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The Crystal Structure of Diphenyl-Δ²-Pyrazoline

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Diphenyl-Δ^2-pyrazoline crystallizes in the monoclinic space group $P2_1/c$ with unit-cell dimensions $a=5\cdot404$, $b=10\cdot162$, $c=21\cdot683$ Å, $\beta=92\cdot85°$, and with $Z=4$. The crystal structure has been determined by the symbolic addition method and refined by least-squares analysis of three-dimensional data collected by both film and counter techniques. The molecule is not completely planar; the dihedral angle between the planes of the phenyl rings is 11°. Both phenyl groups are planar with an average C–C distance of 1·385 Å and an average C–H bond length of 0·99 Å. The pyrazoline ring appears to be significantly non-planar, the maximum deviation from the least-squares plane defined by the ring being 0·053 Å. The intermolecular separations correspond to van der Waals interactions.

Introduction

Many derivatives of diphenyl-Δ^2-pyrazoline (DPP) exhibit strong fluorescent activity and can be used as whitening agents for nylon fabrics in commercial detergents. The crystal structure of the parent compound has been investigated in order to determine the degree of conjugation and the extent of coplanarity of the three rings in the molecule as these factors will influence the physical properties of the fluorescer.

Experimental

Two sets of three-dimensional data were used in this work. A set of data collected on a diffractometer replaced data obtained by photographic methods in the final cycles of refinement.

Crystals of DPP were obtained as pale green needles from ethanol solution. With the use of Cu $K\alpha$ radiation and the multiple film technique, equi-inclination Weissenberg photographs were taken of the levels $0kl \rightarrow 2kl$ for the crystal rotating about the a crystallographic axis and of the levels $h0l \rightarrow h4l$ for the b-axis rotation. The intensities of the reflexions were measured by visual comparison with a calibrated scale, and after application of Lorentz and polarization corrections, common reflexions observed on both rotation axes enabled all the data to be placed on a common relative scale. A total of 1301 independent reflexions were obtained and a further 402 reflexions were below the minimum observable value. No correction was made for absorption.

Data were collected for a second crystal on a Hilger and Watts automatic four-circle diffractometer in the normal beam arrangement. Integrated intensities were obtained for $\theta \le 56°$ by the θ–2θ scanning method using Cu $K\alpha$ radiation and a scintillation counter with pulse height discrimination. Each reflexion was integrated in eighty steps of intervals of 0·01°. Observed structure factor values were derived from the integrated intensities in a data reduction program which also corrected for variation in the integrated intensity of a standard reflexion, measured at frequent intervals during the data collection. No correction was made for absorption. A total of 1552 independent reflexions were measured and of these, 212 reflexions were below the threshold value for an observed reflexion.

Lattice parameters were obtained from measurements of diffraction angles on the diffractometer and the systematic extinctions were determined from the Weissenberg diagrams. The crystal data are as follows:

Diphenyl-Δ^2-pyrazoline, $C_{15}N_2H_{14}$ F.W. 222·3
Monoclinic, $a=5\cdot404(2)$, $b=10\cdot162(4)$, $c=21\cdot683(10)$Å, $\beta=92\cdot85(0\cdot25)°$
$U=1189\cdot3$ Å^3, $Z=4$, $D_x=1\cdot24$ g.cm^{-3}, D_m (not measured)
Cu $K\alpha$ radiation ($\lambda=1\cdot5418$ Å).
Space group, $P2_1/c$ (No. 14).

Structure determination

An examination of the Patterson map obtained using the $0kl$ film data reflexions showed peaks which could be interpreted as representing both intra- and intermolecular ring-to-ring vectors. Several structure factor trials, based on molecular positions derived from the map and assuming the molecule to lie close to the (123) plane, which was represented by the most intense reflexion, failed to indicate a correct trial structure. Further attempts to solve the structure in the $0kl$ projection by the application of minimum functions (Buerger, 1959) were unsuccessful. A three-dimensional vector map, calculated with the use of all the available film data, could not be fully interpreted in terms of molecular orientation, owing to the poor resolution of peaks in the region of the origin.

The symbolic addition procedure (Karle & Karle, 1963) seemed an ideal method of phase determination for the structure and a computer program was used to place the film data on an absolute scale by Wilson's (1942) method and to determine the value of the overall temperature factor as $B=4\cdot03$ Å^2. Normalized structure factor magnitudes, $|E|$, were computed from the observed structure factor values, $|F|$, with thc formula (Karle & Karle, 1964):

$$E_{\mathbf{h}}^2 = F_{\mathbf{h}}^2/\varepsilon \sum_{j=1}^{N} f_{j\mathbf{h}}^2$$

where N is the number of atoms in the unit cell, f_j is the atomic scattering factor for the jth atom corrected for the overall temperature factor B and $\varepsilon = 1$ except for $h0l$ and $0k0$ where $\varepsilon = 2$. The program listed all triplet interactions $E_{\mathbf{h}}$, $E_{\mathbf{k}}$ and $E_{\mathbf{h}-\mathbf{k}}$ with $E_{\mathbf{h}}$ values greater than the chosen limit $|E| = 1{\cdot}5$ which allowed 210 reflexions to be used for the symbolic addition procedure. A lower limit of $|E| = 2{\cdot}0$ was used for the $E_{\mathbf{k}}$ and $E_{\mathbf{h}-\mathbf{k}}$ values to ensure a number of statistically preferable $E_{\mathbf{k}}$, $E_{\mathbf{h}-\mathbf{k}}$ pairs related to each $E_{\mathbf{h}}$.

