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Crystal and Molecular Structure of 3,4′-Biisoxazole

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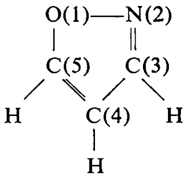
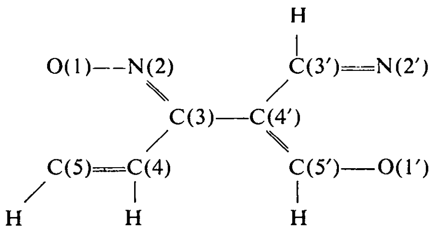
The crystal structure of 3,4′-biisoxazole has been determined from three-dimensional X-ray data. The atomic coordinates and anisotropic temperature factors have been refined by the block-diagonal least-squares method to give a final R index of 0·10 for 816 observed reflexions. The space group is $P2_1/c$, with $Z=4$ and unit-cell dimensions $a=3{\cdot}797$, $b=11{\cdot}175$, $c=14{\cdot}790$ Å and $\beta=94{\cdot}8°$. Bond distances and angles are within the expected values and suggest that there is no conjugation between the rings. The rings are not coplanar, the angle between their planes being 2·8°. Some values of the intermolecular distances are very close to the van der Waals limits.

Introduction

When two isoxazole rings (I) are bonded together six isomers are expected. Four out of the six have so far been synthesized: 3,3′ (Quilico, Gaudiano & Ricca, 1957); 5,5′ (Quilico, Gaudiano & Merlini, 1958); 3,5′ (Ricca & Gaudiano, 1959) and 3,4′ (Maggioni, Gaudiano & Bravo, 1966). The infrared and ultraviolet spectra of these isomers suggested a different conjugation effect between the rings, according to the carbon atoms involved in the inter-ring bond. This effect has been shown to be negligible in 3,3′, limited in 3,4′, bigger in 3,5′ and very strong in 5,5′ (Speroni, 1962; Maggioni, Gaudiano & Bravo, 1966). On the other hand structural studies of the two symmetrical isomers, performed by X-ray analysis (Cannas & Marongiu, 1967, 1968), suggested that the conjugation effect might be greater in 3,3′ than in 5,5′ at least in the solid state. Structural investigations have now been extended to the asymmetrical members of the series and in this paper the results of a three-dimensional analysis of 3,4′-biisoxazole (II) are reported.



(I)



(II)

Experimental

Crystal data: (Cu $K\alpha = 1{\cdot}5418$ Å)
3,4′-Biisoxazole, $C_6O_2N_2H_4$; F.W. 136·11; m.p. 86 °C.
$a = 3{\cdot}797 \pm 0{\cdot}008$, $b = 11{\cdot}175 \pm 0{\cdot}017$,
$c = 14{\cdot}790 \pm 0{\cdot}028$ Å, $\beta = 94{\cdot}8° \pm 0{\cdot}4°$.
$V = 625{\cdot}3$ Å^3 $Z = 4$
$D_m = 1{\cdot}43$ g.cm^{-3}, $D_x = 1{\cdot}445$ g.cm^{-3}.
Space group $P2_1/c$.

The crystals, in the form of colourless prisms elongated along **a**, were supplied by Professor Gaudiano, Politecnico di Milano (Italy). Two crystals, of approximate dimensions 0·45 × 0·20 × 0·30 mm and 0·35 × 0·30 × 0·30 mm were selected for data collection around [100] and [001] respectively. Multifilm equi-inclination Weissenberg techniques with Ni-filtered Cu $K\alpha$ radiation were used to collect intensities on layers 0 to 3 around the a axis and 0 to 4 around the c axis. Of 1420 possible reflexions within the copper sphere 1215 were recorded; of these 827 were observed above the background of the film and were estimated visually by comparison with a standard scale.

The reflexions were reduced to structure factor amplitudes using a series of IBM 1620 data reduction programs, written in this laboratory, which take into account Lp factor, the shape of the spots on non-equatorial layers and the incipient but incomplete α_1–α_2 splitting. An estimate of the standard deviation in each value of F_o was derived according to the range of the standard scale in which the pertinent intensity was evaluated. The standard scale was divided into five ranges and within each one a constant fractional error was assumed; with this assumption the standard deviation is given by $\sigma^2 = \alpha F_o^2$, where $\alpha = 1{\cdot}0$, 1·5, 2·0, 2·5 and 3·0 in the five ranges. Absorption correction was neglected. The interlayer correlation scales were obtained by the method of Rae (1965). Scattering factors were interpolated from the values given in *International Tables for X-ray Crystallography* (1962).

