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The Crystal Structure of the Copper(II) Complex with *o*-Hydroxyacetophenone-isobutylimine

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The crystal structure of the copper(II) complex with *o*-hydroxyacetophenone-isobutylimine, $Cu(C_{12}H_{16}NO)_2$, has been determined from 2583 independent reflections, measured with Mo $K\alpha$ radiation by diffractometer, and refined by least squares to $R=0{\cdot}035$. Crystal data are: $a_0=11{\cdot}286$ (3), $b_0=15{\cdot}241$ (4), $c_0=6{\cdot}804$ (2) Å, $\beta=103{\cdot}75$ (2)°, $Z=2$, space group $P2_1/a$. Coordination of the copper ion is planar, with Cu–N = 2·003 (2) Å and Cu–O = 1·891 (2) Å. The molecule has a 'step-structure' with 2·24 Å perpendicular distance between the phenyl planes. The C–C bond adjacent to the phenyl ring is significantly lengthened, to 1·465 (4) Å, by a twisting which reduces the π-orbital overlap.

Introduction

N-Substituted *o*-hydroxyacetophenone-imines form bis-chelates with copper(II),

$$(\text{–O–}C_6H_4\text{–}C(CH_3)\text{=}N(R)\text{–})_2Cu,$$

which are green or brown in the solid depending on the substituent, R. This 'color isomerism' of Cu(II) complexes has been extensively investigated by Professors D. Hall and T. N. Waters (*cf.* Hall, Sheat & Waters, 1968). As a part of an investigation of the structures of these compounds we have now completed the study of the isobutyl compound.

Experimental

The red-brown crystals were prepared by Professor F. D. Thomas II of the University of Montana. A series of oscillation and Weissenberg photographs showed the crystals to be monoclinic needles bounded by {100} and {010} and the systematic absence of reflections $h0l$ with $h\neq 2n$ and $0k0$ with $k\neq 2n$ identified the space group as $P2_1/a$. For collection of diffractometer data, a section of length 0·52 mm was cut from a needle having cross-section dimensions of 0·096 mm along **b** and 0·20 mm along **a***. All measurements were made with Mo $K\alpha$ radiation, filtered through 0·001 inch of niobium foil, on a Picker automated diffractometer equipped with a scintillation counter and pulse-height discriminator. The take-off angle was set at 3·5°. The cell constants and their estimated standard deviations were obtained by least-squares fit of 25 2θ values (averages of $+2\theta$ and -2θ). The final values are: $a_0=11{\cdot}286$ (3), $b_0=15{\cdot}241$ (4), $c_0=6{\cdot}804$ (2) Å, $\beta=103{\cdot}75$ (2)°. The density measured by flotation, 1·298 g.cm^{-3}, agrees with the calculated density of 1·295 g.cm^{-3} for two molecules per cell.

The intensity data were collected by the ω–2θ scan method using the formula of Alexander & Smith (1964): scan range = $1{\cdot}3+1{\cdot}0\tan\theta$. Stationary background measurements were made at the start and finish of each scan. Between each set of about 100 reflections, the intensities of four standard reflections were remeasured and these values were used to calculate a scale factor for each set. The scale factors showed no systematic variation with time and the maximum variation was ±1·4%. Intensities were measured for the 2583 unique reflections in the range $0<2\theta<55°$. Measurements were recorded in truncated dekacounts and the intensities and their estimated standard deviations were calculated from:

$$I=S-\frac{t_s}{2t_B}(B_1+B_2)-0{\cdot}45\left(2\frac{t_s}{2t_B}-1\right)$$

$$\sigma_I^2=\frac{1}{10}\left[S+\left(\frac{t_s}{2t_B}\right)^2(B_1+B_2)\right]+k^2\left[S+\frac{t_s}{2t_B}(B_1+B_2)\right]^2+0{\cdot}25$$

S = dekacounts recorded during scan time t_s
B_i = dekacounts recorded during background time t_B
k = empirical stability constant, set at 0·01

The terms 0·45 and 0·25 are corrections for the truncation of the counts to dekacounts. 586 reflections having intensity less than twice their estimated standard deviation were coded as 'unobserved' and assigned intensity values of $I+2\sigma_I$ for special treatment in the least-squares refinement. Lorentz and polarization factors were applied, but no corrections were made for absorption ($\mu=10{\cdot}2$ cm^{-1}; range of transmission factors, 0·8 to 0·92) nor for anomalous dispersion.

Structure determination

All calculations were carried out on an IBM 7094-7040 direct-coupled system using the set of programs written

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Table 1.

(*a*) Positional parameters ($\times 10^4$) and thermal parameters ($\times 10^2$)

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cu^{2+}	0	0	0	423 (2)	345 (1)	303 (1)	10 (2)	82 (1)	−17 (1)
O	1398 (1)	710 (1)	199 (2)	527 (8)	535 (9)	362 (7)	−83 (6)	124 (6)	−65 (6)
N	−406 (2)	716 (1)	2220 (3)	473 (8)	353 (8)	383 (8)	20 (7)	127 (7)	−4 (7)
C(1)	1713 (2)	960 (1)	3774 (3)	566 (12)	304 (9)	379 (10)	16 (9)	27 (9)	−12 (8)
C(2)	2133 (2)	826 (1)	1990 (4)	502 (12)	303 (9)	482 (11)	−2 (9)	66 (10)	−15 (8)
C(3)	3407 (2)	845 (2)	2158 (4)	494 (12)	496 (13)	614 (14)	−41 (10)	107 (11)	−14 (11)
C(4)	4216 (2)	943 (2)	4006 (5)	492 (13)	560 (14)	857 (20)	−52 (11)	7 (14)	46 (15)
C(5)	3810 (3)	1042 (2)	5747 (5)	646 (16)	640 (15)	581 (15)	−55 (13)	−92 (13)	27 (13)
C(6)	2583 (3)	1064 (2)	5629 (4)	719 (15)	470 (12)	431 (12)	−11 (11)	15 (11)	7 (10)
C(7)	409 (2)	1056 (1)	3685 (3)	615 (13)	338 (9)	328 (10)	52 (9)	117 (9)	14 (8)
C(8)	85 (3)	1587 (2)	5365 (4)	901 (17)	674 (14)	409 (14)	189 (14)	159 (13)	−121 (11)
C(9)	−1715 (2)	888 (2)	2085 (4)	545 (12)	405 (11)	499 (12)	−14 (9)	226 (10)	−20 (9)
C(10)	−2169 (2)	1730 (2)	978 (5)	490 (12)	413 (12)	776 (16)	47 (9)	185 (12)	11 (11)
C(11)	−3503 (2)	1878 (2)	973 (5)	548 (14)	693 (16)	1073 (24)	104 (12)	245 (16)	−78 (16)
C(12)	−2006 (3)	1713 (2)	−1153 (5)	949 (20)	968 (23)	796 (20)	331 (18)	390 (17)	422 (17)

(*b*) Positional parameters ($\times 10^3$) and thermal parameters ($\times 10$)

H(3)	371 (2)	79 (2)	83 (4)	64 (7)
H(4)	505 (2)	95 (2)	402 (4)	66 (7)
H(5)	443 (3)	114 (2)	709 (5)	97 (9)
H(6)	232 (2)	113 (2)	679 (4)	57 (7)
H(81)	50 (4)	220 (4)	544 (7)	162 (18)
H(82)	26 (3)	131 (2)	651 (6)	92 (11)
H(83)	−66 (3)	188 (3)	504 (6)	107 (12)
H(91)	−218 (2)	38 (2)	141 (3)	47 (5)
H(92)	−180 (2)	89 (2)	356 (4)	68 (7)
H(10)	−163 (2)	226 (2)	172 (4)	66 (7)
H(111)	−394 (2)	131 (2)	26 (4)	73 (8)
H(112)	−357 (3)	189 (2)	243 (5)	85 (10)
H(113)	−386 (3)	249 (2)	20 (5)	90 (9)
H(121)	−250 (4)	127 (3)	−183 (6)	111 (17)
H(122)	−104 (4)	167 (3)	−119 (8)	169 (17)
H(123)	−225 (3)	227 (3)	−178 (6)	114 (12)

