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# The Structure of *N*-Methyl-*p*-chlorobenzaldoxime and Refinement of the Structures of '*syn*'- and '*anti*'-*p*-Chlorobenzaldoxime

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*N*-Methyl-*p*-chlorobenzaldoxime, prepared from '*syn*'-*p*-chlorobenzaldoxime by use of dimethyl sulfate, is shown by a three-dimensional X-ray diffraction study to have the *anti* configuration. There are four molecules of $ClC_6H_4CHN(CH_3)O$ in a unit cell having parameters $a=7{\cdot}50$, $b=9{\cdot}91$, $c=11{\cdot}68$ Å and $\beta=108°\ 1'$. The space group is $P2_1/a$. For the 936 observed diffraction maxima the value of $R=\Sigma||F_o|-|F_c||/\Sigma|F_o|$ is 0·085.

Refinement of the previously determined structure of '*syn*'-*p*-chlorobenzaldoxime by three-dimensional least-squares methods has yielded $R=0{\cdot}116$ for the 1238 observed maxima. A refinement of two-dimensional data for '*anti*'-*p*-chlorobenzaldoxime has also been made, but better data are needed for an accurate structure. A comparison of C=N and N−O distances in this C=N−O system is presented.

This study of the crystal and molecular structure of *N*-methyl-*p*-chlorobenzaldoxime and refinements of the structures of '*syn*'- and '*anti*'-*p*-chlorobenzaldoxime form part of a series of studies of the C=N−O bond system in oximes RR'C=NOH and their alkylated or acylated derivatives. It is expected that detailed information on bond angles and distances will elucidate the nature of bonding in oximes and amine oxides, the questions of relative stabilities of '*syn*' and '*anti*' isomers, the relation of structure to *N*- *versus* *O*-alkylation, and problems relating to planarity, conjugation, charge transfer and spectra of these oximes and their derivatives.

The configuration of the *N*-methyl derivative of oximes has been studied earlier by chemical methods. Methylation by use of dimethyl sulfate, which has never yielded both of the theoretically possible *N*-methyl isomers, has been said to yield the *anti* configuration (Brady & Dunn, 1926; Sutton & Taylor, 1931). On the other hand, use of diazomethane as a methylating agent is stated to yield this same *N*-methyl isomer from the '*syn*' oxime, but a different *N*-methyl isomer from the '*anti*' oxime (Thompson & Baer, 1940). Our study shows conclusively that dimethyl sulfate yields the '*anti*' configuration for the *N*-methyl derivative obtained from '*syn*'-*p*-chlorobenzaldoxime.

The relation of this new structure to the known structures of oximes and amine oxides is exhibited below, but a comparison of *O*-methyl and *N*-methyl derivatives, both of which are known (Brady, Dunn & Goldstein, 1926), will have to await a projected crystal structure study of an *O*-methyl oxime.

In addition, we describe least-squares refinements of the X-ray data which were used previously (Jerslev, 1958) in the elucidation of the structures of '*syn*'- and '*anti*'-*p*-chlorobenzaldoximes, which are closely related to the new structure of *N*-methyl-*p*-chlorobenzaldoxime. The three-dimensional data of the '*syn*'-*p*-chlorobenzaldoxime have yielded an accurate structure. However, as already pointed out (Jerslev, 1958), the slight inconsistencies in the data probably arising from cutting of the crystals of '*anti*'-*p*-chlorobenzaldoxime to a suitable shape for X-ray study have limited the accuracy of refinement of the two-dimensional data for this substance. Nevertheless, these new refinements are presented for comparison of the results of crystallographic studies on these three compounds, and for addition to the review of data available on these and related compounds (Jerslev, 1958).

## Experimental study and X-ray data

*N*-Methyl-*p*-chlorobenzaldoxime was prepared by reacting *p*-chlorobenzaldehyde with hydroxylamine hydrochloride (Erdmann & Schwechten, 1890) and methylating the resulting '*syn*'-*p*-chlorobenzaldoxime (Brady, Dunn & Goldstein, 1926). Single crystals suitable for X-ray diffraction work were obtained by slow evaporation of a saturated solution in benzene. The melting point was 128–129·5°. Microanalysis confirmed the formula $ClC_8H_8NO$ (Cl: calc. 20·9, found 21·1; C: calc. 56·7, found 56·8; H: calc. 4·8, found 4·9; N: calc. 8·3, found 8·2%).

Single crystals no larger than 0·07 mm in cross section were chosen for study ($\mu=35{\cdot}95$ cm$^{-1}$ for Cu $K\alpha$). From oscillation and Weissenberg photographs the reciprocal lattice symmetry was established

Table 1. *List of observed and calculated structure factors for N-methyl-p-chlorobenzaldoxime*

The three columns of each group contain from left to right the values of $l$, 10 $F_o$, and 10 $F_c$. The values of $l$ followed by an asterisk indicate reflections below the observable limit. The value of $F_o$ in this case is the minimum observed value. After this study was completed we found that the $18\bar{5}$ reflection was indeed present on the films

0, 0, L

| | | |
|---|---|---|
| 1 | 113 | -110 |
| 2 | 402 | 382 |
| 3 | 133 | 155 |
| 4 | 147 | 122 |
| 5 | 191 | -180 |
| 6 | 90 | 76 |
| 7 | 28 | 35 |
| 8 | 208 | -219 |
| 9 | 127 | 126 |
| 10 | 184 | -215 |
| 11 | 35 | -41 |
| 12 | 104 | -107 |
| 13 | 60 | -68 |
| 14 | 63 | -77 |

0, 1, L

| | | |
|---|---|---|
| 2 | 167 | 182 |
| 3 | 514 | -518 |
| 4 | 26 | 2 |
| 5 | 560 | -559 |
| 6 | 93 | -76 |
| 7 | 201 | -211 |
| 8 | 93 | -93 |
| 9 | 167 | -177 |
| 10 | 49 | -51 |
| 11 | 29 | 32 |
| 13 | 34 | 31 |

0, 2, L

| | | |
|---|---|---|
| 0 | 142 | 150 |
| 1 | 83 | 80 |
| 2 | 154 | -153 |
| 3 | 57 | 55 |
| 4 | 124 | -119 |
| 5 | 167 | -160 |
| 6 | 43 | -32 |
| 7 | 39 | -37 |
| 8 | 167 | 170 |
| 9 | 44 | 49 |
| 10 | 69 | -68 |
| 11 | 25 | -19 |
| 12 | 26 | 30 |

0, 3, L

| | | |
|---|---|---|
| 1 | 210 | -222 |
| 2 | 151 | -144 |
| 3 | 176 | 188 |
| 4 | 232 | -225 |
| 5 | 78 | -72 |
| 6 | 282 | -268 |
| 7 | 125 | -124 |
| 8 | 139 | -145 |
| 9 | 31 | -33 |
| 10* | 20 | -4 |
| 11* | 21 | -5 |
| 12 | 23 | 23 |

0, 4, L

| | | |
|---|---|---|
| 0 | 161 | -165 |
| 1 | 279 | -319 |
| 2 | 306 | -329 |
| 3 | 377 | -396 |
| 4 | 36 | -32 |
| 5 | 51 | 46 |
| 6 | 39 | -39 |
| 7 | 113 | 105 |
| 8 | 94 | 81 |
| 9 | 42 | 43 |
| 10 | 37 | 42 |
| 11 | 54 | 57 |
| 12 | 42 | 43 |
| 13 | 49 | 58 |

0, 5, L

| | | |
|---|---|---|
| 1* | 16 | 22 |
| 2 | 42 | 45 |
| 3 | 158 | 167 |
| 4 | 111 | 99 |
| 5 | 122 | 127 |
| 6 | 151 | 144 |
| 7 | 176 | 183 |
| 8 | 81 | 71 |
| 9 | 55 | 67 |
| 10 | 24 | 24 |
| 11 | 28 | 24 |

0, 6, L

| | | |
|---|---|---|
| 0 | 47 | -52 |
| 1 | 229 | 232 |
| 2 | 70 | -61 |
| 3* | 19 | -15 |
| 4 | 77 | 71 |
| 5* | 21 | 22 |
| 6 | 30 | -26 |
| 7 | 55 | 57 |
| 8 | 49 | 50 |
| 9 | 62 | -69 |
| 10* | 20 | 12 |
| 11 | 55 | -52 |
| 12 | 27 | -25 |

0, 7, L

| | | |
|---|---|---|
| 1 | 61 | 58 |
| 2 | 125 | -129 |
| 3 | 97 | 108 |
| 4 | 57 | -52 |
| 5 | 95 | 114 |
| 6 | 56 | -54 |
| 7 | 134 | 167 |

0, 8, L

| | | |
|---|---|---|
| 0 | 139 | 147 |
| 1 | 85 | 96 |
| 2 | 143 | 165 |
| 3 | 67 | -68 |
| 4 | 32 | 35 |
| 5* | 22 | 8 |
| 6 | 70 | -83 |
| 7 | 33 | 34 |
| 8 | 20 | 22 |
| 9* | 18 | -4 |
| 10 | 49 | -52 |

0, 9, L

| | | |
|---|---|---|
| 1 | 104 | -123 |
| 2* | 22 | 9 |
| 3 | 28 | -29 |
| 4 | 24 | -22 |
| 5 | 97 | -88 |
| 6 | 25 | -13 |
| 7 | 85 | -82 |
| 8* | 17 | -18 |
| 9 | 20 | -19 |

0, 10, L

| | | |
|---|---|---|
| 0* | 21 | 22 |
| 1 | 43 | 50 |
| 2 | 114 | -117 |
| 3* | 21 | 2 |
| 4 | 60 | -54 |
| 5 | 36 | -34 |
| 6* | 18 | 20 |
| 7* | 16 | -1 |
| 8 | 18 | 18 |

0, 11, L

| | | |
|---|---|---|
| 1* | 14 | 6 |
| 2 | 22 | 24 |

0, 12, L

| | | |
|---|---|---|
| 0* | 15 | -22 |
| 1 | 24 | -9 |
| 2 | 23 | -27 |
| 3 | 26 | 23 |

1, 1, L

| | | |
|---|---|---|
| -9 | 164 | -163 |
| -8 | 196 | -199 |
| -7 | 231 | -225 |
| -6 | 165 | -145 |
| -5 | 163 | -152 |
| -4 | 51 | -39 |
| -3 | 274 | 274 |
| -2 | 364 | 377 |
| -1 | 333 | 311 |
| 0 | 420 | -430 |
| 1 | 27 | -34 |
| 2 | 317 | 270 |
| 3 | 285 | 242 |
| 4 | 188 | 167 |
| 5 | 405 | 383 |
| 6 | 254 | 239 |
| 7 | 63 | 64 |
| 8 | 43 | 44 |
| 9* | 22 | -3 |
| 10* | 22 | -9 |
| 11 | 25 | -23 |
| 12 | 50 | -38 |
| 13 | 36 | -34 |

1, 2, L

| | | |
|---|---|---|
| -13 | 42 | -54 |
| -12 | 41 | -42 |
| -11 | 71 | -81 |
| -10 | 100 | 112 |
| -9 | 98 | -96 |
| -8* | 20 | 15 |
| -7 | 161 | 154 |
| -6 | 73 | 64 |
| -5 | 18 | -13 |
| -4 | 443 | 430 |
| -3 | 404 | 389 |
| -2 | 51 | -14 |
| -1 | 1025 | 1304 |
| 0 | 219 | 226 |
| 1 | 421 | 448 |
| 2 | 197 | 169 |
| 3 | 19 | 8 |
| 4 | 282 | -272 |
| 5 | 43 | 28 |
| 6 | 152 | -143 |
| 7 | 253 | -246 |
| 8 | 28 | -23 |
| 9 | 150 | -160 |
| 10 | 48 | -54 |
| 11 | 99 | -96 |
| 12 | 60 | -68 |
| 13 | 61 | -66 |

1, 3, L

| | | |
|---|---|---|
| -11 | 60 | 59 |
| -10 | 139 | 152 |
| -9 | 111 | 114 |
| -8 | 87 | 89 |
| -7 | 38 | 32 |
| -6 | 302 | 307 |
| -5 | 58 | -57 |
| -4 | 239 | 245 |
| -3 | 82 | 71 |
| -2 | 123 | 132 |
| -1 | 387 | -429 |
| 0 | 267 | -281 |
| 1 | 117 | -102 |
| 2 | 335 | -350 |
| 3 | 90 | -67 |
| 4 | 367 | -371 |
| 5 | 68 | -70 |
| 6 | 177 | -177 |
| 7 | 45 | -36 |
| 8 | 195 | -207 |
| 9 | 54 | -53 |
| 10 | 41 | 44 |
| 11 | 30 | 34 |
| 12 | 40 | 41 |
| 13 | 30 | 34 |

1, 4, L

| | | |
|---|---|---|
| -12 | 46 | 42 |
| -11 | 38 | -41 |
| -10 | 37 | 36 |
| -7 | 43 | 48 |
| -6 | 132 | -118 |
| -5 | 133 | -118 |
| -4 | 66 | -66 |
| -3 | 87 | 76 |
| -2 | 131 | -126 |
| -1 | 21 | -6 |
| 0 | 270 | -290 |
| 1 | 88 | 85 |
| 2 | 153 | 151 |
| 3 | 186 | -191 |
| 4 | 20 | -12 |
| 5 | 70 | 61 |
| 6 | 95 | -87 |
| 7* | 21 | 23 |
| 8 | 145 | 141 |
| 9 | 27 | -31 |
| 10* | 21 | 18 |
| 11 | 36 | 38 |

1, 5, L

| | | |
|---|---|---|
| -11 | 60 | 52 |
| -10 | 38 | 38 |
| -8 | 108 | 112 |
| -7 | 35 | 34 |
| -6 | 124 | 113 |
| -5* | 19 | 7 |
| -4 | 33 | -22 |
| -3 | 55 | -46 |
| -2 | 160 | 170 |
| -1 | 31 | -23 |
| 0 | 238 | -273 |
| 1 | 137 | -137 |
| 2 | 18 | -6 |
| 3 | 33 | 24 |
| 4 | 92 | -87 |
| 5* | 20 | -6 |
| 6 | 110 | -101 |
| 7 | 118 | -112 |
| 8 | 64 | -70 |
| 9 | 25 | 26 |
| 10* | 20 | 16 |
| 11 | 29 | 30 |
| 12 | 34 | 34 |

1, 6, L

| | | |
|---|---|---|
| -12 | 20 | 22 |
| -11 | 49 | 45 |
| -10* | 20 | 14 |
| -9 | 36 | -34 |
| -8 | 48 | -50 |
| -7 | 34 | 37 |
| -6 | 40 | -39 |
| -5 | 69 | -77 |
| -4 | 58 | -56 |
| -3 | 164 | -171 |
| -2 | 265 | -283 |
| -1 | 179 | -179 |
| 0 | 95 | -100 |
| 1 | 172 | -165 |
| 2 | 78 | -83 |
| 3 | 40 | -42 |
| 4 | 69 | 68 |
| 5 | 100 | 111 |
| 6 | 114 | 121 |
| 7* | 22 | 0 |
| 8 | 38 | 42 |
| 9 | 78 | 73 |
| 10 | 67 | 67 |
| 11 | 23 | 29 |
| 12 | 22 | 31 |