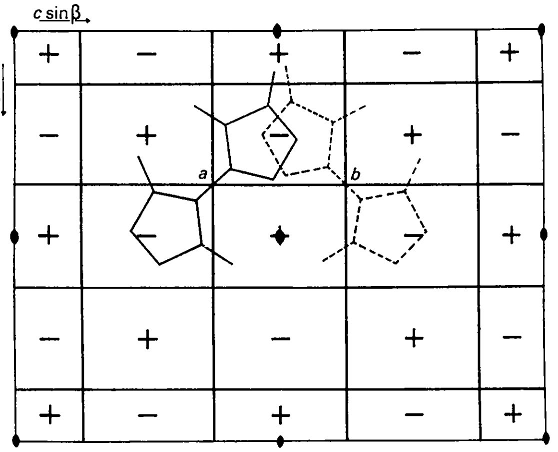


Fig. 1. Trial arrangements; the most probable molecular dispositions (a,b) are shown superimposed on the graph of the cos $2\pi 2y$ cos$2\pi 2z$ function.

Structure analysis and refinement

Since the short d_{100} value (3·78 Å) suggested a molecular packing approximately parallel to (100), the analysis was started with the a projection, with $0kl$ reflexion data. Trial and error methods were used, assuming a molecular model with double bonds C(4′)–C(5′) and C(3)–N(2) in the *trans* position in analogy with similar molecules reported in the literature: 3,3′ and 5,5′-biisoxazole, 3,3′-bi-2-isoxazoline and 2,2′-bithienyl. Bond angles and distances were derived according to the 3,3′ and 5,5′ isomers. Fig. 1 shows two trial arrangements, which were based on the very high intensity of the 022 reflexion. Assuming that the molecular packing is governed by C–H···N and C–H···O contacts between molecules related by symmetry, the two given arrangements are the only ones possible; position a was preferred to b as a result of a structure factor calculation for the more intense reflexions in the sin θ range between 0 and 0·60. Three Fourier syntheses were performed and the R value dropped to 0·21 for all observed $0kl$ reflexions.

Positional and thermal parameters of oxygen, nitrogen and carbon atoms were refined by four cycles of block-diagonal least squares, using the program written by van der Helm (1961). At this stage of the refinement ($R = 0{\cdot}14$) C(3)–C(4) and C(3′)–C(4′) bonds in the projection examined appeared to be shortened compared with the expected values. This suggested that the molecule is rotated about 30° around an axis perpendicular to the direction of these bonds. On the basis of this consideration and by means of structure factor graphs, x coordinates suitable for three-dimensional refinement were obtained, and the least-squares refinement was continued. The quantity minimized was $\Sigma\, w(kF_o - F_c)^2$, where w is equal to $1/\sigma^2$ for estimated reflexions and equal to zero for the unobserved and for those observed but not estimated. In the first cycle positional parameters of oxygen, nitrogen and carbon atoms were refined. The scale factor and isotropic thermal parameters of the last $0kl$ computation were kept constant. A discrepancy in the scale factor increasing with h was immediately evident. Since the possibility of an incorrect rescaling procedure could be disregarded, an anisotropic effect of thermal parameters seemed to be the more reasonable explanation.

Further cycles were performed introducing anisotropic temperature factors of the form

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

for heavy atoms. Hydrogen atoms were also introduced but not refined; their positional parameters were calculated assuming a bond distance of 1·1 Å directed along the bisector of the pertinent carbon atom angle; the isotropic thermal parameters of the hydrogen atoms were given the value 5·0 Å^2. After the second cycle the R value was 0·19 for observed reflexions. When the observed and calculated structure factors were compared at this stage, some low angle, high intensity reflexions were found systematically to have values of F_o smaller than F_c, possibly due to secondary extinction effects. Another explanation of these discrepancies might be that the intensities of these reflexions were measured on the last film in the multiple-

Table 1. *Final positional and thermal parameters*

The estimated standard deviations given in parentheses refer to the last decimal position.

	X	Y	Z
O(1)	0·0092 (9)	0·2490 (3)	0·5624 (2)
N(2)	−0·1466 (11)	0·3403 (3)	0·5052 (3)
C(3)	−0·1385 (11)	0·2985 (3)	0·4218 (3)
C(4)	0·0158 (11)	0·1820 (4)	0·4220 (3)
C(5)	0·0998 (13)	0·1575 (4)	0·5099 (3)
O(1′)	−0·4305 (11)	0·4360 (3)	0·2022 (2)
N(2′)	−0·5042 (11)	0·5300 (3)	0·2625 (3)
C(3′)	−0·4085 (13)	0·4891 (4)	0·3447 (3)
C(4′)	−0·2764 (10)	0·3727 (3)	0·3441 (3)
C(5′)	−0·2942 (14)	0·3454 (4)	0·2534 (3)
H(4)	0·0560	0·1230	0·3730
H(5)	0·1980	0·0850	0·5570
H(3′)	−0·4700	0·5410	0·3990
H(5′)	−0·2100	0·2780	0·2150

