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The Crystal Structure of *ortho*-Bromobenzene-*anti*-diazocyanide

I. BØ, B. KLEWE and CHR. RØMMING

Department of Chemistry, University of Oslo, Oslo 3, Norway

The crystal structure of *o*-bromobenzene-diazocyanide, $Br \cdot C_6H_4 \cdot N_2 \cdot CN$, has been determined by X-ray methods, using 1203 reflections with intensities above background level collected by counter methods. The crystals are monoclinic, space group $P2_1/c$, with cell dimensions $a = 4.60_9$ Å; $b = 12.71_2$ Å, $c = 13.73_4$ Å; $\beta = 105.1_2°$; final conventional R factor 0.059. The molecule is essentially planar and the configuration is *trans* with respect to the N–N double bond; the C–N=N bond angles are 113.6° and 113.0°. The following bond lengths (corrected for thermal vibration) were found: C–Br, 1.914 Å; C(phenyl)–N, 1.403 Å; N=N, 1.276 Å; =N–C≡, 1.364 Å; C≡N, 1.142 Å; C–C, 1.380 Å. A short *inter*-molecular contact between the hydrogen atom in *ortho* position to the azo group and a nitrile nitrogen atom indicates the presence of a weak hydrogen bond.

The structure determination of *p*-chlorobenzene-*anti*-azocyanide[1] revealed a statistical disorder in the crystals resulting in an apparent mirror plane normal to the molecular plane. The molecular dimensions were accordingly measured with low accuracy, especially in the most interesting part – the diazocyanide group. It was assumed that a corresponding compound with the halogen in *ortho* position was less likely to exhibit disorder. A crystal structure determination of *o*-bromo-benzene-*anti*-diazocyanide was therefore carried out.

EXPERIMENTAL

o-Bromobenzenediazonium chloride was synthetized by the Knoevenagel method[2] using glacial acetic acid as solvent. The salt was precipitated with an excess of ether and the synthesis of the corresponding diazocyanide proceeded according to the method given by Le Fevre and Vine.[3] Recrystallization from acetone yielded dark orange needle-formed crystals suitable for the X-ray experiments. (Found: C 40.43; H 1.98; N 18.68; Br 38.91. Calc. for $C_7H_4N_3Br$: C 40.03; H 1.92; N 20.00; Br 38.05.)

Oscillation and Weissenberg photographs showed the crystals to have monoclinic symmetry. The systematic absences are $h0l$ with l odd and $0k0$ with k odd, which is consistent with the space group $P2_1/c$. The crystallographic a axis is parallel to the needle axis.

Unit cell parameters were determined from diffractometer measurements for 16 reflections on a Picker diffractometer, using Mo$K\alpha_1$, Mo$K\alpha_2$ and Mo$K\beta$ radiation (λ: 0.70926, 0.71354, and 0.63225 Å, respectively). The take-off angle was 0.5°. The computer program used in the least-squares treatment of these measurements as well as programs used during the subsequent calculations, are described in Ref. 4.

Three-dimensional intensity data were recorded on an automatic Picker diffractometer using Nb-filtered MoK-radiation and a take-off angle of 3°. A crystal with dimensions $0.20 \times 0.20 \times 0.24$ mm^3 was mounted with the crystallographic b axis along the diffractometer ϕ-axis. The $\omega - 2\theta$ scan technique with a 2θ scan speed of 1° min^{-1} was applied. Background counts were taken for 60 sec at each of the scan range limits. Reflections for which the count rate exceeded 10^4 cps were remeasured with reduced primary beam intensity. The intensities of three standard reflections were measured for every 50 reflections of the data set; during the data collection they showed a decrease to 85 % of the initial intensity, and the intensities of the reflections were accordingly adjusted. The estimated standard deviations were taken as the square root of the total counts with a 2 % addition for the uncertainty in rescaling.

Out of the 1667 unique reflections with $\sin\theta/\lambda < 0.64$, 1203 were measured with an intensity larger than twice the standard deviation. These were regarded as "observed" reflections, whereas the remaining reflections were excluded from the further calculations.

The intensity data were corrected for Lorentz, polarization, and absorption effects.

