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The Crystal Structure of *p*-Chlorobenzene *anti*-Diazoimidoglyoxynitrile

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The crystal structure of *p*-benzene-*anti*-diazoimidoglyoxynitrile, $ClC_6H_4{\cdot}N_2{\cdot}C(NH)CN$, has been determined by three-dimensional X-ray methods. The crystals are monoclinic, space group $P2_1/c$, and the unit cell, containing four molecules, has the dimensions:

$a = 19.223$ Å; $b = 5.943$ Å; $c = 7.523$ Å; $\beta = 91.47°$

The intensity data were collected using integrating Weissenberg techniques. 1273 independent reflections were used for the structure determination. Least-squares refinement procedures resulted in a conventional R factor of 9 %.

The molecule is nearly planar; the diazo group is of the *anti* type with bond angles at the diazo nitrogen atoms of 113 and 111°. Hydrogen bonds are present between imide groups of neighbouring molecules.

A diazocyanide is formed when a solution of KCN is mixed with an acid solution of a diazonium salt. If a neutral solution of the diazonium salt is added to a concentrated solution of potassium cyanide another compound is formed, a compound first prepared by Gabriel [1] in 1879 and having the composition $Ar{\cdot}N_2{\cdot}CN_2HCN{\cdot}$ It was regarded as a "double cyanide", but Hantzsch and Schulze,[2] resp. Hantzsch and Danziger [3] claimed that the compound probably was an imidocyanide, $Ar{\cdot}N_2{\cdot}C(NH)CN$, of the diazocarboxylic acid because it may be reduced to a derivative of phenylhydrazine. Even in recent publications, however, the compound has been described as a true diazonium salt [4] and thus as the third of the isomers in the diazocyanide series. The present report deals with the crystal structure of the *p*-chlorobenzene diazo double cyanide, or, as it actually turned out, the *p*-chlorobenzene-*anti*-diazoimidonitrile.

EXPERIMENTAL

The compound was prepared by adding an aqueous solution of *p*-chlorobenzenediazonium chloride to a concentrated solution of potassium cyanide in a water-alcohol mixture at temperatures below −20°C. After stirring for about half an hour the com-

pound could be collected by filtration, and it was then purified by recrystallisation from absolute alcohol. (Found: C 50.11; H 2.65; N 29.51; Cl 17.99. Calc. for $C_8H_5N_4Cl$: C 49.88; H 2.62; N 29.09; Cl 18.41).

The unit cell dimensions were determined from Guinier films taken at room temperatures with $CuK\alpha_1$ radiation ($\lambda = 1.54050$ Å) and using powdered potassium chloride as a standard of calibration. The position of 13 well defined lines were used in a least-squares computation of the cell constants.

The crystals are of a brownish red colour and of prismatic form. The two specimens used in the X-ray experiments had the dimensions $0.08 \times 0.12 \times 0.20$ mm³ and $0.09 \times 0.08 \times 0.16$ mm³, respectively. They were mounted for rotation about the direction of the largest extensions, which turned out to be parallel to the b and c axes. The intensity data were collected using multifilm integrating equi-inclination Weissenberg techniques with (Ni-filtered) $CuK\alpha$-radiation. The ($hk0$)-zone and the layers ($h0l$) to ($h4l$) were recorded and the intensities of all but the weakest reflections were measured photometrically. The weakest reflections were estimated visually using a calibrated scale. Of 1273 independent reflections which might be expected to appear on these films 816 were actually observed. The Lorentz and polarization factors were applied to the intensity data, but no absorption correction was performed.

The full-matrix least-squares program employed in the parameter refinement procedure was a modified version of the program written by Gantzel, Sparks and Trueblood (IUCr World List No. 384). The program minimizes the function $\sum w^2(F_{obs} - G \cdot F_{calc})^2$; the weight applied to the structure factors was constant for $|F_{obs}|$ less than 6 and proportional to $|F_{obs}|^{-1/2}$ for larger structure factor values. Non-observed reflections were included in the calculations with a structure factor corresponding to the most probable value [5] and with the weight one third of that given to the observed reflections.

The atomic form factors used in the calculations were those derived by Hanson *et al.*[6]

CRYSTAL DATA

p-Chlorobenzene-*anti*-diazoimido glyoxynitrile $Cl \cdot C_6H_4 \cdot N_2 \cdot C(NH)CN$: Monoclinic, unit cell dimensions:

$a = 19.223$ (.006) Å; $b = 5.943$ (.002) Å; $c = 7.523$ (.001) Å; $\beta = 91.47$(.03)°

Figures in parenthesis are standard deviations.

$V = 859.2$ Å³, M = 192.62, $F(000) = 392$, $Z = 4$.

The density obtained by flotation is 1.48 g·cm^{-3}, the calculated density is 1.489 g·cm^{-3}.

Absent reflections: $h0l$ when $l = 2n + 1$
$0k0$ when $k = 2n + 1$

Space group: $P2_1/c$

STRUCTURE DETERMINATION

The structure determination was started by taking advantage of the known approximate geometry of the *p*-chlorophenyl part of the molecule. The orientation and position of this group could easily be ascertained from the sharpened Patterson projections along the b and c axes. Subsequent Fourier syntheses yielded valuable information regarding the structure of the rest of the molecule. After a couple of Fourier refinements in the two projections a three-dimensional least-squares refinement was carried out.

After four cycles with variation of both positional and (isotropic) thermal parameters of the 13 non-hydrogen atoms, the conventional R index was 0.16.

The individual layer line data were rescaled, three additional cycles including also the benzene hydrogen atoms lowered R to 0.15. A new series of five least-squares cycles in which all non-hydrogen atoms were assigned thermal parameters, led to an R value of 0.10.

The position of the remaining hydrogen atom belonging to the imide group was determined by inspection of a three-dimensional Fourier difference map. A final least-squares refinement of all parameters except the thermal parameters of the hydrogen atoms ($B=3.5$ Å^{-2}) resulted in an R value of 0.09. 1273 structure factors were included in these calculations, giving an over-determination ratio of 9.6.