The three linearly independent reflexions, 123, 2,5,$\overline{13}$ and 16$\bar{4}$ were given positive signs to specify the origin and a further reflexion, 1,1,$\overline{18}$, was assigned the symbol a. All four reflexions had large values for $|E|$ and had many relationships applicable to the Σ_2 formula (Hauptman & Karle, 1953):

$$sE_{\mathbf{h}} \sim s \sum_{\mathbf{k}} E_{\mathbf{k}} E_{\mathbf{h}-\mathbf{k}} \, .$$

The starting set of four phased reflexions was used in the symbolic addition procedure to determine the phases, in terms of signs and the symbol a, for the 72 reflexions with $|E_{\mathbf{h}}| > 2{\cdot}0$. From the many indications of the phase of symbol a found in the individual phase determinations, the symbol a was assigned the negative sign. This set of 72 phases was used to determine the signs of the remaining reflexions with $|E_{\mathbf{h}}| > 1{\cdot}5$. A three-dimensional E map was computed with the use of 202 E values as coefficients in a Fourier synthesis and is shown in Fig. 1. The map shows two molecules of DPP in the asymmetric unit, but with several atoms common to both molecules and represented by peaks of double weight in the E map. This unusual result is probably due to rational dependence of the atomic coordinates (Hauptman & Karle, 1959) causing a non-uniform distribution of $\langle |E_{\mathbf{h}}|^2 \rangle$ throughout reciprocal space. In the case of DPP, only the mean statistical average for all the normalized structure factors was computed and any need for renormalization was unnoticed.

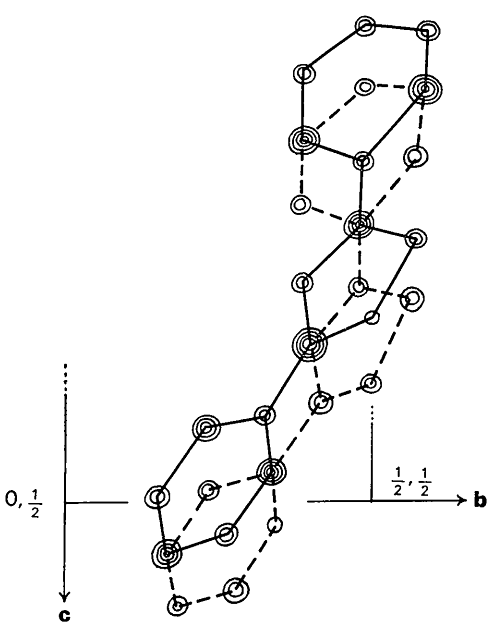

Fig. 1. Sections of the three-dimensional E map projected along [100]. Two molecular positions are indicated, one by a full line, the other by a dashed line. The final atomic coordinates correspond to the mean of the two positions.

Two structure-factor calculations, using the 574 strongest reflexions selected from the full three-dimensional film data and fractional atomic coordinates derived from the E map, gave R values of 0·43 for each of the two molecular positions. Fractional atomic coordinates were now calculated from the mean molecular position and a further structure factor calculation gave an agreement index of 0·27. In these calculations all non-hydrogen atoms were calculated as carbon atoms with isotropic temperature factors of $B = 4{\cdot}03$ Å^2 as determined by the Wilson plot. Nitrogen atoms were distinguished from carbon atoms in the pyrazoline ring by relative peak height comparison on a $0kl$ electron density map and one cycle of $0kl$ Fourier refinement reduced the reliability index R from 0·25 to 0·20.

Refinement

Refinement was continued by the method of least squares with block-diagonal approximation. One cycle of refinement of the scale factor, and the coordinates and individual isotropic temperature factors of the 17 non-hydrogen atoms, reduced R from 0·23 to 0·18 for the 1301 observed film data reflexions. All observations during this first cycle were given unit weights. Two further cycles using the weighting scheme suggested by Cruickshank, Pilling, Bujosa, Lovell & Truter (1961) improved the agreement to 0·16. Seven intense low-order reflexions, assumed to be affected by extinction, were given zero weight in the further refinement. The coordinates of the ten phenyl hydrogen atoms were calculated assuming them to lie on the ring diagonals with C–H equal to 1·05 Å; their contribution (as fixed atoms) to the total scattering was included, with an assumed isotropic B of 5·0 Å^2, in two cycles of anisotropic refinement of the heavy atoms. The agreement R was now 0·13 and the coordinates of the four hydrogen atoms in the pyrazoline ring were obtained from a three-dimensional difference Fourier synthesis. One further refinement cycle with fixed isotropic hydrogen atoms gave an agreement R of 0·120.