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O(1)	0·17439 (381)	0·00998 (30)	0·00344 (15)	−0·00603 (169)	0·00356 (104)	0·00126 (33)
N(2)	0·14863 (399)	0·00829 (32)	0·00389 (18)	0·00112 (167)	0·00899 (128)	−0·00035 (36)
C(3)	0·08772 (321)	0·00739 (28)	0·00369 (19)	−0·00706 (148)	0·00529 (112)	0·00018 (36)
C(4)	0·10109 (404)	0·00851 (37)	0·00446 (23)	0·00311 (167)	0·00486 (141)	−0·00110 (43)
C(5)	0·11930 (419)	0·00810 (36)	0·00536 (25)	−0·00290 (191)	0·00116 (155)	0·00228 (48)
O(1′)	0·19167 (412)	0·01227 (36)	0·00386 (17)	−0·00063 (194)	−0·00226 (123)	0·00076 (38)
N(2′)	0·13805 (384)	0·00819 (30)	0·00483 (20)	0·00145 (164)	−0·00329 (132)	0·00092 (41)
C(3′)	0·12176 (435)	0·00898 (38)	0·00442 (23)	0·00210 (198)	0·00370 (140)	−0·00174 (47)
C(4′)	0·08655 (344)	0·00800 (33)	0·00403 (19)	−0·00711 (155)	0·00665 (121)	−0·00033 (41)
C(5′)	0·15954 (509)	0·00943 (40)	0·00333 (21)	0·00112 (218)	0·00856 (157)	0·00004 (41)

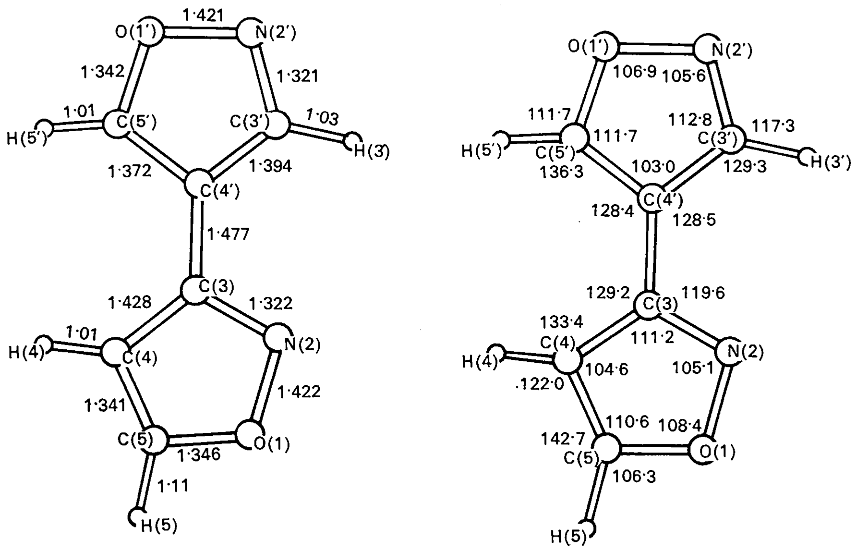


Fig. 2. Bond distances and angles.

film set and were too strong to be estimated with any accuracy. Eleven reflexions were excluded from the data on this basis. The refinement continued to an *R* value of 0·11, when a difference-Fourier synthesis was calculated, based on the heavy atom parameters. The four hydrogen atom peaks were found at reasonable locations with respect to the carbon atoms; no other significant residual electron density peaks were present. A final refinement was done using these hydrogen positions, but not refining them. The reliability index was reduced to 0·10 (omitting unobserved reflexions and the 11 suspected of secondary extinction effects). The

Table 2. *Observed and calculated structure factors*

Columns are: k, l, $10F_{obs}$, $10F_{calc}$. Unobserved reflexions are marked with an asterisk and have been given values corresponding to half the estimated minimum observable intensity. Reflexions marked with two asterisks are apparently affected by extinction.