Atomic form factors used were those of Hanson *et al.*[5] for bromine, nitrogen and carbon and of Stewart *et al.*[6] for hydrogen.

CRYSTAL DATA

o-Bromobenzene-*anti*-diazocyanide, $Br{\cdot}C_6H_4{\cdot}N_2{\cdot}CN$, m.p. 109°C, monoclinic. $a = 4.609(0.001)$ Å; $b = 12.712(0.002)$ Å; $c = 13.734(0.003)$ Å; $\beta = 105.12°$ (0.01).

Figures in parentheses are estimated standard deviations. $V = 776.9$ Å^3; $M = 210.0$; $F(000) = 408$; $\mu = 5.54$ mm^{-1}; $Z = 4$. The density obtained by flotation is 1.76 g cm^{-3}, the calculated density is 1.795 g cm^{-3}.

Absent reflections: $h0l$ for l odd and $0k0$ for k odd; space group $P2_1/c$.

STRUCTURE DETERMINATION

The position of the bromine atom was determined from standard Patterson methods. A Fourier map based on signs given by the bromine position revealed all non-hydrogen atomic peaks; structure factor calculation with a common isotropic temperature factor yielded a conventional R-factor of 0.15. The refinement of the structure model proceeded by least-squares methods to an R-value of 0.127; anisotropic temperature factors were introduced, and further refinement brought R down to 0.067. Introduction of hydrogen atoms in fixed positions (C – H = 1.03 Å, B for H atoms 3.5 Å^2) gave $R = 0.062$.

The agreement between observed and calculated structure factors seemed to be poorer for the low angle data than for the rest of the reflections, the observed values being systematically higher than the calculated structure factors. This may be caused by too low background counts at the lower 2θ scan limit, which may happen at low scattering angles if the cut-off in intensity due to the β-filter falls within the scan range. Least-squares refinements omitting the 131 innermost reflections lowered the conventional R-factor to 0.059 as well as the weighted R-factor (to 0.050) and also the estimated standard deviations. There was no indication of secondary extinction.

