49	−38
		6−	<20	16
		5−	67	−68
		4−	56	39
		3−	97	−105
		2−	71	61
		1−	134	−148
		0	26	8
		1	<20	5
		2	67	66
		3	134	−154
		4	45	−50
		5	34	39
		6	<17	−11
		7	49	28
8	8	6−	57	−33
		5−	46	46
		4−	67	−59
		3−	106	107
		2−	86	−56
		1−	86	79
		0	76	−80
		1	59	54
		2	<20	6
		3	97	−101
		4	<17	8
		5	26	−22
		6	67	69
8	10	6−	<17	−8
		5−	39	−38
		4−	34	21
		3−	29	−27
		2−	39	38
		0	<22	−6
		1−	26	−26
		1	39	−33
		2	<22	9
		3	<20	−8
		4	20	−23
		5	7	4
8	12	3−	<17	3
		2−	<17	−6
		1−	30	2
		0	29	−29
		1	39	−30
		2	<17	9
9	1	9−	<27	9
		8−	<29	−3
		7−	<29	−7
		6−	34	30
		5−	31	−48
		4−	18	−28
		3−	39	−61
		2−	111	133
		1−	159	−188
		0	60	69
		1	57	−89
		2	16	−11
		3	57	−72
		4	<15	34
		5	29	−23
		6	21	−41
		7	<29	6
		8	<27	−11
9	3	9−	<32	21
		8−	79	35
		7−	45	29
		6−	40	43
		5−	<19	1
		4−	54	−56
		3−	68	65
		2−	<17	−21
		1−	56	−74
		0	<17	15
		1	71	88
		2	16	−23
		3	<17	−14
		4	55	−59
		5	<17	16
		6	<17	−3
		7	57	−35
		8	<32	15
9	5	8−	<34	12
9	5	7−	56	−30
		6−	37	43
		5−	67	−58
		4−	<17	26
		3−	<20	29
		2−	26	27
		1−	18	−21
		0	<20	−3
		1	<20	26
		2	32	−53
		3	79	−102
		4	116	−114
		5	53	59
		6	<17	−14
		7	<34	−11
9	7	6−	64	−64
		5−	<21	0
		4−	62	−66
		3−	20	28
		2−	<10	45
		1−	46	−52
		0	75	74
		1	116	−92
		2	147	156
		3	147	−159
		4	49	63
		5	18	33
		6	40	40
9	9	6−	40	40
		5−	20	−24
		4−	23	19
		3−	33	27
		2−	<20	−9
		1−	28	5
		0	122	−119
		1	34	30
		2	53	−49
		3	<20	−3
		4	22	−25
		5	16	24
9	11	4−	<15	−4
		3−	<17	5
		2−	<21	−1
		1−	29	−24
		0	<22	−6
		1	<20	−3
		2	17	22
		3	<17	5
10	0	8−	60	73
		6−	18	−4
		4−	<15	3
		2−	23	−39
		9	87	−110
		2	28	−33
		4	46	−62
		6	46	−69
10	2	8−	<32	13
		7−	<32	−9
		6−	<17	−11
		5−	37	63
		4−	55	57
		3−	<20	20
		2−	134	124
		1−	87	104
		0	81	92
		1	22	41
		2	17	19
		3	18	−26
		4	<17	3
		5	37	−56
		6	<17	−7
		7	<32	2
10	4	8−	33	18
		7−	<37	−8
		6−	<20	−24
		5−	<20	4
		4−	53	−55
		3−	59	−76
		2−	<20	−24
		1−	<20	7
		0	11	14
		1	81	−92
		2	84	−111
		3	<23	0
		4	16	−14
		5	17	−28
		6	<17	17
		7	<35	0
10	6	7−	32	−23
		6−	<19	−1
		5−	27	−30
		4−	17	39
		3−	87	−89
10	6	2−	67	50
		1−	71	−70
		0	101	86
		1	159	−186
		2	24	−42
		3	42	45
		4	<17	−16
		5	40	58
		6	16	−17
10	8	6−	<17	−8
		5−	67	61
		4−	<17	7
		3−	93	85
		2−	70	−58
		1−	116	97
		0	<22	10
		1	46	−46
		2	20	16
		3	<20	−7
		4	32	33
		5	45	−40
10	10	4−	<15	−3
		3−	51	−43
		2−	59	−46
		1−	39	−29
		0	<22	−15
		1	26	20
		2	<20	5
		3	<17	5
11	1	8−	27	−9
		7−	29	−48
		6−	32	−41
		5−	50	−61
		4−	34	−46
		3−	42	−68
		2−	29	35
		1−	88	82
		0	17	14
		1	23	28
		2	48	80
		3	31	44
		4	<17	−33
		5	<17	32
		6	<17	20
		7	<27	15
11	3	8−	<37	54
		7−	<34	29
		6−	59	70
		5−	55	61
		4−	122	102
		3−	27	−40
		2−	116	111
		1−	50	46
		0	64	−88
		1	122	−163
		2	76	−106
		3	42	−62
		4	43	−67
		5	21	−37
		6	<17	−20
		7	<17	1
11	5	7−	<34	−34
		6−	53	−50
		5−	<20	3
		4−	16	−17
		3−	75	−62
		2−	<20	10
		1−	<20	17
		0	46	−38
		1	55	−51
		2	56	−62
		3	72	98
		4	<17	21
		5	<17	6
		6	16	5
11	7	6−	17	−23
		5−	50	37
		4−	<17	9
		3−	<20	10
		2−	<20	11
		1−	42	−33
		0	97	94
		1	73	−82
		2	34	43
		3	23	32
		4	50	58
		5	21	27
11	9	5−	18	18
		4−	<15	11
		3−	<20	−11
		2−	76	−66
		1−	33	−20
		0	22	14
11	9	1	43	−44
		2	49	−45
		3	<17	−1
12	0	8−	12	−5
		6−	24	0
		4−	73	−75
		2−	16	6
		9	49	−65
		2	29	−28
		4	16	−25
		6	46	45
12	2	7−	<32	−14
		6−	39	34
		5−	<20	4
		4−	76	55
		3−	134	132
		2−	134	125
		1−	97	125
		0	<23	−1
		1	21	32
		2	<20	6
		3	20	29
		4	35	−58
		5	<17	−4
		6	<17	−14
12	4	7−	33	25
		6−	<17	−4
		5−	35	−40
		4−	22	36
		3−	67	−79
		2−	32	29
		1−	159	−156
		0	147	−121
		1	89	−116
		2	<20	14
		3	20	−24
		4	<17	12
		5	<17	17
		6	<15	28
12	6	6−	<17	2
		5−	27	3
		4−	<17	11
		3−	<20	−9
		2−	26	−22
		1−	59	−43
		0	<22	−6
		1	31	35
		2	23	22
		3	<20	10
		4	<16	0
		5	<15	4
12	8	5−	<15	7
		4−	<15	−8
		3−	<20	4
		2−	26	18
		1−	53	−31
		0	29	24
		1	<20	22
		2	<20	23
		3	<17	−5
12	10	1−	31	−21
		0	<20	15
13	1	7−	<28	−1
		6−	40	−51
		5−	<20	3
		4−	33	20
		3−	46	38
		2−	<20	−19
		1−	<20	−21
		0	<22	11
		1	51	30
		2	<20	−15
		3	20	38
		4	22	52
		5	<15	9
13	3	7−	<32	8
		6−	66	64
		5−	<17	−11
		4−	122	111
		3−	29	30
		2−	<20	−7
		1−	134	−104
		0	42	−25
		1	21	−30
		2	58	−92
		3	<20	−18
		4	40	−71
		5	<15	−18
13	5	6−	<17	−13
		5−	31	−29
		4−	16	−24
		3−	<20	−3