Table 1. Final positional parameters and their standard deviations (in parenthesis). The values have been multiplied by 10^4.

	x	y	z
Cl	516 (1)	1948 (4)	1748 (3)
N(1)	2958 (3)	7943 (13)	1280 (10)
N(2)	3519 (3)	7248 (15)	1999 (10)
N(3)	3857 (3)	2485 (16)	0011 (11)
N(4)	4651 (3)	8280 (14)	2514 (10)
C(1)	2388 (3)	6378 (14)	1464 (9)
C(2)	2448 (3)	4283 (14)	2246 (10)
C(3)	1871 (3)	2951 (14)	2369 (10)
C(4)	1236 (3)	3703 (13)	1669 (10)
C(5)	1168 (3)	5787 (15)	0893 (10)
C(6)	1752 (3)	7164 (14)	0762 (10)
C(7)	4074 (3)	8845 (14)	1776 (11)
C(8)	3939 (3)	0856 (18)	0777 (13)
H(1)	2900 (47)	3540 (176)	2790 (125)
H(2)	1900 (49)	1440 (182)	2920 (122)
H(3)	0670 (44)	5890 (170)	0460 (112)
H(4)	1760 (55)	8610 (202)	0420 (151)
H(5)	4950 (57)	9520 (194)	2470 (140)

Table 2. Final thermal parameters for non-hydrogen atoms. The temperature factor is of the form $\exp -(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)$. The values have been multiplied by 10^5.

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cl	213	4045	2967	−459	−109	648
N(1)	191	3204	2332	21	−136	535
N(2)	205	3468	2283	180	−225	119
N(3)	301	3450	3244	− 81	−306	−325
N(4)	222	4824	3186	255	−294	404
C(1)	191	2503	2092	−175	− 70	−189
C(2)	172	2847	2445	− 11	−184	−252
C(3)	218	3555	2270	288	− 96	−817
C(4)	212	2186	2493	55	75	−485
C(5)	168	3365	2375	43	− 61	−949
C(6)	209	2914	2344	222	−141	−184
C(7)	188	2984	2706	−129	− 32	−975
C(8)	191	3078	3005	− 18	−138	−1550

Table 3. Observed and calculated structure factors. The columns are h, k, l, 10 $|F_{obs}|$, 10 F_{calc}. Asterisks indicate non-observed reflections.