At this stage the diffractometer data became available and the final least-squares cycles were performed with this data. The starting parameters were those obtained in the final cycle of film data refinement. Three cycles of anisotropic refinement of the heavy atoms, with fixed isotropic (B=5·0 Å^2) hydrogen atoms, reduced R for the observed reflexions from 0·124 to 0·099. Two further cycles, allowing the hydrogen positional parameters to vary also, improved the agreement to 0·095 for the 1330 observations. The shift in any heavy atom parameter was now less than one quarter of its estimated standard deviation and each hydrogen parameter shift was less than one half of its e.s.d. The refinement was thus discontinued.

The weighting scheme used for the diffractometer data was that suggested by Cruickshank *et al.* (1961), and during the final cycles ten strong low-order reflexions, assumed to be affected by extinction, were given zero weight in the refinement. In all structure-factor and least-squares calculations, the atomic scattering factors for C, N and H were those given in *International Tables for X-ray Crystallography* (1962).

The final non-hydrogen positional parameters and anisotropic temperature factors are given in Tables 1 and 2 respectively, together with their estimated standard deviations. Hydrogen atomic coordinates are presented in Table 3 and a list of F_o and F_c values for the counter data is shown in Table 4.

Table 1. *Fractional atomic coordinates*

(e.s.d.'s × 10^4 in parentheses)

	x/a	y/b	z/c
N(1)	−0·0580 (7)	0·4078 (4)	0·3998 (2)
N(2)	0·0954 (7)	0·3884 (4)	0·3541 (2)
C(1)	−0·0243 (10)	0·1711 (5)	0·5592 (2)
C(2)	0·1482 (9)	0·1578 (5)	0·5147 (2)
C(3)	0·1433 (8)	0·2343 (5)	0·4629 (2)
C(4)	−0·0450 (8)	0·3274 (4)	0·4536 (3)
C(5)	−0·2218 (8)	0·3415 (5)	0·4980 (2)
C(6)	−0·2103 (9)	0·2616 (6)	0·5498 (2)
C(7)	0·0443 (8)	0·4748 (5)	0·3090 (2)
C(8)	−0·1520 (8)	0·5700 (5)	0·3248 (2)
C(9)	−0·2424 (8)	0·5138 (5)	0·3846 (2)
C(10)	0·1917 (8)	0·4814 (5)	0·2573 (2)
C(11)	0·3714 (8)	0·3848 (5)	0·2478 (2)
C(12)	0·5172 (9)	0·3946 (5)	0·1975 (2)
C(13)	0·4910 (10)	0·4981 (5)	0·1563 (2)
C(14)	0·3144 (10)	0·5919 (5)	0·1652 (2)
C(15)	0·1637 (9)	0·5855 (5)	0·2153 (2)

Table 3. *Fractional coordinates of the hydrogen atoms*

	x/a	y/b	z/c
H(1)	−0·020	0·119	0·596
H(2)	0·278	0·093	0·521
H(3)	0·272	0·228	0·431
H(4)	−0·355	0·408	0·489
H(5)	−0·344	0·260	0·578
H(6)	−0·289	0·577	0·286
H(7)	−0·082	0·667	0·332
H(8)	−0·427	0·487	0·378
H(9)	−0·260	0·584	0·422
H(10)	0·394	0·305	0·279
H(11)	0·626	0·316	0·193
H(12)	0·581	0·496	0·118
H(13)	0·309	0·658	0·136
H(14)	0·030	0·653	0·223

Assumed isotropic temperature factor B=5·0 Å^2 for all hydrogen atoms.

Average estimated standard deviations:

$\sigma(x/a)=0{\cdot}009\,,\quad \sigma(y/b)=0{\cdot}005\,,\quad \sigma(z/c)=0{\cdot}002\,.$

Discussion

The interatomic bond lengths and angles are shown in Fig. 2. Bond lengths involving hydrogen atoms are given in Table 5. The estimated standard deviations (σ) in atomic positions given in Table 1 and calculated from the inverse diagonal elements of the final least-squares matrix result in an average σ of 0·005 Å for the heavy atom positional parameters and average values of 0·007 Å and 0·5° for bond lengths and angles respectively involving these atoms. These values are probably too small because series-termination errors have not been included.

Both phenyl rings in the molecule have dimensions in agreement with literature values; the mean C–C and C–H bond lengths are 1·385 Å and 0·99 Å respectively and bond angles are not significantly different from 120°.