TABLE 1. (*Continued.*)

h	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c
13	5	2−	44	−41	14	0	6−	16	−17	14	4	3−	55	−49	15	1	3−	20	−18	15	5	1	<18	1
		1−	68	−43			4−	95	97			2−	34	−27			2−	34	−33			2	<17	9
		0	<22	20			2−	26	−34			1−	90	−87			1−	<20	−3					
		1	21	32			0	<15	1			0	37	29			0	29	−28	16	0	4−	42	−40
		2	44	60			2	22	−21			1	<20	−33			1	<20	−8			2−	<15	6
		3	<20	9			4	28	18			2	<20	−17			2	<20	21			0	<15	−3
		4	21	35	14	2	6−	<17	−5			3	<17	−11			3	<17	−14			2	<12	3
13	7	5−	29	−28			5−	29	19			4	<15	9	15	3	4−	<15	−1	16	2	3−	21	−19
		4−	<15	−12			4−	57	44	14	6	4−	<15	−6			3−	60	−42			2−	12	13
		3−	28	−22			3−	20	13			3−	<17	4			2−	18	22			1−	<20	15
		2−	37	20			2−	57	−57			2−	<20	−10			1−	<20	7			0	26	−16
		1−	<20	−12			1−	<20	15			1−	20	6			0	<20	10			1	<17	11
		0	35	−26			0	<21	1			0	<18	−1			1	<20	19	16	4	2−	<17	11
		1	20	21			1	44	78			1	<20	−5			2	<20	11			1−	<17	13
		2	<17	−1			2	<20	−31			2	<17	−5			3	<17	2			0	<17	−9
		3	<17	−11			3	<20	27			3	<17	−5	15	5	3−	<17	−3					
13	9	2−	55	37			4	7	6								2−	20	14					
		1−	54	−29	14	4	5−	16	−16	15	1	5−	16	11			1−	18	−5					
		0	<20	−14			4−	<17	1			4−	<15	−8			0	<17	−3					

$$\Sigma F_o = 75{,}800. \quad \Sigma F_c = 75{,}989. \quad R = (\Sigma F_o - \Sigma F_c)/\Sigma F_c = 0{\cdot}20_6.$$

Values suffering from extinction such as 110, 111, and 20$\bar{2}$ are not included in the data when calculating the residual, but unobserved reflections are included as half their minimum observable value. The scale of the *F*'s tabulated here is $\frac{1}{2} \times F_{\text{absolute}} \times 10$. The observed values are thought to be accurate to about 10%.

TABLE 2.

Atom	*x*	*y*	*z*	*X*	*Y*	*Z*	Atom	*x*	*y*	*z*	*X*	*Y*	*Z*
a	$0{\cdot}143_7$	$0{\cdot}205_5$	$0{\cdot}041_0$	$2{\cdot}10_1$	$2{\cdot}65_1$	$0{\cdot}38_4$	*h*	$0{\cdot}229_2$	$\overline{0{\cdot}053_8}$	$0{\cdot}269_0$	$3{\cdot}17_2$	$\overline{0{\cdot}69_4}$	$2{\cdot}52_0$
b	$0{\cdot}051_3$	$0{\cdot}227_4$	$0{\cdot}041_8$	$0{\cdot}72_6$	$2{\cdot}93_3$	$0{\cdot}39_1$	*i*	$0{\cdot}289_2$	$0{\cdot}026_8$	$0{\cdot}253_8$	$4{\cdot}07_7$	$0{\cdot}34_6$	$2{\cdot}37_8$
c	$\overline{0{\cdot}012_5}$	$0{\cdot}148_1$	$0{\cdot}044_7$	$\overline{0{\cdot}22_5}$	$1{\cdot}91_0$	$0{\cdot}41_9$	*j*	$0{\cdot}258_0$	$0{\cdot}112_6$	$0{\cdot}172_6$	$3{\cdot}68_4$	$1{\cdot}45_3$	$1{\cdot}61_7$
d	$0{\cdot}023_3$	$0{\cdot}040_9$	$0{\cdot}035_9$	$0{\cdot}31_4$	$0{\cdot}52_8$	$0{\cdot}33_7$	*k*	$0{\cdot}027_6$	$0{\cdot}331_1$	$0{\cdot}041_0$	$0{\cdot}37_4$	$4{\cdot}27_1$	$0{\cdot}38_4$
e	$0{\cdot}113_4$	$0{\cdot}031_5$	$0{\cdot}118_7$	$1{\cdot}58_2$	$0{\cdot}40_6$	$1{\cdot}11_2$	*l*	$\overline{0{\cdot}060_6}$	$0{\cdot}357_8$	$0{\cdot}053_9$	$\overline{0{\cdot}94_8}$	$4{\cdot}61_6$	$0{\cdot}50_5$
f	$0{\cdot}171_3$	$0{\cdot}113_5$	$0{\cdot}109_8$	$2{\cdot}45_1$	$1{\cdot}46_4$	$1{\cdot}02_9$	*m*	$\overline{0{\cdot}125_0}$	$0{\cdot}281_0$	$0{\cdot}069_2$	$\overline{1{\cdot}92_0}$	$3{\cdot}62_5$	$0{\cdot}68_4$
g	$0{\cdot}139_7$	$0{\cdot}052_0$	$0{\cdot}205_3$	$1{\cdot}89_7$	$\overline{0{\cdot}67_1}$	$1{\cdot}92_4$	*n*	$\overline{0{\cdot}099_0}$	$0{\cdot}176_6$	$0{\cdot}064_1$	$\overline{1{\cdot}52_8}$	$2{\cdot}27_8$	$0{\cdot}60_0$

Atomic co-ordinates for β-9,9′-bixanthenylidene, half-molecule centred on origin at $\bar{1}$ on glide plane *c*. First three columns are fractional co-ordinates on crystallographic axes; second three columns are distances in Å along orthogonal axes oriented as given in the Text.