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

h	k	l	F_o	F_c
-5	8	5	78	84
-5	7	3	82	- 81
-5	7	2	116	- 120
-5	7	1	64	- 57
-5	7	0	137	- 152
-5	6	0	83	85
-5	6	2	85	- 75
-5	6	4	145	- 142
-5	6	7	78	- 71
-5	6	8	99	103
-5	5	7	113	120
-5	5	3	106	- 98
-5	5	2	90	86
-5	4	0	85	65
-5	4	1	61	66
-5	4	2	101	- 111
-5	4	3	55	60
-5	4	4	137	- 139
-5	4	9	79	58
-5	4	10	69	- 54
-5	3	6	137	- 148
-5	3	4	126	- 116
-5	3	2	157	151
-5	3	0	141	144
-5	2	1	127	128
-5	2	2	78	74
-5	2	3	98	- 77
-5	2	4	158	153
-5	2	5	186	- 191
-5	2	9	94	94
-5	1	12	86	- 50
-5	1	7	86	- 74
-5	1	6	89	- 93
-5	1	3	69	75
-5	1	2	89	87
-5	0	0	197	- 198
-5	0	2	94	82
-5	0	4	69	84
-5	0	6	139	- 134
-5	0	8	82	- 102
-5	0	12	70	39
-4	0	12	61	- 85
-4	0	8	259	269
-4	0	6	122	115
-4	0	4	260	- 267
-4	0	2	141	- 134
-4	0	0	198	189
-4	1	0	78	- 78
-4	1	2	144	- 147
-4	1	3	327	- 328
-4	1	4	56	51
-4	1	5	121	- 129
-4	1	6	117	107
-4	1	7	155	159
-4	1	9	87	91
-4	1	11	107	- 112
-4	2	15	75	72
-4	2	14	74	- 67
-4	2	13	122	134
-4	2	12	75	- 50
-4	2	9	156	- 163
-4	2	7	82	82
-4	2	5	223	230
-4	2	4	102	- 101
-4	2	3	56	17
-4	2	2	66	- 46
-4	2	1	258	- 259
-4	3	0	94	- 98
-4	3	2	170	- 183
-4	3	3	191	193
-4	3	4	147	144
-4	3	5	74	59
-4	3	6	256	247
-4	3	7	75	- 76
-4	3	8	77	- 77
-4	3	9	56	24
-4	3	10	153	- 176
-4	4	12	89	107
-4	4	9	98	- 84
-4	4	8	144	- 137
-4	4	7	96	104
-4	4	6	92	- 91
-4	4	5	133	132
-4	4	4	133	134
-4	4	3	194	- 206
-4	4	2	211	210
-4	4	1	371	- 369
-4	5	1	71	- 82
-4	5	2	63	- 67
-4	5	3	196	202
-4	5	5	65	61
-4	5	7	150	- 149
-4	5	9	93	- 99
-4	5	10	65	- 47
-4	5	11	77	76
-4	6	9	74	89
-4	6	8	144	- 152
-4	6	6	97	- 82
-4	6	5	86	- 96
-4	6	4	154	157
-4	6	3	119	110
-4	6	1	185	195
-4	6	0	214	- 213
-4	7	0	164	183
-4	7	2	72	72
-4	7	3	149	148
-4	7	4	104	- 109
-4	7	5	58	70
-4	7	6	106	- 105
-4	7	7	80	- 78
-4	7	8	70	81
-4	7	10	144	179
-4	7	11	77	105
-4	8	11	80	75
-4	8	9	100	87
-4	8	7	87	- 89
-4	8	6	123	127
-4	8	5	180	- 186
-4	8	4	71	58
-4	8	2	88	- 88
-4	8	1	241	238
-4	8	0	59	- 24
-4	9	2	126	123
-4	9	3	132	- 145
-4	9	6	138	- 143
-4	9	7	98	101
-4	9	11	100	- 111
-4	10	8	80	76
-4	10	7	78	- 71
-4	10	4	86	- 94
-4	10	2	68	- 66
-4	10	1	61	33
-4	10	0	107	110
-4	11	0	81	- 84
-4	11	1	69	48
-4	11	3	126	- 129
-4	11	4	84	95
-4	11	7	121	109
-3	13	6	85	- 97
-3	13	2	133	137
-3	13	1	87	- 86
-3	13	0	70	85
-3	12	6	112	- 99
-3	12	1	100	106
-3	12	4	120	103
-3	12	5	122	- 111
-3	12	9	85	104
-3	11	9	117	- 106
-3	11	8	95	69
-3	11	7	103	- 100
-3	11	5	138	128
-3	11	4	89	- 92
-3	11	3	153	152
-3	11	1	96	- 87
-3	11	0	58	72
-3	10	0	247	- 235
-3	10	2	189	197
-3	10	3	102	- 104
-3	10	4	293	280
-3	10	7	80	67
-3	10	8	163	- 154
-3	10	12	73	73
-3	9	10	107	- 118
-3	9	7	124	- 131
-3	9	6	218	213
-3	9	4	51	46
-3	9	3	188	174
-3	9	2	211	- 206
-3	9	1	79	76
-3	9	0	141	- 134
-3	8	0	91	- 86
-3	8	1	257	- 252
-3	8	3	183	- 174
-3	8	4	132	- 127
-3	8	5	257	240
-3	8	6	165	- 165
-3	8	7	167	169
-3	8	9	147	- 153
-3	8	11	126	- 129
-3	7	14	95	96
-3	7	13	121	- 122
-3	7	12	77	65
-3	7	10	150	- 156
-3	7	9	72	67
-3	7	8	167	- 176
-3	7	7	59	- 63
-3	7	6	145	137
-3	7	5	134	- 134
-3	7	4	116	121
-3	7	3	136	- 135
-3	7	2	294	- 293
-3	7	1	55	46
-3	7	0	130	- 133
-3	6	8	423	418
-3	6	1	191	- 192
-3	6	2	75	- 70