1, 7, L

| | | |
|---|---|---|
| -8 | 126 | -150 |
| -7 | 70 | -77 |
| -6 | 97 | -111 |
| -5 | 23 | -26 |
| -4 | 58 | -50 |
| -3 | 130 | 128 |
| -2* | 20 | -1 |
| -1 | 39 | -37 |
| 0 | 83 | 87 |
| 1 | 50 | 55 |
| 2 | 118 | 118 |
| 3 | 83 | 85 |
| 4 | 117 | 130 |
| 5 | 63 | 71 |
| 6 | 155 | 173 |
| 7 | 35 | 37 |

1, 8, L

| | | |
|---|---|---|
| -10 | 27 | -29 |
| -8 | 51 | 42 |
| -7* | 21 | 11 |
| -6* | 22 | 14 |
| -5* | 48 | 54 |
| -4* | 22 | -19 |
| -3 | 143 | 150 |
| -2 | 44 | 31 |
| -1 | 62 | -66 |
| 0* | 21 | 1 |
| 1 | 91 | 100 |
| 2* | 22 | -1 |
| 3 | 32 | 35 |
| 4 | 31 | 29 |
| 5 | 33 | -29 |
| 6* | 21 | 10 |
| 7 | 29 | -17 |
| 8 | 31 | -27 |
| 9* | 16 | -10 |
| 10 | 20 | -23 |

1, 9, L

| | | |
|---|---|---|
| -8 | 65 | -65 |
| -7* | 20 | -12 |
| -6 | 35 | -41 |
| -5* | 21 | 11 |
| -4 | 48 | -49 |
| -3 | 96 | 108 |
| -2 | 32 | 29 |
| -1 | 30 | 29 |
| 0 | 33 | -29 |
| 1 | 81 | -76 |
| 2* | 22 | 22 |
| 3* | 22 | -8 |
| 4 | 73 | 78 |
| 5* | 21 | -7 |
| 6 | 73 | 68 |
| 7 | 32 | -29 |

1, 10, L

| | | |
|---|---|---|
| -9 | 13 | -19 |
| -8 | 36 | 40 |
| -7 | 35 | -34 |
| -6 | 31 | 32 |
| -5* | 19 | 24 |
| -4* | 20 | -17 |
| -3 | 68 | 68 |
| -2 | 40 | 46 |
| -1* | 21 | 23 |
| 0 | 29 | 29 |
| 1 | 127 | 129 |
| 2 | 74 | -73 |
| 3* | 20 | 4 |
| 4* | 19 | -1 |
| 5* | 18 | -22 |
| 6* | 16 | -13 |
| 7 | 36 | -35 |

1, 11, L

| | | |
|---|---|---|
| -4 | 37 | 41 |
| -3* | 18 | -11 |
| -2 | 32 | 33 |
| -1* | 19 | -17 |
| 0 | 37 | -43 |
| 1 | 51 | -45 |
| 2* | 18 | -5 |
| 3 | 45 | -45 |
| 4 | 28 | -27 |

1, 12, L

| | | |
|---|---|---|
| -1 | 35 | -25 |
| 0 | 22 | 24 |
| 1 | 23 | -12 |

2, 0, L

| | | |
|---|---|---|
| -13 | 60 | 53 |
| -12 | 76 | 86 |
| -11 | 169 | -195 |
| -10 | 41 | -32 |
| -9 | 44 | -49 |
| -8 | 33 | -27 |
| -7 | 237 | -232 |
| -6 | 68 | 82 |
| -5 | 477 | -464 |
| -4 | 187 | -165 |
| -3 | 74 | -41 |
| -2 | 1282 | -1307 |
| -1 | 203 | -179 |
| 0 | 338 | -326 |
| 1 | 204 | 212 |
| 2 | -87 | -85 |
| 3 | 808 | 778 |
| 4 | 38 | -39 |
| 5 | 209 | 210 |
| 6 | 143 | 146 |
| 7 | 148 | 147 |
| 8 | 252 | 262 |
| 9 | 44 | 41 |
| 10 | 94 | 88 |
| 11 | 42 | 43 |
| 12 | 77 | 63 |

2, 1, L

| | | |
|---|---|---|
| -12 | 47 | -43 |
| -11* | 24 | -12 |
| -10 | 171 | -185 |
| -9 | 67 | -65 |
| -8 | 97 | -94 |
| -7 | 120 | -118 |
| -6* | 16 | -1 |
| -5 | 61 | -65 |
| -4 | 120 | 115 |
| -3 | 164 | -173 |
| -2 | 934 | 1025 |
| 0 | 240 | 229 |
| 1 | 86 | 74 |
| 2 | 300 | 286 |
| 3 | 225 | 176 |
| 4 | 279 | 257 |
| 5 | 233 | 235 |
| 6 | 97 | -87 |
| 7 | 134 | 130 |
| 8 | 42 | -39 |
| 9* | 23 | -2 |
| 10 | 101 | -90 |
| 11 | 53 | -50 |
| 12 | 71 | -67 |

2, 2, L

| | | |
|---|---|---|
| -12 | 59 | 55 |
| -11 | 39 | -35 |
| -10 | 52 | -51 |
| -8 | 50 | -50 |
| -7 | 176 | 177 |
| -6 | 48 | -43 |
| -5 | 318 | 305 |
| -4 | 113 | -97 |
| -3 | 89 | -79 |
| -2 | 542 | -599 |
| -1 | 422 | 450 |
| 0 | 251 | 270 |
| 1 | 206 | -206 |
| 2 | 199 | 198 |
| 3 | 104 | -100 |
| 4 | 80 | 77 |
| 5 | 82 | -75 |
| 6* | 22 | 17 |
| 7 | 148 | -149 |
| 8 | 71 | 64 |
| 9* | 24 | -5 |
| 10 | 34 | -33 |

2, 3, L

| | | |
|---|---|---|
| -12 | 54 | -44 |
| -11 | 32 | -27 |
| -10 | 26 | -23 |
| -9 | 103 | -100 |
| -8 | 95 | -81 |
| -7 | 185 | -177 |
| -6 | 53 | -46 |
| -5 | 145 | 130 |
| -4 | 94 | 90 |
| -3 | 153 | 147 |
| -2 | 116 | 107 |
| -1 | 209 | 193 |
| 0 | 94 | 69 |
| 1 | 253 | 243 |
| 2 | 59 | 49 |
| 3 | 98 | 93 |
| 4 | 121 | 108 |
| 5 | 49 | 48 |
| 6 | 146 | 137 |
| 7 | 54 | 48 |
| 8 | 43 | -42 |
| 9 | 83 | -70 |
| 10 | 29 | -26 |
| 11 | 67 | -60 |
| 12 | 41 | -40 |

2, 4, L

| | | |
|---|---|---|
| -10 | 35 | -24 |
| -9 | 55 | 57 |
| -8 | 153 | 141 |
| -7 | 80 | 69 |
| -6 | 94 | 93 |
| -5 | 173 | 148 |
| -4 | 280 | 270 |
| -3 | 65 | 63 |
| -2 | 263 | 256 |
| -1 | 75 | 68 |
| 0 | 260 | 250 |
| 1 | 180 | 171 |
| 2 | 116 | -105 |
| 3 | 98 | -87 |
| 4 | 187 | -178 |
| 5 | 245 | -237 |
| 6 | 239 | -238 |
| 7 | 43 | -42 |
| 8 | 69 | -63 |
| 9 | 69 | -59 |
| 10 | 59 | -55 |
| 11 | 51 | -48 |
| 12 | 28 | -28 |

2, 5, L

| | | |
|---|---|---|
| -13 | 24 | 28 |
| -12* | 19 | 11 |
| -11 | 60 | 60 |
| -9 | 127 | 132 |
| -8* | 23 | -6 |
| -7 | 127 | 129 |
| -6 | 26 | -21 |
| -5 | 74 | 74 |
| -4 | 122 | -117 |
| -3 | 154 | -133 |
| -2* | 17 | 17 |
| -1 | 245 | -227 |
| 0 | 22 | -18 |
| 1 | 318 | -326 |
| 2 | 56 | -49 |
| 3 | 189 | -179 |
| 4 | 37 | -37 |
| 5 | 199 | -191 |
| 6 | 27 | -31 |
| 7* | 24 | -5 |
| 8* | 23 | -27 |
| 9 | 43 | 42 |
| 10 | 34 | 28 |
| 11 | 47 | 48 |

2, 6, L

| | | |
|---|---|---|
| -10 | 46 | -37 |
| -6 | 95 | -89 |
| -5 | 88 | 84 |
| -4 | 116 | -119 |
| -3 | 90 | -85 |
| -2 | 30 | -28 |
| -1 | 102 | -97 |
| 0 | 82 | 68 |
| 1 | 85 | 82 |
| 2 | 26 | -30 |
| 3 | 75 | -77 |
| 4 | 139 | 136 |
| 5 | 115 | -116 |
| 6* | 24 | -12 |
| 7 | 90 | 89 |
| 8 | 50 | 38 |
| 9 | 43 | 35 |
| 10 | 49 | 43 |

2, 7, L

| | | |
|---|---|---|
| -9 | 77 | 66 |
| -8* | 23 | 24 |
| -7 | 30 | 32 |
| -6* | 24 | -12 |
| -5 | 82 | 78 |
| -4 | 167 | -157 |
| -3* | 22 | 6 |
| -2 | 83 | -79 |
| -1 | 65 | 60 |
| 0 | 76 | -81 |
| 1 | 32 | -35 |
| 2 | 36 | 24 |
| 3 | 39 | -44 |
| 4 | 37 | 22 |
| 5 | 80 | -88 |
| 6* | 23 | 7 |
| 7 | 48 | -41 |
| 8 | 47 | 49 |
| 9 | 53 | -10 |
| 10 | 27 | 27 |

2, 8, L

| | | |
|---|---|---|
| -9 | 94 | -95 |
| -8 | 59 | 60 |
| -7 | 57 | -56 |
| -6* | 24 | 3 |
| -5 | 24 | -17 |
| -4* | 24 | 8 |
| -3 | 203 | -217 |
| -2 | 77 | -80 |
| -1* | 23 | -8 |
| 0 | 79 | -83 |
| 1 | 91 | 77 |
| 2 | 38 | -27 |
| 3 | 92 | 96 |
| 4* | 24 | 12 |
| 5 | 104 | 91 |
| 6* | 22 | 0 |
| 7 | 51 | 49 |
| 8 | 36 | 33 |
| 9 | 32 | 35 |

2, 9, L

| | | |
|---|---|---|
| -8 | 69 | -65 |
| -7* | 21 | -14 |
| -6* | 22 | 1 |
| -5 | 45 | -36 |
| -4 | 82 | 89 |
| -3 | 42 | -40 |
| -2 | 49 | 32 |
| -1 | 44 | 42 |
| 0 | 118 | 124 |
| 1 | 25 | -29 |
| 2 | 84 | 90 |
| 3 | 83 | 73 |
| 4* | 22 | -9 |
| 5 | 46 | 39 |
| 6 | 31 | 34 |
| 7 | 39 | 33 |
| 8 | 34 | -32 |

2, 10, L

| | | |
|---|---|---|
| -9 | 24 | 27 |
| -8* | 15 | -4 |
| -7 | 49 | 38 |
| -6 | 40 | 32 |
| -5* | 21 | 16 |
| -4 | 44 | 34 |
| -3* | 22 | 16 |
| -2* | 22 | -6 |
| -1 | 46 | -55 |
| 0 | 47 | 48 |
| 1* | 22 | -7 |
| 2 | 50 | 48 |
| 3* | 21 | 6 |
| 4* | 19 | 3 |
| 5 | 39 | -48 |
| 6 | 37 | -26 |

2, 11, L

| | | |
|---|---|---|
| -1 | 57 | 48 |
| 0 | 64 | -53 |
| 1 | 29 | -23 |

2, 12, L

| | | |
|---|---|---|
| 0 | 49 | 53 |
| 1 | 22 | -25 |

Table 1 (*cont.*)

3, 1, L

| | | |
|---|---|---|
| -11 | 106 | 114 |
| -10 | 98 | 88 |
| -9 | 61 | 54 |
| -8 | 26 | 15 |
| -7 | 96 | 93 |
| -6 | 141 | -134 |
| -5 | 239 | -236 |
| -4 | 97 | -85 |
| -3 | 314 | -323 |
| -2 | 132 | 145 |
| -1 | 213 | -201 |
| 0 | 166 | -144 |
| 1 | 167 | -159 |
| 2 | 28 | 12 |
| 3 | 300 | -289 |
| 4 | 90 | -86 |
| 5 | 85 | 65 |
| 6* | 24 | 6 |
| 7 | 66 | 66 |
| 8* | 25 | 18 |
| 9 | 84 | 69 |
| 10 | 73 | 66 |
| 11 | 75 | 67 |

3, 2, L

| | | |
|---|---|---|
| -12 | 77 | -82 |
| -11 | 46 | -37 |
| -10 | 62 | -60 |
| -9 | 110 | -100 |
| -8 | 130 | -122 |
| -7 | 34 | 30 |
| -6 | 245 | -223 |
| -5 | 191 | -159 |
| -4 | 55 | 60 |
| -3 | 276 | -282 |
| -2 | 117 | -130 |
| -1 | 220 | -221 |
| 0 | 77 | 80 |
| 1 | 105 | 95 |
| 2 | 338 | 353 |
| 3 | 151 | 147 |
| 4 | 288 | 286 |
| 5 | 174 | 173 |
| 6 | 102 | 108 |
| 7 | 153 | 160 |
| 8 | 32 | 33 |
| 9 | 51 | 40 |
| 10* | 24 | 25 |
| 11 | 32 | 20 |

3, 3, L

| | | |
|---|---|---|
| -11 | 41 | -36 |
| -10 | 43 | -40 |
| -9 | 48 | 11 |
| -8 | 80 | -79 |
| -7 | 48 | 37 |
| -6* | 24 | 1 |
| -5 | 29 | 21 |
| -4 | 77 | -71 |
| -3 | 425 | 447 |
| -2 | 372 | 379 |
| -1 | 134 | 122 |
| 0 | 52 | 55 |
| 1 | 121 | 116 |
| 2 | 110 | 110 |
| 3 | 25 | -4 |
| 4 | 150 | 154 |
| 5* | 25 | -12 |
| 6 | 55 | 44 |
| 7 | 46 | -37 |
| 8 | 47 | -50 |
| 9 | 99 | -91 |
| 10 | 65 | -62 |
| 11 | 69 | -64 |

3, 4, L

| | | |
|---|---|---|
| -11 | 43 | 39 |
| -10 | 30 | -8 |
| -9 | 57 | -53 |
| -8 | 89 | 86 |
| -7 | 60 | 56 |
| -6 | 36 | 34 |
| -5 | 35 | 39 |
| -4 | 116 | 116 |
| -3 | 247 | -232 |
| -2 | 161 | 161 |
| -1 | 30 | 32 |
| 0 | 138 | -129 |
| 1 | 62 | 58 |
| 2 | 42 | -32 |
| 3 | 104 | -102 |
| 4 | 51 | 27 |
| 5 | 101 | 93 |
| 6 | 145 | -156 |

3, 5, L

| | | |
|---|---|---|
| -8 | 97 | -82 |
| -7* | 27 | 10 |
| -6 | 35 | -36 |
| -5 | 80 | 78 |
| -4 | 37 | 33 |
| -3 | 158 | 182 |
| -2 | 34 | 25 |
| -1 | 127 | 117 |
| 0 | 95 | 94 |
| 1 | 70 | -63 |
| 2 | 41 | 34 |
| 3 | 38 | -37 |
| 4 | 44 | -34 |
| 5 | 77 | 71 |
| 6 | 126 | 129 |
| 7 | 68 | -70 |
| 8 | 79 | -68 |
| 9 | 33 | -28 |
| 10 | 64 | -53 |