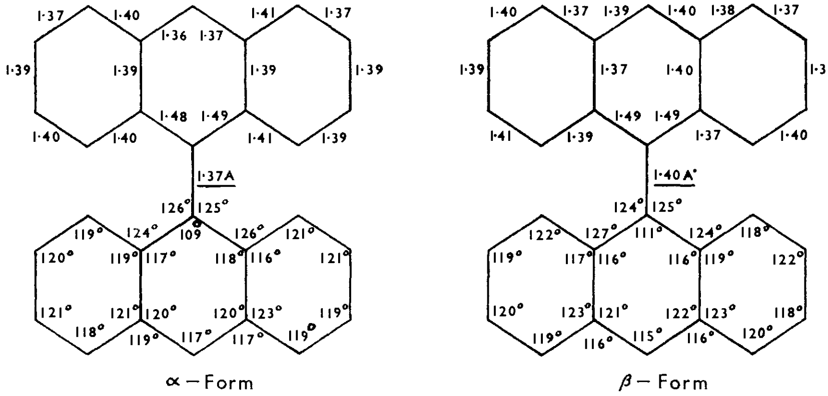

FIG. 3. Bond lengths and interbond angles in the two polymorphs.

the results to be fully refined a comparison of the two molecular structures would be of interest, such refinement was outside the scope of our immediate programme.

Both analyses obey the usual qualitative critera for reliability in that in neither case is there any bad agreement between F_o and F_c, there are no spurious electron-density peaks or large negative voids and there are no intermolecular atomic distances of unusually short length. The bond lengths and interbond angles calculated from the atomic co-ordinates are shown for both molecules in Fig. 3. What differences there are between the molecules probably lie within the limits of experimental error. Certainly the two molecules appear to be virtually identical in conformation. Since in the absence of unusual intermolecular contacts, the effect of intermolecular environment on bond lengths is generally very small it seems reasonable to assume that the observed differences between the molecules in the present analyses are due entirely to errors. If so, a comparison between the two suggests a probable error in bond lengths of about ±0·02 Å, being half the largest difference between the lengths of corresponding bonds, which occurs in bonds *eg*. Similar considerations suggest a probable error in bond angles of about ±1·5°.

TABLE 3.

Observed and calculated structure factors for α-9,9′-bixanthenylidene.

(Values five times absolute.)

h	*k*	*l*	F_o	F_c
0	0	2	587	753
		4	102	−76
		6	221	−203
		8	306	210
		10	289	240
		12	17	−11
		14	<9	15
		16	26	−20
		18	<9	15
		20	<9	7
0	1	1	179	−201
		3	272	262
		5	221	−158
		7	26	7
		9	204	160
		11	60	97
		13	60	−46
		15	68	70
		17	34	14
		19	43	−46
0	2	0	340	360
		2	323	311
		4	102	124
		6	94	−72
		8	221	−214
		10	<12	5
		12	51	30
		14	145	123
		16	68	−64
		18	9	13
		20	77	70
0	3	1	77	68
		3	170	−128
		5	179	−185
		7	196	−190
		9	34	−29
		11	17	28
		13	34	22
		15	<17	20
		17	77	−70
		19	51	−48
0	4	0	23	34
		2	34	−44
		4	34	−23
		6	51	−37
		8	26	9
		10	85	−85
		12	187	−175
		14	<17	15
		16	<17	1
0	5	1	68	103
		3	111	91
		5	162	−158
		7	68	−81
		9	17	−8
		11	<17	26
		13	17	−19
		15	43	−24
0	6	0	17	−29
		2	51	−70
		4	77	105
		6	<17	31
		8	43	54
		10	26	−29
0	7	1	26	−29
1	1	21−	<6	17
		19−	−68	−60
		17−	255	−201
		15−	<22	9
		13−	145	108
		11−	136	117
		9−	476	361
		7−	<8	9
		5−	94	109
		3−	264	−231
		1−	349	297
		1	43	−37
		3	187	−189
		5	34	22
		7	153	118
		9	128	136
		11	136	−133
		13	77	63
		15	111	−76
		17	94	−73
		19	<26	12
1	2	20−	51	33
		18−	111	67
		16−	119	87
		14−	162	125
		12−	17	25
		10−	85	93
		8−	102	−123
		6−	94	124
		4−	44	53
		2−	289	−287
		0	51	95
		2	238	293
		4	94	−63
		6	128	160
		8	102	79
		10	<9	2
		12	34	−4
		14	60	−62
		16	94	81
		18	43	45
		20	<26	20
1	3	19−	<21	8
		17−	<22	10
		15−	77	53
		13−	34	32
		11−	213	221
		9−	111	88
		7−	204	178
		5−	77	102
		3−	26	−31
		1−	68	95
		1	179	−194
		3	272	−326
		5	445	140
		7	51	36
		9	119	75
		11	<20	42
		13	51	−67
		15	43	84
		17	43	48
1	3	19	77	67
1	4	16−	<22	6
		14−	119	73
		12−	196	180
		10−	<14	4
		8−	26	60
		6−	68	−99
		4−	77	56
		2−	34	19
		0	126	102
		2	<1	13
		4	<17	24
		6	<17	24
		8	<9	6
		10	102	−58
		12	238	−209
		14	<21	11
		16	<21	1
1	5	13−	<22	8
		11−	77	−77
		9−	<21	3
		7−	26	6
		5−	170	202
		3−	<17	5
		1−	68	−39
		1	77	−62
		3	85	88
		5	85	74
		7	26	−12
		9	34	32
		11	26	28
		13	<22	33
1	6	10−	<29	11
		8−	−60	37
		6−	77	−77
		4−	111	−107
		2−	77	64
		0	51	18
		2	60	46
		4	43	−51
		6	60	67
		8	60	−15
		10	34	40
1	7	1−	43	−30
		1	34	−24
2	0	20−	43	46
		18−	34	66
		16−	34	−14
		14−	43	78
		12−	115	−111
		10−	68	91
		8−	213	−236
		6−	204	300
		4−	221	−224
		2−	315	382
		0	476	521
		2	323	−306
		4	553	635
		6	94	−88
		8	221	212
		10	136	144
2	0	12	247	269
		14	162	154
		16	<21	3
		18	77	−104
		20	<43	53
2	1	19−	77	68
		17−	60	−65
		15−	26	23
		13−	60	53
		11−	43	−32
		9−	213	−267
		7−	136	−164
		5−	36	−23
		3−	230	276
		1−	485	−585
		1	757	−1084
		3	179	−199
		5	60	48
		7	196	232
		9	128	−164
		11	162	−155
		13	<17	17
		15	85	92
		17	60	−75
		19	<17	5
2	2	20−	17	24
		18−	<16	4
		16−	162	172
		14−	102	94
		12−	85	−97
		10−	<10	3
		8−	111	105
		6−	196	173
		4−	51	39
		2−	85	56
		0	128	139
		2	128	−136
		4	26	−80
		6	43	4
		8	51	−18
		10	17	−12
		12	102	94
		14	111	171
		16	50	−75
		18	<17	31
		20	51	−56
2	3	17−	17	−14
		15−	26	22
		13−	<17	13
		11−	20	−20
		9−	68	−85
		7−	136	138
		5−	179	214
		3−	85	−104
		1−	247	−232
		1	153	−150
		3	264	−302
		5	199	−108
		7	111	115
		9	128	137
		11	221	−196
		13	153	−207
		15	43	−62
		17	<17	4
2	4	16−	34	25
		14−	26	58
		12−	34	−4
		10−	26	9
		8−	85	−114
		6−	43	17
		4−	77	77
		2−	60	−59
		0	60	−78
		2	43	−48
		4	<17	29
		6	<21	7
		8	68	66
		10	<15	17
		12	<14	21
		14	<17	0
		16	<17	28
2	5	13−	34	38
		11−	43	42
		9−	<21	2
		7−	26	−18
		5−	145	150
		3−	26	49
		1−	17	21
		1	<25	24
		3	111	−121
		5	51	101
		7	60	60
		9	<21	14
		11	26	−33
		13	<21	32
2	6	10−	17	11
		8−	43	−55
		6−	<21	21
		4−	77	−88
		2−	43	−31
		0	34	−13
		2	34	−53
		4	77	72
		6	<21	1
		8	110	68
		10	17	17
2	7	1−	0	17
		1	<21	20
3	1	19−	<19	5
		17−	34	22
		15−	247	152
		13−	43	−55
		11−	102	90
		9−	175	132
		7−	43	31
		5−	43	32
		3−	230	−222
		1−	578	647
		1	459	455
		3	510	602
		5	264	272
		7	196	176
		9	94	−101
		11	84	−51
		13	<18	26
		15	153	−124