In the pyrazoline ring, the N(1)–C(9) bond length of 1·493 Å agrees well with the C–N distance of 1·498 Å in pyrazoline hydrochloride (Nardelli & Fava, 1962) but is longer, though not significantly, than the accurate value of 1·476 Å obtained by Becka & Cruick-

Table 2. *Anisotropic temperature factors and their estimated standard deviations* (*in parentheses*) × 10^4

	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
N(1)	468 (17)	139 (5)	37 (1)	−13 (4)	69 (7)	88 (15)
N(2)	447 (16)	125 (5)	31 (1)	0 (4)	91 (7)	84 (14)
C(1)	504 (21)	147 (6)	30 (1)	10 (5)	46 (8)	−27 (20)
C(2)	491 (21)	126 (6)	35 (1)	4 (5)	46 (9)	76 (18)
C(3)	409 (18)	127 (6)	31 (1)	−8 (5)	49 (8)	36 (17)
C(4)	368 (16)	101 (5)	25 (1)	−5 (4)	37 (7)	−32 (14)
C(5)	363 (17)	153 (6)	31 (1)	−2 (5)	66 (7)	5 (17)
C(6)	455 (20)	175 (7)	36 (1)	13 (5)	112 (9)	12 (20)
C(7)	402 (17)	109 (5)	27 (1)	20 (4)	83 (7)	90 (16)
C(8)	390 (17)	111 (5)	31 (1)	−10 (4)	41 (8)	66 (15)
C(9)	360 (17)	123 (6)	34 (1)	−9 (4)	14 (8)	72 (16)
C(10)	399 (17)	107 (5)	29 (1)	−11 (4)	13 (7)	−9 (15)
C(11)	436 (18)	107 (5)	30 (1)	−9 (4)	47 (8)	58 (16)
C(12)	482 (20)	133 (6)	30 (1)	−14 (4)	63 (8)	19 (18)
C(13)	551 (22)	147 (6)	32 (1)	−9 (5)	86 (9)	−65 (20)
C(14)	574 (23)	137 (6)	32 (1)	23 (5)	59 (9)	−31 (20)
C(15)	475 (20)	110 (5)	31 (1)	10 (4)	5 (8)	27 (17)

Table 4. *Observed and calculated structure factors* (×10)

Reflexions indicated by asterisks have intensity values less than the threshold value for an observed reflexion and were omitted from the least-squares and final R calculations.