3, 6, L

| | | |
|---|---|---|
| -10 | 96 | 89 |
| -8 | 100 | 95 |
| -7* | 28 | 19 |
| -6 | 58 | 56 |
| -5 | 62 | -43 |
| -4 | 183 | 185 |
| -3 | 138 | 138 |
| -2 | 39 | -39 |
| -1* | 24 | -1 |
| 0 | 44 | -36 |
| 1 | 31 | 27 |
| 2 | 139 | -140 |
| 3 | 75 | -78 |
| 4 | 172 | -176 |
| 5 | 81 | -90 |
| 6 | 74 | -74 |
| 7* | 26 | -19 |
| 8 | -29 | -36 |

3, 7, L

| | | |
|---|---|---|
| -10 | 39 | 33 |
| -9 | 47 | 40 |
| -8 | 34 | 32 |
| -7 | 37 | -31 |
| -6 | 51 | 54 |
| -5 | 189 | -188 |
| -4 | 89 | -76 |
| -3* | 27 | 11 |
| -2* | 27 | -8 |
| -1 | 103 | -103 |
| 0 | 106 | -106 |
| 1 | 84 | -88 |
| 2 | 100 | -105 |
| 3 | 32 | 17 |
| 4 | 71 | -67 |
| 5* | 28 | 13 |
| 6* | 26 | 13 |
| 7* | 24 | 24 |
| 8 | 54 | 31 |

3, 8, L

| | | |
|---|---|---|
| -11 | 32 | 20 |
| -10 | 44 | -39 |
| -7 | 48 | -33 |
| -6* | 28 | 18 |
| -5 | 69 | -67 |
| -4* | 28 | -17 |
| -3* | 27 | -8 |
| -2 | 34 | 26 |
| -1* | 28 | -39 |
| 0 | 71 | 76 |
| 1* | 28 | -10 |
| 2 | 38 | -38 |
| 3 | 41 | 29 |
| 4* | 27 | 15 |
| 5 | 64 | 53 |

3, 9, L

| | | |
|---|---|---|
| -10 | 42 | 37 |
| -9 | 27 | 17 |
| -8* | 22 | 17 |
| -7* | 24 | -14 |
| -6* | 26 | -1 |
| -5 | 51 | -44 |
| -4* | 27 | -25 |
| -3* | 27 | -21 |
| -2* | 28 | -3 |
| -1* | 28 | 7 |
| 0* | 27 | 10 |
| 1 | 34 | 23 |
| 2 | 34 | -30 |
| 3 | 32 | -15 |
| 4 | 45 | -29 |
| 5 | 53 | 40 |

3,10, L

| | | |
|---|---|---|
| -8 | 23 | -3 |
| -7* | 20 | -17 |
| -6* | 22 | -21 |
| -5* | 23 | -4 |
| -4* | 24 | -21 |
| -3* | 25 | 13 |
| -2* | 25 | -21 |
| -1 | 75 | -70 |
| 0 | 59 | 46 |
| 1* | 24 | 19 |
| 2 | 42 | 43 |
| 3* | 22 | 13 |
| 4 | 44 | 41 |

3,11, L

| | | |
|---|---|---|
| -2 | 41 | 16 |
| -1 | 82 | 72 |
| 0 | 29 | -23 |
| 1 | 31 | 33 |

4, 0, L

| | | |
|---|---|---|
| -13 | 54 | 74 |
| -12 | 83 | 102 |
| -10 | 65 | 59 |
| -9 | 76 | 76 |
| -8 | 29 | -32 |
| -7 | 73 | 67 |
| -6 | 128 | 114 |
| -5 | 141 | 136 |
| -4 | 43 | 35 |
| 0 | 175 | -181 |
| 1 | 278 | -295 |
| 2 | 174 | -186 |
| 3 | 219 | -239 |
| 4 | 54 | -52 |

4, 0, L

| | | |
|---|---|---|
| 5 | 93 | -122 |
| 6 | 147 | -148 |
| 7 | 42 | -46 |
| 8 | 46 | 39 |

4, 1, L

| | | |
|---|---|---|
| -11 | 44 | 31 |
| -10 | 51 | 41 |
| -9 | 38 | -26 |
| -8 | 58 | 55 |
| -7 | 166 | -167 |
| -6 | 55 | -51 |
| -5* | 18 | -10 |
| -4 | 216 | -223 |
| -3 | 179 | -195 |
| -2 | 191 | -208 |
| -1 | 38 | 41 |
| 0 | 158 | -167 |
| 1 | 160 | 156 |
| 2 | 138 | -138 |
| 3* | 24 | 0 |
| 4 | 31 | -17 |
| 5 | 65 | 70 |
| 6 | 84 | 81 |
| 7* | 30 | 35 |
| 8 | 99 | 84 |
| 9 | 56 | 56 |
| 10 | 87 | 72 |

4, 2, L

| | | |
|---|---|---|
| -7 | 71 | -63 |
| -6* | 23 | 7 |
| -5 | 65 | -72 |
| -4 | 344 | 367 |
| -3 | 206 | -215 |
| -2 | 56 | -50 |
| -1 | 64 | -61 |
| 0 | 75 | 62 |
| 1 | 85 | -84 |
| 2 | 115 | 116 |
| 3 | 112 | 102 |
| 4 | 95 | -81 |
| 5 | 63 | 66 |
| 6 | 66 | -50 |
| 7 | 61 | 53 |
| 8* | 29 | -8 |
| 9 | 30 | 17 |
| 10 | 24 | -11 |

4, 3, L

| | | |
|---|---|---|
| -12 | 54 | 34 |
| -9 | 66 | 57 |
| -8* | 30 | -11 |
| -7* | 28 | 2 |
| -6 | 127 | -121 |
| -5 | 26 | -13 |
| -4 | 359 | -368 |
| -3 | 116 | 116 |
| -2 | 112 | -94 |
| -1 | 122 | -101 |
| 0 | 34 | 37 |
| 1 | 26 | -27 |
| 2* | 26 | -11 |
| 3 | 47 | 33 |
| 4 | 75 | 74 |
| 5 | 114 | -111 |
| 6 | 95 | 96 |
| 7 | 60 | 50 |
| 8 | 54 | 47 |
| 9 | 69 | 56 |
| 10 | 51 | 45 |

4, 4, L

| | | |
|---|---|---|
| -11 | 89 | -82 |
| -10 | 49 | -36 |
| -9 | 120 | -120 |
| -8* | 30 | 15 |
| -7 | 121 | -109 |
| -6 | 48 | 34 |
| -5 | 66 | 63 |
| -4 | 69 | -60 |
| -3* | 23 | -7 |
| -2 | 146 | -143 |
| -1 | 140 | 145 |
| 0* | 29 | 9 |
| 1 | 208 | 203 |
| 2 | 79 | 69 |
| 3 | 266 | 265 |
| 4 | 147 | 151 |
| 5 | 50 | 40 |

4, 5, L

| | | |
|---|---|---|
| -8 | 51 | 45 |
| -7 | 77 | -76 |
| -6 | 178 | 170 |
| -5 | 39 | 25 |
| -4 | 95 | 82 |
| -3 | 103 | 85 |
| -2 | 103 | 95 |
| -1 | 105 | 98 |
| 0* | 26 | 18 |
| 1 | 112 | 114 |
| 2 | 110 | -95 |
| 3 | 114 | 108 |
| 4 | 80 | -72 |
| 5* | 30 | -17 |
| 6 | 42 | -34 |
| 7 | 39 | -34 |
| 8 | 86 | -72 |
| 9 | 60 | -52 |

4, 6, L

| | | |
|---|---|---|
| -10 | 61 | -56 |
| -9 | 47 | 38 |
| -8* | 30 | 26 |
| -7 | 54 | -37 |
| -6 | 53 | 52 |
| -5 | 95 | 87 |
| -4* | 29 | 7 |
| -3 | 77 | -65 |
| -2* | 29 | 35 |
| -1 | 136 | -135 |
| 0 | 46 | 35 |
| 1 | 32 | -11 |
| 2 | 40 | -35 |
| 3 | 40 | 24 |
| 4 | 35 | 18 |
| 5 | 53 | -59 |
| 6 | 53 | -60 |
| 7* | 26 | 1 |
| 8 | 43 | -38 |

4, 7, L

| | | |
|---|---|---|
| -9 | 35 | -15 |
| -8* | 29 | -13 |
| -7* | 30 | 6 |
| -6* | 30 | 12 |
| -5 | 90 | 78 |
| -4 | 55 | 37 |
| -3* | 30 | -25 |
| -2 | 45 | 41 |
| -1 | 59 | 56 |
| 0 | 51 | -48 |
| 1 | 46 | -43 |
| 2 | 54 | 47 |
| 3 | 40 | -30 |
| 4* | 30 | -25 |
| 5 | 35 | 25 |

4, 8, L

| | | |
|---|---|---|
| -11 | 28 | 35 |
| -6 | 61 | -59 |
| -5 | 104 | 103 |
| -4* | 30 | 6 |
| -3 | 35 | 27 |
| -2 | 59 | -50 |
| -1* | 30 | -26 |
| 0* | 30 | 25 |
| 1 | 70 | -71 |
| 2* | 30 | -14 |
| 3 | 51 | -51 |
| 4* | 27 | -19 |
| 5 | 99 | -83 |

4, 9, L

| | | |
|---|---|---|
| -9 | 45 | -16 |
| -8* | 23 | -11 |
| -7* | 25 | 7 |
| -6* | 27 | -7 |
| -5 | 64 | -56 |
| -4 | 41 | -34 |
| -3 | 33 | -8 |
| -2 | 61 | -56 |
| -1 | 43 | 36 |
| 0 | 71 | -80 |
| 1* | 28 | -15 |
| 2* | 27 | -8 |
| 3 | 42 | 29 |

4,10, L

| | | |
|---|---|---|
| -2 | 35 | 25 |
| -1* | 25 | -3 |

4,11, L

| | | |
|---|---|---|
| -5 | 29 | 23 |
| -4 | 28 | -24 |
| -3 | 35 | -34 |
| -2* | 19 | 9 |
| -1 | 29 | 27 |
| 0* | 18 | 3 |
| 1* | 16 | 1 |
| 2 | 24 | 26 |

5, 1, L

| | | |
|---|---|---|
| -9 | 58 | -56 |
| -8 | 105 | 103 |
| -7 | 112 | 107 |
| -6* | 25 | -14 |
| -5 | 118 | 113 |
| -4 | 52 | -52 |
| -3 | 148 | 157 |
| -2 | 38 | 33 |
| -1 | 36 | 37 |
| 0 | 93 | -92 |
| 1 | 105 | 100 |
| 2 | 91 | -79 |
| 3 | 67 | -72 |
| 4* | 33 | -17 |
| 5 | 77 | -81 |
| 6* | 34 | -15 |
| 7 | 58 | -47 |
| 8 | 46 | -48 |
| 9 | 58 | -48 |

5, 2, L

| | | |
|---|---|---|
| -10 | 55 | 47 |
| -8 | 50 | 31 |
| -7* | 29 | -6 |
| -6* | 27 | -5 |
| -5* | 24 | -18 |
| -4 | 47 | -38 |
| -3* | 19 | -2 |
| -2 | 42 | -28 |
| -1 | 76 | -78 |

5, 2, L

| | | |
|---|---|---|
| 0 | 140 | -154 |
| 1 | 106 | -105 |
| 2 | 203 | -205 |
| 3 | 79 | -80 |
| 4 | 44 | -27 |
| 5 | 46 | -41 |
| 6* | 34 | -8 |
| 7 | 39 | 35 |

5, 3, L

| | | |
|---|---|---|
| -7 | 54 | -55 |
| -6* | 27 | -13 |
| -5 | 107 | -110 |
| -4 | 154 | -163 |
| -3 | 92 | -91 |
| -2 | 35 | 27 |
| -1 | 30 | -19 |
| 0 | 42 | 27 |
| 1 | 99 | 102 |
| 2* | 31 | -14 |
| 3 | 56 | -42 |
| 4 | 41 | 38 |
| 5 | 81 | 94 |
| 6 | 42 | 49 |
| 7 | 68 | 59 |
| 8 | 48 | 47 |
| 9 | 34 | 38 |

5, 4, L

| | | |
|---|---|---|
| -11 | 52 | 36 |
| -10* | 33 | -23 |
| -6 | 124 | -124 |
| -5 | 113 | 113 |
| -4 | 61 | 63 |
| -3* | 28 | -28 |
| -2* | 28 | 16 |
| -1 | 32 | 11 |
| 0* | 36 | -11 |
| 1 | 41 | 53 |
| 2 | 54 | 60 |
| 3 | 81 | -63 |
| 4 | 43 | 29 |
| 5* | 34 | 12 |
| 6 | 40 | 30 |

5, 5, L

| | | |
|---|---|---|
| -7 | 45 | 41 |
| -6 | 40 | -30 |
| -5 | 230 | -225 |
| -4* | 31 | 28 |
| -3* | 31 | 10 |
| -2* | 31 | -19 |
| -1* | 31 | 20 |
| 0 | 57 | 52 |
| 1* | 32 | -20 |
| 2 | 69 | 59 |
| 3 | 45 | 41 |
| 4 | 65 | -77 |
| 5 | 43 | 34 |
| 6 | 41 | 36 |
| 7 | 37 | 20 |
| 8 | 33 | 34 |

5, 6, L

| | | |
|---|---|---|
| -10 | 76 | -60 |
| -8 | 63 | -56 |
| -7 | 103 | 100 |
| -6 | 57 | -46 |
| -5 | 36 | 22 |
| -4 | 42 | -33 |
| -3 | 41 | 45 |
| -2 | 60 | 51 |
| -1* | 33 | -3 |
| 0 | 116 | 121 |
| 1* | 34 | 7 |
| 2 | 82 | 77 |
| 3 | 39 | 26 |
| 4 | 120 | 109 |
| 5 | 43 | -22 |

5, 7, L

| | | |
|---|---|---|
| -7 | 62 | 54 |
| -6 | 63 | 57 |
| -5* | 34 | 13 |
| -4* | 34 | -12 |
| -3 | 61 | 53 |
| -2* | 34 | -18 |
| -1 | 59 | 59 |
| 0 | 39 | 35 |
| 1 | 39 | -13 |
| 2 | 38 | -15 |
| 3 | 69 | -70 |
| 4 | 37 | -33 |

5, 8, L

| | | |
|---|---|---|
| -2 | 62 | -67 |
| -1* | 33 | -31 |

6, 0, L

| | | |
|---|---|---|
| -9 | 120 | 135 |
| -4 | 66 | 80 |
| -3* | 21 | -27 |
| -2 | 68 | 72 |
| -1 | 47 | 51 |
| 0 | 60 | 68 |
| 1 | 62 | 65 |
| 2 | 83 | 97 |

6, 1, L

| | | |
|---|---|---|
| 0* | -22 | -46 |
| 1 | 66 | -78 |
| 2* | 23 | 17 |
| 3* | 22 | -22 |
| 4 | 65 | -70 |

6, 2, L

| | | |
|---|---|---|
| -1 | 49 | 58 |
| 0 | 50 | -55 |
| 1 | 53 | -61 |
| 2* | 27 | -13 |
| 3 | 60 | 54 |

6, 3, L

| | | |
|---|---|---|
| -8 | 54 | -11 |
| -7* | 32 | -29 |
| -6 | 173 | 188 |
| -5* | 32 | -67 |
| -4* | 32 | 53 |
| -3* | 32 | -45 |
| -2* | 32 | 37 |
| -1 | 104 | -120 |
| 0 | 73 | 77 |
| 1 | 79 | -85 |
| 2 | 64 | -65 |
| 3* | 32 | 43 |
| 4 | 62 | -58 |

6, 4, L

| | | |
|---|---|---|
| -8 | 100 | -93 |
| -7* | 41 | -11 |
| -6 | 92 | -88 |
| -5 | 87 | 76 |
| -4* | 41 | -15 |
| -3* | 41 | -40 |
| -2* | 41 | -8 |
| -1 | 84 | -96 |
| 0* | 41 | -12 |
| 1 | 121 | -134 |

7, 1, L

| | | |
|---|---|---|
| -6 | 58 | 42 |
| -5* | 22 | -58 |
| -4* | 22 | -14 |
| -3* | 22 | -1 |
| -2 | 58 | 67 |
| -1 | 42 | -43 |
| 0 | 71 | 93 |

as $2/m$ ($C_{2h}$). The systematic extinction of $h0l$ reflections when $h$ is odd and of $0k0$ when $k$ is odd identified the space group as $P2_1/a$ ($C_{2h}^5$). The unit-cell dimensions, determined from zero level Weissenberg photographs calibrated with sodium chloride, are

$$a=7{\cdot}50\pm0{\cdot}02,\ b=9{\cdot}91\pm0{\cdot}02,\ c=11{\cdot}677\pm0{\cdot}005\ \text{Å};$$
$$\beta=108{\cdot}02\pm0{\cdot}14°.$$

The assumption of four molecules per unit cell leads to a calculated density of 1·365 g.cm$^{-3}$ in good agreement with the observed value of 1·354 g.cm$^{-3}$ measured by flotation in potassium iodide solution.