		H=0	
0	2	55	49
0	4	451	435
0	6	136	-132
0	8	16	-11
0	10	66	-57
0	12	226	219
0	14	158	138
0	16	61	60
0	18	7*	-14
1	1	166	127
1	2	169	162
1	3	169	-163
1	4	23	-17
1	5	54	-58
1	6	306	-293
1	7	231	230
1	8	56	-56
1	9	47	-50
1	10	116	-101
1	11	56	-62
1	12	36	-35
1	13	149	140
1	14	85	79
1	15	80	-79
1	16	11*	5
1	17	31	30
1	18	25	-19
2	0	241	-211
2	1	457	453
2	2	837	-833
2	3	191	-188
2	4	5*	-13
2	5	54	-40
2	6	250	227
2	7	109	89
2	8	150	130
2	9	36	28
2	10	8*	-2
2	11	25	-28
2	12	94	-89
2	13	11*	7
2	14	74	-64
2	15	11*	5
2	16	75	-65
2	17	9*	8
2	18	7*	-5
3	1	26	24
3	2	45	31
3	3	67	-55
3	4	178	172
3	5	93	-85
3	6	129	-115
3	7	26	-24
3	8	215	199
3	9	24	-27
3	10	66	-58
3	11	59	-52
3	12	96	-88
3	13	11*	12
3	14	11*	-8
3	15	43	-36
3	16	43	-37
3	17	8*	2
3	18	6*	2
4	0	40	36
4	1	29	-15
4	2	93	-73
4	3	94	78
4	4	144	-137
4	5	182	-155
4	6	312	-255
4	7	198	161
4	8	211	-196
4	9	148	-141
4	10	105	-99
4	11	92	94
4	12	45	-45
4	13	70	-66
4	14	11*	15
4	15	34	31
4	16	10*	-3
4	17	8*	-8
4	18	5*	8
5	1	93	88
5	2	311	-292
5	3	78	-72
5	4	26	26
5	5	69	62
5	6	185	-173
5	7	45	-38
5	8	136	128
5	9	32	31
5	10	53	64
5	11	10*	3
5	12	121	110
5	13	24	27
5	14	39	34
5	15	10*	8
5	16	9*	15
5	17	6*	1
6	0	267	244
6	1	85	-73
6	2	56	61
6	3	104	95
6	4	143	141
6	5	29	-28
6	6	86	81
6	7	76	73
6	8	244	225
6	9	113	-109
6	10	10*	-5
6	11	75	69
6	12	166	150
6	13	52	-48
6	14	11*	4
6	15	10*	15
6	16	8*	1
7	1	57	58
7	2	105	92
7	3	166	-151
7	4	208	194
7	5	142	133
7	6	17	7
7	7	40	37
7	8	34	-36
7	9	10*	-7
7	10	127	-119
7	11	25	-25
7	12	55	-53
7	13	11*	13
7	14	32	-32
7	15	8*	-8
8	0	14	8
8	1	23	-24
8	2	13	-6
8	3	26	-23
8	4	55	-53
8	5	63	-62
8	6	73	70
8	7	21	16
8	8	25	25
8	9	54	50
8	10	136	-117
8	11	11*	
8	12	11*	14
8	13	10*	13
8	14	8*	-9
8	15	6*	-8
9	1	61	61
9	2	41	-39
9	3	81	-81
9	4	150	-146
9	5	10*	-1
9	6	64	-62
9	7	21	-22
9	8	84	73
9	9	54	-43
9	10	32	32
9	11	15	12
9	12	27	24
9	13	18	17
10	0	84	-91
10	1	11*	-11
10	2	11*	-13
10	3	87	90
10	4	122	-121
10	5	63	-62
10	6	23	-21
10	7	34	32
10	8	58	-51
10	9	11*	-8
10	10	10*	-7
10	11	44	-36
10	12	8*	-1
10	13	6*	-6
11	1	11*	-13
11	2	85	83
11	3	11*	7
11	4	26	30
11	5	19	18
11	6	26	25
11	7	19	16
11	8	25	23
11	9	9*	2
11	10	36	-30
11	11	7*	3
12	0	36	32
12	1	39	-41
12	2	48	48
12	3	37	33
12	4	42	40
12	5	39	-40
12	6	21	21
12	7	16	19
12	8	8*	9
12	9	7*	-3
13	1	9*	-7
13	2	9*	-13
13	3	9*	5
13	4	19	-21
13	5	8*	-6
13	6	7*	-9
14	0	5*	-4
14	1	10*	-9
14	2	10*	-7
14	3	5*	-6
14	4	8*	-3
		H=1	
0	0	122**	159
0	2	495**	677
0	4	66	-93
0	6	7*	-8
0	8	174	-198
0	10	96	107
0	12	117	146
0	14	12	17
0	16	46	51
0	18	5*	21
0	-2	837**	1265
0	-4	5*	16
0	-6	164	-197
0	-8	20	-16
0	-10	9*	-15
0	-12	10*	5
0	-14	168	167
0	-16	58	54
1	0	343**	427
1	1	336**	-453
1	2	47	-55
1	3	83	104
1	4	23	-28
1	5	137	175
1	6	70	-72
1	7	70	-84
1	8	87	-103
1	9	21	24
1	10	52	-56
1	11	103	114
1	12	80	91