-3	6	3	54	37
-3	6	4	308	- 301
-3	6	5	197	196
-3	6	7	114	118
-3	6	8	297	309
-3	6	9	67	- 49
-3	6	11	59	- 66
-3	6	12	76	- 97
-3	5	15	83	82
-3	5	11	61	- 38
-3	5	9	289	307
-3	5	8	68	67
-3	5	7	408	418
-3	5	6	53	- 55
-3	5	5	186	- 177
-3	5	4	68	59
-3	5	3	437	- 426
-3	5	2	41	51
-3	5	1	213	206
-3	5	0	75	- 85
-3	4	0	323	321
-3	4	1	377	368
-3	4	2	153	- 148
-3	4	3	104	103
-3	4	4	271	- 273
-3	4	5	186	- 179
-3	4	6	122	120
-3	4	7	226	- 230
-3	4	8	127	121
-3	4	10	170	- 169
-3	4	11	203	210
-3	4	12	172	- 171
-3	4	15	76	- 87
-3	3	14	107	- 116
-3	3	13	92	- 91
-3	3	11	98	- 98
-3	3	10	223	235
-3	3	8	105	99
-3	3	7	57	- 35
-3	3	6	364	- 353
-3	3	5	70	- 55
-3	3	4	71	- 64
-3	3	3	90	- 98
-3	3	2	422	418
-3	3	1	133	- 129
-3	3	0	215	213
-3	2	0	205	- 211
-3	2	1	440	438
-3	2	3	163	172
-3	2	4	217	216
-3	2	5	455	- 426
-3	2	6	48	17
-3	2	7	225	- 220
-3	2	8	115	- 129
-3	2	9	189	190
-3	2	10	73	51
-3	2	11	143	160
-3	2	12	69	80
-3	2	13	81	- 81
-3	2	15	71	- 71
-3	1	13	167	174
-3	1	11	93	88
-3	1	10	132	142
-3	1	9	201	- 216
-3	1	8	63	- 30
-3	1	7	162	- 155
-3	1	6	379	- 366
-3	1	5	192	186
-3	1	4	104	- 91
-3	1	3	434	425
-3	1	2	448	440
-3	1	1	70	- 71
-3	1	0	395	381
-3	0	0	415	- 415
-3	0	2	166	143
-3	0	4	449	455
-3	0	6	156	- 151
-3	0	8	327	- 339
-3	0	10	115	103
-3	0	12	176	171
-3	0	16	89	- 73
-2	0	16	86	92
-2	0	12	199	- 200
-2	0	10	145	160
-2	0	8	609	605
-2	0	6	121	- 107
-2	0	4	856	- 781
-2	0	2	294	- 256
-2	0	0	843	819
-2	1	0	326	- 306
-2	1	1	498	473
-2	1	2	366	- 343
-2	1	3	802	- 735
-2	1	4	52	41
-2	1	5	393	- 364
-2	1	6	477	452
-2	1	7	358	345
-2	1	9	163	164
-2	1	10	373	- 385
-2	1	11	141	- 134
-2	1	12	96	- 101
-2	1	13	170	- 162
-2	1	14	108	109
-2	2	16	75	29
-2	2	15	124	127
-2	2	13	56	67
-2	2	12	127	- 127
-2	2	11	205	- 204
-2	2	10	50	52
-2	2	9	251	- 247
-2	2	8	181	177
-2	2	7	46	24
-2	2	6	317	- 305
-2	2	5	281	263
-2	2	4	362	- 327
-2	2	3	317	- 302
-2	2	1	628	- 626
-2	2	0	179	190
-2	3	0	333	- 308
-2	3	1	113	- 97
-2	3	2	801	- 766
-2	3	3	391	354
-2	3	5	405	375
-2	3	6	737	713
-2	3	7	71	- 72
-2	3	8	173	174
-2	3	9	47	- 11
-2	3	10	320	- 326
-2	3	11	141	130
-2	3	12	57	- 59
-2	3	14	140	133
-2	4	16	71	- 69
-2	4	15	96	90
-2	4	12	158	156
-2	4	11	138	- 156
-2	4	9	174	- 184
-2	4	8	248	- 248
-2	4	7	291	298
-2	4	6	233	215
-2	4	5	674	644
-2	4	4	701	625
-2	4	3	275	- 258
-2	4	2	241	231
-2	4	1	425	- 405
-2	4	0	447	- 431
-2	5	1	250	- 240
-2	5	3	339	327
-2	5	4	204	- 195
-2	5	5	61	- 40
-2	5	7	416	- 405
-2	5	8	173	177
-2	5	9	279	- 271
-2	5	11	93	83
-2	5	12	64	- 22
-2	5	13	161	176
-2	6	16	88	- 76
-2	6	13	102	- 111
-2	6	12	172	168
-2	6	11	60	55
-2	6	10	111	- 118
-2	6	9	247	251
-2	6	8	476	- 463
-2	6	7	76	- 89
-2	6	5	288	- 274
-2	6	4	556	514
-2	6	1	84	73
-2	6	0	618	- 598
-2	7	0	191	189
-2	7	1	270	- 257
-2	7	2	511	480
-2	7	3	274	260
-2	7	4	91	- 74
-2	7	5	245	236
-2	7	6	416	- 389
-2	7	8	115	- 110
-2	7	10	129	128
-2	7	11	58	59
-2	7	14	114	- 116
-2	8	14	78	66
-2	8	13	64	- 25
-2	8	11	166	172
-2	8	9	109	116
-2	8	7	259	- 244
-2	8	6	88	95
-2	8	5	308	- 291
-2	8	4	114	- 98
-2	8	3	260	242
-2	8	2	47	- 54
-2	8	1	481	470
-2	8	0	119	117
-2	9	0	66	63
-2	9	1	68	54
-2	9	2	327	316
-2	9	3	165	- 157
-2	9	4	73	53
-2	9	5	63	- 55