L	FO	FC
H#	0, K#	0
2	129	-104
4	320	321
6	323	301
8	72	-55
10	176	-180
12	39	39
14	125	-124
16	55	60
18	51	51
20	117	133
22	19*	-8
H#	0, K#	1
1	307	326
2	166	164
3	127	-109
4	102	-98
5	320	-348
6	178	-176
7	83	90
8	75	66
9	164	165
10	171	171
11	109	105
12	59	-48
13	121	-119
14	71	75
15	28	-22
16	35	-35
17	83	88
18	92	-103
19	48	-48
20	42	-42
21	70	78
22	33	-32
23	18*	3
H#	0, K#	2
0	482	491
1	357	369
2	914	-1177
3	293	318
4	497	-532
5	409	422
6	122	113
7	105	-114
8	212	224
9	204	184
10	181	178
11	50	41
12	171	170
13	25	19
14	71	-71
15	47	-42
16	22	13
17	100	105
18	33	-28
19	100	-115
20	20*	-7
21	82	92
22	48	-56
H#	0, K#	3
1	90	86
2	287	305
3	146	-132
4	50	-31
5	97	82
6	176	-170
7	280	-265
8	77	-78
9	139	139
10	29	19
11	69	63
12	138	-140
13	137	-139
14	54	59
15	26	17
16	19*	6
17	65	66
18	57	-59
19	78	-86
20	47	35
21	32	-29
22	30	28
H#	0, K#	4
0	246	-255
1	54	-59
2	67	71
3	77	-70
4	116	-112
5	59	-58
6	120	-114
7	261	-252
8	90	-84
9	15*	-4
10	114	-112
11	38	39
12	104	-112
13	44	48
14	109	-117
15	22	-11
16	20*	8
17	55	60
18	20*	-3
19	87	-95
20	27	-30
21	18*	8
H#	0, K#	5
1	45	-56
2	14	-13
3	57	-50
4	29	-16
5	239	233
6	132	129
7	207	-195
8	67	-70
9	110	-109
10	62	55
11	18*	-15
12	56	50
13	85	-90
14	36	30
15	21	21
16	40	52
17	61	60
18	20*	11
19	19*	-7
20	18*	3
H#	0, K#	6
0	16*	-17
1	114	-110
2	165	166
3	105	-102
4	98	91
5	17*	12
6	32	19
7	26	16
8	114	121
9	81	-83
10	29	-29
11	19*	-5
12	30	25
13	22	-14
14	36	26
15	21	-14
16	78	94
17	20*	-4
18	19*	18
19	27	21
H#	0, K#	7
1	18*	-1
2	104	-104
3	18*	5
4	45	-35
5	51	42
6	132	134
7	31	-21
8	65	-53
9	58	64
10	20*	-15
11	45	-42
12	24	20
13	22	-22
14	64	-73
15	23	13
16	19*	-10
17	18*	2
H#	0, K#	8
0	115	114
1	72	68
2	66	-61
3	29	-27
4	21	-11
5	117	115
6	36	-26
7	150	154
8	81	88
9	26	18
10	55	-51
11	31	-18
12	20*	15
13	20*	-3
14	26	-26
15	18*	-14
H#	0, K#	9
1	20*	-4
2	44	-30
3	24	9
4	49	-46
5	89	-89
6	46	40
7	23	14
8	28	15
9	44	38
10	46	-38
11	19*	-10
12	18*	12
13	18*	15
H#	0, K#	10
0	69	70
1	94	91
2	109	-104
3	125	-132
4	64	-57
5	54	-52
6	35	-22
7	22	-19
8	28	16
9	19	-18
H#	1, K#	0
0	828	931
2	264	274
4	424	413
6	94	76
8	104	-80
10	49	-54
12	295	-285
14	147	-142
16	28	15
18	20*	6
20	71	-72
22	74	93
H#	1, K#	1
0	82	71
1	676	745
2	368	381
3	1043	-1272
4	122	-117
5	367	340
6	89	80
7	118	119
8	157	-159
9	55	50
10	185	193
11	72	-62
12	16*	5
13	19	-21
14	38	38
15	28	26
16	25	13
17	28	16
18	20*	-1
19	32	-38
20	30	-26
21	66	79
22	30	23
H#	1, K#	2
0	215	-219
1	116	126
2	750	-846
3	1111	1451
4	191	-204
5	46	-40
6	169	-182
7	153	153
8	41	-24
9	266	269
10	224	214
11	35	31
12	26	21
13	42	-39
14	38	34
15	51	55
16	104	103
17	35	-30
18	20*	-2
19	20*	2
20	55	-51
21	46	53
22	39	44
H#	1, K#	3
0	112	-107
1	58	48
2	277	305
3	245	-229
4	152	-143
5	270	267
6	75	66
7	39	38
8	238	-252
9	85	-81
10	174	176
11	28	-23
12	63	-62
13	42	39
14	19*	7
15	25	-15
16	36	-35
17	35	27
18	20*	-1
19	51	-57
20	30	-21
21	29	-24
H#	1, K#	4
0	115	-123
1	316	-321
2	109	-99
3	31	29
4	350	360
5	394	-416
6	254	266
7	178	-196
8	40	-30
9	26	-21
10	78	59
11	20	17
12	35	-28
13	33	28
14	52	-45
15	48	48
16	39	42
17	36	-28
18	54	-57
19	42	-43
20	19	-17
21	18*	1
H#	1, K#	5
0	96	-89
1	206	-218
2	23	15
3	15*	14
4	29	27
5	141	146
6	41	30
7	17*	-4
8	56	-57
9	96	-90
10	18*	-9
11	51	-57
12	31	24
13	48	47
14	41	39
15	31	-25
16	20*	-10
17	42	47
18	20*	9
19	18*	-7
20	18*	10
H#	1, K#	6
0	49	34
1	61	50
2	56	49
3	130	-129
4	40	-29
5	68	75
6	19	9