1	13	66	-81
1	14	36	33
1	15	46	45
1	16	30	29
1	17	8*	-16
1	18	5*	-26
1	-1	166**	217
1	-2	153	-183
1	-3	10	-18
1	-4	131	154
1	-5	5*	12
1	-6	38	44
1	-7	76	-81
1	-8	185	210
1	-9	41	49
1	-10	9*	8
1	-11	24	-23
1	-12	67	67
1	-13	80	-80
1	-14	12*	-4
1	-15	78	68
1	-16	11*	-13
1	-17	9*	-23
2	0	744**	-887
2	1	164**	203
2	2	5*	
2	3	69	91
2	4	27	-35
2	5	104	105
2	6	169	206
2	7	94	102
2	8	21	28
2	9	65	72
2	10	23	20
2	11	27	22
2	12	50	-53
2	13	100	113
2	14	77	-77
2	15	11*	-9
2	16	10*	-7
2	17	8*	3
2	-1	369**	519
2	-2	54	-64
2	-3	87	95
2	-4	136	-157
2	-5	42	41
2	-6	91	89
2	-7	182	193
2	-8	165	197
2	-9	54	-54
2	-10	62	57
2	-11	46	49
2	-12	72	-70
2	-13	92	90
2	-14	77	-72
2	-15	12*	11
2	-16	48	-39
2	-17	21	24
3	0	98	114
3	1	145	166
3	2	5*	-5
3	3	34	36
3	4	32	-34
3	5	37	43
3	6	215	268
3	7	133	-157
3	8	52	-59
3	9	9*	-4
3	10	10*	-7
3	11	59	-59
3	12	35	-35
3	13	28	-27
3	14	55	-52
3	15	10*	12
3	16	9*	-11
3	17	7*	-6
3	-1	121	-140
3	-2	220	-263
3	-3	88	101
3	-4	24	32
3	-5	96	-103
3	-6	88	-105
3	-7	49	45
3	-8	69	75
3	-9	9*	-8
3	-10	43	-37
3	-11	118	136
3	-12	34	34
3	-13	68	-61
3	-14	69	63
3	-15	47	47
3	-16	10*	-
3	-17	27	-26
4	0	94	-98
4	1	185	-219
4	2	110	113
4	3	58	-63
4	4	141	-153
4	5	11	11
4	6	115	-134
4	7	80	-93
4	8	113	-134
4	9	10*	5
4	10	14	-18
4	11	99	-119
4	12	52	-51
4	13	12*	-
4	14	23	20
4	15	45	-42
4	16	8*	10
4	-1	166	-190
4	-2	63	67
4	-3	71	-91
4	-4	34	-33
4	-5	158	-178
4	-6	136	-142
4	-7	136	138
4	-8	191	-204
4	-9	111	-111
4	-10	51	-52
4	-11	10*	12
4	-12	23	-24
4	-13	86	-82
4	-14	12*	-12
4	-15	23	-18
4	-16	31	35
4	-17	8*	-8
5	0	102	-111
5	1	77	-72
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5	3	24	27
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6	-9	62		-50
6	-10	10	*	-5
6	-11	26		-24
6	-12	8	*	2
6	-13	32		28
7	0	17		12
7	1	43		35
7	2	11	*	-3
7	3	11	*	-6
7	4	11	*	-
7	5	10	*	-5
7	6	33		27
7	7	10	*	-7
7	8	22		-17
7	9	17		-15
7	10	6	*	-5
7	-1	15		-9
7	-2	26		-21
7	-3	11	*	-13
7	-4	45		-38
7	-5	41		-33
7	-6	11	*	-6
7	-7	10	*	-10
7	-8	10	*	8
7	-9	10	*	-12
7	-10	8	*	-13
7	-11	6	*	7
7	-12	6	*	8
8	0	25		23
8	1	35		31
8	2	10	*	4
8	3	10	*	-1
8	4	10	*	-3
8	5	39		37
8	6	9	*	-3
8	7	6	*	4
8	8	7	*	-
8	9	5	*	15
8	-1	11	*	-11
8	-2	11	*	1
8	-3	31		25
8	-4	10	*	-8
8	-5	10	*	1
8	-6	10	*	15
8	-7	42		40
8	-8	19		-17
8	-9	6	*	4
8	-10	7	*	-10
8	-11	16		13
9	0	23		-19
9	1	16		12
9	2	10		4
9	3	9	*	-6
9	4	8	*	-10
9	5	8	*	-3
9	6	7	*	-3
9	7	6	*	6
9	8	5	*	-2
9	-1	19		16
9	-2	10	*	-4
9	-3	10	*	4
9	-4	10	*	6
9	-5	9	*	11
9	-6	8	*	-1
9	-7	6	*	7
9	-8	7	*	2
9	-9	6	*	6
9	-10	5	*	-1
10	0	8	*	-5
10	1	8	*	12
10	2	8	*	-12
10	3	7	*	1
10	4	13		14
10	5	6	*	2
10	-1	25		-24
10	-2	8	*	12
10	-3	8	*	-7
10	-4	8	*	1
10	-5	7	*	-9
10	-6	25		22
10	-7	6	*	-10
10	-8	4	*	15
11	0	5	*	12
11	1	23		-24
11	2	5	*	7
11	-1	6	*	
11	-2	5	*	7
11	-3	5	*	5
11	-4	5	*	15