Table 1. Continued.

h	k	l	F_o	F_c
-2	9	6	285	- 275
-2	9	7	145	145
-2	9	8	88	- 68
-2	9	10	188	204
-2	9	11	148	- 145
-2	9	12	85	85
-2	9	13	66	- 46
-2	10	12	133	- 147
-2	10	8	273	260
-2	10	6	76	81
-2	10	4	370	- 334
-2	10	3	159	144
-2	10	2	137	- 149
-2	10	1	139	125
-2	10	0	285	268
-2	11	0	86	- 82
-2	11	1	159	146
-2	11	2	74	- 72
-2	11	3	183	- 177
-2	11	4	91	100
-2	11	5	157	- 159
-2	11	6	86	74
-2	11	7	123	128
-2	11	9	146	133
-2	12	9	85	- 100
-2	12	8	66	54
-2	12	6	124	- 122
-2	12	5	93	96
-2	12	4	175	- 160
-2	12	3	117	- 101
-2	12	1	255	- 257
-2	12	0	178	169
-2	13	0	68	- 68
-2	13	2	272	- 259
-2	13	3	71	- 45
-2	13	5	83	- 50
-2	13	6	181	187
-2	14	7	75	94
-2	14	5	91	94
-2	14	4	108	94
-2	14	3	76	- 43
-2	14	2	95	94
-2	14	1	85	- 59
-2	15	0	60	42
-2	15	3	106	119
-1	15	6	83	- 76
-1	15	5	81	- 83
-1	15	3	111	- 101
-1	15	2	96	67
-1	15	1	75	70
-1	14	0	58	85
-1	14	1	129	111
-1	14	3	158	133
-1	14	4	115	- 108
-1	14	5	64	- 59
-1	14	7	110	- 110
-1	14	8	80	78
-1	13	10	100	119
-1	13	9	64	- 25
-1	13	6	170	- 175
-1	13	2	252	227
-1	13	0	75	80
-1	12	0	198	- 186
-1	12	1	274	266
-1	12	2	65	16
-1	12	3	184	168
-1	12	4	203	200
-1	12	5	118	- 117
-1	12	6	78	82
-1	12	7	151	- 150
-1	12	8	84	- 81
-1	12	9	86	95
-1	12	10	79	- 63
-1	12	11	124	119
-1	11	10	73	67
-1	11	9	204	- 208
-1	11	8	100	104
-1	11	7	154	- 159
-1	11	6	59	- 65
-1	11	5	221	218
-1	11	4	57	- 52
-1	11	3	255	238
-1	11	2	54	61
-1	11	1	322	- 291
-1	11	0	67	- 76
-1	10	0	358	- 352
-1	10	2	64	- 41
-1	10	3	230	- 231
-1	10	4	290	274
-1	10	7	120	127
-1	10	8	244	- 257
-1	10	9	63	6
-1	10	10	59	- 40
-1	10	11	93	- 90
-1	10	12	136	124
-1	9	13	79	79
-1	9	12	78	- 86
-1	9	11	58	76
-1	9	10	137	- 159
-1	9	9	131	- 126
-1	9	6	151	153
-1	9	5	319	312
-1	9	4	56	- 40
-1	9	3	325	292
-1	9	2	382	- 347
-1	9	1	140	- 128