1 0 0	119	116	3 4 0	148	139
2 0 0	105	-96	4 4 0	12	-18 *
3 0 0	43	41	5 4 0	35	56
4 0 0	336	-353	6 4 0	207	188
5 0 0	254	275	7 4 0	81	-85
6 0 0	320	-337	8 4 0	146	-126
7 0 0	264	-278	9 4 0	140	-128
8 0 0	49	-36	10 4 0	13	-26 *
9 0 0	487	-504	11 4 0	119	121
10 0 0	188	-195	12 4 0	28	-44
11 0 0	252	-257	13 4 0	42	-38
12 0 0	312	-321	14 4 0	13	21 *
13 0 0	89	-85	15 4 0	84	-79
14 0 0	342	-341	16 4 0	12	-19 *
15 0 0	73	59	17 4 0	52	-50
16 0 0	159	156	18 4 0	10	14 *
17 0 0	451	452	19 4 0	9	19 *
18 0 0	108	99	20 4 0	22	20
19 0 0	60	-52	1 5 0	20	-27
20 0 0	171	167	2 5 0	139	121
21 0 0	85	74	3 5 0	45	53
22 0 0	14	12	4 5 0	139	94
23 0 0	51	43	5 5 0	133	-111
24 0 0	16	24	6 5 0	61	-69
1 1 0	149	142	7 5 0	82	70
2 1 0	489	-544	8 5 0	20	-23
3 1 0	495	-541	9 5 0	35	-39
4 1 0	313	-323	10 5 0	13	5 *
5 1 0	307	-326	11 5 0	13	-0 *
6 1 0	690	-755	12 5 0	87	75
7 1 0	78	74	13 5 0	128	-95
8 1 0	372	-378	14 5 0	78	-71
9 1 0	9	-1 *	15 5 0	16	14
10 1 0	10	6 *	16 5 0	33	-22
11 1 0	11	-13 *	0 6 0	28	-43
12 1 0	208	206	1 6 0	59	50
13 1 0	211	194	2 6 0	59	37
14 1 0	258	247	3 6 0	13	-18 *
15 1 0	350	352	4 6 0	103	75
16 1 0	56	-55	5 6 0	54	-54
17 1 0	63	51	6 6 0	27	14
18 1 0	200	189	7 6 0	11	-21
19 1 0	73	-70	8 6 0	126	-84
20 1 0	75	68	9 6 0	11	5 *
21 1 0	62	-62	10 6 0	79	-46
22 1 0	76	-74	11 6 0	10	-8 *
23 1 0	87	-86	12 6 0	15	-19
24 1 0	62	-63	13 6 0	9	9 *
0 2 0	905	-956	14 6 0	41	42
1 2 0	82	-88	1 7 0	15	-6
2 2 0	67	-58	2 7 0	44	-23
3 2 0	239	-262	3 7 0	21	-8
4 2 0	59	56	4 7 0	42	-22
5 2 0	146	-131	5 7 0	31	-32
6 2 0	270	-256	6 7 0	47	-39
7 2 0	252	234	7 7 0	20	-21
8 2 0	420	390	-24 0 2	13	18
9 2 0	373	365	-23 0 2	43	53
10 2 0	188	175	-22 0 2	53	-58
11 2 0	60	-50	-21 0 2	15	15 *
12 2 0	232	229	-20 0 2	106	-108
13 2 0	78	68	-19 0 2	63	-59
14 2 0	28	30	-18 0 2	233	240
15 2 0	19	42	-17 0 2	278	-270
16 2 0	19	-21	-16 0 2	293	-283
17 2 0	143	-153	-15 0 2	293	-280
18 2 0	48	-53	-14 0 2	180	-172
19 2 0	28	-35	-13 0 2	48	51
20 2 0	63	-74	-12 0 2	122	-117
21 2 0	66	-88	-11 0 2	118	116
22 2 0	57	-68	-10 0 2	426	423
23 2 0	63	-73	-9 0 2	12	4 *
1 3 0	80	-90	-8 0 2	443	450
2 3 0	169	148	-7 0 2	192	201
3 3 0	287	272	-6 0 2	206	222
4 3 0	10	23 *	-5 0 2	340	349
5 3 0	215	181	-4 0 2	170	-150
6 3 0	222	186	-3 0 2	510	523
7 3 0	11	1 *	-2 0 2	163	154
8 3 0	119	96	-1 0 2	1008	1021
9 3 0	21	-32	0 0 2	762	-791
10 3 0	65	53	1 0 2	1204	-1232
11 3 0	53	59	2 0 2	311	-296
12 3 0	70	-63	3 0 2	404	-407
13 3 0	31	-33	4 0 2	772	-769
14 3 0	121	-110	5 0 2	72	-69
15 3 0	119	-104	6 0 2	200	-214
16 3 0	19	-28	7 0 2	274	269
17 3 0	62	-64	8 0 2	54	-50
18 3 0	100	-92	9 0 2	138	-130
19 3 0	18	15	10 0 2	317	337
20 3 0	10	16 *	11 0 2	58	52
21 3 0	28	-30	12 0 2	238	225
22 3 0	44	57	13 0 2	241	248
0 4 0	46	-33	14 0 2	213	210
1 4 0	12	-11 *	15 0 2	172	174
2 4 0	14	-7	16 0 2	254	261
17 0 2	129	-137	-8 0 8	14	26 *
18 0 2	50	-57	-7 0 8	15	25 *
19 0 2	26	-23	-6 0 8	15	13 *
20 0 2	135	-119	-5 0 8	15	31 *
21 0 2	148	-146	-4 0 8	103	72
22 0 2	67	-74	-3 0 8	22	-5
23 0 2	10	0 *	-2 0 8	44	-39
24 0 2	23	-43	-1 0 8	66	-48
-22 0 4	26	-42	0 0 8	31	-28
-21 0 4	12	-11 *	1 0 8	22	-25
-20 0 4	13	-25 *	2 0 8	74	-60
-19 0 4	90	79	3 0 8	21	-38
-18 0 4	71	-68	4 0 8	15	12 *
-17 0 4	69	-66	5 0 8	69	-61
-16 0 4	105	103	6 0 8	15	-3 *
-15 0 4	127	106	7 0 8	14	14 *
-14 0 4	120	112	8 0 8	13	-4 *
-13 0 4	173	158	9 0 8	13	20 *
-12 0 4	168	153	10 0 8	12	22 *
-11 0 4	323	303	11 0 8	43	36
-10 0 4	128	-110	12 0 8	26	36
-9 0 4	140	-122	13 0 8	82	74
-8 0 4	15	17 *	-24 1 1	16	26
-7 0 4	204	-192	-23 1 1	83	78
-6 0 4	130	-122	-22 1 1	27	36
-5 0 4	89	-86	-21 1 1	13	24 *
-4 0 4	268	-255	-20 1 1	119	116
-3 0 4	279	-260	-19 1 1	15	-20 *
-2 0 4	39	29	-18 1 1	27	-25
-1 0 4	442	-414	-17 1 1	162	-153