shifts in the positional parameters were now much less than the standard deviations, indicating that with the present data no significant improvement by least-squares refinement could be expected. The final positional and thermal parameters and their standard deviations are listed in Table 1. The observed and calculated structure factors are shown in Table 2.

Discussion

The molecular configuration, shown in (II), presents the bonds C(3)–N(2) and C(4′)–C(5′) in *trans* positions. The best molecular plane was calculated according to Schomaker, Waser, Marsh & Bergman (1959), assigning unit weights to all the atoms. Since the deviations of some atoms from this plane are higher than the expected standard deviations, the planes for the individual rings were also calculated. The results of this analysis, summarized in Table 3, suggest that the rings are planar, but that the molecule is significantly out of planarity, the angle between the planes of the two rings being 2·84°.

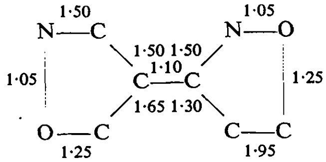
Table 3. *Least squares planes*

	Plane and deviations ($\times 10^4$) from the plane		
	I	II	III
O(1)	−27	−14	
N(2)	312	20	
C(3)	174	−19	
C(4)	−160	9	
C(5)	−274	4	
O(1′)	21		25
N(2′)	−276		7
C(3′)	−201		−34
C(4′)	220		48
C(5′)	212		−46

Plane	
I	$3{\cdot}493X+4{\cdot}336Y-1{\cdot}999Z=-0{\cdot}0153$
II	$3{\cdot}454X+4{\cdot}594Y-2{\cdot}014Z=0{\cdot}0419$
III	$3{\cdot}527X+4{\cdot}084Y-2{\cdot}025Z=-0{\cdot}1448$

Bond distances and angles are shown in Fig. 2 and Table 4, where the standard deviations, derived from the uncertainties of the final atomic parameters and cell dimensions, are indicated in parentheses. Owing to the block-diagonal approximation and to additional uncertainties introduced by the effects of thermal libration and, to some extent, of systematic errors in the data, their values should probably be increased by 50%. Bond angles and distances involving hydrogen atoms should have a standard deviation of 0·1 Å and 3°, because of the lack of accuracy in localizing the maxima of the peaks in the difference-Fourier synthesis.

The value of the C(3)–C(4′) bond, connecting the two rings, is that of a C(sp^2)–C(sp^2) bond; it suggests an irrelevant conjugation effect between the rings. A resonance effect is, on the other hand, evident within the ring, where all the bonds, except N–O, have a significant partial double bond character. Bond numbers, calculated according to Pauling (1960) are as follows:



where the sum of double bond character is 4·10, which is very close to the expected value 4·00.

The magnitudes and orientations of the principal axes of thermal motion are listed in Table 5; a projection along an axis normal to Z and making an angle of

−10° with the $Z-Y$ plane is shown in Fig. 3 (Johnson, 1965). It can be seen that the higher vibration amplitudes are perpendicular to the molecular plane.

The structure viewed along the *a* and *b* axes is shown in Figs. 4 and 5.

As in the 3,3′ and 5,5′ isomers, the main feature of the packing is a network of C–H···O and C–H···N contacts. Their values, which are very close to the van der Waals limiting distances, are shown in Fig. 4.

The computations in the present analysis were carried out on the IBM 1620/II of this University, exclud-

Table 4. *Bond distances and angles with their standard deviations*

Corresponding bond angles and distances in the rings are given side by side.

C(3)—C(4′)	1·477 Å	0·006 Å			
C(4′)–C(3′)	1·394	0·006	C(4)–C(3)	1·428 Å	0·007 Å
C(4′)–C(5′)	1·372	0·005	C(4)–C(5)	1·341	0·007
C(5′)–O(1′)	1·342	0·006	C(5)–O(1)	1·346	0·006
N(2′)–O(1′)	1·421	0·006	N(2)–O(1)	1·422	0·006
C(3′)–N(2′)	1·321	0·007	C(3)–N(2)	1·322	0·007
C(5′)–H(5′)	1·01		C(5)–H(5)	1·11	
C(3′)–H(3′)	1·03				
			C(4)–H(4)	1·01	
C(5′)–C(4′)–C(3)	128·40°	0·37°			
C(3′)–C(4′)–C(3)	128·54	0·40			
C(4)—C(3) –C(4′)	129·22	0·40			
N(2) –C(3) –C(4′)	119·56	0·36			
N(2′)–C(3′)–H(3′)	117·29				
C(4′)–C(3′)–H(3′)	129·31				
C(3)—C(4) –H(4)	133·38				
C(5)—C(4) –H(4)	121·96				
C(5′)–C(4′)–C(3′)	103·02	0·39	C(5)–C(4)–C(3)	104·63°	0·43°
C(4′)–C(3′)–N(2′)	112·85	0·41	C(4)–C(3)–N(2)	111·22	0·39
C(3′)–N(2′)–O(1′)	105·57	0·35	C(3)–N(2)–O(1)	105·10	0·35
N(2′)–O(1′)–C(5′)	106·89	0·35	N(2)–O(1)–C(5)	108·39	0·34
O(1′)–C(5′)–C(4′)	111·66	0·40	O(1)–C(5)–C(4)	110·65	0·42
O(1′)–C(5′)–H(5′)	111·73		O(1)–C(5)–H(5)	106·29	
C(4′)–C(5′)–H(5′)	136·30		C(4)–C(5)–H(5)	142·75	