h	k	l	F_o	F_c
-1	9	0	48	- 48
-1	8	1	582	- 550
-1	8	2	127	120
-1	8	3	463	- 420
-1	8	4	158	161
-1	8	5	418	426
-1	8	6	133	- 128
-1	8	7	371	388
-1	8	8	78	- 69
-1	8	9	188	- 199
-1	8	10	57	41
-1	8	11	243	- 243
-1	8	15	115	103
-1	7	15	67	- 33
-1	7	14	105	91
-1	7	13	85	- 100
-1	7	12	92	- 59
-1	7	11	55	42
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Table 1. Continued.

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5	1	1	120	- 124
5	0	2	139	- 182

Table 2. Fractional atomic coordinates and thermal parameters with estimated standard deviations ($\times 10^5$). The temperature factor is given by $\exp-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)$. The numbering of atoms is given in Fig. 1.

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Br	47206	40491	61618	5612	608	634	923	89	−176
	14	5	5	44	5	5	23	17	8
N1	73730	19814	57074	3642	452	561	229	887	189
	98	37	36	240	32	32	146	144	51
N2	85429	10993	55999	4826	683	706	404	989	146
	117	46	42	285	42	37	186	174	67
N3	132057	6167	69156	6449	750	770	1181	978	255
	151	48	51	368	42	41	201	214	67
C1	48082	22158	49341	2947	569	517	−654	798	71
	120	49	44	260	38	37	172	163	63
C2	36763	16156	40822	4583	647	611	−414	884	−140
	139	53	50	353	44	43	196	208	72
C3	11895	19013	33649	4984	894	508	−1029	526	−112
	155	62	51	369	59	40	248	201	78
C4	−2662	28307	34656	3813	943	596	−420	317	483
	140	67	55	322	59	44	241	192	90
C5	8149	34565	42885	4136	742	594	223	471	116
	142	56	53	332	49	43	208	194	82
C6	33425	31501	50242	3239	404	580	−219	563	91
	118	43	45	282	37	38	158	170	59
C7	110857	8950	63413	4461	539	652	623	1251	256
	145	53	51	329	39	41	218	204	77

A comparison of observed and calculated structure factors is given in Table 1; the final parameters are listed in Table 2. A difference Fourier map showed no electron densities exceeding 0.2 e Å^{-3}.

Magnitudes and directions of the principal axes of the ellipsoids of vibration are given in Table 3. The r.m.s. discrepancy between the atomic vibration tensor components arrived at in the structure analysis and those calculated from the rigid-body parameters obtained by analysis of the librational, translational and screw motion of the molecule, is 0.0042 Å^2. The corresponding value when omitting the atoms of the $=N-C\equiv N$ group is 0.0031 Å^2, indicating that the benzene ring and the atoms directly attached to it may be regarded as a rigid body whereas the "tail" of the molecule does not fit well into this approximation. The L values of the latter model were applied in the libration corrections of bond distances in this part of the molecule. The eigenvalues of T are 0.22, 0.19, and 0.16 Å while the r.m.s. librational amplitudes are 6.1, 3.4, and 2.5°. The major axis of libration is nearly parallel to the axis having the least moment of inertia.

Bond distances, interbond angles, short *intra-* and *inter*-molecular contacts are given in Table 4. In Fig. 1 bond distances and angles as well as the numbering of atoms may be found.