Multiple film equi-inclination Weissenberg photographs were collected, using Cu $K\alpha$ radiation, of levels $Hkl$ for $0\leq H\leq 5$ and $hKl$ for $0\leq K\leq 5$. The intensities were estimated visually with the use of standard scales. A total of 936 independent diffraction maxima were observed; within this range 197 reflections were below the minimum observed intensity $M$; they were assigned the value $M$, and are identified with an asterisk in Table 1. The preliminary data-processing, scaling and correction for Lorentz and polarization factors were carried out with the use of programs written for the IBM 7090. Correlation of the two axes sets were made with the use of reflections common to the two sets. The weighting scheme for the least-squares refinements were chosen as $w=1/\sigma^2$, where $\sigma=0{\cdot}20\,I_o$ for $I_o\leq 150$ and $\sigma=0{\cdot}09\,I_o$ for $I_o>150$. No corrections for absorption or extinction were made. Preliminary scale and temperature factors were estimated by Wilson's method.

## Structure determination

A three-dimensional Patterson function was computed with the use of coefficients sharpened so that the average $F_{hkl}^2$ was independent of sin $\theta$. The origin peak was partially removed, and the unobserved reflections were included in this calculation in order to improve the resolution of the map. Examination of the Harker plane at $y=\frac{1}{2}$ and the Harker line at $x=\frac{1}{2}$, $z=0$ gave three possible sets of coordinates for the Cl atom. Inspection of the area around the origin clearly indicated the positions of the six C atoms of the benzene ring, and also atom C(8), but this examination did not fix atoms 9, 10, and 11 with certainty (Fig. 1). Packing considerations and calculations of interatomic vectors for this part of the molecule then quickly eliminated the two incorrect sets of Cl coordinates. A structure factor calculation for the 480 largest reflections including contributions from Cl and 7 C atoms of the molecule gave $R=\Sigma||F_o|-|F_c||/\Sigma|F_o|=0{\cdot}42$. A three-dimensional Fourier synthesis was then calculated using only those $F_{hkl}$'s whose signs were regarded as probably correct in the above calculation. This map showed the three additional atoms in the structure having peak heights (on a relative scale) at 49, 26 and 28, respectively, as compared with 115 for Cl and 34–42 for the C atoms. It was not possible at this stage to distinguish between the carbon atom of the $CH_3$ group and the oxygen atom, but packing considerations led us to assign atom 10 as O and atom 11 as C. This choice was independently tested later, as described below.

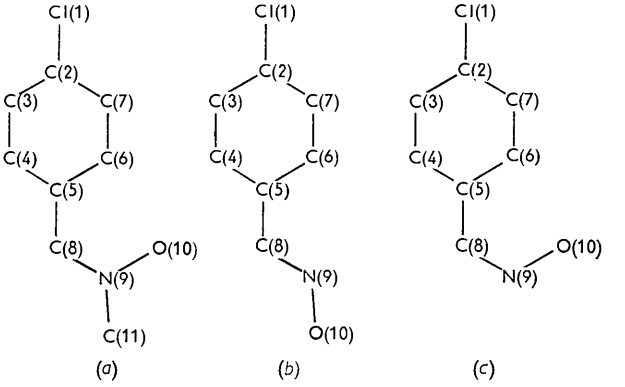


Fig. 1. Numbering diagram for the three oximes discussed in this paper. (*a*) *N*-Methyl-*p*-chlorobenzaldoxime, (*b*) '*syn*'-*p*-chlorobenzaldoxime and (*c*) '*anti*'-*p*-chlorobenzaldoxime

All observed reflections were then introduced. Three cycles of least-squares refinement with individual isotropic temperature factors reduced the disagreement index to $R=0{\cdot}184$. Four H atoms on the benzene ring were introduced at expected positions, and were included in the refinements as fixed atoms. Two cycles of anisotropic refinement then yielded $R=0{\cdot}097$, $R_{F^2}=\Sigma|F_o^2-F_c^2|/\Sigma F_o^2=0{\cdot}159$, and $R_{wF^2}=\sqrt{(\Sigma w|F_o^2-F_c^2|^2/\Sigma wF_o^4)}=0{\cdot}25$. All distance parameter shifts were less than their respective standard deviations in the final cycle of this full matrix refinement. Only one coordinate shifted by as much as 0·007 Å, and the average absolute shift of coordinates was less than 0·002 Å in this final cycle of refinement. The final shifts of diagonal thermal parameters ($\beta_{ii}$) were less than the standard deviations of the $\beta_{ii}$ by a factor of four.

This low disagreement factor suggested that our choice of the identities of atoms 10 and 11 had been correct. Furthermore, the individual atomic thermal parameters had behaved normally throughout the refinements. Nevertheless, we made a further impartial test of the assignment of atom 10 as O and atom 11 as C (Table 2). Fixed atom contributions were calculated for atoms 1 to 9, and atoms 10 and 11 were each given the form factor of oxygen. The individual isotropic temperature factors were then refined. It would thus be expected that a correct choice of the identities of atoms 10 and 11 would lower the disagreement factor and improve the temperature factor, while the wrong choice would have just the opposite effect. Indeed this was the case: choice II increased the refined $R$ and gave an ab-

*Table 2. Test of the assignment of atoms* 10 *and* 11

| Condition | Isotropic temperature factor Initial | Final | $R=\Sigma\|F_o\|-\|F_c\|/\Sigma\|F_o\|$ Initial | Final |
|---|---|---|---|---|
| I Atom 10=oxygen | 4·0 | 5·06 | 0·178 | 0·111 |
| Atom 11=oxygen | 4·0 | 8·93 | | |
| II Atom 10=carbon | 5·06 | 1·75 | 0·148 | 0·134 |
| Atom 11=oxygen | 8·93 | 10·42 | | |
| III Atom 10=oxygen | 5·06 | 5·24 | 0·118 | 0·103 |
| Atom 11=carbon | 8·93 | 5·10 | | |

normally large thermal parameter for atom 11, while choice III resulted in reasonable temperature factors and a decrease in the refined value of $R$ (Table 2).

We then felt confident that the structure was determined correctly, and that the configuration was that of the '*anti*' oxime. Confirmatory evidence of an independent kind was obtained from a final three-dimensional difference electron density map that showed all the H atoms of the structure. A composite projection of this map is shown in Fig. 2. No false peaks higher than 0·2 e.Å$^{-3}$ occurred in this map. Introduction of these eight H atoms, followed by one cycle of isotropic least-squares refinement with the use of observed reflections only lowered $R$ to 0·085, $R_{F^2}$ to 0·143, and $R_{wF^2}$ to 0·224. In Table 1 we list the observed and calculated structure factors including unobserved reflections which were not used in the least-squares refinement.

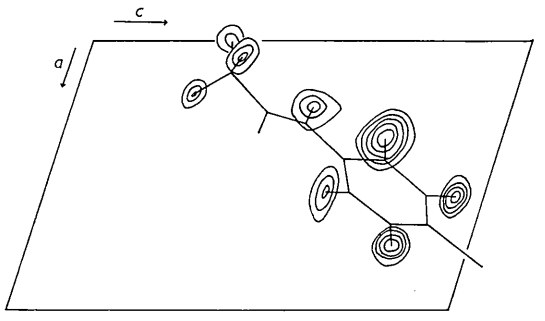


Fig. 2. Composite map on (010) of the electron density through the centers of the hydrogen atoms from the final difference map. A skeleton of the molecule is superposed. The contours are at intervals of 0·1 e.Å$^{-3}$, starting at 0·3 e.Å$^{-3}$.

## Discussion of the structure

The final structure parameters are given in Tables 3 and 4, and a summary of the disagreement factor $R$ is given in Table 5. The molecular packing as viewed along the $b$ axis is shown in Fig. 3 and the numbering diagram in Fig. 1(*a*). Bond distances and angles with their standard deviations were computed using the full variance–covariance matrix from the

Table 3. *Final atomic parameters and their standard deviations for N-methyl-p-chlorobenzaldoxime*

| Atom | $x$ | $y$ | $z$ | $\sigma(x)$ | $\sigma(y)$ | $\sigma(z)$ |
|---|---|---|---|---|---|---|
| Cl(1) | 0·8464 | 0·1341 | 1·0410 | 0·0004 | 0·0002 | 0·0002 |
| C(2) | 0·6914 | 0·1122 | 0·8936 | 0·0010 | 0·0006 | 0·0005 |
| C(3) | 0·5834 | −0·0011 | 0·8675 | 0·0011 | 0·0006 | 0·0006 |
| C(4) | 0·4617 | −0·0198 | 0·7521 | 0·0010 | 0·0006 | 0·0005 |
| C(5) | 0·4499 | 0·0780 | 0·6621 | 0·0008 | 0·0005 | 0·0005 |
| C(6) | 0·5675 | 0·1907 | 0·6930 | 0·0009 | 0·0006 | 0·0005 |
| C(7) | 0·6883 | 0·2081 | 0·8093 | 0·0010 | 0·0006 | 0·0006 |
| C(8) | 0·3133 | 0·0468 | 0·5452 | 0·0009 | 0·0006 | 0·0005 |
| N(9) | 0·2685 | 0·1280 | 0·4522 | 0·0008 | 0·0004 | 0·0004 |
| O(10) | 0·3439 | 0·2434 | 0·4480 | 0·0007 | 0·0004 | 0·0004 |
| C(11) | 0·1196 | 0·0849 | 0·3397 | 0·0011 | 0·0007 | 0·0006 |
| | | | | $B$* | | |
| H(3) | 0·604 | −0·065 | 0·934 | 2·2 | | |
| H(4) | 0·361 | −0·097 | 0·724 | 0·6 | | |
| H(6) | 0·553 | 0·257 | 0·629 | 3·1 | | |
| H(7) | 0·777 | 0·285 | 0·827 | 1·3 | | |
| H(8) | 0·244 | −0·053 | 0·536 | 2·8 | | |
| H(11′) | 0·075 | −0·013 | 0·337 | 6·5 | | |
| H(11″) | 0·175 | 0·100 | 0·268 | 5·6 | | |
| H(11‴) | 0·016 | 0·155 | 0·327 | 5·4 | | |

Scale factor: 2·208 ± 0·009†

* Little significance can be attached to the isotropic temperature factors $B$ given for the H atoms. In general the standard errors were comparable to the $B$'s themselves.

† The scale factor relates the absolute scale of the calculated $F$'s and the relative scale of the observed $F$'s, $|F_{rel}|=|F_{abs}|.s$.

Table 4. *Final anisotropic thermal parameters for N-methyl-p-chlorobenzaldoxime*

The anisotropic thermal parameters are in the form:
$\exp\{-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})\}$

| Atom | $\beta_{11}$ | $\beta_{22}$ | $\beta_{33}$ | $\beta_{12}$ | $\beta_{13}$ | $\beta_{23}$ |
|---|---|---|---|---|---|---|
| Cl(1) | 0·0395 | 0·0176 | 0·0085 | −0·0017 | −0·0017 | −0·0010 |
| C(2) | 0·0269 | 0·0122 | 0·0077 | 0·0009 | 0·0028 | −0·0006 |
| C(3) | 0·0337 | 0·0104 | 0·0096 | 0·0039 | 0·0063 | 0·0006 |
| C(4) | 0·0287 | 0·0096 | 0·0107 | −0·0016 | 0·0065 | −0·0003 |
| C(5) | 0·0195 | 0·0094 | 0·0093 | 0·0020 | 0·0059 | 0·0003 |
| C(6) | 0·0207 | 0·0093 | 0·0096 | −0·0010 | 0·0050 | 0·0012 |
| C(7) | 0·0241 | 0·0100 | 0·0114 | −0·0009 | 0·0043 | −0·0007 |
| C(8) | 0·0253 | 0·0097 | 0·0095 | 0·0020 | 0·0062 | −0·0002 |
| N(9) | 0·0233 | 0·0100 | 0·0095 | 0·0011 | 0·0062 | −0·0002 |
| O(10) | 0·0258 | 0·0122 | 0·0113 | −0·0015 | 0·0045 | 0·0027 |
| C(11) | 0·0303 | 0·0159 | 0·0088 | 0·0030 | 0·0025 | 0·0015 |

least-squares refinement and are given in Table 6, and shown in Figs. 4 and 5. We feel that the reported standard deviations should be multiplied by a factor of 2 or 3 to obtain a reasonable level of significance.

Table 5. *Final values of* $R=\Sigma||F_o|-|F_c||/\Sigma|F_o|$ *for N-methyl-p-chlorobenzaldoxime*

| *hkl* | *R* | Range of sin θ | *R* |
|---|---|---|---|
| All orders | 0·089 | 0·00 to 0·40 | 0·074 |
| *h* even | 0·088 | 0·40 to 0·50 | 0·065 |
| *h* odd | 0·090 | 0·50 to 0·60 | 0·060 |
| *k* even | 0·088 | 0·60 to 0·65 | 0·077 |
| *k* odd | 0·090 | 0·65 to 0·70 | 0·096 |
| *l* even | 0·086 | 0·70 to 0·75 | 0·105 |
| *l* odd | 0·092 | 0·75 to 0·80 | 0·121 |
| *k*+*l* even | 0·087 | 0·80 to 0·85 | 0·136 |
| *k*+*l* odd | 0·091 | 0·85 to 0·90 | 0·149 |
| *h*+*l* even | 0·093 | 0·90 to 1·00 | 0·177 |
| *h*+*l* odd | 0·086 | | |
| *h*+*k* even | 0·083 | | |
| *h*+*k* odd | 0·096 | | |
| *h*+*k*+*l* even | 0·094 | | |
| *h*+*k*+*l* odd | 0·085 | | |

Overall disagreement $R=0·085$ for $F_o$ only, and $R=0·108$ if 197 unobserved $F$'s are included.