0 0 4	37	-41	-16 1 1	22	-27
1 0 4	35	-43	-15 1 1	121	111
2 0 4	65	62	-14 1 1	65	-59
3 0 4	34	37	-13 1 1	14	7 *
4 0 4	299	289	-12 1 1	148	-147
5 0 4	472	432	-11 1 1	23	10
6 0 4	336	330	-10 1 1	107	-99
7 0 4	68	-51	-9 1 1	420	-433
8 0 4	55	56	-8 1 1	10	31 *
9 0 4	165	162	-7 1 1	263	268
10 0 4	66	53	-6 1 1	95	93
11 0 4	16	18 *	-5 1 1	339	374
12 0 4	30	-39	-4 1 1	41	24
13 0 4	106	-101	-3 1 1	650	730
14 0 4	238	-228	-2 1 1	181	-192
15 0 4	17	26 *	-1 1 1	484	-523
16 0 4	158	-155	0 1 1	36	-28
17 0 4	99	-94	1 1 1	5	-19 *
18 0 4	63	-60	2 1 1	479	533
19 0 4	99	-106	3 1 1	169	-173
20 0 4	13	-12 *	4 1 1	28	-39
21 0 4	50	58	5 1 1	51	47
22 0 4	48	65	6 1 1	177	-182
-19 0 6	9	21 *	7 1 1	296	-291
-18 0 6	18	21	8 1 1	338	-357
-17 0 6	13	19 *	9 1 1	90	-73
-16 0 6	14	5 *	10 1 1	239	238
-15 0 6	15	-10 *	11 1 1	121	-123
-14 0 6	16	-10 *	12 1 1	140	142
-13 0 6	16	-2 *	13 1 1	130	126
-12 0 6	29	-31	14 1 1	99	91
-11 0 6	226	-190	15 1 1	15	-2 *
-10 0 6	119	-110	16 1 1	111	-101
-9 0 6	27	-24	17 1 1	92	85
-8 0 6	17	-25 *	18 1 1	71	62
-7 0 6	38	-28	19 1 1	40	39
-6 0 6	17	16 *	20 1 1	14	7 *
-5 0 6	17	-21 *	21 1 1	13	17 *
-4 0 6	17	-19 *	22 1 1	12	7 *
-3 0 6	274	226	23 1 1	20	-32
-2 0 6	120	91	24 1 1	75	-80
-1 0 6	116	102	-24 1 2	14	-14
0 0 6	74	71	-23 1 2	72	-68
1 0 6	40	40	-22 1 2	72	-81
2 0 6	100	90	-21 1 2	70	-69
3 0 6	100	89	-20 1 2	14	-24 *
4 0 6	17	7 *	-19 1 2	156	-155
5 0 6	23	11	-18 1 2	82	-79
6 0 6	248	-216	-17 1 2	16	21 *
7 0 6	86	-82	-16 1 2	41	42
8 0 6	17	-3 *	-15 1 2	57	52
9 0 6	109	-112	-14 1 2	178	182
10 0 6	75	-72	-13 1 2	169	163
11 0 6	86	-77	-12 1 2	132	115
12 0 6	90	-81	-11 1 2	306	319
13 0 6	38	-38	-10 1 2	174	166
14 0 6	128	123	-9 1 2	304	312
15 0 6	20	17	-8 1 2	86	-83
16 0 6	13	16 *	-7 1 2	91	-83
17 0 6	16	25	-6 1 2	58	56
18 0 6	27	35	-5 1 2	186	-193
-14 0 8	8	-7 *	-4 1 2	172	-186
-13 0 8	10	9 *	-3 1 2	131	129
-12 0 8	19	-32	-2 1 2	1242	-1461
-11 0 8	17	21	-1 1 2	477	-500
-10 0 8	53	46	0 1 2	139	134
-9 0 8	32	26	1 1 2	311	-332
2 1 2	314	307			
3 1 2	269	-275			
4 1 2	149	135			
5 1 2	150	152			
6 1 2	273	276			
7 1 2	442	470			
8 1 2	280	294			
9 1 2	12	23 *			
10 1 2	270	282			
11 1 2	96	85			
12 1 2	231	221			
13 1 2	11	6 *			
14 1 2	86	-79			
15 1 2	309	-327			
16 1 2	232	-230			
17 1 2	98	102			
18 1 2	158	-152			
19 1 2	90	-82			
20 1 2	82	-72			
21 1 2	76	-72			
22 1 2	11	-12 *			
23 1 2	9	20 *			
-23 1 3	8	10 *			
-22 1 3	30	-32			
-21 1 3	30	34			
-20 1 3	107	-99			
-19 1 3	103	-100			
-18 1 3	26	-35			
-17 1 3	15	2 *			
-16 1 3	87	81			
-15 1 3	39	-31			
-14 1 3	15	1 *			
-13 1 3	48	44			
-12 1 3	14	-12 *			
-11 1 3	24	19			
-10 1 3	83	-62			
-9 1 3	154	147			
-8 1 3	298	300			
-7 1 3	56	-57			
-6 1 3	174	171			
-5 1 3	18	-18			
-4 1 3	10	11 *			
-3 1 3	294	-303			
-2 1 3	373	-383			
-1 1 3	89	77			
0 1 3	9	8 *			
1 1 3	57	-60			
2 1 3	44	-44			
3 1 3	35	-28			
4 1 3	27	13			
5 1 3	46	-35			
6 1 3	134	-120			
7 1 3	104	84			
8 1 3	125	120			
9 1 3	228	225			
10 1 3	13	3 *			
11 1 3	101	92			
12 1 3	86	72			
13 1 3	15	-22 *			
14 1 3	100	-82			
15 1 3	39	-39			
16 1 3	15	3 *			
17 1 3	15	-33 *			
18 1 3	50	-40			
19 1 3	45	-26			
20 1 3	13	25 *			
21 1 3	46	-45			
22 1 3	9	-27 *			
23 1 3	16	-20			
-22 1 4	25	34			
-21 1 4	59	62			
-20 1 4	62	-61			
-19 1 4	69	68			
-18 1 4	139	135			
-17 1 4	116	114			
-16 1 4	97	90			
-15 1 4	32	-26			
-14 1 4	16	12 *			
-13 1 4	15	-29 *			
-12 1 4	110	-105			
-11 1 4	37	-26			
-10 1 4	150	-149			
-9 1 4	266	-266			
-8 1 4	102	-89			
-7 1 4	148	-139			
-6 1 4	23	18			
-5 1 4	140	-142			
-4 1 4	12	7 *			
-3 1 4	316	-305			
-2 1 4	94	99			
-1 1 4	624	647			
0 1 4	219	196			
1 1 4	190	183			
2 1 4	202	196			
3 1 4	104	93			
4 1 4	153	151			
5 1 4	12	5 *			