Standard deviations were calculated from the correlation matrix ignoring the standard deviations in cell parameters. For the C−Br bond the estimated standard deviation is 0.006 Å, for C−C bonds less than 0.011 Å and for C−N

Table 3. The root mean square amplitudes of vibration $(\bar{u}^2)^{\frac{1}{2}}$ (Å) and B-values (Å^2) along the principal axes given by the components of a unit vector in fractional coordinates ($\times 10^3$).

Atom	$(\bar{u}^2)^{\frac{1}{2}}$	B	e_x	e_y	e_z
	.289	6.58	129	36	−38
Br	.218	3.74	126	31	64
	.199	3.11	135	−63	9
	.232	4.26	61	34	68
N1	.189	2.83	191	24	−14
	.181	2.59	101	−67	30
	.258	5.26	60	41	64
N2	.235	4.36	91	56	−34
	.213	3.57	197	−38	19
	.284	6.39	152	54	34
N3	.266	5.60	84	1	−60
	.211	3.50	143	−57	31
	.229	4.16	−56	66	30
C1	.212	3.55	100	−27	69
	.153	1.86	194	33	−4
	.245	4.75	78	−55	53
C2	.225	4.01	−92	39	48
	.206	3.35	190	41	25
	.284	6.35	99	−71	5
C3	.226	4.03	105	21	−52
	.199	3.12	173	28	54
	.306	7.38	−32	65	37
C4	.206	3.34	51	44	−54
	.192	2.91	217	−1	38
	.252	5.00	9	69	36
C5	.235	4.36	77	35	−54
	.199	3.13	211	−16	39
	.233	4.28	−8	18	70
C6	.188	2.79	165	−52	28
	.171	2.31	153	57	3
	.256	5.19	104	39	63
C7	.207	3.39	138	33	−37
	.188	2.79	144	−60	20

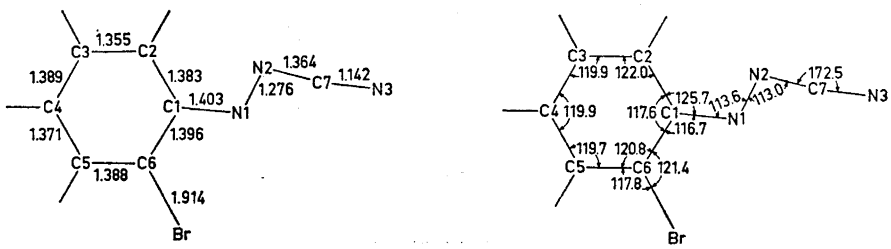

Fig. 1. Inter-atomic distances and bond angles.

Table 4. Bond distances, bond angles, *intra*-molecular contacts, and *inter*-molecular distances less than 3.4 Å (equivalent position numbers in parentheses as defined below). Bond distances in parentheses are corrected for anisotropic thermal motion.

Bond distances (Å)			Bond angles (°)	
Br–C6	1.906	(1.914)	C1–C2–C3	122.0
C1–C2	1.380	(1.383)	C2–C3–C4	119.9
C2–C3	1.352	(1.355)	C3–C4–C5	119.9
C3–C4	1.383	(1.389)	C4–C5–C6	119.7
C4–C5	1.366	(1.371)	C5–C6–C1	120.8
C5–C6	1.384	(1.388)	C6–C1–C2	117.6
C6–C1	1.388	(1.396)	N1–C1–C2	125.7
C1–N1	1.400	(1.403)	N1–C1–C6	116.7
N1–N2	1.269	(1.276)	C1–N1–N2	113.6
N2–C7	1.362	(1.364)	N1–N2–C7	113.0
C7–N3	1.140	(1.142)	N2–C7–N3	172.5
			Br–C6–C1	121.4
			Br–C6–C5	117.8

Intra-molecular contacts (Å)		Other contacts (Å)	
Br–N1	3.04	N1–N3(1)	3.33
C2–N2	2.71	N1–C5(2)	3.38
		C1–C7(1)	3.35

1: $-1+x, y, z$ 2: $1+x, y, z$

and N–N bonds less than 0.008 Å. Standard deviations in bond angles are 0.7° or less.