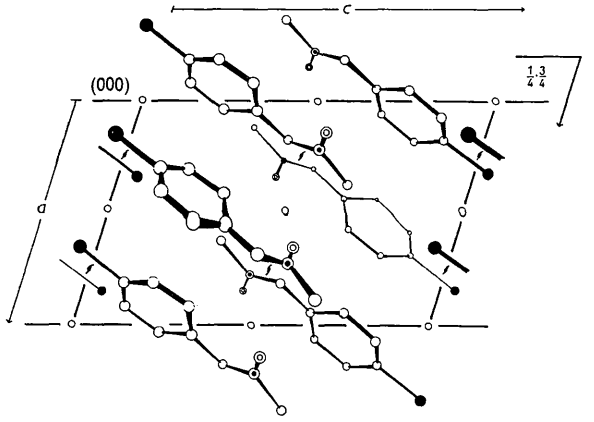


Fig. 3. The structure of *N*-methyl-*p*-chlorobenzaldoxime viewed along the *b* axis. Black circles represent Cl atoms, open circles C atoms, circles with a dot N atoms and double circles O atoms. The origin is at (000).

Table 6. *Intramolecular bonds and angles in N-methyl-p-chlorobenzaldoxime*

| Atoms | Distance | Standard deviation* |
|---|---|---|
| Cl(1)–C(2) | 1·768 Å | ±0·007 |
| C(2)–C(3) | 1·363 | 0·009 |
| C(3)–C(4) | 1·388 | 0·009 |
| C(4)–C(5) | 1·411 | 0·008 |
| C(5)–C(6) | 1·401 | 0·008 |
| C(6)–C(7) | 1·391 | 0·008 |
| C(7)–C(2) | 1·363 | 0·008 |
| C(5)–C(8) | 1·464 | 0·008 |
| C(8)–N(9) | 1·309 | 0·007 |
| N(9)–O(10) | 1·284 | 0·006 |
| N(9)–C(11) | 1·501 | 0·008 |
| C(3)–H(3) | 0·98 | |
| C(4)–H(4) | 1·06 | |
| C(6)–H(6) | 0·98 | |
| C(7)–H(7) | 0·99 | |
| C(8)–H(8) | 1·11 | |
| C(11)–H(11′) | 1·02 | |
| C(11)–H(11″) | 1·05 | |
| C(11)–H(11‴) | 1·01 | |

| Atoms | Angle | Standard deviation* |
|---|---|---|
| Cl(1)–C(2)–C(3) | 119·1° | ±0·5° |
| C(2)–C(3)–C(4) | 119·6 | 0·5 |
| C(3)–C(4)–C(5) | 120·2 | 0·5 |
| C(4)–C(5)–C(6) | 117·8 | 0·5 |
| C(5)–C(6)–C(7) | 121·3 | 0·5 |
| C(6)–C(7)–C(2) | 118·6 | 0·6 |
| C(7)–C(2)–C(3) | 122·5 | 0·6 |
| Cl(1)–C(2)–C(7) | 118·5 | 0·5 |
| C(6)–C(5)–C(8) | 127·8 | 0·5 |
| C(4)–C(5)–C(8) | 114·5 | 0·5 |
| C(5)–C(8)–N(9) | 124·7 | 0·5 |
| C(8)–N(9)–O(10) | 125·2 | 0·5 |
| C(8)–N(9)–C(11) | 118·8 | 0·5 |
| O(10)–N(9)–C(11) | 115·9 | 0·5 |

* The standard deviations are calculated using the full variance–covariance matrix from the least-squares refinement. The values should be multiplied by 2 or 3 for significant uncertainty.

The Cl atom and the benzene ring are coplanar to within 0·01 Å. C(8) is 0·04 Å from the approximate plane through Cl and the ring ($X-0·58Y-0·30Z+1·67=0$), while N(9), O(10), and C(11) are removed 0·17, 0·22, and 0·24 Å, respectively, from this plane.

The oxime group has caused a slight displacement of the C(5)–C(8) bond that is significantly out of

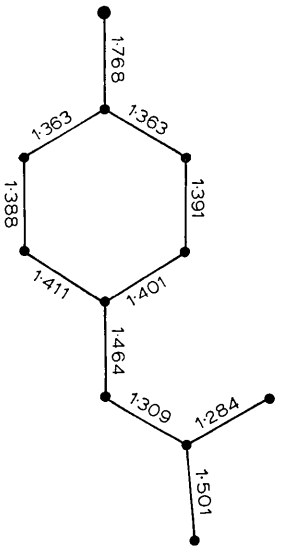


Fig. 4. Bond lengths in *N*-methyl-*p*-chlorobenzaldoxime.

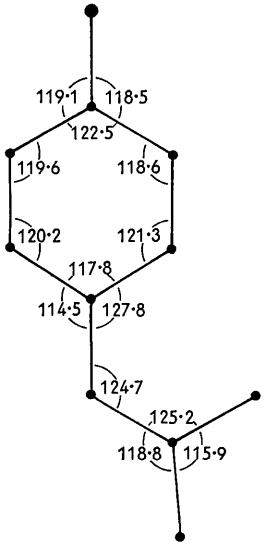


Fig. 5. Bond angles in *N*-methyl-*p*-chlorobenzaldoxime.

the line bisecting the benzene ring: the angle C(6)–C(5)–C(8) is 127·8°. The C–C bonds of the ring are essentially normal; the average value for the C–C distances of this ring is 1·386 Å and the average of those angles not involving H atoms of the ring is 120·0°. The C(5)–C(8) distance is slightly larger in the *N*-methyl compound than in either '*syn*' or '*anti*'-*p*-chlorobenzaldoxime. The N(9)–O(10) distance of 1·284 Å, remarkably shorter than that reported for non-alkylated oximes, indicates multiple bonding of a larger degree than that in the oximes themselves. The distance N(9)–C(11) = 1·501 Å is only slightly longer than the value reported for 3-covalent nitrogen (1·472 Å). Finally, the bond distances N(9)–O(10) = 1·284 Å and N(9)–C(11) = 1·501 Å provide additional strong support for the assignment of the '*anti*' configuration of *N*-methyl-*p*-chlorobenzaldoxime.

Short intramolecular distances are C(5) · · · O(10) = 2·889, C(6) · · · O(10) = 2·883, C(8) · · · O(10) = 2·302, C(11) · · · O(10) = 2·364, C(8) · · · C(11) = 2·421 and C(6) · · · C(8) = 2·417 Å, all ±0·008 Å. The first two distances may be compared to the non-bonded distance of 1·40 + 1·55 = 2·95 Å which might be expected from van der Waals radii of O and C. No abnormally short intermolecular contacts are present. The closest Cl · · · Cl distance is 3·66 Å, and the closest intermolecular C · · · O distance is 3·24 Å. The angle between normals to the benzene ring and the C(8)N(9)O(10) plane is 5° 37′. This small twist is distributed between the C(5)–C(8) and C(8)–N(9) bonds. The angle between the normals to the C(5)C(8)N(9) plane and the benzene plane indicates that the C(8)N(9) bond is twisted around C(5)C(8) by 6° 4′. Also, the angle between the normals to the benzene ring and the average plane defined by C(5)C(8)N(9)O(10) is 4° 27′. These twists place atoms C(8), N(9), O(10) and C(11) slightly above the plane of the ring, provided that we define the positive direction by a vector from the origin of the unit cell toward the plane. If a coordinate $Z$ is defined perpendicular to the crystallographic $x=X$ and $y=Y$ axes, the equation of the plane of the ring is

$$X - 0{\cdot}5749\,Y - 0{\cdot}3026\,Z + 1{\cdot}6709 = 0$$

where the units are in Å. The distances of atoms from this plane are C(8), 0·04 Å; N(9), 0·17 Å; O(10), 0·22 Å; and C(11), 0·24 Å.

## Refinement of '*syn*'- and '*anti*'-*p*-chlorobenzaldoximes

The three-dimensional structure determination of '*syn*'-*p*-chlorobenzaldoxime, reported by Jerslev (1957), was carried out by means of three-dimensional Fourier syntheses. It was felt that these original data merited further refinement so that comparison could be made between the structures of '*syn*'-*p*-chlorobenzaldoxime, *N*-methyl-*p*-chlorobenzaldoxime, and an *O*-methyl derivative of a closely related oxime to be studied in the near future. The present refinement of '*syn*'-*p*-chlorobenzaldoxime was done by the least-squares method starting from the values reported by Jerslev (1958). The weighting scheme is as follows: $w = 1/\sigma^2$, where $\sigma = F_o^2$ for $F_o^2 \leq 4.F_{\min}^2$ and $\sigma = 0{\cdot}10.F_o^2$ for $F_o^2 > 4.F_{\min}^2$. Two cycles of three-dimensional least-squares refinement in which individual isotropic temperature factors were assumed reduced $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ to 0·151. Introduction of the four

Table 7. *List of observed and calculated structure factors for 'syn'-p-chlorobenzaldoxime*

The three columns of each group contain from left to right the values of $l$, 10 $F_o$ and 10 $F_c$.
The values of $l$ followed by an asterisk indicate reflections below the observable limit

-7,0,L

| | | |
|---|---|---|
| 2 | 33 | 27 |
| 4 | 49 | 51 |
| 6 | 17 | -21 |
| 8 | 44 | 43 |
| 10 | 42 | -42 |
| 12 | 27 | 30 |
| 14* | 0 | 7 |
| 16 | 23 | -41 |

-6,0,L

| | | |
|---|---|---|
| 2* | 0 | 19 |
| 4 | 101 | -58 |
| 6 | 148 | -174 |
| 8 | 143 | 157 |
| 10 | 95 | 48 |
| 12 | 11 | 20 |
| 14 | 19 | -16 |
| 16* | 0 | -17 |
| 18 | 43 | 51 |
| 20 | 29 | -26 |
| 22 | 29 | 30 |

-5,0,L

| | | |
|---|---|---|
| 2 | 133 | 157 |
| 4 | 119 | -134 |
| 6 | 53 | 63 |
| 8 | 33 | 17 |
| 10 | 262 | -332 |
| 12 | 74 | 81 |
| 14 | 30 | -41 |
| 16 | 20 | 24 |
| 18 | 69 | 74 |
| 20 | 75 | -83 |
| 22 | 72 | 57 |

-4,0,L

| | | |
|---|---|---|
| 2 | 201 | 229 |
| 4 | 101 | -111 |
| 6 | 198 | 234 |
| 8 | 95 | 97 |
| 10 | 79 | -81 |
| 12 | 96 | 87 |
| 14 | 147 | -171 |
| 16 | 21 | 38 |
| 18 | 62 | -66 |
| 20 | 82 | -85 |
| 22 | 39 | 44 |
| 24 | 105 | -77 |
| 26 | 59 | 60 |

-3,0,L

| | | |
|---|---|---|
| 2 | 91 | 99 |
| 4 | 191 | -255 |
| 6 | 211 | 234 |
| 8 | 281 | -287 |
| 10 | 102 | 105 |
| 12 | 448 | 457 |
| 14 | 145 | -143 |
| 16 | 140 | 147 |
| 18 | 80 | -92 |
| 20 | 37 | -27 |
| 22 | 179 | 161 |
| 24 | 72 | -72 |
| 26 | 59 | -63 |
| 28 | 75 | -75 |

-2,0,L

| | | |
|---|---|---|
| 2 | 113 | 166 |
| 4 | 177 | -168 |
| 6 | 305 | 300 |
| 8 | 247 | -219 |
| 10 | 202 | 220 |
| 12 | 167 | -167 |
| 14 | 157 | -146 |
| 16 | 292 | 255 |
| 18 | 143 | -129 |
| 20 | 209 | 218 |
| 22 | 25 | -35 |
| 24 | 125 | -137 |
| 26 | 202 | 193 |
| 28* | -0 | -6 |
| 30 | 23 | 20 |

-1,0,L

| | | |
|---|---|---|
| 2 | 92 | -93 |
| 4 | 239 | -276 |
| 6 | 56 | -66 |
| 8 | 334 | -462 |
| 10 | 152 | 159 |
| 12 | 163 | -161 |
| 14* | 0 | 23 |
| 16 | 112 | 112 |
| 18 | 152 | -165 |
| 20 | 145 | 150 |
| 22 | 97 | -94 |
| 24* | 0 | 7 |
| 26 | 41 | 41 |
| 28 | 50 | -50 |
| 30 | 83 | 76 |
| 32 | 39 | -23 |

0,0,L

| | | |
|---|---|---|
| 2 | 119 | -150 |
| 4 | 334 | -360 |
| 6 | 210 | 198 |
| 8 | 508 | -534 |
| 10 | 501 | 465 |
| 12 | 247 | -255 |
| 14 | 267 | -271 |
| 16 | 118 | -127 |
| 18 | 106 | -105 |
| 20 | 138 | 129 |
| 22 | 128 | -133 |
| 24 | 123 | 123 |
| 26 | 55 | 54 |
| 28 | 106 | -110 |
| 30 | 25 | 20 |
| 32 | 34 | -35 |

1,0,L

| | | |
|---|---|---|
| 0 | 197 | -398 |
| 2 | 580 | -732 |
| 4 | 744 | 885 |
| 6 | 180 | 234 |
| 8 | 327 | 412 |
| 10 | 393 | 423 |
| 12 | 290 | -312 |
| 14 | 334 | 417 |
| 16 | 35 | 30 |
| 18 | 112 | -104 |
| 20* | 0 | 4 |
| 22 | 177 | -171 |
| 24 | 51 | 47 |
| 26 | 106 | -115 |
| 28 | 30 | 19 |
| 30 | 70 | 71 |

2, 0,L

| | | |
|---|---|---|
| 0 | 27 | -11 |
| 2 | 891 | -919 |
| 4 | 207 | 173 |
| 6 | 267 | -291 |
| 8 | 62 | -50 |
| 10 | 59 | 61 |
| 12 | 243 | -254 |
| 14 | 225 | 227 |
| 16 | 113 | 96 |
| 18 | 170 | 181 |
| 20 | 60 | 67 |
| 22 | 25 | -30 |
| 24 | 63 | 70 |
| 26 | 55 | -52 |
| 28 | 15 | 20 |
| 30 | 34 | -29 |

3,0,L

| | | |
|---|---|---|
| 0 | 114 | -122 |
| 2 | 168 | 191 |
| 4 | 286 | 301 |
| 6 | 420 | -454 |
| 8 | 103 | 98 |
| 10 | 209 | -219 |
| 12* | 0 | 10 |
| 14 | 277 | 278 |
| 16 | 392 | -413 |
| 18 | 65 | -85 |
| 20 | 46 | 48 |
| 22 | 31 | 30 |
| 24 | 123 | 123 |
| 26 | 29 | -30 |
| 28 | 17 | 18 |

4,0,L

| | | |
|---|---|---|
| 0* | 0 | -6 |
| 2* | 0 | 12 |
| 4 | 39 | 44 |
| 6 | 103 | -124 |
| 8 | 122 | 126 |
| 10 | 48 | -49 |
| 12 | 53 | 63 |
| 14 | 108 | -108 |
| 16 | 51 | -52 |
| 18 | 174 | 179 |
| 20 | 115 | -107 |
| 22 | 18 | -16 |
| 24 | 61 | -49 |
| 26 | 80 | -66 |

5,0,L

| | | |
|---|---|---|
| 0 | 127 | -133 |
| 2 | 76 | 93 |
| 4 | 72 | 93 |
| 6* | 0 | -1 |
| 8 | 138 | 128 |
| 10 | 115 | -79 |
| 12 | 46 | 50 |
| 14 | 43 | -33 |
| 16 | 58 | 72 |
| 18 | 53 | 77 |
| 20 | 15 | -22 |
| 22 | 35 | 15 |