The temperature factors are calculated as:

$$\exp\left(-\tfrac{1}{4}\{B_{11}a^{*2}h^2+B_{22}b^{*2}k^2+B_{33}c^{*2}l^2+2B_{12}a^*b^*hk+2B_{13}a^*c^*hl+2B_{23}b^*c^*kl\}\right)$$

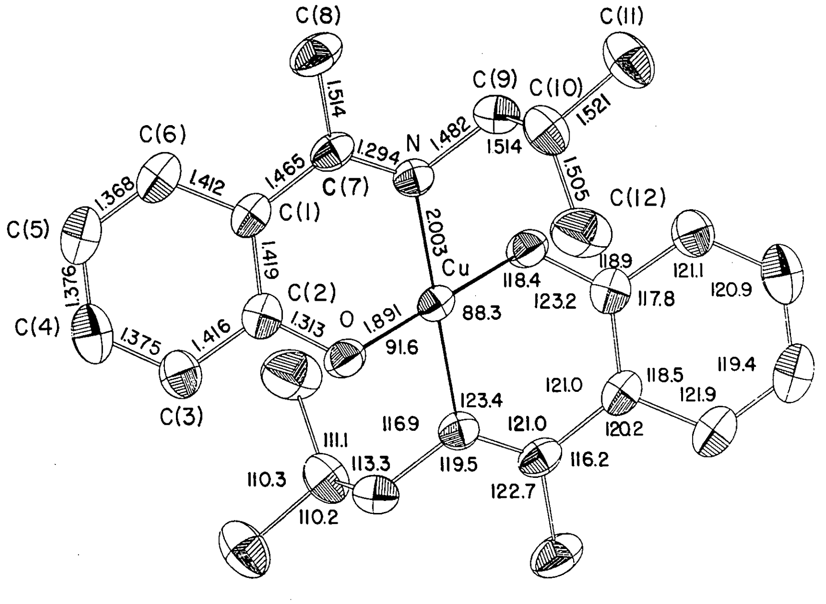

Fig. 1. Interatomic distances and angles and 30% probability thermal ellipsoids. Estimated standard deviations are 0·002 Å for Cu–O and Cu–N, 0·003 Å for O–C and N–C, 0·004 Å for C–C, and 0·2° for bond angles.

Table 2. *Observed and calculated structure factors*

Columns are h, $10F_o$ and $10F_c$. Unobserved reflections are marked with *.