Table. 3. Continued.

6	1	4	48	-47
7	1	4	93	85
8	1	4	294	-295
9	1	4	37	-33
10	1	4	118	-101
11	1	4	166	-164
12	1	4	157	-138
13	1	4	148	-140
14	1	4	192	-190
15	1	4	50	37
16	1	4	158	158
17	1	4	14	-3 x
18	1	4	52	52
19	1	4	75	68
20	1	4	84	76
21	1	4	21	31
-21	1	5	13	-29
-20	1	5	9	-21 x
-19	1	5	46	48
-18	1	5	25	36
-17	1	5	13	28 x
-16	1	5	14	27 x
-15	1	5	15	13 x
-14	1	5	32	31
-13	1	5	15	-25 x
-12	1	5	16	-20 x
-11	1	5	15	7 x
-10	1	5	15	18 x
-9	1	5	56	41
-8	1	5	107	-85
-7	1	5	33	-33
-6	1	5	59	-58
-5	1	5	78	-67
-4	1	5	95	-89
-3	1	5	14	-19 x
-2	1	5	49	47
-1	1	5	28	39
0	1	5	13	9 x
1	1	5	106	85
2	1	5	113	97
3	1	5	14	-6 x
4	1	5	14	27 x
5	1	5	14	11 x
6	1	5	38	39
7	1	5	47	42
8	1	5	15	10 x
9	1	5	59	-57
10	1	5	15	-9 x
11	1	5	64	-63
12	1	5	15	-17 x
13	1	5	64	-59
14	1	5	15	-25 x
15	1	5	27	-10
16	1	5	36	-36
17	1	5	41	34
18	1	5	26	24
19	1	5	21	29
20	1	5	8	-1 x
-19	1	6	26	41
-18	1	6	24	-25
-17	1	6	67	-53
-16	1	6	76	-67
-15	1	6	52	-53
-14	1	6	46	-30
-13	1	6	106	-92
-12	1	6	15	2 x
-11	1	6	66	52
-10	1	6	15	-29 x
-9	1	6	106	83
-8	1	6	62	62
-7	1	6	56	53
-6	1	6	63	62
-5	1	6	88	75
-4	1	6	15	4 x
-3	1	6	159	141
-2	1	6	156	129
-1	1	6	32	-26
0	1	6	89	-63
1	1	6	101	-68
2	1	6	76	-70
3	1	6	166	-151
4	1	6	177	-167
5	1	6	29	-25
6	1	6	41	36
7	1	6	121	-107
8	1	6	16	-7 x
9	1	6	15	-15 x
10	1	6	15	16 x
11	1	6	15	28 x
12	1	6	88	85
13	1	6	42	35
14	1	6	89	84
15	1	6	109	106
16	1	6	11	-3 x
17	1	6	28	34
-16	1	7	9	-6 x
-15	1	7	10	-19 x
-14	1	7	33	-22 x
-13	1	7	12	-12 x
-12	1	7	13	-6 x
-11	1	7	14	-1 x
-10	1	7	14	-4 x
-9	1	7	15	-12 x
-8	1	7	15	3 x
-7	1	7	15	-2 x
-6	1	7	48	36
-5	1	7	15	7 x
-4	1	7	15	14 x
-3	1	7	15	15 x
-2	1	7	15	11 x
-1	1	7	15	37 x
0	1	7	15	21 x
1	1	7	15	1 x
2	1	7	15	-28 x
3	1	7	23	-24
4	1	7	15	-1 x
5	1	7	56	-42
6	1	7	15	-22 x
7	1	7	15	4 x
8	1	7	21	-33
9	1	7	14	15 x
10	1	7	14	-17 x
11	1	7	13	27 x
12	1	7	12	5 x
13	1	7	11	-23 x
14	1	7	10	20 x
15	1	7	9	12 x
-13	1	8	57	55
-12	1	8	53	59
-11	1	8	10	-2 x
-10	1	8	11	5 x
-9	1	8	12	-2 x
-8	1	8	12	-3 x
-7	1	8	13	-8 x
-6	1	8	13	-0 x
-5	1	8	36	-38
-4	1	8	14	-23 x
-3	1	8	10	-19 x
-2	1	8	61	-50
-1	1	8	14	-25 x
0	1	8	14	-21 x
1	1	8	51	-35
2	1	8	14	-15 x
3	1	8	14	-5 x
4	1	8	105	87
5	1	8	119	87
6	1	8	47	-39
7	1	8	40	26
8	1	8	31	29
9	1	8	11	22 x
10	1	8	32	34
11	1	8	9	13 x
-8	1	9	7	0 x
-7	1	9	8	14 x
-6	1	9	9	-7 x
-5	1	9	9	-21 x
-4	1	9	10	2 x
-3	1	9	10	0 x
-2	1	9	10	4 x
-1	1	9	10	-5 x
0	1	9	10	-16 x
1	1	9	10	2 x
2	1	9	10	-11 x
3	1	9	10	-9 x
4	1	9	9	-11 x
5	1	9	9	4 x
6	1	9	8	31 x
7	1	9	7	-2 x
-23	2	1	11	-0 x
-22	2	1	14	8 x
-21	2	1	16	-8 x
-20	2	1	33	-47 x
-19	2	1	19	-22 x
-18	2	1	136	-141
-17	2	1	75	-66
-16	2	1	104	-92
-15	2	1	64	-56
-14	2	1	80	-70
-13	2	1	34	37
-12	2	1	60	-52
-11	2	1	56	55
-10	2	1	189	172
-9	2	1	219	211
-8	2	1	97	88
-7	2	1	280	256
-6	2	1	292	254
-5	2	1	56	39
-4	2	1	89	83
-3	2	1	105	-115
-2	2	1	8	-1 x
-1	2	1	222	-233
0	2	1	91	85
1	2	1	476	-505
2	2	1	207	-209
3	2	1	425	-435
4	2	1	21	-33
5	2	1	193	-172
6	2	1	167	-145
7	2	1	369	319
8	2	1	125	110
9	2	1	84	64
10	2	1	267	232
11	2	1	168	153
12	2	1	95	81
13	2	1	19	35 x
14	2	1	19	-9 x
15	2	1	184	162
16	2	1	20	-17 x
17	2	1	37	53
18	2	1	156	-164
19	2	1	129	-107
20	2	1	33	-45
21	2	1	96	-84
22	2	1	30	-21
-22	2	2	13	23 x
-21	2	2	15	5 x
-20	2	2	61	70
-19	2	2	72	80
-18	2	2	19	2 x
-17	2	2	131	132
-16	2	2	137	132
-15	2	2	99	93
-14	2	2	56	66
-13	2	2	19	-8 x
-12	2	2	111	-96
-11	2	2	59	53
-10	2	2	50	-53
-9	2	2	16	19 x
-8	2	2	338	-309
-7	2	2	474	-454
-6	2	2	76	-57
-5	2	2	219	-201
-4	2	2	244	-222
-3	2	2	11	32 x
-2	2	2	99	101
-1	2	2	116	-100
0	2	2	244	227
1	2	2	413	404
2	2	2	251	239
3	2	2	81	76
4	2	2	293	290
5	2	2	12	5 x
6	2	2	284	263
7	2	2	231	200
8	2	2	109	-96
9	2	2	16	14 x
10	2	2	253	-240
11	2	2	100	-76
12	2	2	168	-140
13	2	2	296	-279
14	2	2	20	-17 x
15	2	2	20	6 x
16	2	2	207	-190
17	2	2	64	56
18	2	2	19	7 x
19	2	2	18	7 x
20	2	2	44	45
21	2	2	15	30 x
22	2	2	73	70
-22	2	3	21	-49
-21	2	3	14	-33 x
-20	2	3	16	11 x
-19	2	3	17	22 x
-18	2	3	49	49
-17	2	3	19	-3 x