6,0,L

| | | |
|---|---|---|
| 0 | 110 | -111 |
| 2 | 113 | 119 |
| 4 | 119 | -134 |
| 6 | 65 | -74 |
| 8 | 72 | 92 |
| 10* | 0 | -15 |
| 12 | 59 | 69 |
| 16 | 82 | 58 |
| 18 | 14 | 3 |

7,0,L

| | | |
|---|---|---|
| 0 | 17 | -25 |
| 2 | 20 | 28 |
| 4 | 55 | -51 |
| 6 | 42 | 38 |
| 8 | 34 | -34 |
| 10 | 67 | -63 |
| 12 | 20 | 17 |

-7,1,L

| | | |
|---|---|---|
| 2 | 77 | -70 |
| 3 | 69 | 50 |
| 8 | 14 | -1 |
| 9 | 43 | 33 |
| 10 | 75 | 69 |
| 12 | 50 | -43 |

-6, 1,L

| | | |
|---|---|---|
| 1 | 102 | -90 |
| 2 | 34 | -38 |
| 3 | 65 | 64 |
| 4 | 20 | 26 |
| 5 | 33 | -38 |
| 6 | 67 | -67 |
| 7 | 33 | 30 |
| 8 | 51 | 53 |
| 9 | 27 | -23 |
| 10 | 50 | -40 |
| 11 | 45 | -42 |
| 12 | 72 | -70 |
| 13 | 66 | 70 |
| 14 | 124 | 103 |
| 16 | 34 | -29 |
| 17 | 33 | 29 |
| 18 | 11 | 17 |
| 19 | 25 | -18 |
| 20 | 11 | 3 |

-5,1,L

| | | |
|---|---|---|
| 1 | 84 | 92 |
| 2 | 65 | -69 |
| 3* | 0 | 11 |
| 4 | 171 | 138 |
| 5 | 241 | -253 |
| 6 | 24 | 23 |
| 7 | 72 | 72 |
| 8* | 0 | 17 |
| 9 | 41 | -45 |
| 10 | 50 | -43 |
| 11 | 34 | -16 |
| 12 | 24 | -23 |
| 13 | 20 | 7 |
| 14 | 40 | 34 |
| 15 | 98 | -93 |
| 16 | 66 | -56 |
| 17 | 53 | 52 |
| 18 | 37 | 35 |
| 19* | 0 | -10 |
| 20 | 41 | -39 |
| 21 | 39 | 30 |
| 22* | 0 | -1 |
| 23 | 13 | 15 |
| 24 | 42 | 37 |
| 25 | 25 | -19 |

-4, 1,L

| | | |
|---|---|---|
| 1 | 76 | 79 |
| 2 | 82 | -70 |
| 3* | 0 | -8 |
| 4* | 0 | -2 |
| 5 | 103 | 106 |
| 6 | 190 | -207 |
| 7 | 167 | 171 |
| 8 | 144 | 152 |
| 9 | 219 | -171 |
| 10 | 23 | -28 |
| 11 | 86 | 78 |
| 12 | 129 | 127 |
| 13* | 0 | -5 |
| 14 | 96 | 90 |
| 15 | 98 | -96 |
| 16 | 126 | -133 |
| 17 | 24 | 25 |
| 18 | 90 | 78 |
| 19 | 106 | -106 |
| 20 | 19 | -25 |
| 21* | 0 | 14 |
| 22 | 18 | 24 |
| 23* | 0 | -0 |
| 24* | 0 | 14 |
| 25 | 11 | -12 |
| 26 | 81 | -68 |
| 27* | 0 | 3 |
| 28 | 18 | 17 |

-3,1,L

| | | |
|---|---|---|
| 1 | 211 | 171 |
| 2 | 317 | 347 |
| 3* | 0 | -3 |
| 4 | 72 | 68 |
| 5 | 52 | 53 |
| 6 | 239 | -208 |
| 7 | 243 | 213 |
| 8 | 32 | 16 |
| 9 | 79 | -73 |
| 10 | 166 | -143 |
| 11 | 189 | 175 |
| 12 | 34 | 32 |
| 13 | 66 | -76 |
| 14 | 116 | -111 |
| 15 | 63 | 63 |
| 16* | 0 | -1 |
| 17 | 154 | 139 |
| 18 | 88 | 88 |
| 19 | 75 | -74 |
| 20 | 62 | -53 |
| 21* | 0 | 31 |
| 22 | 114 | 108 |
| 23 | 88 | -87 |
| 24 | 50 | -55 |
| 25 | 22 | -21 |
| 26* | 0 | 4 |
| 27 | 33 | 38 |
| 28 | 85 | 81 |
| 29 | 25 | -25 |
| 30 | 35 | -54 |

-2,1,L

| | | |
|---|---|---|
| 1 | 34 | 17 |
| 2 | 216 | 174 |
| 3 | 221 | -181 |
| 4 | 196 | -159 |
| 5 | 154 | 143 |
| 6 | 170 | 147 |
| 7 | 330 | -274 |
| 8 | 392 | 355 |
| 9 | 152 | -122 |
| 10 | 209 | -187 |
| 11 | 316 | 287 |
| 12 | 129 | 121 |
| 13 | 124 | -106 |
| 14 | 107 | -108 |
| 15 | 171 | 161 |
| 16* | 0 | -15 |
| 17 | 43 | -39 |
| 18 | 102 | 102 |
| 19 | 93 | -90 |
| 20 | 125 | -115 |
| 21 | 232 | 207 |
| 22 | 56 | -54 |
| 23* | 0 | 1 |
| 24 | 51 | -49 |
| 25 | 35 | 33 |
| 26 | 56 | 57 |
| 27* | 0 | -21 |
| 28* | 0 | 16 |
| 29 | 41 | -41 |
| 30* | 0 | -7 |
| 31 | 27 | 33 |

-1,1,L

| | | |
|---|---|---|
| 1 | 607 | 575 |
| 2 | 524 | 524 |
| 3 | 671 | -655 |
| 4 | 835 | -932 |
| 5 | 452 | -444 |
| 6 | 28 | 21 |
| 7 | 91 | -77 |
| 8* | 0 | -28 |
| 9 | 50 | -45 |
| 10 | 157 | -135 |
| 11 | 17 | 14 |
| 12 | 270 | 255 |
| 13 | 189 | -179 |
| 14 | 215 | -197 |
| 15* | 0 | 14 |
| 16 | 113 | 125 |
| 17 | 92 | -91 |
| 18 | 122 | 8 |
| 20 | 42 | -45 |
| 21* | 0 | 26 |
| 22 | 148 | 143 |
| 23 | 127 | -125 |
| 24 | 65 | -59 |
| 25 | 119 | 104 |
| 26* | 0 | 24 |
| 27* | 0 | -9 |
| 28 | 56 | -53 |
| 29* | 0 | 13 |
| 30 | 62 | -64 |
| 31 | 29 | 29 |

0,1,L

| | | |
|---|---|---|
| 1 | 174 | 171 |
| 2 | 164 | -221 |
| 3 | 33 | -49 |
| 4 | 195 | 204 |
| 5 | 947 | 880 |
| 6 | 612 | 602 |
| 7 | 117 | -131 |
| 8 | 183 | -157 |
| 9 | 79 | -80 |
| 10 | 95 | 99 |
| 11 | 105 | -117 |
| 12* | 0 | -41 |
| 13 | 270 | -266 |
| 14 | 202 | -199 |
| 15 | 139 | 122 |
| 16 | 233 | 222 |
| 17 | 123 | -128 |
| 18 | 171 | -161 |
| 19 | 22 | -27 |
| 20 | 69 | -65 |
| 21* | 0 | -28 |
| 22 | 50 | 44 |
| 23 | 65 | -63 |
| 24* | 0 | -14 |
| 25 | 39 | 42 |
| 26 | 69 | 69 |
| 27 | 44 | -46 |
| 28 | 29 | -33 |
| 29 | 48 | 47 |
| 30 | 46 | 44 |
| 31* | 0 | 15 |

1,1,L

| | | |
|---|---|---|
| 0 | 371 | -341 |
| 1 | 61 | -55 |
| 2 | 147 | -152 |
| 3 | 111 | -76 |
| 4 | 374 | -403 |
| 5 | 344 | 352 |
| 6 | 448 | 437 |
| 7 | 125 | -111 |
| 8 | 198 | -191 |
| 9 | 427 | 469 |
| 10 | 84 | -70 |
| 11 | 181 | 194 |
| 12 | 53 | 45 |
| 13* | 0 | 5 |
| 14 | 62 | 48 |
| 15 | 100 | 102 |
| 16 | 212 | 188 |
| 17 | 91 | -90 |
| 18 | 82 | -67 |
| 19 | 58 | 58 |
| 20 | 96 | 95 |
| 21 | 122 | -129 |
| 22* | 0 | -20 |
| 23* | 0 | -6 |
| 24 | 112 | -104 |
| 24* | 0 | -104 |
| 25 | 49 | 50 |
| 26* | 0 | 21 |
| 27 | 83 | -72 |
| 28* | 0 | -27 |
| 29 | 33 | 19 |
| 30 | 25 | 26 |
| 31 | 52 | -47 |

2, 1,L

| | | |
|---|---|---|
| 0 | 382 | 385 |
| 1 | 365 | -315 |
| 2 | 24 | 49 |
| 3* | 0 | -5 |
| 4 | 212 | 206 |
| 5 | 90 | -77 |
| 6 | 54 | -57 |
| 7 | 394 | -363 |
| 8 | 359 | -353 |
| 9 | 156 | 140 |
| 10 | 232 | 237 |
| 11 | 119 | -117 |
| 12 | 133 | -129 |
| 13 | 149 | 151 |
| 14* | 0 | -7 |
| 15 | 176 | 168 |
| 16 | 34 | -19 |
| 17 | 108 | -99 |
| 18 | 225 | -211 |
| 19 | 95 | 91 |
| 20 | 163 | 153 |
| 21 | 76 | 82 |
| 22* | 0 | 21 |
| 23 | 59 | 66 |
| 24 | 25 | 39 |
| 25 | 22 | -28 |
| 26 | 55 | 47 |
| 27 | 33 | -39 |
| 28 | 55 | -54 |
| 29* | 0 | 6 |
| 30 | 28 | 24 |

3,1,L

| | | |
|---|---|---|
| 0 | 208 | 191 |
| 1 | 269 | -266 |
| 2 | 144 | -135 |
| 3 | 48 | 48 |
| 4 | 25 | -15 |
| 5 | 106 | -111 |
| 6 | 49 | 41 |
| 7* | 0 | -26 |
| 8 | 79 | -84 |
| 9 | 128 | 118 |
| 10 | 144 | 154 |
| 11 | 279 | -276 |
| 12 | 23 | 45 |
| 13 | 21 | -35 |
| 14 | 24 | 32 |
| 15 | 80 | -81 |
| 16 | 79 | -81 |
| 17* | 0 | 4 |
| 18 | 46 | 39 |
| 19 | 133 | 133 |
| 20 | 27 | 33 |
| 21 | 95 | -100 |
| 22 | 132 | -127 |
| 23* | 0 | 10 |
| 24 | 22 | 20 |
| 25 | 17 | 18 |
| 26 | 20 | -21 |
| 27 | 30 | 24 |
| 28 | 14 | 4 |

4,1,L

| | | |
|---|---|---|
| 0 | 156 | 151 |
| 1 | 17 | -6 |
| 2 | 180 | -191 |
| 3 | 155 | 155 |
| 4 | 129 | 139 |
| 5 | 18 | -16 |
| 6 | 31 | -78 |
| 7 | 23 | 21 |
| 8 | 23 | 8 |
| 9 | 64 | -64 |
| 10 | 112 | 97 |
| 11 | 56 | -52 |
| 12 | 229 | -243 |
| 13 | 146 | 165 |
| 14 | 20 | 23 |
| 15 | 108 | -101 |
| 16 | 56 | 53 |
| 17 | 44 | -39 |
| 18 | 33 | 29 |
| 19 | 27 | -17 |
| 20 | 73 | 65 |
| 21 | 54 | -53 |
| 22 | 42 | -41 |
| 23 | 49 | 44 |
| 24* | 0 | 1 |
| 25 | 38 | -31 |

5,1,L

| | | |
|---|---|---|
| 0* | 0 | 14 |
| 1 | 34 | -34 |
| 2 | 34 | -40 |
| 3 | 122 | 137 |
| 4 | 102 | 98 |
| 5 | 73 | -73 |
| 6 | 85 | -87 |
| 7 | 20 | 26 |
| 8 | 83 | 91 |
| 9 | 90 | 98 |
| 10 | 24 | 34 |
| 11 | 17 | 17 |
| 12 | 28 | -21 |
| 13* | 0 | 7 |
| 14 | 72 | 65 |
| 15 | 38 | -40 |
| 16 | 62 | -68 |
| 17 | 51 | 47 |
| 18* | 0 | 6 |
| 19 | 31 | -19 |
| 20 | 82 | -50 |
| 21 | 25 | -21 |
| 22 | 19 | -20 |

6,1,L

| | | |
|---|---|---|
| 0* | 0 | -16 |
| 1 | 69 | -63 |
| 2 | 39 | -39 |
| 3* | 0 | 8 |
| 4* | 0 | -5 |
| 5 | 63 | -53 |
| 6 | 42 | -41 |
| 7 | 77 | 67 |
| 8 | 41 | 38 |
| 9 | 41 | -35 |
| 10 | 71 | -67 |
| 11 | 11 | 3 |
| 12* | 0 | -6 |
| 13 | 35 | 38 |
| 14 | 38 | 71 |
| 15 | 28 | -25 |
| 16* | 0 | -8 |
| 17 | 51 | 44 |
| 18* | 0 | 17 |

7,1,L

| | | |
|---|---|---|
| 0 | 75 | -62 |
| 1 | 31 | 36 |
| 2* | 0 | 5 |
| 3* | 0 | -6 |
| 4 | 35 | 38 |
| 5 | 55 | -58 |
| 6* | 0 | -11 |
| 7 | 40 | 32 |
| 8 | 43 | 39 |
| 9 | 23 | -25 |
| 10 | 41 | -36 |
| 11 | 35 | -23 |

-7,2,L

| | | |
|---|---|---|
| 1 | 13 | -25 |
| 2 | 13 | -11 |
| 3 | 13 | 8 |
| 4* | 0 | 5 |
| 5 | 49 | 44 |
| 6 | 12 | 16 |
| 7 | 52 | -51 |
| 8* | 0 | -3 |
| 9 | 54 | 52 |
| 10* | 0 | -3 |
| 11 | 37 | -46 |

-6,2,L

| | | |
|---|---|---|
| 1 | 69 | -62 |
| 2 | 19 | 17 |
| 3* | 0 | 23 |
| 4 | 17 | -29 |
| 5* | 0 | -15 |
| 6* | 0 | 19 |
| 7 | 56 | -56 |
| 8 | 11 | -14 |
| 9 | 76 | 74 |
| 10* | 0 | -11 |
| 11 | 67 | -66 |
| 12* | 0 | 7 |
| 13 | 48 | 49 |
| 14 | 24 | -20 |
| 15* | 0 | -7 |
| 16 | 21 | 22 |
| 17 | 59 | -51 |
| 18 | 15 | 10 |
| 19 | 61 | 58 |