TABLE 3. (*Continued.*)

h	*k*	*l*	F_o	F_c
	1	17	<19	8
		19	34	43
3	2	20−	19	21
		18−	68	63
		16−	221	160
		14−	204	106
		12−	51	29
		10−	119	−125
		8−	60	−85
		6−	264	319
		4−	68	75
		2−	60	80
		0	85	43
		2	238	−213
		4	196	−210
		6	111	133
		8	51	−72
		10	<11	9
		12	128	−128
		14	<17	47
		16	94	110
		18	51	47
3	3	19−	17	14
		17−	53	−24
		15−	26	−32
		13−	51	17
		11−	111	62
		9−	16	6
		7−	43	63
		5−	145	−163
		3−	51	57
		1−	238	270
		1	85	71
		3	77	69
		5	77	49
		7	162	−142
		9	17	24
		11	138	−211
		13	34	−57
		15	51	−69
		17	26	−32
3	4	16−	<19	8
		14−	179	123
		12−	43	−68
		10−	162	−170
		8−	60	−46
		6−	68	94
		4−	213	227
		2−	17	−43
		0	68	68
		2	51	21
		4	51	81
		6	68	−90
		8	51	−37
		10	204	−168
		12	68	−35
		14	<19	51
		16	<19	8
3	5	13−	102	−77
		11−	43	−39
		9−	68	−83
		7−	77	−75
		5−	34	5
		3−	145	−113
		1−	43	−9
		1	<26	29
		3	111	109
		5	34	−51
		7	43	−15
		9	17	−16
		11	34	26
		13	<19	6
3	6	10−	17	11
		8−	60	15
		6−	34	−26
		4−	77	−66
		2−	<26	28
		0	43	37
		2	34	8
		4	56	47
		6	94	−94
		8	17	−89
		10	51	−40
4	0	20−	<43	20
		18−	<43	40
		16−	<43	42
		14−	<43	11
		12−	34	−33
		10−	94	110
		8−	60	−80
		6−	51	35
		4−	51	−13
		2−	757	1002
4	0	0	638	−935
		2	782	−1148
		4	468	−460
		6	179	162
		8	281	309
		10	196	−166
		12	26	−43
		14	<22	18
		16	<22	22
		18	<22	19
		20	<22	8
4	1	19−	34	−32
		17−	<17	4
		15−	187	174
		13−	51	43
		11−	43	−12
		9−	26	39
		7−	196	200
		5−	17	−38
		3−	111	−50
		1−	459	−451
		1	94	67
		3	340	−332
		5	196	−172
		7	43	49
		9	60	−30
		11	136	−119
		13	34	−42
		15	<22	25
		17	<22	14
		19	34	41
4	2	20−	<22	17
		18−	−77	−88
		16−	43	35
		14−	<24	28
		12−	<11	17
		10−	60	61
		8−	213	223
		6−	298	369
		4−	238	211
		2−	119	126
		0	94	117
		2	187	−209
		4	153	−167
		6	196	−215
		8	85	82
		10	77	60
		12	43	39
		14	102	−135
		16	60	−78
		18	34	59
4	3	17−	<21	29
		15−	<23	2
		13−	43	50
		11−	17	34
		9−	26	48
		7−	51	47
		5−	<17	22
		3−	170	−182
		1−	77	−74
		1	170	−140
		3	111	−113
		5	17	20
		7	111	123
		9	34	35
		11	119	−89
		13	43	−71
		15	51	−78
		17	<23	28
4	4	16−	11	1
		14−	51	−59
		12−	68	71
		10−	153	171
		8−	60	50
		6−	77	75
		4−	42	25
		2−	111	126
		0	68	78
		2	17	−31
		4	<21	5
		6	<21	18
		8	17	16
		10	17	−31
		12	119	78
		14	34	53
		16	<32	21
4	5	13−	<21	4
		11−	17	−7
		9−	34	56
		7−	51	69
		5−	51	−56
		3−	204	−239
		1−	17	8
		1	34	−11
4	5	3	<26	14
		5	43	41
		7	26	7
		9	34	51
		11	43	−38
		13	<22	37
4	6	10−	17	−9
		8−	8	−8
		6−	26	42
		4−	51	−74
		2−	34	−34
		0	60	−66
		2	60	90
		4	22	18
		6	60	−86
		8	17	−2
5	1	19−	34	−28
		17−	162	150
		15−	323	266
		13−	128	114
		11−	145	109
		9−	264	−230
		7−	60	−18
		5−	26	−28
		3−	281	302
		1−	306	285
		1	306	−256
		3	34	−9
		5	179	143
		7	221	196
		9	77	−73
		11	43	39
		13	<19	13
		15	<22	28
		17	68	87
		19	34	17
5	2	18−	26	−16
		16−	34	−28
		14−	26	−23
		12−	26	28
		10−	153	−152
		8−	<10	3
		6−	60	−57
		4−	60	−61
		2−	119	151
		0	18	−1
		2	374	−147
		4	238	−299
		6	128	−156
		8	26	−19
		10	68	−30
		12	162	−169
		14	26	5
		16	34	40
		18	17	−13
5	3	17−	<20	2
		15−	<20	12
		13−	68	68
		11−	17	−34
		9−	17	−17
		7−	68	−85
		5−	43	48
		3−	102	88
		1−	26	−3
		1	51	−70
		3	204	211
		5	26	6
		7	26	−8
		9	94	−83
		11	136	−95
		13	60	105
		15	43	−44
		17	<19	16
5	4	16−	<19	5
		14−	34	24
		12−	153	−143
		10−	102	−70
		8−	111	−143
		6−	43	31
		4−	<17	−14
		2−	26	−3
		0	17	4
		2	85	−144
		4	77	−97
		6	<21	7
		8	17	60
		10	60	−57
		12	51	53
		14	<19	4
		16	<17	16
5	5	13−	77	−66
		11−	26	47
		9−	<21	2
5	5	7−	43	48
		5−	136	−161
		3−	179	−188
		1−	94	79
		1	26	19
		3	<26	13
		5	94	−116
		7	51	−24
		9	26	−7
		11	34	51
		13	<19	11
5	6	8−	17	2
		6−	17	13
		4−	42	34
		2−	51	37
		0	43	47
		2	60	−53
		4	68	−53
		6	<21	1
		8	17	24
6	0	20−	<43	0
		18−	68	79
		16−	153	156
		14−	119	97
		12−	77	54
		10−	9	−28
		8−	102	157
		6−	94	89
		4−	144	113
		2−	94	46
		0	493	−556
		2	136	173
		4	136	−199
		6	179	174
		8	9	2
		10	60	−47
		12	51	−47
		14	170	−171
		16	128	−141
		18	<26	26
6	1	19−	51	−51
		17−	<25	21
		15−	68	52
		13−	77	60
		11−	26	28
		9−	77	53
		7−	51	153
		5−	232	256
		3−	60	−75
		1−	102	−114
		1	430	443
		3	145	120
		5	102	140
		7	221	−225
		9	111	−108
		11	9	10
		13	34	51
		15	77	85
		17	<26	13
		19	<26	18
6	2	18−	34	42
		16−	<25	1
		14−	128	−124
		12−	<12	13
		10−	102	72
		8−	85	81
		6−	34	24
		4−	17	−82
		2−	170	190
		0	42	5
		2	17	23
		4	196	233
		6	128	140
		8	68	75
		10	51	−65
		12	<13	14
		14	128	−153
		16	51	−69
		18	60	−55
6	3	17−	<25	14
		15−	77	70
		13−	26	5
		11−	34	25
		9−	51	48
		7−	17	9
		5−	94	−121
		3−	111	−118
		1−	94	120
		1	26	2
		3	<21	4
		5	94	93
		7	111	103
		9	153	−136
6	3	11	153	135
		13	43	37
		15	60	90
		17	<23	28
6	4	16−	<25	1
		14−	77	−56
		12−	<13	12
		10−	34	−58
		8−	68	54
		6−	51	90
		4−	94	−113
		2−	60	−58
		0	<19	18
		2	111	157
		4	94	98
		6	<21	12
		8	34	−35
		10	26	15
		12	17	13
		14	<26	19
6	5	13−	34	25
		11−	51	−56
		9−	60	−81
		7−	17	−2
		5−	60	−40
		3−	85	−74
		1−	60	−57
		1	102	−103
		3	34	72
		5	17	23
		7	43	45
		9	77	−86
		11	94	−84
6	6	8−	<17	14
		6−	26	−36
		4−	34	−11
		2−	26	9
		0	26	27
		2	60	67
		4	34	−37
		6	<17	5
		8	43	−18
7	1	19−	<43	7
		17−	102	61
		15−	<43	51
		13−	<43	1
		11−	102	58
		9−	17	−9
		7−	93	75
		5−	119	74
		3−	502	447
		1−	204	179
		1	145	−151
		3	306	−338
		5	332	−336
		7	230	−204
		9	51	−50
		11	43	53
		13	102	−79
		15	43	−49
		17	<43	14
7	2	18−	<43	7
		16−	60	−70
		14−	119	−87
		12−	136	−117
		10−	34	−5
		8−	221	206
		6−	34	−43
		4−	111	−109
		2−	179	−185
		0	162	−163
		2	102	−96
		4	51	45
		6	111	−123
		8	102	−64
		10	196	−162
		12	<13	19
		14	43	41
		16	85	−87
		18	<43	66
7	3	17−	<43	16
		15−	19	26
		13−	<43	44
		11−	43	−33
		9−	<21	46
		7−	<17	9
		5−	111	118
		3−	119	116
		1−	43	36
		1	51	33
		3	68	64
		5	94	−96
		7	43	66