Deviations of atomic positions from two least-squares planes are presented in Table 5. Fig. 2 is a composite Fourier map as seen along the *a* axis.

Table 5. Deviations of atoms from two least squares planes (Å). Plane No. 1 is through all atoms and plane No. 2 is through the phenyl group and the atoms attached to it. Deviations of atoms not defining the plane in parentheses.

Br	.002	.001
N1	−.010	(−.006
N2	.050	(.057)
N3	−.024	(−.018)
C1	.006	.010
C2	−.010	−.004
C3	−.002	.004
C4	−.002	.002
C5	−.011	−.009
C6	−.004	−.001
C7	−.006	(.001)

DISCUSSION

The structure of *o*-bromobenzene-*anti*-diazocyanide is ordered, and is thus more accurately determined than the structure of the corresponding *p*-chloro compound.[1]

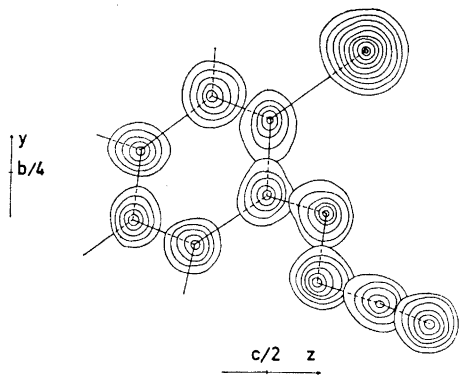

Fig. 2. Composite three-dimensional electron density map as viewed along the *a* axis. Contour intervals are 1.5 e.Å^{-3} for the light atoms.

As may be seen from Table 5, the molecule is nearly planar. The atoms of the benzene ring and the Br and N1 atoms are all situated in a distance less than the estimated standard deviation from a least-squares plane defined by these atoms. N2 lies on one side of this plane (0.057 Å) and N3 on the opposite side (0.018 Å). The dihedral angles C6 – C1 – N1 – N2 and C1 – N1 – N2 – C7 are 177.5° and 178.2°, respectively.

The bond lengths and angles observed in the bromobenzene part of the molecule are in close agreement with the expected values found for instance in *trans-p,p'*-dibromoazobenzene.[7] The slight difference between the external angles at C6 is probably due to a repulsion between Br and N1, the N1 – Br separation being only 3.04 Å.

The C1 – N1 bond (1.403 Å) is found to be approximately the same as in the *p*-chloro compound. The value of the C2 – C1 – N1 angle *cis* to the N – N double bond (126°) seems to be normal for this kind of configuration; a similar effect has been observed in several substituted azobenzenes and has been ascribed to repulsion between C2 and N2.

The N = N bond length (1.276 Å, corrected) is found to be the same as in *trans-p,p'*-dibromoazobenzene (1.276 Å)[7] and in *p*-chlorobenzene-*anti*-diazoimidoglyoxynitrile (1.264 Å).[8] The bond angles at the nitrogen atoms, 113.6° (N1) and 113.0° (N2), are also equal to the corresponding angles in these compounds, and indeed equal to those found in various *trans*-azobenzenes.

The N2 – C7 bond length (1.364 Å) is 0.04 Å shorter than the C1 – N1 bond. This is nearly the expected difference between the carbon single bond covalent radii for *sp* and sp^2 hybridized carbon atoms. The corrected C≡N bond length is 1.142 Å.

As may be seen from Table 4 the intermolecular contacts are normal. There is, however, a fairly short separation (3.59 Å) between C2 in one molecule and the nitrile nitrogen atom (N3) in another. This corresponds to a distance of 2.59 Å between the (calculated) atomic position of the hydrogen atom attached to C2 and the nitrogen atom. A similar somewhat short contact was observed in the structure of *p*-chlorobenzene-*anti*-imidoglyoxynitrile.[8] The non-linearity of the N2 – C7≡N3 arrangement may possibly be related to

this weak interaction, the distortion relative to linearity causing the contact to be shorter. The indicated hydrogen bond links molecules together forming a centrosymmetrical dimer.

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