h	10Fo	10Fc
H,0,0		
2	62	54
4	356	156
6	534	526
8	186	192
10	51	45
12	64	62
14	43	50
H,1,0		
1	1038	1017
2	129	128
3	380	369
4	68	-68
5	807	794
6	124	125
7	221	227
8	105	-104
9	135	135
10	25*	-12
11	53	51
12	26*	5
13	31	29
14	25*	3
H,2,0		
0	895	904
1	432	-419
2	570	535
3	289	-285
4	311	305
5	36	25
6	327	322
7	83	84
8	180	181
9	47	-43
10	89	80
11	28*	22
12	65	69
13	27*	-2
14	35*	31
H,3,0		
1	384	368
2	261	260
3	304	293
4	208	202
5	61	60
6	26	-20
7	345	345
8	24	26
9	110	110
10	47	-49
11	79	80
12	32*	3
13	59	57
14	32*	13
H,4,0		
0	882	-931
1	997	-1035
2	551	546
3	61	55
4	302	296
5	250	243
6	247	246
7	64	64
8	182	186
9	26*	-14
10	130	134
11	29*	18
12	64	64
13	32*	26
H,5,0		
1	304	314
2	29	-31
3	493	486
4	224	216
5	137	145
6	36	30
7	127	123
8	102	100
9	226	227
10	40	-36
11	110	113
12	26*	12
13	64	64
H,6,0		
0	403	-409
1	322	326
2	420	418
3	52	63
4	522	528
5	108	102
6	174	180
7	38	34
8	204	210
9	27*	14
10	169	171
11	26*	-5
12	44	51
13	26*	2
H,7,0		
1	240	240
2	112	-111
3	450	459
4	62	-62
5	207	210
6	177	179
7	182	179
8	51	-50
9	146	147
10	35	28
11	105	105
12	25*	4
13	66	63
H,8,0		
0	37*	-9
1	423	433
2	397	401
3	21	-17
4	336	346
5	33	29
6	87	85
7	55	-52
8	159	158
9	28*	-4
10	112	116
11	26*	-1
12	73	70
H,9,0		
1	157	156
2	40	-36
3	287	294
4	93	-97
5	194	195
6	41	-43
7	186	185
8	29*	-14
9	39	42
10	32	34
11	82	82
12	33*	-11
H,10,0		
0	591	601
1	18*	-11
2	147	149
3	24*	15
4	141	140
5	110	-109
6	181	183
7	39	-39
8	72	73
9	22*	-5
10	57	54
11	25*	-12
12	60	59
H,11,0		
1	251	256
2	14*	-4
3	141	142
4	40	-43
5	287	295
6	106	-104
7	81	77
8	48	-45
9	86	85
10	30*	7
11	47	45
H,12,0		
0	511	515
1	34	-38
2	121	121
3	53	-54
4	158	162
5	119	-120
6	185	181
7	29*	-16
8	69	65
9	27*	-12
10	56	56
11	28*	-3
H,13,0		
1	253	254
2	42	43
3	104	105
4	50	-49
5	163	159
6	31	-28
7	104	104
8	43	-40
9	87	84
10	29*	-11
H,14,0		
0	104	98
1	53	-55
2	185	142
3	35	-30
4	97	100
5	22*	6
6	120	118
7	36*	-13
8	68	71
9	24*	-0
10	35	34
H,15,0		
1	128	124
2	29*	20
3	135	133
4	28	37
5	32	31
6	29*	-10
7	71	73
8	49	45
9	48	52
H,16,0		
0	56*	32
1	27	-20
2	127	126
3	45	45
4	75	78
5	39	34
6	56	53
7	31*	20
8	69	61
H,17,0		
1	55	92
2	31*	-5
3	133	132
4	38	33
5	49	40
6	35	28
7	39	38
H,18,0		
0	62	57
1	35	33
2	95	88
3	32*	13
4	90	83
5	33	24
H,19,0		
1	59	61
2	26*	0
3	74	69
H,0,1		
0	533	490
2	77	-77
-2	175	-185
4	236	235
-4	40	-36
6	456	452
-6	410	405
8	239	246
-8	148	152
10	51	52
-10	147	150
12	32	33
-12	33	41
-14	61	57
H,1,1		
0	589	-588
1	553	528
-1	813	775
2	113	-105
-2	209	205
3	211	198
-3	39	46
4	15*	7
-4	229	227
5	312	307
-5	178	175
6	93	96
-6	410	-392
7	189	184
-7	201	199
8	61	66
-8	83	78
9	226	237
-9	181	184
10	26*	26
-10	40	41
11	64	72
-11	61	58
12	27*	13
-12	29*	-19
13	60	57
-13	61	60
-14	24*	-3
H,2,1		
0	357	334
1	400	392
-1	901	-919
2	822	822
-2	490	469
3	36	44
-3	28	34
4	380	381
-4	380	366
5	33	36
-5	435	-415
6	324	318
-6	365	358
7	75	81
-7	62	60
8	271	279
-8	164	162
9	26*	-12
-9	36	25
10	80	76
-10	141	146
11	29*	26
-11	34	38
12	54	55
-12	77	73
13	37	35
-13	30*	17
-14	35	37
H,3,1		
0	856	-876
1	640	629
-1	811	809
2	255	-241
-2	226	214
3	586	580
-3	608	591
4	27	-22
-4	23	20
5	371	370
-5	244	244
6	23	-22
-6	304	-297
7	372	374
-7	364	363
8	193	198
-8	51	51
9	76	76
-9	129	132
10	22*	-0
-10	75	65
11	83	84
-11	87	95
12	29	14
-12	32*	-19
13	46	53
-13	52	54
-14	34*	3
H,4,1		
0	887	910
1	654	-655
-1	66	-67
2	582	570
-2	575	575
3	159	161
-3	30	22
4	407	401
-4	434	437
5	31	26
-5	386	-381
6	252	252
-6	231	230
7	27*	-8
-7	33	21
8	163	162
-8	232	235
9	21	-19
-9	86	89
10	128	126
-10	78	76
11	23*	9
12	77	76
-12	84	88
13	40	41
-13	27*	-5
-14	44	41
H,5,1		
0	443	-437
1	405	406
-1	28	20
2	138	-134
-2	147	144
3	427	428
-3	435	437
4	33	20
-4	121	-122
5	384	383
-5	546	542
6	75	-73
-6	21	-22
7	179	176
-7	125	132
8	40	38
-8	23	16
9	80	78
-9	133	132
10	29*	-7
-10	48	51
11	76	75
-11	118	119
12	24*	14
-12	23*	-7
13	27*	23
-13	53	49
-14	24*	6
H,6,1		
0	574	583
1	133	-132
-1	200	207
2	52	47
-2	375	383
3	148	-155
-3	18	-17
4	316	312
-4	412	423
5	25	-29
-5	31	34
6	143	149
-6	94	105
7	62	64
-7	18*	0
8	28*	15
-8	233	232
9	42	-38
-9	25*	7
10	151	146
-10	70	65
11	23*	6
-11	28*	-1
12	58	61
-12	65	69
13	24*	-3
-13	27*	4
H,7,1		
0	194	194
1	239	245
-1	95	97
2	93	93
-2	17*	-0
3	228	231
-3	401	402
4	23*	-5
-4	28	-27
5	208	204
-5	347	349
6	98	103
-6	64	60
7	98	95
-7	198	200
8	184	-182
-8	46	43
9	78	76
-9	138	137
10	37	28
-10	29	-35
11	71	70
-11	90	94
12	31*	11
-12	26	17
-13	60	52
H,8,1		
0	72	-66
1	35	45
-1	214	214
2	350	363
-2	567	567
3	142	-133
-3	43	40
4	207	212
-4	421	426
5	71	71
-5	152	155
6	91	89
-6	223	231
7	25*	-20
-7	25*	14
8	63	61
-8	164	161
9	23*	7
-9	28*	-14
10	35	46
-10	116	133
11	39*	11
-11	31	-32
12	63	56
-12	70	70
-13	24*	2
H,9,1		
0	413	420
1	252	259
-1	288	294
2	23*	15
-2	123	-124
3	256	257
-3	416	425
4	24*	22
-4	36	-21
5	67	67
-5	222	218
6	28	21
-6	159	165
7	107	102
-7	242	242
8	64	-66
-8	31	-23
9	109	106
-9	137	141
10	23*	-6
-10	41	-42
11	67	72
-11	91	89
12	34*	-14
-12	35	24
H,10,1		
0	80	86
1	64	63
-1	28	30
2	239	243
-2	304	306
3	50	46
-3	23*	-18
4	191	194
-4	246	247
5	27	24
-5	122	128
6	174	166
-6	269	267
7	78	-76
-7	21*	-13
8	95	95
-8	125	130
9	38	37
-9	26*	-10
10	50	53
-10	118	120
11	34*	-18
-11	27*	-5
-12	45	45
H,11,1		
0	93	99
1	206	208
-1	278	282
2	19*	-4
-2	42	-44
3	180	176
-3	200	203
4	25*	13
-4	25	25
5	140	139
-5	165	166
6	37	-23
-6	26*	18
7	[illegible]	44
-7	[illegible]	65
8	45	37
-8	34	-23
9	129	124
-9	166	111
10	24*	-16
-10	24*	-4
11	54	47
-11	56	50
-12	24*	-7
H,12,1		
0	249	253
1	159	163
-1	157	-164
2	161	160
-2	50	56
3	51	54
-3	20*	5
4	177	177
-4	126	131
5	48	-47
-5	20*	0
6	109	107
-6	115	114
7	22*	2
-7	22*	-4
8	134	132
-8	55	54
9	24*	10
-9	27*	-24
10	29	35
-10	70	73
-11	24*	4
H,13,1		
0	102	-106
1	139	140
-1	245	254
2	77	79
-2	21	15
3	170	173
-3	54	57
4	47	-46
-4	73	73
5	150	146
-5	79	76
6	22*	-1
-6	68	-71
7	123	118
-7	64	57
8	50	50
-8	22	0
9	50	53
-9	41	40
10	27*	-2
-10	24*	0
H,14,1		
0	237	238
1	22*	17
-1	47	-42
2	195	188
-2	97	99
3	53	54
-3	44	46
4	128	130
-4	54	55
5	25	-34
-5	50	-53
6	97	96
-6	87	77
7	33*	16
-7	29*	3
8	124	116
-8	34	25
9	34*	-10
-9	30*	5
-10	31*	26
H,15,1		
0	80	-79
1	152	148
-1	140	143
2	34	37
-2	43	45
3	158	156
-3	80	75
4	28*	-4
-4	22	20
5	116	118
-5	83	79
6	38	-40
-6	29*	2
7	130	129
-7	68	65
8	34*	26
-8	23	-0
-9	34*	21
H,16,1		
0	172	173
1	71	-68
-1	74	70
2	102	102
-2	115	111
3	32*	5
-3	27*	-7
4	82	77
-4	62	60
5	32	-25
-5	26*	5
6	101	103
-6	62	59
7	31*	15
-7	30*	8
-8	63	55
H,17,1		
0	36*	-20
1	122	120
-1	55	50
2	29	-20
-2	38	31
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4	26*	7
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6	27*	-5
-6	24*	14
-7	62	58
H,18,1		
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5	26*	13
-5	33*	-3
H,19,1		
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2	30	-30
-2	27*	-17
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-3	72	73
H,0,2		
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-10	133	134
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-14	68	63
H,1,2		
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-2	348	356
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-4	15	20
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-5	204	207
6	20	16
-6	86	-88
7	415	418
-7	277	285
8	158	-161
-8	56	49