-16	2	3	20	21 x
-15	2	3	20	28 x
-14	2	3	144	144
-13	2	3	48	59
-12	2	3	41	48
-11	2	3	138	147
-10	2	3	38	38
-9	2	3	115	-119
-8	2	3	43	-42
-7	2	3	144	-152
-6	2	3	138	-131
-5	2	3	276	-276
-4	2	3	184	-177
-3	2	3	210	183
-2	2	3	85	-90
-1	2	3	84	88
0	2	3	345	-327
1	2	3	13	33 x
2	2	3	119	107
3	2	3	179	181
4	2	3	203	193
5	2	3	127	129
6	2	3	304	310
7	2	3	163	155
8	2	3	173	-169
9	2	3	83	83
10	2	3	65	-66
11	2	3	171	-158
12	2	3	66	-74
13	2	3	128	-121
14	2	3	152	143
15	2	3	120	-112
16	2	3	102	-95
17	2	3	96	-105
18	2	3	34	-44
19	2	3	17	53 x
20	2	3	15	7 x
21	2	3	83	39
22	2	3	63	80
-20	2	4	14	35 x
-19	2	4	16	-14 x
-18	2	4	17	-20 x
-17	2	4	18	-27 x
-16	2	4	19	-54 x
-15	2	4	85	-108
-14	2	4	37	-62
-13	2	4	84	-98
-12	2	4	20	-18 x
-11	2	4	66	-72
-10	2	4	19	-23 x
-9	2	4	19	44 x
-8	2	4	18	-56 x
-7	2	4	110	107
-6	2	4	103	105
-5	2	4	31	-40
-4	2	4	332	360
-3	2	4	256	257
-2	2	4	59	-59
-1	2	4	185	178
0	2	4	16	-45 x
1	2	4	16	25 x
2	2	4	72	-71
3	2	4	144	-132
4	2	4	35	-44
5	2	4	190	-193
6	2	4	173	-172
7	2	4	18	-20 x
8	2	4	79	-74
9	2	4	86	-91
10	2	4	72	-78
11	2	4	71	-68
12	2	4	20	51 x
13	2	4	124	132
14	2	4	159	170
15	2	4	19	-34 x
16	2	4	57	73
17	2	4	79	94
18	2	4	16	9 x
19	2	4	39	42
20	2	4	12	23 x
-19	2	5	13	13 x
-18	2	5	39	-43
-17	2	5	16	2 x
-16	2	5	18	14 x
-15	2	5	19	5 x
-14	2	5	19	-14 x
-13	2	5	20	-52 x
-12	2	5	20	1 x
-11	2	5	20	-51 x
-10	2	5	157	-178
-9	2	5	20	-12 x
-8	2	5	67	-56
-7	2	5	19	-2 x
-6	2	5	19	11 x
-5	2	5	49	79
-4	2	5	277	296
-3	2	5	18	-15 x
-2	2	5	18	-14 x
-1	2	5	18	-21 x
0	2	5	58	62
1	2	5	114	114
2	2	5	18	21 x
3	2	5	18	0 x
4	2	5	19	44 x
5	2	5	19	-39 x
6	2	5	51	-74
7	2	5	232	-252
8	2	5	20	-39 x
9	2	5	20	-16 x
10	2	5	97	-105
11	2	5	20	40 x
12	2	5	20	28 x
13	2	5	105	118
14	2	5	19	-34 x
15	2	5	18	-39 x
16	2	5	17	49 x
17	2	5	15	50 x
18	2	5	13	28 x
-17	2	6	18	6 x
-16	2	6	14	-4 x
-15	2	6	16	9 x
-14	2	6	17	45 x
-13	2	6	18	17 x
-12	2	6	19	26 x
-11	2	6	81	98
-10	2	6	20	56 x
-9	2	6	20	11 x
-8	2	6	20	-17 x
-7	2	6	20	8 x
-6	2	6	20	12 x
-5	2	6	20	54 x
-4	2	6	20	17 x
-3	2	6	147	-173
-2	2	6	90	-79
-1	2	6	20	-44 x
0	2	6	80	-83
1	2	6	20	-28 x
2	2	6	20	-47 x
3	2	6	20	-34 x
4	2	6	20	20 x
5	2	6	20	-25 x
6	2	6	143	146
7	2	6	58	83
8	2	6	20	-61 x
9	2	6	20	65 x
10	2	6	19	39 x
11	2	6	41	66
12	2	6	73	94
13	2	6	17	-4 x
14	2	6	30	-49
15	2	6	15	-5 x
16	2	6	13	-26 x
-15	2	7	11	-0 x
-14	2	7	13	9 x
-13	2	7	15	-0 x
-12	2	7	16	-3 x
-11	2	7	34	-49
-10	2	7	56	51
-9	2	7	58	53
-8	2	7	19	31 x
-7	2	7	19	50 x
-6	2	7	19	37 x
-5	2	7	36	61
-4	2	7	52	-69
-3	2	7	82	-79
-2	2	7	20	3 x
-1	2	7	20	-16 x
0	2	7	20	-30 x
1	2	7	20	-30 x
2	2	7	20	-22 x
3	2	7	20	24 x
4	2	7	19	-54 x
5	2	7	19	-57 x
6	2	7	19	-45 x
7	2	7	19	56 x
8	2	7	89	70
9	2	7	17	11 x
10	2	7	17	47 x
11	2	7	16	44 x
12	2	7	15	32 x
13	2	7	13	-30 x
14	2	7	12	-14 x
-9	2	8	14	-18 x
-8	2	8	15	-13 x
-7	2	8	15	-6 x
-6	2	8	16	3 x
-5	2	8	16	-29 x
-4	2	8	17	-24 x
-3	2	8	17	1 x
-2	2	8	17	12 x
-1	2	8	17	26 x
0	2	8	17	10 x
1	2	8	17	35 x
2	2	8	17	34 x
3	2	8	17	21 x
4	2	8	16	19 x
5	2	8	30	34
6	2	8	15	4 x
7	2	8	47	-46
8	2	8	14	-1 x
-20	3	1	100	-126
-19	3	1	21	-9 x
-18	3	1	22	15 x
-17	3	1	39	73
-16	3	1	24	53 x
-15	3	1	60	28
-14	3	1	40	54
-13	3	1	108	107
-12	3	1	268	252
-11	3	1	83	78
-10	3	1	69	83
-9	3	1	140	130
-8	3	1	18	10 x
-7	3	1	172	-177
-6	3	1	29	28
-5	3	1	284	-290
-4	3	1	227	-239
-3	3	1	365	-415
-2	3	1	72	-79
-1	3	1	10	-12 x
0	3	1	55	-63
1	3	1	63	-58
2	3	1	179	-169
3	3	1	86	88
4	3	1	186	159
5	3	1	425	410
6	3	1	118	99
7	3	1	203	192
8	3	1	256	237
9	3	1	155	119
10	3	1	119	-81
11	3	1	110	76
12	3	1	131	-97
13	3	1	117	-94
14	3	1	132	-112
15	3	1	81	-77
16	3	1	40	-51
17	3	1	104	-109
18	3	1	41	-68
19	3	1	86	-94
-19	3	2	48	57
-18	3	2	22	6 x
-17	3	2	23	-43 x
-16	3	2	23	-17 x
-15	3	2	24	-15 x
-14	3	2	40	-51
-13	3	2	23	-49 x
-12	3	2	43	-48
-11	3	2	143	-115
-10	3	2	101	-100
-9	3	2	138	-129
-8	3	2	115	114
-7	3	2	134	134
-6	3	2	17	-42 x
-5	3	2	115	101
-4	3	2	29	18
-3	3	2	27	13
-2	3	2	253	281
-1	3	2	64	71
0	3	2	13	-22 x