7	21	-6
8	156	-159
9	126	-128
10	102	-100
11	37	-32
12	33	-33
13	24	-12
14	43	37
15	44	-43
16	61	66
17	19*	4
18	18*	3
H#	1, K#	7
0	55	-59
1	31	20
2	31	-26
3	76	-77
4	23	4
5	80	76
H#	3, K#	0
0	222	-220
H#	1, K#	7
6	49	47
7	35	27
8	48	45
9	22	16
10	68	-68
11	77	-82
12	24	17
13	72	75
14	20*	1
15	19	-13
16	26	-26
17	18*	15
H#	1, K#	8
0	79	-79
1	96	96
2	70	65
3	59	-59
4	43	-35
5	187	197
6	78	80
7	134	138
8	80	82
9	20*	-4
10	20*	6
11	54	57
12	33	-16
13	27	22
14	19	14
15	18*	-17
H#	1, K#	9
0	85	-85
1	98	91
2	55	52
3	113	-115
4	64	-63
5	20*	11
6	49	45
7	36	23
8	20*	-2
9	27	18
10	34	-29
11	50	-54
12	18*	8
H#	1, K#	10
0	45	-42
1	19*	10
2	27	-20
3	63	-65
4	45	-40
5	46	-44
6	36	-35
7	28	-25
8	33	33
H#	2, K#	0
0	21	16
2	88	-85
4	244	-237
6	70	-71
8	92	-83
10	26	-12
12	213	-214
14	19*	-1
16	46	-34
18	57	-53
20	19*	0
H#	2, K#	1
0	98	-90
1	44	-50
2	24	-11
3	135	-123
4	268	247
5	46	37
6	323	-331
7	68	-65
8	56	60
9	23	-11
10	58	51
11	117	103
12	18*	-6
13	137	-129
14	19*	8
15	54	53
16	20*	-4
17	24	-9
18	21	7
19	33	-17
20	19*	8
21	18*	14
H#	2, K#	2
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-2	81	-68
-1	31	22
H#	3, K#	6
-15	18*	13
-14	19*	10
-13	44	41
-12	174	-186
-11	101	-105
-10	20*	-9
-9	69	65
-8	88	-85
-7	62	-66
-6	31	8
-5	37	28
-4	20*	1
-3	63	-65
-2	39	-43
-1	27	-16
H#	3, K#	7
-13	21	-8
-12	41	-38
-11	19*	2
-10	171	-182
-9	82	-82
-8	33	-14
-7	58	47
-6	44	-37
-5	46	53
-4	21	-10
-3	84	-98
-2	20*	-11
-1	44	-36
H#	3, K#	8
-10	34	28
-9	30	26
-8	19*	3
-7	43	-35
-6	36	26
-5	47	46
-4	27	23
-3	43	-31
-2	83	-90
-1	20*	1
H#	3, K#	9
-6	18*	-16
-5	31	27
-4	18*	-7
-3	42	-40
-2	22	-19
-1	23	-19
H#	4, K#	0
-16	52	-49
-14	35	30
-12	51	-54
-10	93	95
-8	79	-78
-6	28	18
-4	72	77
-2	85	88
H#	4, K#	1
-17	18*	0
-16	33	-27
-15	19*	12
-14	104	-120
-13	48	-46
-12	41	-41
-11	22	15
-10	20*	-2
-9	164	171
-8	20*	-8
-7	145	-143
-6	20	11
-5	151	141
-4	19*	13
-3	105	111
-2	115	104
-1	19	6
H#	4, K#	2
-17	27	22
-16	24	-9
-15	30	-15
-14	40	38
-13	43	-37
-12	40	-32
-11	54	-50
-10	46	43
-9	154	158
-8	34	-22
-7	214	-217
-6	65	-62
-5	64	-55
-4	39	-39
-3	39	-36
-2	73	-67
-1	46	-37
H#	4, K#	3
-16	56	59
-15	61	70
-14	72	-76
-13	39	-33
-12	37	28
-11	66	-67
-10	20*	2
-9	74	70
-8	52	-49
-7	157	-163
-6	88	-89
-5	113	-125
-4	137	-108
-3	164	-175
-2	53	38
-1	37	-27
H#	4, K#	4
-15	18*	-9
-14	18*	-2
-13	22	-11
-12	41	-36
-11	20*	-3
-10	20*	-1
-9	122	131
-8	103	111
-7	50	-36
-6	33	22
-5	48	49
-4	25	-11
-3	67	71
-2	84	-92
-1	27	16
H#	4, K#	5
-13	64	-68
-12	63	71
-11	69	-75
-10	70	73
-9	77	80
-8	101	114
-7	73	76
-6	43	44
-5	38	28
-4	37	37
-3	33	-28
-2	34	-37
-1	84	90
H#	4, K#	6
-11	18*	15
-10	25	-23
-9	45	-50
-8	19*	-3
-7	43	39
-6	36	26
-5	23	-21
-4	44	-36
-3	20*	-6
-2	43	-46
-1	39	-34
H#	4, K#	7
-8	27	-26
-7	43	53
-6	57	-72
-5	19*	14
-4	43	48
-3	19*	-2
-2	24	-29
-1	19*	11
H#	5, K#	0
-12	27	-5
-10	62	60
-8	42	19
-6	160	-164
-4	235	244
-2	47	33
H#	5, K#	1
-12	51	-49
-11	59	57
-10	42	-35
-9	43	39
-8	48	59
-7	73	-75
-6	23*	1
-5	105	110
-4	102	102
-3	35	26
-2	125	124
-1	67	58
H#	5, K#	2
-12	22	-18
-11	50	48
-10	50	51
-9	25	-10
-8	13*	7
-7	22	-3
-6	128	-133
-5	57	-49
-4	77	68
-3	20*	-4
-2	58	-52
-1	41	34
H#	5, K#	3
-10	34	20
-9	22	-14
-8	46	47
-7	66	-76
-6	76	-87
-5	70	-75
-4	45	-39
-3	55	-50
-2	26	-16
-1	35	-25
H#	5, K#	4
-9	29	-3
-8	25	4
-7	25	13
-6	19	-3
-5	22	-5
-4	33	-27
-3	22	3
-2	21	5
-1	26	16
H#	5, K#	5
-6	30	10
-5	29	-16
-4	23	-23
-3	24	22
-2	29	-15
-1	18*	-3