TABLE 3. (*Continued.*)

h	*k*	*l*	*F*o	*F*c
7	3	9	111	−108
		11	111	80
		13	43	49
		15	<43	5
7	4	14−	68	−58
		12−	111	−105
		10−	<15	18
		8−	60	78
		6−	60	102
		4−	238	−260
		2−	187	−173
		0	136	−126
		2	98	−62
		4	94	−110
		6	<21	17
		8	60	52
		10	26	42
		12	34	72
		14	<43	7
7	5	11−	60	47
		9−	34	38
		7−	85	115
		5−	<21	19
		3−	34	33
		1−	26	28
		1	60	55
		3	34	−25
		5	51	−31
		7	26	−28
		9	<17	19
		11	<11	13
7	6	6−	26	29
		4−	26	31
		2−	26	16
		0	<33	25
		2	85	−82
		4	<21	10
		6	<17	24
8	0	18−	<10	34
		16−	111	131
		14−	77	102
		12−	289	296
		10−	136	140
		8−	85	78
		6−	51	−86
		4−	221	310
		2−	119	−141
		0	119	−145
		2	68	110
		4	162	193
		6	170	196
		8	213	−204
		10	<43	35
		12	17	32
		14	95	−69
		16	<34	17
		18	<34	41
8	1	19−	<31	6
		17−	85	72
		15−	128	−93
		13−	179	−118
		11−	68	−58
		9−	111	104
		7−	20	1
		5−	94	−93
		3−	153	−161
		1−	306	295
		1	68	69
		3	51	38
		5	94	79
		7	26	−29
		9	43	−41
		11	43	−35
		13	<34	11
		15	<34	6
		17	26	11
8	2	18−	51	42
		16−	51	35
		14−	94	66
		12−	85	78
		10−	51	45
		8−	<13	7
		6−	136	−177
		4−	153	−164
		2−	26	39
		0	26	−133
		2	<26	12
		4	51	−21
		6	145	148
		8	128	101
		10	<14	4
		12	43	−69
		14	43	−46
8	3	16	60	87
		17−	<31	7
		15−	<34	16
		13−	68	−40
		11−	60	−42
		9−	51	−72
		7−	<17	0
		5−	102	−112
		3−	−94	−84
		1−	43	−29
		1	17	9
		3	68	54
		5	26	−5
		7	111	−99
		9	153	−125
		11	<12	1
		13	43	48
		15	43	31
8	4	14−	34	17
		12−	34	39
		10−	60	71
		8−	17	−29
		6−	60	−71
		4−	43	−37
		2−	68	−55
		0	119	−160
		2	17	−22
		4	<21	6
		6	<21	6
		8	26	−42
		10	<15	13
		12	<13	4
8	5	11−	17	−7
		9−	<21	10
		7−	77	−77
		5−	119	−144
		3−	<26	34
		1−	43	52
		1	<26	2
		3	34	−55
		5	60	−71
		7	<17	18
		9	60	−43
8	6	4−	17	−14
		2−	51	−53
		0	34	43
		2	<21	7
		4	60	−75
9	1	17−	85	55
		15−	85	−86
		13−	68	−64
		11−	136	−135
		9−	68	57
		7−	94	64
		5−	85	78
		3−	119	118
		1−	272	−266
		1	94	−98
		3	34	27
		5	43	47
		7	94	−94
		9	111	−85
		11	68	−41
		13	60	−53
		15	<34	44
		17	60	−74
9	2	16−	<34	1
		14−	<34	0
		12−	95	−95
		10−	43	44
		8−	43	44
		6−	34	34
		4−	26	20
		2−	68	−89
		0	102	−106
		2	43	47
		4	145	166
		6	136	158
		8	26	19
		10	145	−137
		12	26	53
		14	<17	10
		16	<34	22
9	3	15−	26	28
		13−	34	−39
		11−	34	−34
		9−	33	−70
		7−	26	28
		5−	34	48
		3−	<26	18
		1−	60	−75
		1	51	46
		3	<26	3
9	3	5	<21	38
		7	51	42
		9	<21	11
		11	128	135
		13	34	−48
		15	<34	4
9	4	14−	68	−43
		12−	26	42
		10−	43	14
		8−	85	81
		6−	26	−22
		4−	102	−94
		2	26	−19
		0	94	−115
		2	94	−81
		4	<21	0
		6	<21	42
		8	60	−63
		10	26	−31
		12	34	−51
9	5	9−	26	16
		7−	17	−38
		5−	34	−4
		3−	94	105
		1−	102	84
		1	68	62
		3	26	−32
		5	34	43
		7	60	49
		9	34	26
9	6	2−	<21	14
		0	60	−40
10	0	18−	94	115
		16−	60	32
		14−	77	−91
		12−	68	−85
		10−	34	33
		8−	145	159
		6−	60	42
		4−	26	25
		2−	119	−105
		0	196	273
		2	102	98