Table. 3. Continued.

1	3	2	211	225		16	3	3	22	20 *		0	3	5	23	-41 *		-13	4	1	30	-34 *		12	4	2	60	-58	
2	3	2	51	38		17	3	3	80	77		1	3	5	23	-67 *		-12	4	1	29	69 *		-11	4	3	30	-21 *	
3	3	2	52	-37		18	3	3	20	49 *		2	3	5	67	-103		-11	4	1	28	-14 *		-10	4	3	29	2 *	
4	3	2	130	122		-17	3	4	20	2 *		3	3	5	23	-58 *		-10	4	1	217	-197		-9	4	3	29	64 *	
5	3	2	104	-85		-16	3	4	21	-6 *		4	3	5	96	-113		-9	4	1	170	-142		-8	4	3	57	-70	
6	3	2	227	-200		-15	3	4	22	-6 *		5	3	5	39	-57		-8	4	1	50	-42		-7	4	3	39	38	
7	3	2	268	-236		-14	3	4	23	44 *		6	3	5	24	-19 *		-7	4	1	294	-264		-6	4	3	231	254	
8	3	2	190	-169		-13	3	4	24	4 *		7	3	5	24	-66 *		-6	4	1	262	-230		-5	4	3	183	196	
9	3	2	165	164		-12	3	4	24	-7 *		8	3	5	24	-42 *		-5	4	1	20	-29 *		-4	4	3	124	135	
10	3	2	40	-42		-11	3	4	24	-17 *		9	3	5	130	162		-4	4	1	18	51 *		-3	4	3	25	-6 *	
11	3	2	120	-118		-10	3	4	23	58 *		10	3	5	59	90		-3	4	1	105	128		-2	4	3	24	69 *	
12	3	2	23	1 *		-9	3	4	228	254		11	3	5	39	69		-2	4	1	15	-13 *		-1	4	3	24	39 *	
13	3	2	99	-91		-8	3	4	23	55 *		12	3	5	22	-18 *		-1	4	1	121	168		0	4	3	24	-59 *	
14	3	2	24	22 *		-7	3	4	22	-39 *		13	3	5	22	46 *		0	4	1	97	107		1	4	3	24	-57 *	
15	3	2	24	25 *		-6	3	4	22	32 *		14	3	5	21	77 *		1	4	1	152	166		2	4	3	24	-35 *	
16	3	2	23	29 *		-5	3	4	42	-50		15	3	5	19	14 *		2	4	1	127	130		3	4	3	221	-224	
17	3	2	54	63		-4	3	4	21	-16 *		-13	3	6	19	-32 *		3	4	1	166	156		4	4	3	73	-74	
18	3	2	21	48 *		-3	3	4	20	-58 *		-12	3	6	20	24 *		4	4	1	99	82		5	4	3	75	-82	
19	3	2	109	86		-2	3	4	20	-30 *		-11	3	6	21	9 *		5	4	1	132	134		6	4	3	67	-67	
-18	3	3	59	57		-1	3	4	20	-40 *		-10	3	6	22	60 *		6	4	1	22	33 *		7	4	3	40	-54	
-17	3	3	22	18 *		0	3	4	34	-64		-9	3	6	23	-38 *		7	4	1	24	-53 *		8	4	3	110	-107	
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-15	3	3	23	-6 *		2	3	4	129	-121		-7	3	6	23	31 *		9	4	1	56	-58		10	4	3	29	-8 *	
-14	3	3	24	21 *		3	3	4	21	13 *		-6	3	6	83	-86		10	4	1	308	-259		11	4	3	161	185	
-13	3	3	20	22		4	3	4	77	-71		-5	3	6	57	-84		11	4	1	307	-263		-8	4	4	60	70	
-12	3	3	97	-120		5	3	4	215	-226		-4	3	6	24	17 *		12	4	1	29	33 *		-7	4	4	59	-75	
-11	3	3	106	-134		6	3	4	22	50 *		-3	3	6	24	-10 *		13	4	1	30	-55 *		-6	4	4	72	-76	
-10	3	3	60	-66		7	3	4	22	43 *		-2	3	6	106	90		-13	4	2	30	27 *		-5	4	4	58	48	
-9	3	3	131	-122		8	3	4	193	206		-1	3	6	24	-16 *		-12	4	2	29	78 *		-4	4	4	134	-117	
-8	3	3	229	-227		9	3	4	80	80		0	3	6	24	-14 *		-11	4	2	29	-29 *		-3	4	4	28	-28 *	
-7	3	3	20	-18 *		10	3	4	89	-113		1	3	6	24	62 *		-10	4	2	28	-42 *		-2	4	4	28	27 *	
-6	3	3	66	-75		11	3	4	151	166		2	3	6	24	-26 *		-9	4	2	27	-31 *		-1	4	4	27	31 *	
-5	3	3	32	63		12	3	4	24	-10 *		3	3	6	24	32 *		-8	4	2	93	67		0	4	4	56	75	
-4	3	3	18	-6 *		13	3	4	23	-45 *		4	3	6	24	-5 *		-7	4	2	223	222		1	4	4	28	-27 *	
-3	3	3	195	204		14	3	4	77	88		5	3	6	24	51 *		-6	4	2	24	-32 *		2	4	4	28	-53 *	
-2	3	3	169	183		15	3	4	22	-12 *		6	3	6	80	100		-5	4	2	23	-1 *		3	4	4	28	22 *	
-1	3	3	98	102		16	3	4	49	65		7	3	6	23	21 *		-4	4	2	31	46		4	4	4	28	13 *	
0	3	3	100	107		17	3	4	19	2 *		8	3	6	23	7 *		-3	4	2	82	-98		5	4	4	29	42 *	
1	3	3	103	118		-15	3	5	20	-26 *		9	3	6	22	-39 *		-2	4	2	20	-42 *		6	4	4	29	7 *	
2	3	3	146	148		-14	3	5	21	-23 *		10	3	6	22	11 *		-1	4	2	92	-107		7	4	4	29	13 *	
3	3	3	107	99		-13	3	5	22	-51 *		11	3	6	21	-40 *		0	4	2	19	65 *		8	4	4	30	43 *	
4	3	3	150	138		-12	3	5	23	-14 *		12	3	6	94	-111		1	4	2	19	-7 *		-5	4	5	30	-80 *	
5	3	3	140	-140		-11	3	5	23	39 *		-5	3	7	22	-31 *		2	4	2	20	17 *		-4	4	5	95	-80	
6	3	3	58	-60		-10	3	5	24	4 *		-4	3	7	22	-1 *		3	4	2	21	-5 *		-3	4	5	29	-59 *	
7	3	3	35	-48		-9	3	5	24	-4 *		-3	3	7	65	-36		4	4	2	85	-83		-2	4	5	60	-67	
8	3	3	178	-174		-8	3	5	100	127		-2	3	7	22	-25 *		5	4	2	23	61 *		-1	4	5	60	-96	
9	3	3	273	-282		-7	3	5	102	125		-1	3	7	22	-14 *		6	4	2	101	-102		0	4	5	60	-70	
10	3	3	60	-32		-6	3	5	106	126		0	3	7	22	-17 *		7	4	2	136	-131		1	4	5	29	23 *	
11	3	3	88	-59		-5	3	5	23	-19 *		1	3	7	22	-2 *		8	4	2	27	37 *		2	4	5	73	-91	
12	3	3	24	-14 *		-4	3	5	39	49		2	3	7	22	15 *		9	4	2	28	6 *		3	4	5	30	57 *	
13	3	3	98	-68		-3	3	5	23	22 *		3	3	7	22	22 *		10	4	2	129	129		4	4	5	95	93	
14	3	3	24	6 *		-2	3	5	23	-21 *		4	3	7	22	29 *		11	4	2	29	-10 *		5	4	5	30	25 *	
15	3	3	23	42 *		-1	3	5	23	-27 *		5	3	7	72	40													

A three-dimensional Fourier synthesis was calculated; a composite electron density map as viewed along the *b*-axis is shown in Fig. 1. A corresponding difference Fourier synthesis contained no electron density exceeding 0.5 e·Å^{-3}.

Final positional parameters and their standard deviations are listed in Table 1. The thermal parameters of the heavier atoms are listed in Table 2.

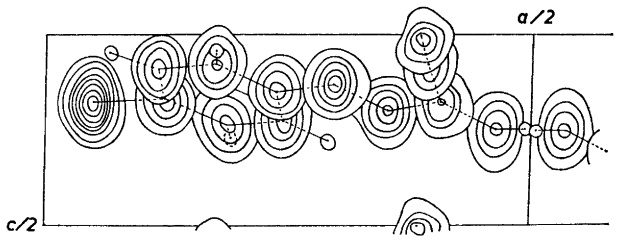

Fig. 1. Composite electron density map as viewed along the *b*-axis. Contour intervals are 1.5 e·Å^{-3}.