Table 5. *Description of thermal ellipsoids*

$B_i=8(\pi U_i)^2$, where U_i is the root mean square displacement corresponding to the *i* axis of the ellipsoid. $\theta_{ia^*}\theta_{ib}\theta_{ic}$ are the angles between the *i* axis and the crystal axes a^*,b,c.

	i	B_i	U_i	θ_{ia^*}	θ_{ib}	θ_{ic}
O(1)	1	2·899	0·1916	92·7	78·2	167·9
	2	5·017	0·2521	83·3	13·3	78·6
	3	10·072	0·3571	172·7	83·9	86·1
N(2)	1	3·262	0·2033	93·3	81·7	9·0
	2	4·159	0·2295	90·7	8·4	98·6
	3	8·529	0·3287	3·6	88·8	86·8
C(3)	1	3·090	0·1978	79·0	69·2	156·2
	2	3·534	0·2116	72·4	30·0	66·5
	3	5·259	0·2581	159·7	69·6	93·3
C(4)	1	3·604	0·2136	95·0	57·6	32·8
	2	4·459	0·2377	98·1	34·1	122·9
	3	5·832	0·2718	9·5	80·5	90·0
C(5)	1	3·538	0·2117	90·7	33·5	123·5
	2	5·082	0·2537	73·9	57·8	36·9
	3	6·978	0·2973	163·9	81·8	76·2
O(1′)	1	3·299	0·2044	98·5	85·0	170·2
	2	6·148	0·2791	88·2	5·1	85·3
	3	11·157	0·3759	171·2	88·9	81·2
N(2′)	1	3·765	0·2184	101·1	45·9	133·8
	2	4·396	0·2360	80·0	44·1	47·6
	3	8·184	0·3219	15·0	89·4	105·0
C(3′)	1	3·496	0·2105	89·1	59·3	30·7
	2	4·812	0·2469	96·3	31·2	120·4
	3	7·000	0·2978	6·4	85·0	94·0
C(4′)	1	3·361	0·2063	75·3	81·2	162·8
	2	4·812	0·2173	68·0	26·5	76·0
	3	5·301	0·2591	153·1	65·2	99·8
C(5′)	1	2·814	0·1888	88·2	90·0	178·2
	2	4·708	0·2442	91·2	1·2	90·0
	3	9·144	0·3403	2·2	88·6	88·3

ing the drawing of the thermal ellipsoids which was produced in the Computer Center of the University of Washington.

The authors wish to thank Professor P. Paoletti and Professor C. Dejak for helpful discussions and for their continued interest in the work.

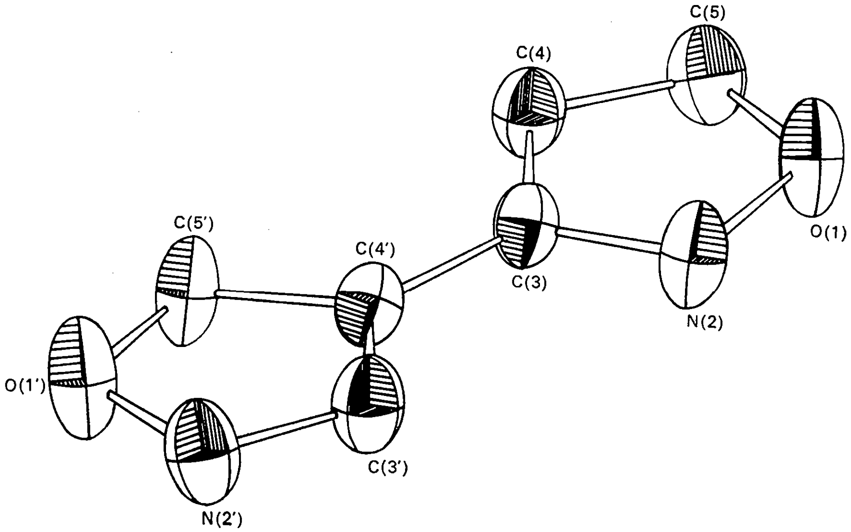


Fig. 3. The thermal ellipsoids.