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-9	197	194
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13	64	63
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-14	34*	-9
H,2,2		
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6	190	191
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7	91	93
-7	154	119
8	252	257
-8	179	179
9	86	-87
-9	23	12
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-11	22*	-11
12	63	62
-12	73	75
-13	26*	-3
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H,3,2		
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2	50	-36
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-4	80	-76
5	232	230
-5	152	153
6	41	43
-6	25	-27
7	174	176
-7	297	296
8	204	-206
-8	125	118
9	98	105
-9	154	150
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12	30*	24
-12	25	9
-13	49	51
-14	30*	5
H,4,2		
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2	170	176
-2	508	505
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-4	287	293
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8	28	24
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9	29	-29
-9	85	93
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-12	79	80
-13	23*	-12
-14	46	37
H,5,2		
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-2	291	293
3	189	206
-3	645	631
4	45	42
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-7	239	237
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9	169	168
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11	86	76
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12	30	15
-12	32*	-6
-13	62	61
-14	25*	9
H,6,2		
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-4	438	434
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-6	344	342
7	30	-27
-7	71	75
8	37	-36
-8	229	227
9	26*	-13
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12	34*	29
-12	76	82
-13	33*	9
H,7,2		
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7	38	35
-7	145	153
8	64	65
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9	105	106
-9	131	127
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-12	30*	-6
-13	49	44
H,8,2		
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5	36	-35
-5	42	-41
6	113	116
-6	189	194
7	70	-67
-7	27*	-26
8	111	108
-8	70	74
9	28	21
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-11	25	17
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H,9,2		
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7	163	162
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8	107	102
-8	88	-92
9	46	42
-9	102	99
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11	33	39
-11	72	75
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-13	27*	15
H,10,2		
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3	145	138
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-4	231	236
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6	79	78
-6	61	64
7	23*	-11
-7	95	-97
8	136	139
-8	85	80
9	45	40
-9	31*	-14
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11	31*	-16
-11	30*	-16
-12	36	27
H,11,2		
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2	161	158
-2	40	-41
3	214	216
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5	113	110
-5	147	144
6	25*	10
-6	43	40
7	156	153
-7	95	97
8	25	18
-8	77	-77
9	55	53
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-11	70	65
-12	26*	-3
H,12,2		
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7	59	61
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8	121	119
-8	105	99
9	34*	-5
-9	38	-44
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-10	85	81
-11	26	-11
H,13,2		
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3	106	100
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5	26*	6
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7	27*	18
-7	38	33
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-8	67	73
-9	24*	-6
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H,15,2		
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3	38	41
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-4	25*	-17
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6	30*	0
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-9	43	44
H,16,2		
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1	31	-22
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-4	23*	19
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-5	21*	-5
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H,17,2		
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-4	26*	24
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H,18,2		
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-5	27*	-9
H,19,2		
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H,0,3		
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-12	72	72
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H,1,3		
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-2	235	231
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-3	465	461
4	101	98
-4	17*	15
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6	27*	2
-6	49	50
7	144	143
-7	495	514
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-9	114	115
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-10	25*	14
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-11	62	65
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-14	28*	-18
H,2,3		
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-4	296	293
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7	110	-103
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8	108	103
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-12	80	76
-13	27*	-5
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H,3,3		
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3	309	299
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-5	351	347
6	32	28
-6	26*	4
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-7	203	198
8	56	-61
-8	147	148
9	81	83
-9	232	234
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-11	102	103
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-14	31*	-4
H,4,3		
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1	56	-59
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3	22	-21
-3	375	373
4	245	245
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6	160	161
-6	264	263
7	142	-136
-7	74	-82
8	173	173
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9	23*	-20
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10	88	89
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-13	27*	-5
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H,5,3		
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2	106	-136
-2	96	108
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4	29	22
-4	41	37
5	209	214
-5	236	244
6	32	39
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7	174	173
-7	20	12
8	77	-76
-8	57	55
9	79	90
-9	130	136
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-10	24*	-14
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-14	31*	1
H,6,3		
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3	22*	-19
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4	221	222
-4	353	353
5	35	36
-5	26*	-25
6	137	143
-6	125	133
7	29	30
-7	23*	-19
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-8	23*	14
9	36*	-20
-9	30*	-9
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-11	33*	-13