A comparison of observed and calculated structure factors is given in Table 3, the figures for non-observed reflections being those used in the least-squares calculations.

Table 4. Interatomic distances (Å) and bond angles (°). Standard deviations in parenthesis.

Cl—C(4)	1.735 (.007)	Cl—C(4)—C(3)	119.1 (.6)
C(1)—C(2)	1.381 (.011)	Cl—C(4)—C(5)	119.3 (.5)
C(2)—C(3)	1.368 (.010)	C(1)—C(2)—C(3)	119.5 (.5)
C(3)—C(4)	1.391 (.009)	C(2)—C(3)—C(4)	119.6 (.7)
C(4)—C(5)	1.374 (.012)	C(3)—C(4)—(5)	121.6 (.6)
C(5)—C(6)	1.395 (.010)	C(4)—C(5)—C(6)	119.4 (.6)
C(1)—C(6)	1.400 (.009)	C(5)—C(6)—C(1)	118.3 (.7)
C(1)—C(4)	2.734 (.009)	C(6)—C(1)—C(2)	121.7 (.6)
C(2)—C(5)	2.786 (.009)	N(1)—C(1)—C(2)	124.4 (.6)
C(3)—C(6)	2.787 (.012)	N(1)—C(1)—C(6)	113.9 (.7)
C(1)—N(1)	1.447 (.010)	C(1)—N(1)—N(2)	112.9 (.7)
N(1)—N(2)	1.264 (.010)	N(1)—N(2)—C(7)	111.2 (.8)
N(2)—C(7)	1.441 (.010)	N(2)—C(7)—N(4)	114.5 (.8)
C(7)—C(8)	1.432 (.013)	N(2)—C(7)—C(8)	119.1 (.6)
C(8)—N(3)	1.136 (.014)	N(4)—C(7)—C(8)	126.4 (.8)
C(7)—N(4)	1.273 (.009)	C(7)—C(8)—N(3)	177.2 (.8)
C(2)—H(1)	1.05 (.09)		
C(3)—H(2)	0.99 (.11)		
C(5)—H(3)	1.01 (.08)		
C(6)—H(4)	0.90 (.12)		
N(4)—H(5)	0.94 (.11)		

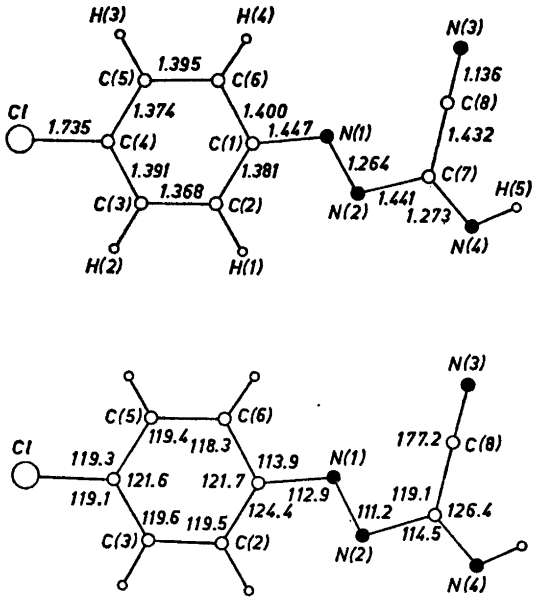

Fig. 2. Interatomic distances and bond angles.

DISCUSSION

The structure determination demonstrates that Gabriel's "double cyanide" is actually an imidoglyoxynitrile of the aryldiazo radical. The bond distances and interbond angles are given in Table 4 together with their standard deviations. Distances and angles are also shown in Fig. 2.

The molecule is essentially planar. A least-squares plane containing the Cl, C, and N atoms has the equation $-3.129\,x+1.811\,y+4.497\,z-1.0=0$, where x, y, and z are fractional coordinate values. The nitrile nitrogen atom, N(3), is situated 0.08 Å from this plane; all other atoms are closer to it. An analysis of the equations of various planes through groups of atoms indicates a small twist (3.5°) about the C(1)—N(1) bond relative to a planar molecule, and an additional twist (2°) about the N(2)—C(7) bond.

Standard deviations of the B_{ij} values given in Table 2 are 5—10 % for i=j and up to 50 % for i≠j. This comparatively large uncertainty and the fact that the intensity data have been collected mainly from one layer line set make a thermal vibrational analysis appear rather inconclusive. The mean of the atomic vibration seems, however, to increase markedly with the distance from the centre of gravity of the molecule, indicating a molecular libration about this point in addition to the lattice vibrations.

The geometry of the *p*-chlorobenzene-*anti*-diazo part of the molecule is in good agreement with the results from investigations of similar systems, *e.g.* *p*-chlorobenzene-*anti*-diazocyanide,[7] azobenzene,[8] and *p*-azotoluene.[9] A small deviation from hexagonal symmetry of the carbon arrangement in the benzene ring is indicated by an opening of the C(2)—C(1)—C(6) and C(3)—C(4)—C(5) angles, and by a significant shortening of the C(1)—C(4) diagonal (2.734 Å) relative to the two other diagonals (2.786 Å and 2.787 Å). The C—N distances in the diazo group are equal to 1.447 Å and 1.441 Å, as compared to 1.426 Å in the diazocyanide, 1.434 Å in azobenzene, and 1.433 Å in *p*-azotoluene; the N=N bond lengths is 1.264 Å, the corresponding values in the other compounds are 1.249 Å, 1.243 Å, and 1.244 Å, respectively. The non-linearity of C(4)—C(1)—N(1) is significant, and probably caused by repulsion between the N(2) and the C(2) and H(1) atoms.

The observed bond angles at the N(1) and N(2) atoms are 112.9° and 111.2°, respectively, which seem to be normal values for nitrogen bonded to *two* other atoms [8-11] with a double and a single bond.

The interbond angles at the C(7) atom may appear somewhat unexpected for an sp^2 hybridized carbon atom. A certain deviation from the usually observed values might, however, be caused by intermolecular interactions. Bond distances in the imidoglyoxynitrile group are, the experimental uncertainty being taken into account, those to be expected.

Chains of hydrogen bonded molecules are present in the crystal; the imide nitrogen atom acts both as donor and acceptor. The N—N distance is 3.261 Å (e.s.d. 0.011 Å), and the hydrogen atom is slightly displaced (0.2 Å) from the line connecting the two nitrogen atoms.

A rather short distance between the C(2) atom in one molecule and the N(3) atom of a neighbouring molecule, 3.53 Å, is observed. The C(2)—(H(1))—

N(3)′ direction is approximately normal to the nitrile triple bond (N(3)′—C(8)′).

No other intermolecular distances are shorter than those corresponding to van der Waals' contacts.

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