-5,2,L

| | | |
|---|---|---|
| 1 | 40 | -35 |
| 2 | 44 | -34 |
| 3 | 24 | 20 |
| 4* | 0 | 13 |
| 5* | 0 | -2 |
| 6* | 0 | 7 |
| 7 | 49 | 39 |
| 8 | 52 | -49 |
| 9 | 69 | 64 |
| 10* | 0 | 2 |
| 11 | 87 | -81 |
| 12* | 0 | -3 |
| 13 | 85 | 80 |
| 14* | 0 | 3 |
| 15 | 66 | -60 |
| 16* | 0 | -8 |
| 17 | 34 | -29 |
| 18 | 44 | -46 |
| 19 | 48 | 45 |
| 20 | 30 | 28 |
| 21 | 61 | -52 |
| 22* | 0 | 5 |
| 23 | 58 | 49 |

-4,2,L

| | | |
|---|---|---|
| 1 | 281 | -333 |
| 2 | 119 | 116 |
| 3 | 259 | 248 |
| 4 | 49 | -38 |
| 5 | 45 | -42 |
| 6 | 53 | -41 |
| 7 | 42 | -27 |
| 8 | 74 | -65 |

Table 7 (*cont.*)

| | | |
|---|---|---|
| -4,2,L | | |
| 9 | 63 | 55 |
| 10 | 37 | 28 |
| 11 | 96 | -90 |
| 12* | 0 | -0 |
| 13 | 134 | 131 |
| 14 | 66 | -70 |
| 15 | 44 | -39 |
| 16 | 20 | -14 |
| 17 | 92 | 91 |
| 18 | 23 | 20 |
| 19* | 0 | 13 |
| 20* | 0 | 2 |
| 21 | 88 | -81 |
| 22 | 43 | -31 |
| 23 | 67 | 57 |
| 24* | 0 | 1 |
| 25 | 86 | -71 |
| 26 | 21 | 11 |
| -3, 2,L | | |
| 1* | 0 | 8 |
| 2 | 32 | -24 |
| 3 | 201 | 149 |
| 4 | 205 | 149 |
| 5 | 238 | -261 |
| 6 | 171 | 131 |
| 7 | 119 | 109 |
| 8 | 97 | 80 |
| 9 | 123 | -107 |
| 10 | 115 | 105 |
| 11 | 79 | -74 |
| 12 | 28 | -35 |
| 13 | 113 | 95 |
| 14 | 30 | 35 |
| 15 | 181 | -160 |
| 16 | 28 | 23 |
| 17 | 110 | 94 |
| 18 | 62 | -50 |
| 19* | 0 | -24 |
| 20* | 0 | -2 |
| 21 | 30 | 24 |
| 22* | 0 | -33 |
| 23 | 83 | 80 |
| 24* | 0 | -3 |
| 25 | 54 | -53 |
| 26* | 0 | -3 |
| 27 | 62 | 64 |
| -2,2,L | | |
| 1 | 150 | 104 |
| 2 | 307 | -247 |
| 3 | 329 | 249 |
| 4 | 347 | -264 |
| 5 | 553 | -486 |
| 6 | 173 | -145 |
| 7 | 301 | 216 |
| 8 | 152 | 136 |
| 9 | 73 | 71 |
| 10 | 88 | 72 |
| 11 | 39 | 37 |
| 12 | 37 | -33 |
| 13 | 112 | 89 |
| 14 | 64 | 64 |
| 15 | 189 | -169 |
| 16 | 86 | 86 |
| 17* | 0 | 6 |
| 18 | 118 | 102 |
| 19 | 100 | -85 |
| 20* | 0 | 17 |
| 21 | 52 | 51 |
| 22* | 0 | -23 |
| 23* | 0 | -17 |
| 24* | 0 | 14 |
| 25 | 45 | -49 |
| 26* | 0 | -12 |
| 27 | 67 | 63 |
| 28* | 0 | -2 |
| 29 | 39 | -40 |
| -1,2,L | | |
| 1 | 265 | 229 |
| 2* | 0 | -1 |
| 3 | 410 | -341 |
| 4 | 201 | 139 |
| 5 | 87 | 87 |
| 6* | 0 | 36 |
| 7 | 522 | 467 |
| 8 | 316 | -241 |
| 9 | 555 | -512 |
| 10 | 315 | -275 |
| 11 | 136 | 126 |
| 12 | 85 | -85 |
| 13 | 79 | -83 |
| 14 | 73 | 61 |
| 15 | 105 | -96 |
| 16 | 64 | -60 |
| 17 | 347 | 354 |
| 18 | 105 | -98 |
| 19 | 126 | -110 |
| 20* | 0 | 10 |
| 21* | 0 | -28 |
| 22 | 75 | 63 |
| 23 | 30 | -23 |
| 24 | 76 | 79 |
| 25 | 34 | -33 |
| 26 | 25 | 20 |
| 27 | 33 | 42 |
| 28* | 0 | -10 |
| 29 | 38 | -41 |
| 0,2,L | | |
| 0* | 0 | -27 |
| 1 | 268 | 253 |
| 2 | 106 | 105 |
| 3 | 94 | -71 |
| 4 | 86 | 91 |
| 5 | 139 | 136 |
| 0, 2,L | | |
| 6 | 50 | 49 |
| 7 | 205 | 167 |
| 8 | 117 | 129 |
| 9 | 131 | -121 |
| 10* | 0 | 0 |
| 11 | 240 | 219 |
| 12 | 29 | 16 |
| 13 | 174 | -160 |
| 14 | 37 | -41 |
| 15 | 59 | 66 |
| 16 | 93 | -88 |
| 17* | 0 | -14 |
| 18* | 0 | 6 |
| 19 | 173 | -158 |
| 20 | 64 | -57 |
| 21 | 166 | 149 |
| 22 | 64 | -55 |
| 23 | 69 | -63 |
| 24 | 28 | -34 |
| 25 | 41 | 43 |
| 26 | 34 | -32 |
| 27 | 23 | 29 |
| 28 | 35 | 31 |
| 29 | 56 | -49 |
| 30 | 24 | 18 |
| 1,2,L | | |
| 0* | 0 | 20 |
| 1 | 236 | 223 |
| 2 | 41 | 55 |
| 3 | 323 | -307 |
| 4 | 131 | 121 |
| 5* | 0 | -21 |
| 6 | 100 | 91 |
| 7 | 189 | -159 |
| 8 | 121 | -122 |
| 9 | 123 | -113 |
| 10 | 74 | 79 |
| 11 | 347 | 309 |
| 12 | 157 | 156 |
| 13 | 129 | -122 |
| 14* | 0 | -13 |
| 15 | 192 | 167 |
| 16* | 0 | 2 |
| 17* | 0 | -8 |
| 18* | 0 | 5 |
| 19 | 132 | -129 |
| 20 | 30 | 39 |
| 21 | 142 | 132 |
| 22 | 65 | 54 |
| 23* | 0 | -21 |
| 24 | 46 | -39 |
| 25* | 0 | 9 |
| 26 | 49 | -50 |
| 27 | 31 | -32 |
| 28* | 0 | -15 |
| 29 | 25 | -30 |
| 2,2,L | | |
| 0 | 121 | -111 |
| 1 | 243 | 197 |
| 2 | 48 | 50 |
| 3 | 314 | -251 |
| 4 | 101 | -85 |
| 5 | 336 | 323 |
| 6 | 142 | -127 |
| 7 | 44 | -37 |
| 8 | 142 | 124 |
| 9 | 74 | -58 |
| 10 | 102 | 102 |
| 11 | 40 | -29 |
| 12 | 33 | -13 |
| 13 | 221 | -203 |
| 14* | 0 | 17 |
| 15 | 112 | 106 |
| 16 | 37 | 32 |
| 17 | 113 | -118 |
| 18 | 46 | 43 |
| 19 | 124 | 122 |
| 20* | 0 | 4 |
| 21 | 67 | 73 |
| 22 | 54 | -49 |
| 23 | 167 | -157 |
| 24* | 0 | 3 |
| 25 | 90 | 88 |
| 26 | 63 | 61 |
| 27* | 0 | -10 |
| 28 | 31 | 27 |
| 3,2,L | | |
| 0 | 25 | 9 |
| 1 | 20 | 11 |
| 2 | 73 | -73 |
| 3 | 167 | -172 |
| 4 | 114 | -104 |
| 5 | 187 | 157 |
| 6 | 49 | -31 |
| 7 | 139 | -121 |
| 8 | 127 | -124 |
| 9 | 97 | 90 |
| 10 | 60 | -64 |
| 11 | 40 | 32 |
| 12 | 25 | -36 |
| 13 | 64 | -52 |
| 14 | 22 | -20 |
| 15 | 104 | 92 |
| 16 | 46 | 43 |
| 17 | 75 | -69 |
| 18* | 0 | 4 |
| 19 | 22 | 24 |
| 20* | 0 | 8 |
| 21 | 56 | -58 |
| 22 | 23 | 26 |
| 23* | 0 | -17 |
| 24* | 0 | 5 |
| 25 | 37 | 34 |
| 26* | 0 | -3 |
| 27 | 17 | -38 |
| 4,2,L | | |
| 0 | 185 | 197 |
| 1 | 41 | 26 |
| 2* | 0 | 7 |
| 3 | 19 | -22 |
| 4* | 0 | 8 |
| 5 | 135 | 135 |
| 6 | 54 | 47 |
| 7 | 241 | -263 |
| 8 | 83 | 75 |
| 9 | 81 | 83 |
| 10* | 0 | 6 |
| 11 | 52 | 41 |
| 12 | 84 | -89 |
| 13 | 28 | -41 |
| 14 | 40 | -46 |
| 15 | 43 | 36 |
| 16 | 43 | -38 |
| 17 | 64 | -65 |
| 18 | 11 | -15 |
| 19 | 52 | 47 |
| 20* | 0 | -11 |
| 21 | 34 | -17 |
| 22 | 34 | -27 |
| 23 | 37 | 23 |
| 24 | 38 | 26 |
| 5, 2,L | | |
| 0 | 80 | -69 |
| 1 | 143 | -149 |
| 2 | 49 | -38 |
| 3 | 63 | 63 |
| 4 | 80 | 71 |
| 5* | 0 | -3 |
| 6 | 52 | 48 |
| 7 | 37 | 30 |
| 8 | 31 | -19 |
| 9 | 153 | 170 |
| 10* | 0 | 6 |
| 11 | 136 | -149 |
| 12 | 52 | 52 |
| 13 | 11 | 24 |
| 14 | 63 | 67 |
| 15 | 38 | 37 |
| 16 | 28 | 21 |
| 17 | 49 | -42 |
| 18 | 45 | -30 |
| 19 | 63 | 52 |
| 20 | 38 | -25 |
| 21* | 0 | -41 |
| 6,2,L | | |
| 0* | 0 | -12 |
| 1 | 96 | -94 |
| 2 | 37 | -33 |
| 3 | 25 | 25 |
| 4 | 18 | 15 |
| 5 | 25 | -22 |
| 6 | 35 | -34 |
| 7 | 34 | -32 |
| 8* | 0 | -0 |
| 9 | 29 | 23 |
| 10* | 0 | -1 |
| 11 | 54 | -46 |
| 12 | 29 | -22 |
| 13 | 41 | 35 |
| 14 | 20 | 10 |
| 15* | 0 | -3 |
| 16* | 0 | 3 |
| 7,2,L | | |
| 0 | 22 | -26 |
| 1* | 0 | -15 |
| 2 | 30 | 24 |
| 3 | 66 | 53 |
| 4* | 0 | 5 |
| 5 | 37 | -30 |
| 6 | 31 | -24 |
| 7 | 21 | 17 |
| -6,3,L | | |
| 1 | 28 | 32 |
| 2* | 0 | -3 |
| 3 | 55 | -55 |
| 4 | 28 | 33 |
| 5 | 37 | 34 |
| 6 | 27 | -24 |
| 7 | 32 | -25 |
| 8 | 58 | 61 |
| 9 | 42 | -39 |
| 10 | 27 | -26 |
| 11 | 50 | 42 |
| 12 | 46 | -34 |
| 13 | 28 | -21 |
| 14 | 35 | 28 |
| -5,3,L | | |
| 1* | 0 | -8 |
| 2 | 24 | 27 |
| 3 | 60 | -62 |
| 4 | 42 | 35 |
| 5 | 86 | 92 |
| 6 | 56 | -53 |
| 7 | 88 | -93 |
| 8 | 33 | 27 |
| 9 | 20 | 16 |
| 10 | 29 | -22 |
| 11 | 28 | 23 |
| 12 | 49 | 48 |
| 13 | 52 | -56 |
| 14 | 21 | 17 |
| 15 | 44 | 43 |
| 16 | 45 | -41 |
| 17 | 48 | -39 |
| 18 | 59 | 56 |
| 19 | 31 | 27 |
| 20 | 38 | -36 |
| -4,3,L | | |
| 1 | 23 | -5 |
| 2* | 0 | -13 |
| 3 | 39 | 37 |
| 4* | 0 | -8 |
| 5 | 39 | 40 |
| 6 | 39 | -43 |
| 7 | 76 | -79 |
| 8 | 82 | 84 |
| 9 | 58 | 51 |
| 10 | 30 | -29 |
| 11 | 79 | -74 |
| 12 | 35 | 30 |
| 13 | 27 | -21 |
| 14 | 22 | -22 |
| 15 | 50 | 45 |
| 16* | 0 | -0 |
| 17 | 61 | -61 |
| 18 | 49 | 45 |
| 19 | 51 | 40 |
| 20 | 63 | -52 |
| 21* | 0 | -13 |
| 22 | 48 | 44 |
| -3,3,L | | |
| 1* | 0 | -18 |
| 2 | 28 | 31 |
| 3 | 45 | 38 |
| 4 | 64 | -55 |
| 5 | 71 | 67 |
| 6 | 72 | -54 |
| 7 | 77 | -75 |
| 8 | 121 | 96 |
| 9 | 275 | 235 |
| 10 | 84 | -67 |
| 11* | 0 | 8 |
| 12* | 0 | 11 |
| 13* | 0 | -20 |
| 14* | 0 | -27 |
| 15* | 0 | -9 |
| 16 | 28 | 29 |
| 17 | 73 | -74 |
| 18* | 0 | 27 |
| 19 | 53 | 50 |
| 20* | 0 | -30 |
| 21 | 60 | -57 |
| 22 | 51 | 53 |
| 23* | 0 | 7 |
| 24 | 41 | -51 |
| 25 | 23 | 29 |
| -2,3,L | | |
| 1 | 334 | -230 |
| 2 | 198 | 148 |
| 3 | 59 | 33 |
| 4 | 97 | -83 |
| 5 | 142 | -123 |
| 6 | 111 | 100 |
| 7 | 46 | 48 |
| 8* | 0 | 9 |
| 9* | 0 | 10 |
| 10 | 263 | -227 |
| 11 | 218 | -190 |
| 12 | 77 | 67 |
| 13 | 294 | 233 |
| 14 | 30 | -25 |
| 15 | 66 | 41 |
| 16* | 0 | 18 |
| 17 | 30 | 33 |
| 18* | 0 | -13 |
| 19 | 86 | 88 |
| 20 | 40 | -48 |
| 21 | 96 | -94 |
| 22 | 37 | 29 |
| 23 | 42 | 54 |
| 24* | 0 | -10 |
| 25* | 0 | -18 |
| 26 | 41 | 45 |
| 27* | 0 | -27 |
| -1,3,L | | |
| 1 | 244 | -200 |
| 2 | 289 | 223 |
| 3 | 138 | 105 |
| 4 | 133 | -98 |
| 5 | 73 | -38 |
| 6 | 107 | 98 |
| 7 | 28 | -40 |
| 8 | 66 | -67 |
| 9* | 0 | -2 |
| 10* | 0 | 11 |
| 11 | 181 | -155 |
| 12 | 213 | 199 |
| 13 | 83 | -79 |
| 14 | 103 | -88 |
| 15 | 104 | -80 |
| 16 | 31 | -31 |
| 17 | 87 | 76 |
| 18 | 53 | -50 |
| 19* | 0 | -8 |
| 20* | 0 | -7 |
| 21 | 22 | -2 |
| 22 | 33 | 39 |
| 23 | 70 | 79 |
| 24 | 49 | -44 |
| 25 | 32 | -33 |
| 26* | 0 | 5 |
| 27 | 48 | 46 |
| 0,3,L | | |
| 1 | 62 | -69 |
| 2 | 63 | -72 |
| 3 | 257 | 234 |
| 4 | 113 | -109 |
| 5 | 174 | -163 |
| 6 | 128 | 109 |
| 7 | 159 | 143 |
| 8 | 105 | -100 |
| 9 | 74 | -71 |
| 0,3,L | | |
| 10 | 113 | 115 |
| 11* | 0 | 12 |
| 12 | 55 | 43 |
| 13 | 299 | 259 |
| 14 | 158 | -148 |
| 15 | 188 | -159 |
| 16 | 149 | 144 |
| 17 | 76 | -63 |
| 18* | 0 | 1 |
| 19 | 75 | -72 |
| 20* | 0 | 23 |
| 21 | 48 | -44 |
| 22 | 32 | 29 |
| 23 | 71 | 62 |
| 24 | 56 | -55 |
| 25 | 52 | -50 |
| 26* | 0 | 5 |
| 27* | 0 | -9 |
| 28* | 0 | -16 |
| 1,3,L | | |
| 0* | 0 | 12 |
| 1 | 223 | 192 |
| 2 | 111 | 118 |
| 3 | 140 | 122 |
| 4 | 48 | -37 |
| 5 | 116 | -89 |
| 6 | 85 | 70 |
| 7 | 165 | 149 |
| 8 | 67 | -73 |
| 9 | 94 | -88 |
| 10 | 73 | 76 |
| 11 | 44 | 47 |
| 12 | 81 | -80 |
| 13 | 29 | -35 |
| 14 | 29 | -38 |
| 15* | 0 | -26 |
| 16 | 88 | -26 |
| 17 | 150 | 139 |
| 18 | 59 | -59 |
| 19 | 50 | -41 |
| 20 | 66 | 60 |
| 21 | 54 | 57 |
| 22 | 37 | -28 |
| 23 | 49 | -46 |
| 24* | 0 | -32 |
| 25 | 64 | -57 |
| 26 | 84 | 69 |
| 27 | 88 | 71 |
| 2,3,L | | |
| 0 | 119 | 89 |
| 1 | 58 | -55 |
| 2 | 52 | -47 |
| 3 | 58 | -48 |
| 4 | 122 | -115 |
| 5* | 0 | 4 |
| 6 | 42 | 49 |
| 7 | 143 | 120 |
| 8 | 67 | -47 |
| 9 | 102 | -82 |
| 10 | 37 | 39 |
| 11 | 123 | 115 |
| 12 | 56 | -45 |
| 13* | 0 | -0 |
| 14 | 54 | 53 |
| 15 | 65 | -61 |
| 16 | 42 | -61 |
| 17 | 59 | 61 |
| 18 | 77 | -75 |
| 19 | 22 | -18 |
| 20* | 0 | 14 |
| 21* | 0 | 16 |
| 22 | 34 | -38 |
| 23* | 0 | -1 |
| 24 | 37 | 35 |
| 25* | 0 | 8 |
| 26 | 29 | 25 |
| 3,3,L | | |
| 0 | 246 | 213 |
| 1 | 274 | 252 |
| 2 | 117 | -109 |
| 3 | 223 | -216 |
| 4 | 22 | -32 |
| 5 | 126 | -123 |
| 6 | 61 | 56 |
| 7 | 22 | -29 |
| 8 | 87 | -86 |
| 9 | 116 | -114 |
| 10 | 46 | 49 |
| 11 | 103 | 94 |
| 12 | 39 | -41 |
| 13 | 85 | -87 |
| 14* | 0 | -14 |
| 15 | 27 | 37 |
| 16 | 37 | -39 |
| 17 | 48 | 51 |
| 18* | 0 | 0 |
| 19 | 56 | -52 |
| 20 | 49 | 54 |
| 21 | 50 | 51 |
| 22 | 27 | -22 |
| 23 | 31 | -25 |
| 24 | 29 | 26 |
| 4,3,L | | |
| 0* | 0 | 9 |
| 1* | 0 | 18 |
| 2 | 110 | -121 |
| 3* | 0 | 27 |
| 4 | 117 | 140 |
| 5 | 142 | 166 |
| 6 | 46 | 46 |
| 7 | 33 | -19 |
| 8* | 0 | -14 |
| 9* | 0 | 0 |
| 10 | 42 | 42 |
| 4,3,L | | |
| 11* | 0 | -11 |
| 12 | 62 | -76 |
| 13 | 85 | -95 |
| 14 | 45 | 44 |
| 15 | 52 | 59 |
| 16 | 22 | 21 |
| 17 | 52 | -54 |
| 18 | 28 | -29 |
| 19 | 48 | -39 |
| 20* | 0 | -9 |
| 21 | 23 | 18 |
| 5,3,L | | |
| 0* | 0 | -0 |
| 1 | 24 | 28 |
| 2* | 0 | -20 |
| 3 | 123 | -127 |
| 4 | 69 | 79 |
| 5 | 51 | 61 |
| 6 | 63 | -74 |
| 7 | 44 | 35 |
| 8 | 28 | -22 |
| 9 | 28 | 16 |
| 10 | 38 | 39 |
| 11 | 42 | 35 |
| 12* | 0 | -7 |
| 13* | 0 | -3 |
| 14 | 59 | 51 |
| 15 | 42 | 31 |
| 16 | 30 | -21 |
| 17* | 0 | -3 |
| 6,3,L | | |
| 0* | 0 | 10 |
| 1 | 20 | -24 |
| 2* | 0 | -3 |
| 3 | 42 | 26 |
| 4* | 0 | 4 |
| 5 | 41 | 36 |
| 6 | 58 | -46 |
| 7 | 101 | -99 |
| 8 | 43 | 39 |
| 9 | 18 | 13 |
| 10 | 12 | -10 |
| 11 | 14 | -10 |
| -5,4,L | | |
| 1* | 0 | -13 |
| 2 | 79 | -76 |
| 3* | 0 | -9 |
| 4 | 44 | 48 |
| 5* | 0 | -23 |
| 6 | 23 | -23 |
| 7 | 58 | 49 |
| 8 | 53 | -54 |
| 9* | 0 | -0 |
| 10 | 62 | 57 |
| 11* | 0 | 8 |
| 12 | 62 | -64 |
| 13* | 0 | 6 |
| 14 | 28 | 23 |
| -4,4,L | | |
| 1 | 59 | 59 |
| 2 | 66 | -72 |
| 3 | 27 | -27 |
| 4 | 69 | 71 |
| 5* | 0 | 4 |
| 6 | 43 | -44 |
| 7 | 21 | 20 |
| 8 | 18 | 19 |
| 9 | 29 | -31 |
| 10 | 30 | 42 |
| 11 | 34 | 45 |
| 12 | 65 | -75 |
| 13 | 31 | -33 |
| 14 | 34 | 38 |
| 15* | 0 | -14 |
| 16 | 37 | -39 |
| 17 | 30 | 33 |
| 18* | 0 | 5 |
| -3,4,L | | |
| 1 | 31 | -46 |
| 2 | 31 | -37 |
| 3 | 28 | -7 |
| 4 | 167 | 136 |
| 5* | 0 | -22 |
| 6 | 43 | -39 |
| 7* | 0 | -10 |
| 8 | 74 | 68 |
| 9* | 0 | 11 |
| 10 | 52 | -40 |
| 11* | 0 | -18 |
| 12 | 50 | -57 |
| 13* | 0 | 2 |
| 14 | 116 | 130 |
| 15 | 61 | 73 |
| 16 | 61 | -53 |
| 17* | 0 | -15 |
| 18* | 0 | 12 |
| 19 | 35 | -44 |
| 20* | 0 | 13 |
| 21* | 0 | 3 |
| -2,4,L | | |
| 1 | 52 | 59 |
| 2 | 30 | -25 |
| 3 | 52 | -53 |
| 4 | 48 | 45 |
| 5 | 37 | 37 |
| 6 | 177 | -135 |
| 7* | 0 | 4 |
| 8 | 94 | 77 |
| 9 | 82 | -91 |
| 10* | 0 | -14 |