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-13	34*	-13
H,7,3		
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1	242	257
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2	231	234
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3	303	305
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-4	22	16
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6	21	14
-6	20	13
7	77	84
-7	25	23
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-8	130	-131
9	95	89
-9	97	91
10	24*	-9
-10	32	30
11	41	42
-11	83	81
-12	24*	-0
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H,8,3		
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1	33	43
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2	295	307
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3	76	74
-3	132	-131
4	234	242
-4	258	303
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6	119	116
-6	214	208
7	96	97
-7	21	17
8	103	102
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9	33*	8
-9	27	-22
10	43	46
-10	117	109
-11	33*	14
-12	54	59
-13	34*	-14
H,9,3		
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1	301	300
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2	125	127
-2	72	-56
3	220	226
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4	30	-34
-4	20*	1
5	183	187
-5	257	258
6	35*	-28
-6	39*	34
7	48	46
-7	136	134
8	61	56
-8	50	-52
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-10	25*	22
-11	63	60
-12	28*	8
H,10,3		
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3	21*	3
-3	80	-83
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-6	147	147
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-7	27*	17
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9	26	18
-9	26*	5
10	33	42
-10	71	70
-11	24*	17
-12	43	41
H,11,3		
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2	31	-41
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3	82	94
-3	211	212
4	34	-35
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6	54	-54
-6	21*	4
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-8	37	27
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-9	47	49
-10	26*	11
-11	43	39
H,12,3		
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1	46	-44
-1	52	-49
2	122	123
-2	206	206
3	39*	-26
-3	35*	-30
4	85	89
-4	75	73
5	31	-28
-5	26*	-25
6	89	90
-6	71	73
7	24*	-13
-7	58	50
8	38	33
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-9	34*	-17
-10	35	34
-11	25*	15
H,13,3		
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1	66	72
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2	55	-56
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3	65	66
-3	117	112
4	22*	13
-4	40	-42
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-5	59	60
6	30*	-3
-6	35	30
7	95	93
-7	130	123
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-8	40	32
-9	81	79
-10	25*	-9
H,14,3		
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3	22	12
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-5	26*	-7
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-6	97	93
7	37*	-20
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-8	92	93
-9	31*	7
H,15,3		
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1	78	72
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6	40	36
-6	34*	4
-7	59	59
-8	34*	23
-9	74	77
H,16,3		
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2	69	61
-2	64	56
3	24*	-9
-3	24*	22
4	38	35
-4	108	104
5	33	28
-5	25	23
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-7	37*	-25
H,17,3		
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1	95	84
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2	25*	21
-2	34*	17
3	51	51
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4	27*	-1
-4	38	29
-5	74	71
-6	26	-11
H,18,3		
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1	31*	21
-1	31*	15
2	52	51
-2	43	46
-3	35*	10
-4	73	70
H,0,4		
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2	222	225
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4	146	138
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6	189	189
-6	269	270
8	47	51
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10	81	77
-10	36	26
-12	79	71
-14	50	45
H,1,4		
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1	313	310
-1	457	459
2	23*	-5
-2	24*	4
3	193	194
-3	405	397
4	19*	6
-4	83	-79
5	128	124
-5	166	175
6	33	33
-6	21	12
7	144	144
-7	283	285
8	22	0
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9	52	52
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-10	43	33
11	39	31
-11	64	67
-12	32*	3
-13	68	67
-14	27*	-15
H,2,4		
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1	39	-34
-1	53	57
2	168	172
-2	258	269
3	36	32
-3	231	-231
4	158	162
-4	246	246
5	82	79
-5	97	-97
6	163	163
-6	273	276
7	25	21
-7	120	-125
8	77	77
-8	207	204
9	23*	8
-9	27*	0
10	54	53
-10	76	77
-11	23*	4
-12	50	50
-13	44	-41
-14	56	49
H,3,4		
0	69	67
1	271	279
-1	327	338
2	36	32
-2	17*	-15
3	171	175
-3	208	213
4	31	31
-4	25*	14
5	290	294
-5	281	285
6	114	115
-6	47	-58
7	90	90
-7	124	122
8	29*	-7
-8	181	-186
9	73	72
-9	137	139
10	28*	-15
-10	27	21
-11	64	64
-12	27*	-15
-13	29	30
-14	27*	-7
H,4,4		
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1	109	112
-1	22*	-13
2	237	242
-2	285	292
3	61	57
-3	44	52
4	219	220
-4	228	237
5	125	132
-5	23*	-11
6	122	127
-6	167	183
7	22*	8
-7	166	-175
8	95	97
-8	68	64
9	29	14
-9	57	57
10	27*	30
-10	133	137
-11	29*	-14
-12	59	50
-13	29	-22
H,5,4		
0	142	147
1	296	305
-1	195	192
2	19*	1
-2	26	26
3	240	244
-3	273	274
4	68	64
-4	105	107
5	178	184
-5	269	263
6	48	52
-6	74	-72
7	105	106
-7	220	215
8	27*	-4
-8	21*	1
9	70	70
-9	25	29
10	34*	1
-10	24*	4
-11	101	96
-12	25*	-15
-13	47	47
H,6,4		
0	144	143
1	65	-64
-1	117	122
2	353	351
-2	288	290
3	24*	22
-3	87	95
4	203	205
-4	260	267
5	31	41
-5	26*	-2
6	107	108
-6	152	148
7	32*	-23
-7	29	29
8	101	106
-8	66	61
9	30*	-16
-9	26*	1
10	38*	41
-10	147	145
-11	30*	-1
-12	88	87
-13	34*	-0
H,7,4		
0	36	-36
1	150	152
-1	128	124
2	55	-57
-2	73	73
3	239	233
-3	195	199
4	50	51
-4	48	-41
5	147	142
-5	203	204
6	92	-91
-6	35	42
7	107	111
-7	137	135
8	25*	1
-8	67	69
9	71	71
-9	94	87
10	26*	-0
-10	24*	2
-11	107	103
-12	27*	2
-13	38	43
H,8,4		
0	96	93
1	114	-119
-1	32	28
2	185	187
-2	57	55
3	21*	8
-3	63	60
4	157	159
-4	198	200
5	80	-77
-5	26	-18
6	69	69
-6	153	148
7	28	-24
-7	84	86
8	91	95
-8	54	51
9	26*	-15
-9	25	-25
-10	117	114
-11	26*	9
-12	65	67
H,9,4		
0	89	-90
1	52*	48
-1	132	135
2	23*	-5
-2	40	42
3	157	162
-3	82	64
4	32*	-11
-4	40	-45
5	85	68
-5	152	152
6	31	-41
-6	23*	3
7	82	79
-7	41	40
8	26	-8
-8	110	104
9	71	69
-9	128	125
-10	33*	-19
-11	73	75
-12	25*	2
H,10,4		
0	66	70
1	63	59
-1	74	-73
2	97	98
-2	120	119
3	29	-21
-3	21*	10
4	126	124
-4	146	138
5	41	-46
-5	24	19
6	75	63
-6	111	106
7	33*	11
-7	47	49
8	55	55
-8	131	129
-9	30*	5
-10	59	53
-11	27*	-3
H,11,4		
0	39	-33
1	144	143
-1	132	138
2	62	57
-2	24	-17
3	124	124
-3	177	177
4	33	-36
-4	30	24
5	56	55
-5	86	68
6	33*	22
-6	29*	18
7	68	61
-7	121	115
8	25*	1
-8	25*	17
-9	87	88
-10	30*	9
-11	48	44
H,12,4		
0	155	157
1	51	52
-1	29*	-19
2	120	116
-2	186	186
3	22*	-14
-3	54	-61
4	60	68
-4	133	130
5	33*	-12
-5	25	34
6	95	90
-6	111	109
7	39	26
-7	28	26
-8	139	134
-9	25*	8
-10	30	21
H,13,4		
0	30*	12
1	183	186
-1	134	134
2	27*	14
-2	35	-36
3	72	69
-3	175	173
4	28	-37
-4	22*	3
5	86	85
-5	96	97