Table 5. *Bond lengths involving hydrogen atoms*

C(1)–H(1)	0·96 Å	C(9)—H(8)	1·04 Å
C(2)–H(2)	0·97	C(9)—H(9)	1·09
C(3)–H(3)	1·00	C(11)–H(10)	1·05
C(5)–H(4)	1·00	C(12)–H(11)	1·00
C(6)–H(5)	0·98	C(13)–H(12)	0·97
C(8)–H(6)	1·09	C(14)–H(13)	0·92
C(8)–H(7)	1·06	C(15)–H(14)	1·02

shank (1963). The N(2)–C(7) bond length is intermediate between values expected for a single and a double bond; from a C–N bond order curve drawn as described by Wheatley (1955), the amount of double bond character was determined as 39%. Although insufficient data are available to draw a similar N–N bond order curve, the N(1)–N(2) bond length is significantly shorter than the single bond distance ($\sim$1·48 Å) and must also have appreciable double bond character. Similarly, bonds C(4)–N(1) and C(7)–C(10), between the pyrazoline and phenyl rings, are both shorter than single-bond distances, suggesting that conjugation between the phenyl rings occurs *via* atoms N(1), N(2) and C(7). The bond angles within the pyrazoline ring agree well with those found in the 1-*p*-iodophenyl-3-phenylpyrazoline adduct of isocolumbin (Cheung, Melville, Overton, Robertson & Sim, 1966).

The equations of the least-squares planes calculated according to Blow (1960) through each ring in the molecule are given in Table 6 together with the atom deviations from the planes. Tests of χ^2 for atoms defining phenyl rings I and III give $\chi^2=7{\cdot}84$ and 1·98 respectively with three degrees of freedom and $p>0{\cdot}01$ in both cases. The phenyl rings are thus planar within experimental error. For the pyrazoline ring, $\chi^2=410{\cdot}0$, $p \leqslant 0{\cdot}01$ indicating that the ring is significantly aplanar.

Table 6. *Least-squares planes*

The equations of the planes are:

I $-0{\cdot}5504X-0{\cdot}6831Y-0{\cdot}4799Z+6{\cdot}5868=0$
II $-0{\cdot}6251X-0{\cdot}6289Y-0{\cdot}4623Z+6{\cdot}1085=0$
III $-0{\cdot}6444X-0{\cdot}5324Y-0{\cdot}5489Z+6{\cdot}1494=0$

where X, Y and Z are orthogonal coordinates in Å.
Atomic deviations (Å) from the planes (* identifies the atoms defining the plane).

	I	II	III
N(1)	0·011	−0·035*	−0·329
N(2)	0·136	−0·003*	−0·247
C(1)	−0·009*	−0·125	−0·950
C(2)	0·006*	−0·207	−0·981
C(3)	−0·003*	−0·196	−0·799
C(4)	0·002*	−0·068	−0·542
C(5)	−0·005*	0·025	−0·500
C(6)	0·008*	0·013	−0·687
C(7)	0·130	0·039*	−0·032
C(8)	−0·101	−0·053*	−0·040
C(9)	−0·028	0·052*	−0·091
C(10)	0·153	−0·019	−0·003*
C(11)	0·382	0·081	0·001*
C(12)	0·374	−0·004	0·003*
C(13)	0·137	−0·193	−0·005*
C(14)	−0·077	−0·279	0·002*
C(15)	−0·075	−0·197	0·001*

The molecule as a whole is non-planar; the sense and magnitude of the non-planarity can be seen from Table 6 using plane II as a reference. Both phenyl rings are bent downwards below plane II by approximately the same amount (0·1 Å) at the ring centres; in addition, rotations about the C(4)–N(1) and C(7)–C(10) bonds give the molecule a slight propeller shape. The dihedral angle between the planes of the phenyl rings is 11·0° and the angles between planes I and II, and planes II and III, are 5·4° and 7·5° respectively. The aplanarity of the molecule is probably due to both intra- and intermolecular close approaches. Atom C(9) deviates from plane II by +0·052 Å; to maintain an equal interaction of H(4) with H(8) and H(9) would involve a small rotation of phenyl ring I about the C(4)–N(1) bond in the direction indicated in Table 6. Similarly, C(8) lies 0·053 Å below plane II and the rotation of phenyl ring III, to prevent close hydrogen interactions, would be in a direction consistent with the observed aplanarity of the molecule.

All intermolecular distances less than 3·60 Å between heavy atoms and those less than 3·00 Å involving hydrogen atoms have been calculated and are given in Table 7. Some of these distances are shown in Fig. 3, the projection of the structure along [100]. None of