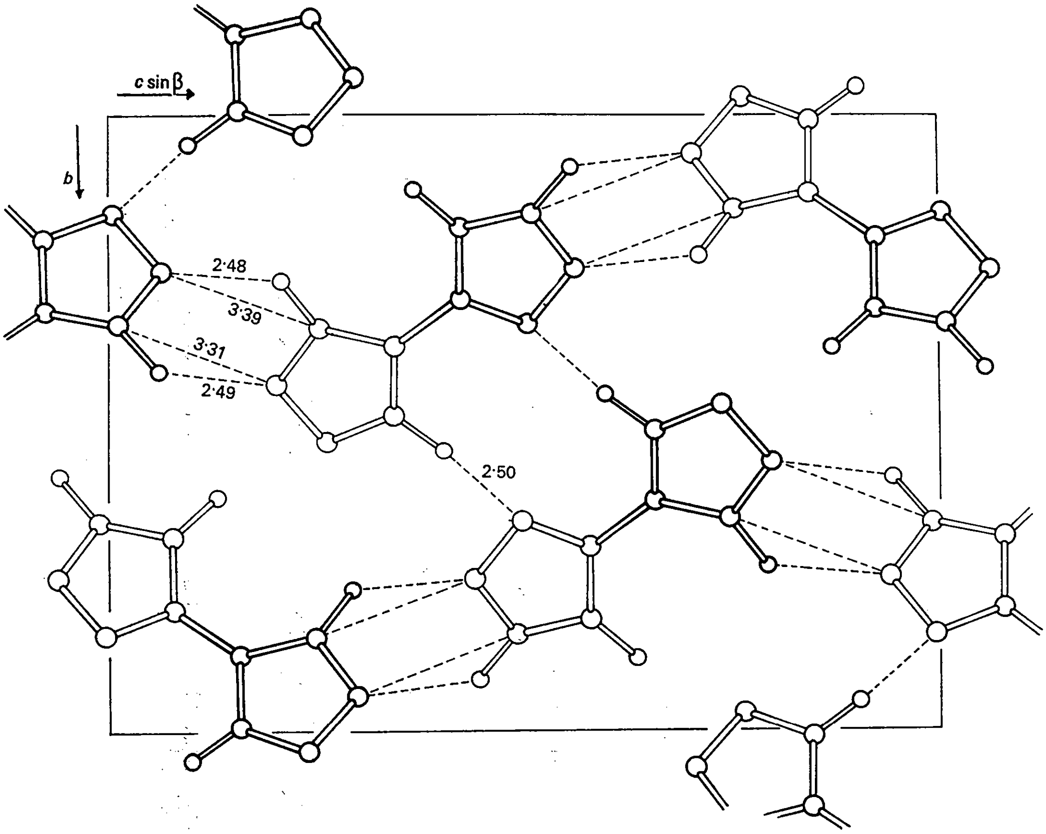


Fig. 4. Projection of the structure along the *a* axis.

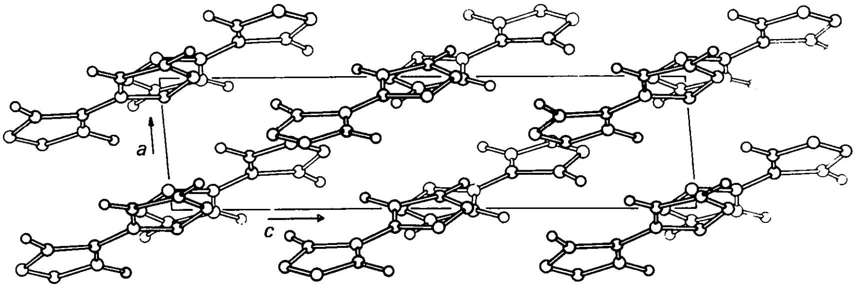


Fig. 5. Projection of the structure along the b axis.

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The Molecular Structure of Fe(CO)₅ in the Gas Phase

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New electron diffraction intensity data have been collected for gaseous iron pentacarbonyl, $Fe(CO)_5$. Full-matrix least-squares refinement confirms the trigonal bipyramid molecular structure found in earlier studies. The structural parameters found are: C–O (mean) $1\cdot145 \pm 0\cdot003$ Å, Fe–C (equatorial) $1\cdot833 \pm 0\cdot004$ Å, Fe–C (axial) $1\cdot806 \pm 0\cdot005$ Å, Fe–C (mean) $1\cdot822 \pm 0\cdot003$ Å. The effects of different weighting schemes, different magnitudes of shrinkage corrections and the use of assumed vibrational amplitudes have been investigated in detail. The results are compared with a parallel study by Almenningen, Haaland & Wahl.

Introduction

The crystal structure of iron pentacarbonyl, $Fe(CO)_5$, has been investigated by A. W. Hanson (1962), and further refined in an alternative space group by Donohue & Caron (1964). These studies showed that the molecular structure is, within experimental error, a trigonal bipyramid, with the carbon atoms of the carbonyl groups bonded to the iron atom. No significant difference between the axial and equatorial Fe–C bond lengths was found. Davis & Hanson (1965, 1967) have studied iron pentacarbonyl in the gas phase by electron diffraction and concluded that the equatorial Fe–C bond length is longer than the axial Fe–C bond length by $0\cdot049 \pm 0\cdot020$ Å. This work has been the subject of comment by Donohue & Caron (1966, 1967),