Table 2 (*cont.*)

H,13,4		
6	25*	11
-6	54	53
-7	99	96
-8	58	-51
-9	59	60
-10	31*	20
H,14,4		
0	202	200
1	24*	6
-1	25*	-9
2	70	71
-2	127	122
3	26*	-5
-3	33	-33
4	66	61
-4	89	93
5	34*	-2
-5	39	33
6	67	72
-6	85	90
-7	25*	-23
-8	56	58
-9	34*	6
H,15,4		
0	57	56
1	122	121
-1	123	117
2	33*	-21
-2	39	-35
3	41	37
-3	72	74
4	31*	-11
-4	24	28
5	75	73
-5	107	100
-6	25*	4
-7	68	59
-8	26	-16
H,16,4		
0	105	98
1	27*	5
-1	23	22
2	63	62
-2	82	82
3	34*	-6
-3	30*	19
-4	66	64
-5	30	15
-6	55	57
H,17,4		
0	34	37
1	48	41
-1	69	73
-2	34*	14
-3	57	49
-4	27*	19
H,0,5		
0	493	501
2	195	196
-2	210	213
4	126	120
-4	90	91
6	189	197
-6	90	120
8	93	95
-8	52	57
-10	54	50
-12	56	51
H,1,5		
0	57	55
1	334	336
-1	266	263
2	20*	18
-2	37	40
3	149	143
-3	163	162
4	62	70
-4	18*	12
5	221	227
-5	139	144
6	32	-24
-6	38	43
7	108	110
-7	153	149
8	33*	19
-8	36	-37
9	55	47
-9	60	61
-10	30	36
-11	38	42
-12	27*	3
-13	26	26
H,2,5		
0	425	427
1	140	-140
-1	202	210
2	163	166
-2	196	196
3	45	44
-3	49	56
4	150	149
-4	184	180
5	64	64
-5	25*	-0
6	150	149
-6	204	208
7	27*	17
-7	29*	-4
8	77	76
-8	105	99
9	34*	-20
-9	25	-5
-10	83	83
-11	31	23
-12	42	48
-13	30	-21
H,3,5		
0	34	29
1	180	177
-1	314	324
2	25	-17
-2	27*	-13
3	162	161
-3	206	206
4	75	79
-4	27*	16
5	134	131
-5	245	247
6	32*	-20
-6	59	-61
7	75	77
-7	131	128
8	27	8
-8	26	-11
9	53	58
-9	168	161
-10	60	55
-11	75	72
-12	27*	-7
-13	47	44
H,4,5		
0	55	53
1	99	-102
-1	23	-17
2	172	170
-2	267	271
3	35	-29
-3	24*	9
4	163	166
-4	237	234
5	38	45
-5	38	-39
6	59	58
-6	189	191
7	33*	1
-7	71	-77
8	52	49
-8	191	189
9	25*	-11
-9	34	16
-10	108	103
-11	27*	10
-12	49	53
-13	28*	-1
H,5,5		
0	83	-86
1	55	56
-1	186	189
2	46	-48
-2	46	-55
3	174	179
-3	252	251
4	31*	18
-4	25	-22
5	34*	9
-5	137	134
6	23*	12
-6	98	-106
7	84	79
-7	191	194
8	25*	-4
-8	27	-18
9	38	47
-9	124	123
-10	42	35
-11	82	82
-12	31*	-2
-13	72	66
H,6,5		
0	124	-124
1	40	-38
-1	38	-48
2	195	194
-2	252	257
3	22*	4
-3	65	-55
4	125	126
-4	214	213
5	22*	15
-5	54	-62
6	33	27
-6	126	117
7	27*	-3
-7	29	-19
8	53	46
-8	182	180
-9	32*	19
-10	87	87
-11	27*	4
-12	72	65
H,7,5		
0	75	-78
1	101	109
-1	83	76
2	30*	10
-2	60	55
3	156	159
-3	239	238
4	26*	9
-4	39	-41
5	40	41
-5	153	147
6	29	21
-6	24	26
7	52	54
-7	137	137
8	27*	12
-8	28	-19
-9	96	93
-10	26	7
-11	40	86
-12	34*	11
H,8,5		
0	139	141
1	72	66
-1	40	-41
2	139	131
-2	131	134
3	48	46
-3	21*	15
4	113	111
-4	191	191
5	27*	-22
-5	23*	7
6	57	56
-6	140	137
7	24*	4
-7	32	33
8	59	55
-8	101	96
-9	27*	19
-10	85	91
-11	34*	6
-12	53	55
H,9,5		
0	25	21
1	177	173
-1	97	94
2	24	27
-2	35	39
3	112	112
-3	136	137
4	33	-36
-4	46	39
5	118	111
-5	196	176
6	25*	15
-6	27	29
7	43	43
-7	87	98
-8	55	56
-9	69	65
-10	42	-32
-11	74	72
H,10,5		
0	217	216
1	90	49
-1	34	-29
2	110	109
-2	89	88
3	25	-23
-3	46	44
4	87	81
-4	127	128
5	40*	-27
-5	45	49
6	73	78
-6	117	113
7	31*	-8
-7	31	26
-8	36	37
-9	24*	6
-10	66	69
-11	36	-13
H,11,5		
0	33	66
1	144	149
-1	98	96
2	23*	2
-2	24*	-23
3	77	72
-3	84	79
4	24	-28
-4	49	49
5	93	91
-5	111	109
6	31*	-18
-6	34	32
-7	81	78
-8	23*	29
-9	47	35
-10	36	-31
H,12,5		
0	166	163
1	26*	4
-1	73	72
2	65	69
-2	62	62
3	27*	-12
-3	26*	12
4	38	40
-4	79	79
5	34*	-5
-5	40	44
-6	90	90
-7	27*	22
-8	33	30
-9	27*	-17
H,13,5		
0	61	56
1	94	86
-1	122	122
2	23*	7
-2	27*	10
3	46	47
-3	62	64
4	31*	15
-4	23*	5
5	67	65
-5	83	81
-6	46	46
-7	44	45
-8	30	-8
H,14,5		
0	106	103
1	25*	-8
-1	51	45
2	51	48
-2	83	78
3	34*	8
-3	24*	1
-4	75	74
-5	23	25
-6	67	71
-7	28*	10
H,15,5		
0	31*	-13
1	45	44
-1	117	113
2	31*	9
-2	27*	4
-3	64	67
-4	25*	-2
-5	50	52
-6	24*	-1
H,16,5		
0	39*	26
-1	35*	-18
-2	99	96
-3	27*	-14
H,0,6		
0	127	124
2	135	135
-2	203	194
4	116	117
-4	140	137
6	48	43
-6	129	164
8	35	45
-8	111	113
-10	124	114
-12	43	41
H,1,6		
0	158	-156
1	106	106
-1	301	294
2	41	-47
-2	34	37
3	92	89
-3	111	111
4	29*	-14
-4	21	-26
5	85	91
-5	120	113
6	31*	-21
-6	31*	-29
7	72	73
-7	125	134
-8	29*	7
-9	122	124
-10	42	31
-11	55	52
-12	24*	-3
H,2,6		
0	171	170
1	107	-105
-1	113	-113
2	104	105
-2	140	146
3	28*	-1
-3	24	25
4	120	118
-4	110	107
5	38	-45
-5	82	-92
6	88	89
-6	132	132
7	36	-26
-7	26*	15
-8	104	103
-9	50	54
-10	87	84
-11	27*	22
-12	43	39
H,3,6		
0	230	-230
1	140	140
2	44	-49
-2	105	106
3	115	117
-3	132	127
4	23*	-6
-4	30*	17
5	117	114
-5	123	121
6	28*	-16
-6	72	-74
7	66	63
-7	94	93
-8	66	56
-9	85	81
-10	65	64
-11	61	54
-12	38*	-3
H,4,6		
0	173	176
1	34	-33
-1	59	-56
2	124	123
-2	141	141
3	29*	13
-3	72	78
4	102	101
-4	138	132
5	30*	-15
-5	24*	-22
6	73	73
-6	92	92
7	31*	-6
-7	24	22
-8	99	101
-9	54	55
-10	55	56
-11	32	28
-12	54	52
H,5,6		
0	44	-37
1	179	175
-1	37	42
2	23*	6
-2	61	60
3	141	140
-3	206	204
4	26*	23
-4	56	57
5	91	87
-5	140	135
6	27*	-2
-6	24*	-20
7	64	61
-7	132	127
-8	33	41
-9	63	58
-10	34	36
-11	64	65
-12	27*	4
H,6,6		
0	135	133
1	32*	-26
-1	63	59
2	152	148
-2	165	164
3	32*	10
-3	36*	22
4	69	71
-4	193	198
5	24*	17
-5	27	24
6	60	55
-6	121	112
7	26*	7
-7	26*	-1
-8	108	106
-9	36*	4
-10	49	48
-11	27*	9
H,7,6		
0	93	95
1	153	148
-1	77	71
2	27*	0
-2	48	-49
3	107	109
-3	189	189
4	26	14
-4	32*	-12
5	44	51
-5	176	174
6	25*	-3
-6	33	34
-7	102	101
-8	33	-24
-9	63	67
-10	34*	-17
-11	58	58
H,8,6		
0	109	105
1	25*	15
-1	35	41
2	107	102
-2	136	132
3	27*	-15
-3	42	-34
4	65	66
-4	165	164
5	26	19
-5	39	32
6	43	42
-6	133	131
-7	32*	-23
-8	89	82
-9	37	-43
-10	43	45
-11	26*	-10
H,9,6		
0	99	101
1	84	88
-1	151	145
2	33*	14
-2	53	-60
3	73	75
-3	119	117
4	25*	-7
-4	23*	-6
5	36	35
-5	147	149
-6	34	24
-7	73	78
-8	29	-23
-9	63	65
-10	41	-29
H,10,6		
0	61	70
1	65	65
-1	32*	3
2	94	91
-2	123	119
3	27*	-11
-3	26*	-15
4	77	68
-4	115	120
5	34*	19
-5	36	31
-6	115	114
-7	33*	6
-8	62	60
-9	31*	-15
H,11,6		
0	23	32
1	62	62
-1	140	137
2	35*	19
-2	23*	-0
3	67	66
-3	82	86
4	31*	-3
-4	26*	9
-5	81	79
-6	34*	25
-7	73	79
-8	27*	-1
-9	64	57
H,12,6		
0	88	77
1	31*	18
-1	30*	-4
2	72	66
-2	63	67
3	23*	15
-3	23*	5
-4	55	61
-5	24*	0
-6	72	69
-7	27	16
-8	38	42
H,13,6		
0	46	-40
1	55	57
-1	64	64
2	34*	9
-2	30	28
-3	40	37
-4	27*	-2
-5	43	35
-6	26*	-3
H,14,6		
0	75	72
-1	31*	-11
-2	25*	23
-3	27*	11
-4	34*	21
H,0,7		
0	69	64
2	65	68
-2	104	102
4	63	55
-4	96	94
-6	54	42
-8	111	122
-10	95	100
H,1,7		
0	30	31
1	59	62
-1	79	76
2	31	22
-2	32	33
3	86	75
-3	103	106
4	33*	14
-4	32*	26
5	55	55
-5	67	60
-6	28*	-5
-7	109	111
-8	36	21
-9	105	107
-10	27*	-9
-11	62	56
H,2,7		
0	94	91
1	23*	4
-1	52	45
2	75	71
-2	137	131
3	27*	21
-3	67	63
4	58	62
-4	106	99
5	30*	13
-5	31*	18
-6	50	66
-7	26*	-10
-8	120	117
-9	33*	15
-10	72	75
-11	26*	-1
H,3,7		
0	30	15
1	100	100
-1	136	139
2	25	13
-2	48	46
3	73	68
-3	122	119
4	27*	15
-4	29*	27
5	60	58
-5	113	113
-6	31	-27
-7	109	107
-8	36*	28
-9	74	72
-10	27*	-10
-11	54	48
H,4,7		
0	122	122
1	27*	-19
-1	32*	-2
2	97	94
-2	135	138
3	36*	-9
-3	35	37
4	74	75
-4	109	110
5	25*	-2
-5	24*	0
-6	131	132
-7	23*	-4
-8	71	64
-9	26*	-0
-10	40	39
H,5,7		
0	33	-32
1	123	121
-1	160	157
2	28	-23
-2	30	33
3	73	75
-3	109	109
4	25*	-7
-4	26*	7
5	62	66
-5	135	130
-6	25	-10
-7	75	70
-8	29	11
-9	40	37
-10	30*	5
H,6,7		
0	138	136
1	30*	-14
-1	24*	-15
2	95	100
-2	97	93
3	22*	-4
-3	44	38
4	86	92
-4	113	113
-5	26*	4
-6	120	122
-7	26*	16
-8	33*	23
-9	24*	16
-10	34*	26
H,7,7		
0	29*	-11
1	114	112
-1	106	100
2	25*	-9
-2	33*	25
3	78	82
-3	104	100
4	31*	-0
-4	39	28
-5	123	123
-6	28	26
-7	46	47
-8	27*	11
-9	32	32
H,8,7		
0	111	107
1	25*	16
-1	27*	1
2	82	82
-2	63	61
3	34*	10
-3	32*	12
-4	112	105
-5	33*	21
-6	73	74
-7	30*	17
-8	49	47
-9	27*	19
H,9,7		
0	30*	6
1	73	74
-1	39	34
2	26*	10
-2	33*	-22
-3	95	90
-4	27*	11
-5	76	73
-6	27*	14
-7	51	52
-9	27	-10
H,10,7		
0	40	41
1	27*	7
-1	27	6
-2	54	51
-3	50	-46
-4	74	76
-5	24*	-5
-6	36	29
-7	30*	-10
H,11,7		
0	34*	16
-1	36	30
-2	38	-43
-3	63	62
-4	36	-30
-5	41	37
-6	34*	-7
H,0,8		
0	97	92
2	69	55
-2	158	151
-4	62	56
-6	94	88
-8	76	66
H,1,8		
0	25*	5
1	89	84
-1	126	124
2	26*	-24
-2	33*	11
-3	101	95
-4	23*	1
-5	70	71
-6	27*	-8
-7	103	94
-8	26*	3
-9	61	59
H,2,8		
0	82	81
1	27*	-0
-1	27*	-1
2	59	55
-2	126	118
-3	45	44
-4	69	67
-5	33*	-7
-6	77	73
-7	26*	-5
-8	94	85
H,3,8		
0	37*	25
1	59	61
-1	69	73
2	34	-23
-2	49	45
-3	94	92
-4	30*	17
-5	69	66
-6	24*	5
-7	75	76
-8	28*	2
H,4,8		
0	65	65
1	31*	10
-1	34*	32
2	52	55
-2	50	44
-3	55	52
-4	46	47
-5	33*	10
-6	62	61
-7	25*	5
-8	62	59
H,5,8		
0	34*	15
1	43	41
-1	48	38
-2	40	33
-3	60	68
-4	30*	20
-5	85	83
-6	30*	4
-7	33	39
H,6,8		
0	57	55
-1	24*	4
-2	24*	14
-3	30*	5
-4	94	90
-5	27*	0
-6	51	59
-7	31*	-20
H,7,8		
-1	32	40
-2	26	-29
-3	41	41
-4	27*	1
-5	74	71
-6	33	-28
H,8,8		
-2	56	46
-3	36	-32
-4	52	52