		4	110	72
		6	170	−167
		8	204	−204
		10	68	−50
		12	77	−77
		14	51	−47
		16	60	62
10	1	17−	<32	8
		15−	<33	31
		13−	85	−76
		11−	136	−127
		9−	60	−53
		7−	−26	−5
		5−	128	−112
		3−	119	−118
		1−	34	28
		1	102	−108
		3	26	40
		5	94	−103
		7	34	17
		9	43	35
		11	<12	20
		13	34	−37
		15	<33	24
10	2	16−	<33	19
		14−	34	25
		12−	68	50
		10−	<8	10
		8−	43	−39
		6−	51	−60
		4−	85	105
		2−	43	−21
		0	102	−101
		2	60	71
		4	26	−50
		6	68	−71
		8	187	−191
		10	17	−17
		12	<14	22
		14	<33	23
10	3	15−	<33	25
		13−	77	−76
		11−	85	−78
		9−	43	−33
		7−	<26	−1
		5−	43	−36
		3−	26	23
		1−	128	−120
		1	162	−156
		3	34	−53
10	3	5	<21	10
		7	136	−109
		9	136	−124
		11	9	14
		13	51	76
10	4	12−	<13	10
		10−	26	18
		8−	17	42
		6−	68	−73
		4−	<21	16
		2−	51	49
		0	85	77
		2	<26	0
		4	102	−125
		6	51	−41
		8	19	0
		10	<15	13
10	5	9−	<17	16
		7−	17	33
		5−	<32	6
		3−	17	25
		1−	34	−38
		1	26	23
		3	34	−40
		5	26	−38
		7	43	−35
11	1	15−	<34	5
		13−	26	46
		11−	<12	34
		9−	94	114
		7−	26	−34
		5−	111	104
		3−	51	−62
		1−	255	−229
		1	94	−99
		3	<26	6
		5	77	101
		7	68	74
		9	43	37
		11	<34	−34
		13	51	−50
		15	34	8
11	2	14−	<43	4
		12−	27	1
		10−	34	41
		8−	68	84
		6−	51	−57
		4−	94	−95
		2−	<26	6
		0	51	33
		2	94	86
		4	42	38
		6	60	68
		8	<13	28
		10	77	69
		12	26	77
		14	<43	24
11	3	13−	43	46
		11−	68	73
		9−	26	18
		7−	17	4
		5−	77	−99
		3−	77	−63
		1−	87	−84
		1	60	−55
		3	128	−142
		5	26	−8
		7	77	−64
		9	85	67
		11	17	29
11	4	10−	33	34
		8−	43	47
		6−	102	−158
		4−	43	−43
		2−	34	35
		0	34	48
		2	<26	15
		4	26	22
		6	<21	20
		8	16	−11
11	5	7−	60	−71
		5−	<17	4
		3−	<26	11
		1−	34	35
		1	<17	7
		3	26	−26
12	0	14−	<43	5
		12−	68	−151
		10−	128	−175
		8−	136	−160
		6−	77	14
		4−	<21	6
12	0	2−	85	−85
		0	68	−73
		2	<43	17
		4	68	68
		6	111	−114
		8	<14	22
		10	<17	3
		12	111	−88
12	1	15−	<26	25
		13−	<26	7
		11−	26	−32
		9−	85	69
		7−	145	−119
		5−	102	−95
		3−	111	123
		1−	51	−55
		1	102	−117
		3	51	−47
		5	26	−24
		7	<17	46
		9	<21	7
		11	34	35
		13	<26	23
12	2	14−	<43	5
		12−	26	−34
		10−	43	−50
		8−	51	−61
		6−	<31	−16
		4−	94	117
		2−	51	−53
		0	26	−19
		2	60	−71
		4	<21	1
		6	<21	6
		8	102	−87
		10	51	−48
		12	26	−45
12	3	11−	<11	20
		9−	26	48
		7−	43	−23
		5−	51	−31
		3−	<23	22
		1−	17	26
		1	<17	1
		3	43	−57
		5	77	−61
		7	<51	−15
		9	<51	55
		11	<17	41
12	4	8−	<13	16
		6−	<21	6
		4−	26	38
		3−	<26	4
		0	85	94
		2	60	52
		4	<21	8
		6	12	24
		8	8	8
12	5	3−	34	41
		1−	<17	2
		1	43	−48
13	1	13−	<17	8
		11−	26	34
		9−	<21	16
		7−	<17	12
		5−	60	66
		3−	111	−118
		1−	60	−37
		1	43	57
		3	60	68
		5	<21	11
		7	51	−61
		9	<21	27
		11	<12	22
13	2	12−	34	30
		10−	<14	17
		8−	60	−58
		6−	94	−90
		4−	102	−87
		2−	<26	4
		0	12	0
		2	26	11
		4	33	47
		6	68	−76
		8	93	−92
		10	43	69
13	3	11−	9	−1
		9−	60	68
		7−	17	18
		5−	17	−21
		3−	<23	23
		1−	<17	2
		1	<17	13