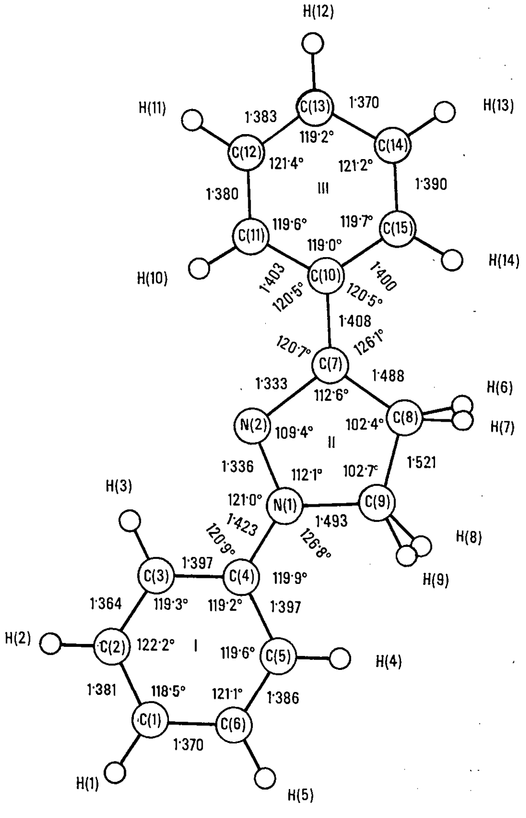

Fig. 2. Interatomic bond lengths and angles.

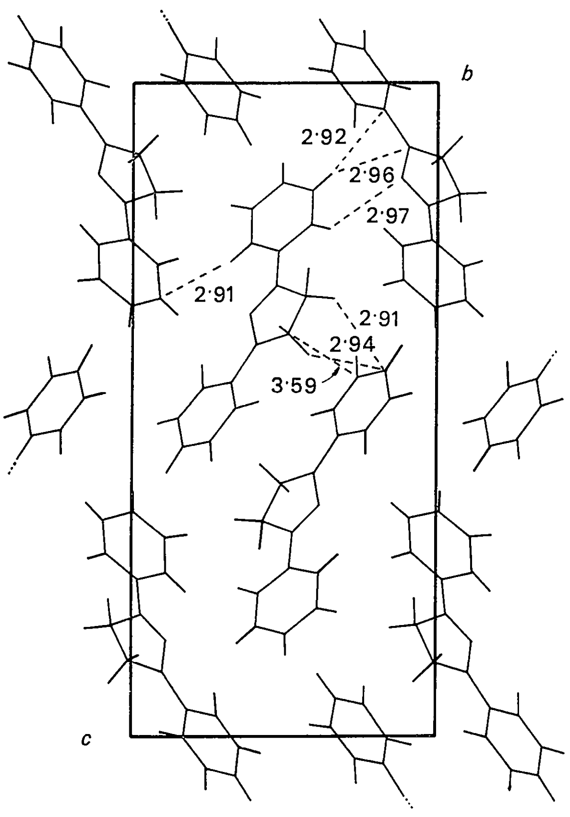

Fig. 3. Projection of the structure along [100].

the contacts is less than the normal van der Waals separation.

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Table 7. *Intermolecular distances*

All distances less than 3·60 Å between heavy atoms and less than 3·00 Å involving hydrogen atoms are included.

From	to	in molecule at	
H(7)	C(1)	$-x,\ 1-y,\ 1-z$	2·91 Å
H(9)	C(1)		2·94 Å
C(9)	C(6)		3·59
N(2)	H(8)	$1+x,\ y,\ z$	2·80
C(11)	C(8)		3·54
C(11)	H(6)		2·78
C(12)	H(6)		2·84
H(13)	C(4)	$1-x,\ \frac{1}{2}+y,\ \frac{1}{2}-z$	2·92
H(13)	N(1)		2·96
H(14)	N(2)		2·97
H(10)	C(14)	$1-x,\ y-\frac{1}{2},\ \frac{1}{2}-z$	2·91

References

Becka, L. N. & Cruickshank, D. W. J. (1963). *Proc. Roy. Soc.* A**273**, 435.
Blow, D. M. (1960). *Acta Cryst.* **13**, 168.
Buerger, M. J. (1959). *Vector Space and its Application in Crystal Structure Investigation*, p. 239. New York: John Wiley.
Cheung, K. K., Melville, D., Overton, K. H., Robertson, J. M. & Sim, G. A. (1966). *J. Chem. Soc.* (*B*), p. 853.
Cruickshank, D. W. J., Pilling, D. E., Bujosa, A., Lovell, F. M. & Truter, M. R. (1961). In *Computing Methods and the Phase Problem in X-ray Crystal Analysis.* Oxford: Pergamon Press.
Hauptman, H. & Karle, J. (1953). *Solution of the Phase Problem* I. *The Centrosymmetric Crystal.* ACA monograph No. 3. Pittsburgh: Polycrystal Book Service.
Hauptman, H. & Karle, J. (1959). *Acta Cryst.* **12**, 846.
International Tables for X-ray Crystallography (1962). Vol. III, p. 202. Birmingham: Kynoch Press.
Karle, I. L. & Karle, J. (1963). *Acta Cryst.* **16**, 969.
Karle, I. L. & Karle, J. (1964). *Acta Cryst.* **17**, 1356.
Nardelli, M. & Fava, G. (1962). *Acta Cryst.* **15**, 214.
Wheatley, P. J. (1955). *Acta Cryst.* **8**, 224.
Wilson, A. J. C. (1942). *Nature, Lond.* **150**, 151.