or adapted by Stewart (1964). Scattering factors for copper, oxygen, nitrogen and carbon atoms were taken from Cromer & Waber (1965) and for hydrogen from Table 2 of Stewart, Davidson & Simpson (1965).

Phases were calculated from the copper atoms in the special positions 0,0,0 and $\frac{1}{2},\frac{1}{2}$,0 and a three-dimensional Fourier synthesis was calculated, in which the oxygen, nitrogen and eight of the twelve carbon atoms could be readily found. A second Fourier synthesis gave positions of all non-hydrogen atoms. One cycle of full-matrix least-squares with isotropic thermal parameters and two cycles with anisotropic thermal parameters lowered R from 0·23 to 0·06 $\left(R=\frac{\sum||F_o|-|F_c||}{\sum|F_o|}\right.$, for unique observed reflections only$\left.\right)$. The function minimized was $\sum w(|F_o|-|F_c|)^2$, with all $w=1$ except that any unobserved reflection with $F_c<F_o$ was given $w=0$ for that cycle.

At this stage a three-dimensional difference Fourier synthesis showed the positions of the 16 hydrogen atoms. Three cycles of refinement of the hydrogen atom positional and isotropic thermal parameters reduced R to 0·043.

Weights were now introduced, equal to the reciprocal of the variance of each F_o. Since the total number of parameters to be refined exceeded the limit set by the capacity of the computer, the final series of refinement cycles consisted of two types. In one type, all parameters of all non-hydrogen atoms were refined, and in the other type the parameters of the hydrogen atoms and those of the attached carbon atoms were refined. The two types were alternated until convergence had been attained. In the final cycles the mean and maximum shifts of the hydrogen atoms were 0·05 and 0·53 standard deviations, respectively, while those for the other atoms were 0·02 and 0·11. The final value of R was 0·035, that of R_w $\left[=\left(\frac{\sum w(F_o-F_c)^2}{\sum wF_o^2}\right)^{1/2}\right]$ was 0·033,

Table 3. *Carbon–hydrogen interatomic distances*

C(3)–H(3)	1·04 (3) Å	C(9)—H(92)	1·04 (3) Å
C(4)–H(4)	0·94 (3)	C(10)–H(10)	1·06 (3)
C(5)–H(5)	1·02 (3)	C(11)–H(111)	1·06 (3)
C(6)–H(6)	0·91 (3)	C(11)–H(112)	1·02 (4)
C(8)–H(81)	1·04 (5)	C(11)–H(113)	1·10 (3)
C(8)–H(82)	0·87 (4)	C(12)–H(121)	0·93 (4)
C(8)–H(83)	0·94 (3)	C(12)–H(122)	1·09 (5)
C(9)–H(91)	0·99 (2)	C(12)–H(123)	0·97 (4)

and that of the standard deviation of an observation of unit weight ($=[\sum w(F_o-F_c)^2/(n-p)]^{1/2}$) was 1·85. There was no evidence of secondary extinction effects.

The final atom parameters are listed in Table 1 and structure factors in Table 2.

Discussion

The structure consists of discrete molecules with a conformation as shown in Figs. 1 and 2. Distances and angles are given in Fig. 1 and Table 3. The molecule is centrosymmetric and the coordination of the copper(II) ion is therefore exactly planar. The Cu–N bond length of 2·003 (2) Å and Cu–O of 1·891 (2) Å are similar to the lengths reported for other compounds with planar coordination, *e.g.* 1·993(4) and 1·878 (3) Å in bis-(*N*-phenylsalicylaldiminato)copper(II) (Wei, Stogsdill & Lingafelter, 1964), 1·989 (12) and 1·901 (12) Å in bis-(*N*-methylsalicylaldiminato)copper(II) (Lingafelter, Simmons, Morosin, Scheringer & Freiburg, 1961). The coordination of the copper(II) ion must be considered to be fourfold, since the nearest other atoms are H(122) at 2·85 (5), H(121) at 3·41 (4), and C(12) at 3·423 (4) Å. Presumably, the presence of this methyl group of the isobutyl substituent prevents the approach of any other possible coordinating atom. The brown color and the planar fourfold coordination agree with the suggestion of Waters & Hall (1959).