TABLE 3. (*Continued.*)

h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc
13	3	3	−42	−24	14	0	8	51	86	14	2	2	<26	50	15	1	5−	77	−72	15	3	3	43	48
		5	43	−37			10	<17	41			4	51	−33			3−	110	−90					
		7	111	−101	14	1	11−	60	63			6	153	−102			1−	<26	26	16	0	6−	34	43
		9	<17	5			9−	51	54			8	<13	23			1	<26	22			4−	34	−48
13	4	6−	60	−61			7−	<17	6	14	3	9−	<52	48			3	<21	14			2−	60	−96
		4−	43	60			5−	26	32			7−	34	−34			5	<17	35			0	<34	1
		2−	<21	10			3−	<17	18			5−	<26	22			7	43	−35			2	<26	47
		0	<33	11			1−	44	37			3−	<26	36	15	2	8−	0	12			4	<21	48
		2	17	21			1	43	63			1−	<26	7			6−	51	−56	16	1	7−	<17	12
		4	<26	33			3	<26	40			1	85	64			4−	<26	1			5−	77	84
							5	<29	12			3	85	84			2−	<26	14			3−	<72	14
14	0	10−	43	81			7	<13	1			5	<17	10			0	<26	14			1−	34	−36
		8−	60	−68			9	<17	4			7	<14	19			2	<26	17			1	<27	23
		6−	94	−83	14	2	10−	<14	6	14	4	2−	16	18			4	51	−44			3	<22	28
		4−	77	−84			8−	17	13			0	<34	8			6	77	−92			5	<17	36
		2−	<26	10			6−	60	72						15	3	5−	34	42	16	2	4−	26	36
		0	68	−99			4−	<31	10								3−	<21	20			2−	<27	13
		2	102	−106			2−	94	−107	15	1	9−	77	−105			1−	<17	34			0	<26	5
		4	136	−140			0	<34	20			7−	68	−70			1	38	40			2	<18	13
		6	60	−47																				

$\Sigma F_o = 98{,}256$. $\Sigma F_c = 99{,}066$. $R = (\Sigma F_o - F_c)/F_o = 0{\cdot}20_8$.

Values suffering from extinction, such as 211, 402, are not included in the data when the residuals are calculated, but unobserved reflections are included as half their minimum observable value. The scale of the F's tabulated here is $\frac{1}{2} \times F_{\text{absolute}} \times 10$. The observed values are thought to be accurate to about 10%.

TABLE 4.

Atom	x	y	z	X	Y	Z	Atom	x	y	z	X	Y	Z
a	$0{\cdot}097_5$	$0{\cdot}447_9$	$0{\cdot}063_0$	$1{\cdot}41_4$	$2{\cdot}80_4$	$1{\cdot}19_1$	*h*	$0{\cdot}081_5$	$\overline{0{\cdot}020}_0$	$0{\cdot}212_1$	$1{\cdot}01_0$	$\overline{0{\cdot}12}_5$	$4{\cdot}00_9$
b	$0{\cdot}123_0$	$0{\cdot}383_3$	$\overline{0{\cdot}001}_7$	$1{\cdot}86_6$	$2{\cdot}39_9$	$\overline{0{\cdot}03}_2$	*i*	$0{\cdot}106_8$	$0{\cdot}184_7$	$0{\cdot}232_8$	$1{\cdot}37_2$	$1{\cdot}15_6$	$4{\cdot}40_0$
c	$0{\cdot}097_0$	$0{\cdot}184_7$	$\overline{0{\cdot}028}_3$	$1{\cdot}44_3$	$1{\cdot}15_6$	$\overline{0{\cdot}53}_5$	*j*	$0{\cdot}110_0$	$0{\cdot}341_3$	$\overline{0{\cdot}182}_3$	$1{\cdot}47_5$	$2{\cdot}13_7$	$\overline{3{\cdot}44}_6$
d	$0{\cdot}034_4$	$0{\cdot}060_7$	$0{\cdot}013_5$	$0{\cdot}50_8$	$0{\cdot}38_0$	$0{\cdot}25_6$	*k*	$0{\cdot}177_7$	$0{\cdot}518_3$	$\overline{0{\cdot}039}_3$	$3{\cdot}12_1$	$3{\cdot}24_5$	$\overline{0{\cdot}74}_3$
e	$0{\cdot}056_8$	$0{\cdot}091_7$	$0{\cdot}089_8$	$0{\cdot}76_7$	$0{\cdot}57_4$	$1{\cdot}69_7$	*l*	$0{\cdot}208_0$	$0{\cdot}445_7$	$\overline{0{\cdot}101}_8$	$3{\cdot}26_9$	$2{\cdot}79_0$	$\overline{1{\cdot}92}_4$
f	$0{\cdot}085_5$	$0{\cdot}292_9$	$0{\cdot}111_8$	$1{\cdot}17_9$	$1{\cdot}83_4$	$2{\cdot}11_3$	*m*	$0{\cdot}192_2$	$0{\cdot}237_5$	$\overline{0{\cdot}124}_7$	$3{\cdot}05_4$	$1{\cdot}48_7$	$\overline{2{\cdot}35}_7$
g	$0{\cdot}055_5$	$\overline{0{\cdot}066}_7$	$0{\cdot}142_2$	$0{\cdot}69_0$	$\overline{0{\cdot}41}_8$	$2{\cdot}68_8$	*n*	$0{\cdot}132_0$	$0{\cdot}111_7$	$\overline{0{\cdot}091}_2$	$2{\cdot}10_3$	$0{\cdot}69_9$	$\overline{1{\cdot}72}_3$

Atomic co-ordinates for α-dixanthenylidene, half-molecule centred on origin at $\bar{1}$ on glide plane *n*. Column headings as for Table 2.

DISCUSSION

The distances between the conflicting carbon atoms *g* and *n′* are 3·01 Å for α- and 2·97 Å for β-bixanthenylidene which in view of the assumed experimental errors could be identical. This is as close as non-bonded carbon atoms can approach and indicates strain in the molecule. This view is confirmed by a comparison with the results of the crystal structure analysis of rubrofusarin [13] (IIIb). Here the heterocyclic ring has a weak boat conformation, atoms C-15 and C-16 being only 0·051 and 0·077 Å, respectively, out of the plane of the other four ring atoms. In both bixanthenylidene molecules this distance is about 0·6 Å (*i.e.*, the oxygen atom *a* is about 0·3 Å from the *bcef* plane) and this, compared with rubrofusarin, indicates strain. The boat conformation is, of course, favoured rather than the chair because both in bixanthenylidene and in rubrofusarin there is a need to preserve planarity of the two halves of the ring formed by the diagonal through the oxygen atom (Fig. 4). This would not be satisfied by a ring in the chair conformation; compare, for example, the chair conformation of 1,4-dithian [14] (IV) with the boat conformation of 1,4-dithiin. (V).

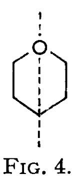

FIG. 4.

Apart from the central bond, the bond lengths are adequately explained on the basis of four bond structures (VI)—(IX). The mean length of 1·49 Å for *cd* and *de* compares well with a similar formally single C–C sp^2-bond of 1·477 Å in the more accurate analysis

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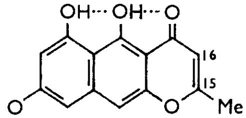
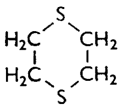
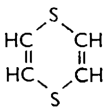
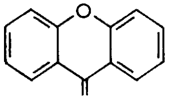
[13] Stout and Jensen, *Acta Cryst.*, 1962, **15**, 451.
[14] Marsh, *Acta Cryst.*, 1955, **8**, 91.