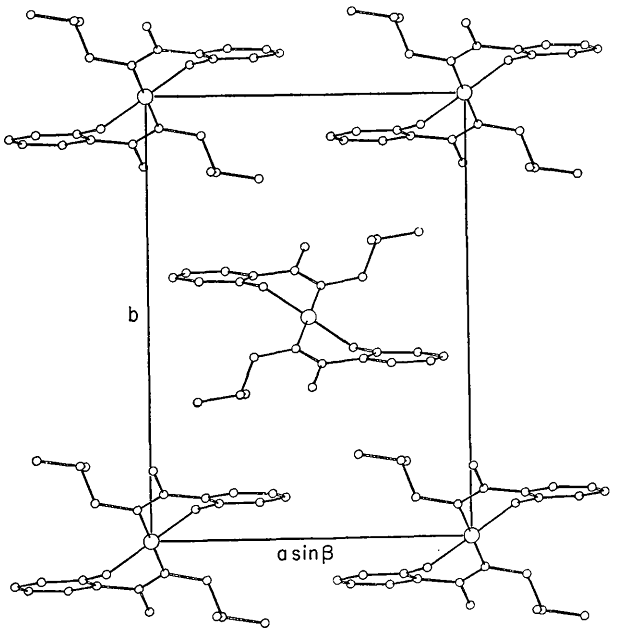

Fig. 2. Projection down *c* axis.

Bond distances within the ligand appear to be normal, except for C(1)–C(7). In particular, all comparable distances, except for C(1)–C(7), agree well with the average values for salicylaldimine chelates reported by Lingafelter & Braun (1966). The C(1)–C(7) bond [1·465 (4) Å] is 0·035 Å longer than the average reported by Lingafelter & Braun. The lengthening does not seem to be due to a steric effect of the methyl group, since: (*a*) C(8)–C(6) (2·89 Å) is slightly greater than C(8)–C(9) (2·84 Å); (*b*) H(82)–H(6) (2·30 Å) is considerably greater than H(83)–H(92) (2·08 Å); and (*c*) C(1)–C(7)–C(8) (116·2°) is considerably smaller than N–C(7)–C(8) (122·7°). However, an analysis of the angles in the system C(2)–C(6)–C(1)–C(7)–C(8)–N shows that the p_z orbitals on C(1) and C(7) are rotated out of alignment about the C(1)–C(7) axis by 24°, and we

Table 4. *Coefficients of least-squares planes equations and distances from these planes*

$$AX+BY+CZ=D$$

X, Y, Z are orthogonal Ångström space coordinates referred to crystallographic a, b and c^* axes

	Plane	*A*	*B*	*C*	*D*
I	Benzene plane	0·147	0·991	−0·137	0·112
II	*o*-Hydroxyacetophenone imine plane	−0·625	0·987	−0·158	0·102
III	Cu-N-C(9)-C(8)-C(7)-C(1)	0·142	0·831	−0·538	0·207
IV	Coordination plane	−0·382	0·627	−0·679	−0·119

Distances from least-squares planes

The planes are determined by the atoms marked by *

	I	II	III	IV
Cu	1·119	1·024	0·021*	0·0*
O	0·042	−0·024*	−1·026	0·0*
N	0·251	0·179*	0·020*	0·0*
C(1)	−0·010*	−0·028*	−0·041*	1·280
C(2)	0·020*	−0·012*	−0·613	0·898
C(3)	−0·014*	−0·023*	−0·777	1·494
C(4)	−0·004*	0·022*	−0·331	2·464
C(5)	0·015*	0·056*	0·268	2·869
C(6)	−0·008*	0·011*	0·392	2·274
C(7)	−0·141	−0·181*	0·013*	0·593
C(8)	−0·781	−0·804	0·028*	0·596
C(9)	0·000	−0·095	−0·041*	−0·781
C(10)	−1·366	−1·477	−1·453	−2·210
C(11)	−1·569	−1·702	−1·430	−2·928
C(12)	−1·542	−1·675	−2·265	−2·949

therefore suggest that the lengthening of the C(1)–C(7) bond is caused by this rotation, which has decreased the overlap between the two p_z orbitals and therefore decreased the double-bond character of the bond.

The molecule is distorted from planarity by an unusual amount. Omitting the 'outside' atoms of the isobutyl group, C(10), C(11), C(12), the distortion of one half of the molecule from complete planarity may be approximately described by a rotation of the (nearly) planar group C(1), C(7), C(8), N, C(9), Cu through an angle of 25° about the line C(1)–C(9). Equations for various least-squares planes and the distances of the several atoms from these planes are given in Table 4. Comparison of these distances with those found in other compounds shows a number of differences. The metal atom is farther (1·12 Å) out of the plane defined by the benzene ring, the farthest heretofore reported being 0·75 Å in bis-(*N*-t-butylsalicylaldiminato)palladium(II) (Day, Glick & Hoard, 1968). The nitrogen atom is also farther (0·25 Å) out of the plane and is out in the same direction as the metal, whereas in other compounds it is out in the opposite direction from the metal atom (*cf.* Table 6, Jain & Lingafelter, 1967). The dihedral angle between the coordination plane CuN_2O_2 and the mean plane of the *o*-hydroxyacetophenone imine residue is 136·6°, which may be compared with the corresponding angle of 144·4° in bis-(*N*-t-butylsalicylaldiminato)palladium.

The packing of the molecules may be seen in Fig. 2. None of the intermolecular contact distances is unusual.

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The Crystal Structure of Compounds with (N-P)$_n$ Rings. VII.* Refinement of the Crystal Structure of Hexabromocyclotriphosphazene, $N_3P_3Br_6$

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The crystal structure of $N_3P_3Br_6$, as determined by Giglio & Puliti (1967), has been refined from three-dimensional intensity data, collected with Mo radiation on an automatic three-circle diffractometer. Two crystals were used. Data processing and refinement were carried out independently with the data obtained from each crystal. The intensities were corrected for absorption and during the refinement a correction for extinction was applied according to Zachariasen (1967, 1968). Atomic coordinates obtained from the two crystals agree within the standard deviations. The molecules $N_3P_3Br_6$ lie on crystallographic mirror planes of the space group *Pnma*, with unit-cell dimensions $a = 14·463$ (0·002), $b = 13·410$ (0·003) and $c = 6·601$ (0·001) Å. There are four short intermolecular Br···Br distances (3·65–3·85 Å) and a short N···Br distance (3·24 Å). The two independent angles P–N–P of a molecule are significantly different: 119·3 (0·6) and 122·4 (0·5)°. Other chemically equivalent bond lengths and angles are equal within experimental error. Mean values (and individual standard deviations) are P–N 1·576 (0·008), P–Br 2·162 (0·004) Å, N–P–N 118·5 (0·5) and Br–P–Br 102·1 (0·1)°. The $(N–P)_3$ ring is non-planar and slightly chair-shaped. The dihedral angles of the ring bonds are −6·7, 6·3 and −5·5° and the largest distance from a ring atom to the least-squares plane through the ring is 0·047 Å.

Introduction

The crystal structures of several chloro- and fluorocyclophosphazenes are known with some precision, *viz.* $N_3P_3Cl_6$ (Wilson & Carroll, 1960), $N_4P_4Cl_8$ [two crystal modifications, *K* form (Hazekamp, Migchelsen & Vos, 1962) and *T* form (Wagner & Vos, 1968)], $N_5P_5Cl_{10}$ (Schlueter & Jacobson, 1968), $N_3P_3F_6$ (Dougill, 1963) and $N_4P_4F_8$ (McGeachin & Tromans, 1961).

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* Part VI: Olthof (1969).