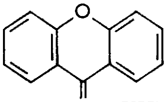

(IIIb)

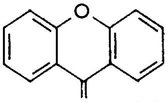

(IV)

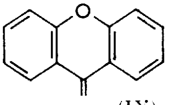

(V)

of *p*-benzoquinone[15] and of 1·478 Å in anthraquinone.[16] The mean C–O distance of 1·38 Å compares well with 1·37 Å for rubrofusarin. Possibly the only surprising bond length is the mean value of $1{\cdot}38_5$ Å for the central *dd'* bond, where one might have expected a length closer to 1·34 Å found in ethylene. However, there may be significant differences in the double-bond lengths found between two carbon atoms depending upon whether these atoms are bonded to other carbon atoms or not. In this connection, as Cruickshank[17] has pointed out, an accurate redetermination of the C=C length in tetramethylethylene would be of interest. In the two-dimensional analysis[1] of bianthronylidene (II) a central bond length of 1·31 Å was recorded. However, this analysis shows other features that do not agree well with ours, such as longer *cd*-type bonds of 1·53 Å. In view of the inevitably lower accuracy of this two-dimensional analysis it is difficult to assign much weight to the measured short central bond. The mean values found for bond lengths in the outer rings, namely, 1·37, $1{\cdot}38_5$ Å in the β-form and 1·39, $1{\cdot}39_3$ Å in the α-form, are in accord with the average of $1{\cdot}38_5$ Å found in anthraquinone, when the errors in our analysis are borne in mind.


(VI)


(VII)


(VIII)


(IX)

The differences in mode of molecular packing for the two polymorphs are shown by Figs. 5 (a) and (b). In both polymorphs the β-angle is close to 90°, so that in the *z*-projection for β-bixanthenylidene (Fig. 5b) and in the *x*-projection for α-bixanthenylidene the *C*-centred and *A*-centred layers, respectively, lie virtually in the plane of projection. In both these Figures the molecules drawn in full lines are those which have their mid-points lying in the centre layer containing the origin. The molecules in broken lines are those which have their midpoints lying in centred layers half a cell above (or below) the origin plane.

In β-bixanthenylidene the molecules lie rather flatter in the plane of projection than they do in the α-form but the molecular orientations are sufficiently similar for direct comparison. The esential difference in packing is that in the β-form the molecular centres all lie along the axes of projection whereas in the α-form they do not. In both cases, however, there is a tendency for the outer rings of the molecules to stack on top of each other in columnar array. In α-bixanthenylidene the heterocyclic rings are also stacked this way, but with the oxygen atoms alternating from left to right. Because of the manner in which the molecules are crossed in β-bixanthenylidene the heterocyclic rings do not lie in columns.

The closest distances between atoms in adjacent molecules which do not exceed 3·70 Å are listed in Tables 5 (a) and (b). In Table 5(a) the atoms listed in the extreme left hand column refer to a molecule centred on the origin and the co-ordinates for these are given in Table 1. The molecule to which the other atom of the pair belongs is indicated by a parenthetic Roman numeral thus: (I) $\frac{1}{2}+x,y-\frac{1}{2}, z$; (II) $x,\bar{y}, \frac{1}{2}+z$; (III) $\frac{1}{2}+x, \frac{1}{2}+y,z$. The

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[15] Trotter, *Acta Cryst.*, 1960, **13**, 86.

[16] Murty, *Z. Krist.*, 1960, **113**, 445.

[17] Cruickshank, *Tetrahedron*, 1962, **17**, 155.

Roman numerals are appropriately marked on Fig. 5(b) together with the letters appropriate to the atoms. To avoid confusion the shortest distances are not marked in.

Analogous remarks apply to Table 5(b) which lists the closest contacts for α-bixanthenylidene. The Roman numerals refer to co-ordinates as follows: (I) $x, y + 1, z$; (II) $x + \frac{1}{2}, \frac{1}{2} - y, z$. The fewer number of short contacts in this case is very marked.

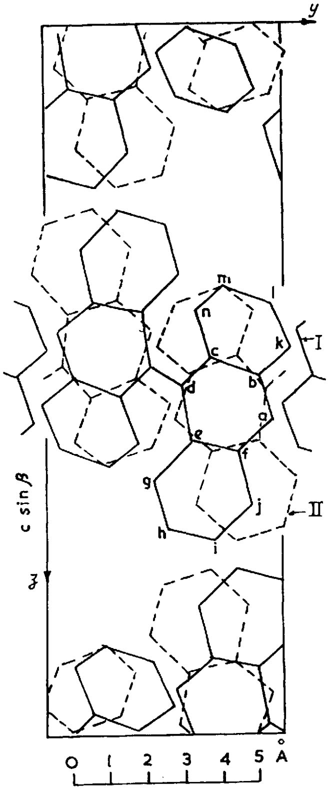

FIG. 5(a). α-Bixanthenylidene in *x*-projection.

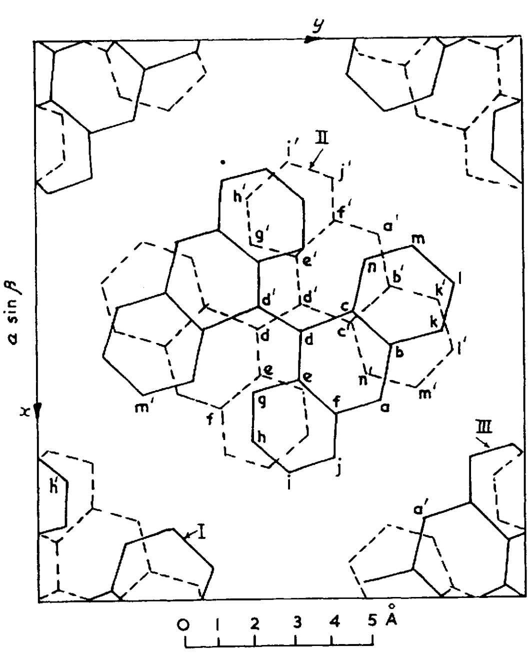

FIG. 5(b). β-Bixanthenylidene in *z*-projection.

TABLE 5(a).

	Å		Å
n–f' (II)	3·44	*h–a* (II)	3·55
h–f (II)	3·48	*e–n'* (II)	3·55
j–a' (III)	3·50	*j–n'* (II)	3·67
a–a' (III)	3·52	*m'–h'* (I)	3·68

TABLE 5(b).

	Å
a–g (I)	3·46
b–a' (I)	3·64
l–e' (II)	3·68

Non-bonded close contacts for (a) β- and (b) α-9,9'-bixanthenylidene, which are smaller than 3·70 Å. Atom *a* is oxygen; the others are carbon. Significance of Roman numerals is given in the Text and in Figs. 5(a) and (b).

We believe the differences in packing cause the differences in colour of the crystals but we are unable to specify what features of the packing are responsible.

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