Table 7 (*cont.*)

| | | |
|---|---|---|
| **-2, 4, L** | | |
| 11* | 0 | 2 |
| 12 | 59 | 64 |
| 13 | 41 | -31 |
| 14 | 22 | 35 |
| 15* | 0 | -23 |
| 16 | 34 | -37 |
| 17* | 0 | -4 |
| 18 | 95 | 96 |
| 19 | 23 | 18 |
| 20 | 38 | -43 |
| 21 | 31 | 42 |
| 22* | 0 | 5 |
| 23* | 0 | -16 |
| **-1, 4, L** | | |
| 1 | 49 | -46 |
| 2* | 0 | -9 |
| 3* | 0 | 9 |
| 4* | 0 | 10 |
| 5 | 82 | 77 |
| 6 | 119 | -96 |
| 7* | 0 | 13 |
| 8 | 49 | 43 |
| 9 | 76 | 65 |
| 10 | 54 | -92 |
| 11* | 0 | 8 |
| 12 | 61 | 51 |
| 13* | 0 | 5 |
| 14* | 0 | -24 |
| 15* | 0 | 12 |
| 16 | 50 | -56 |
| 17 | 73 | -73 |
| 18 | 71 | 67 |
| 19* | 0 | -2 |
| 20 | 40 | -41 |
| 21* | 0 | -15 |
| 22 | 52 | 53 |
| 23* | 0 | -18 |
| **0, 4, L** | | |
| 0 | 201 | -191 |
| 1* | 0 | 37 |
| 2 | 186 | 180 |
| 3 | 71 | 62 |
| 4* | 0 | 5 |
| 5* | 0 | -20 |
| 6 | 76 | -70 |
| 7 | 135 | -111 |
| 8 | 210 | 172 |
| 9 | 41 | -52 |
| 10 | 116 | -104 |
| 11 | 91 | 82 |
| 12 | 35 | -32 |
| 13 | 34 | 39 |
| 14 | 63 | -56 |
| 15* | 0 | -3 |
| 16 | 22 | 21 |
| 17 | 45 | 48 |
| 18 | 50 | 49 |
| 19* | 0 | 10 |
| 20 | 79 | -69 |
| 21* | 0 | -8 |
| 22* | 0 | 18 |
| 23* | 0 | 8 |
| 24 | 60 | -44 |
| **1, 4, L** | | |
| 0 | 81 | -64 |
| 1* | 0 | -23 |
| 2 | 139 | 130 |
| 3* | 0 | 26 |
| 4 | 102 | -100 |
| 5* | -0 | -5 |
| 6 | 96 | 103 |
| 7 | 30 | 27 |
| 8 | 30 | 22 |
| 9 | 91 | 95 |
| 10 | 113 | -97 |
| 11 | 79 | -72 |
| 12 | 179 | 157 |
| 13 | 64 | -61 |
| 14 | 41 | -27 |
| 15* | 0 | 8 |
| 16* | 0 | 18 |
| 17* | 0 | -26 |
| 18* | 0 | -13 |
| 19 | 31 | 30 |
| 20 | 56 | -55 |
| 21* | 0 | 10 |
| 22 | 56 | 44 |
| 23* | 0 | -10 |
| **2, 4, L** | | |
| 0 | 33 | -41 |
| 1* | 0 | 8 |
| 2 | 80 | 71 |
| 3 | 38 | -36 |
| 4 | 215 | -188 |
| 5 | 128 | -134 |
| 6 | 54 | 53 |
| 7 | 38 | 41 |
| 8* | 0 | 5 |
| 9* | 0 | 20 |
| 10 | 27 | -30 |
| 11* | 0 | 16 |
| 12 | 83 | 80 |
| 13 | 25 | 36 |
| 14 | 58 | -51 |
| 15 | 24 | -24 |
| 16 | 46 | 44 |
| 17 | 17 | 21 |
| 18* | 0 | -3 |
| 19* | 0 | 0 |
| 20* | 0 | 5 |
| 21* | 0 | -18 |
| 22 | 28 | 29 |

| | | |
|---|---|---|
| **3, 4, L** | | |
| 0 | 34 | -30 |
| 1 | 28 | 27 |
| 2 | 27 | -13 |
| 3 | 27 | -31 |
| 4 | 27 | -13 |
| 5 | 77 | 32 |
| 6 | 127 | 125 |
| 7* | 0 | 14 |
| 8 | 146 | -136 |
| 9 | 102 | -97 |
| 10* | 0 | 2 |
| 11 | 56 | -43 |
| 12* | 0 | 13 |
| 13* | 0 | -14 |
| 14 | 97 | -93 |
| 15 | 17 | 30 |
| 16 | 33 | 77 |
| 17* | 0 | 15 |
| 18 | 30 | -32 |
| 19 | 23 | -27 |
| 20* | 0 | -2 |
| **4, 4, L** | | |
| 0 | 54 | 52 |
| 1* | 0 | 3 |
| 2 | 43 | 45 |
| 3* | 0 | 20 |
| 4 | 29 | -23 |
| 5* | 0 | 3 |
| 6 | 37 | 34 |
| 7 | 29 | 28 |
| 8 | 32 | -32 |
| 9* | 0 | 10 |
| 10 | 43 | 46 |
| 11 | 37 | 32 |
| 12 | 24 | -26 |
| 13* | 0 | 13 |
| 14* | 0 | 1 |
| 15 | 27 | -24 |
| 16 | 20 | 16 |
| **5, 4, L** | | |
| 0 | 51 | 57 |
| 1* | 0 | 8 |
| 2* | 0 | -1 |
| 3 | 33 | -30 |
| 4* | 0 | -3 |
| 5* | 0 | 22 |
| 6* | 0 | 12 |
| 7 | 44 | -41 |
| 8 | 50 | -45 |
| 9* | 0 | -6 |
| 10 | 54 | 51 |
| 11 | 31 | 30 |
| **-4, 5, L** | | |
| 1 | 50 | -41 |
| 2 | 28 | -13 |
| 3 | 62 | 46 |
| 4* | 0 | -17 |
| 5 | 39 | 25 |
| 6 | 70 | 52 |
| 7 | 38 | -32 |
| 8 | 55 | -46 |
| 9 | 35 | 36 |
| **-3, 5, L** | | |
| 1* | 0 | -13 |
| 2 | 24 | -23 |
| 3 | 17 | 27 |
| 4* | 0 | -3 |
| 5 | 23 | -40 |
| 6* | 0 | 10 |
| 7* | 0 | 14 |
| 8 | 22 | -39 |
| 9* | 0 | 22 |
| 10 | 21 | 41 |
| 11* | 0 | -35 |
| 12* | 0 | -15 |
| 13 | 23 | 58 |
| 14 | 14 | 5 |
| **-2, 5, L** | | |
| 1 | 48 | -48 |
| 2 | 32 | -33 |
| 3 | 22 | 22 |
| 4* | 0 | 0 |
| 5* | 0 | 19 |
| 6* | 0 | -5 |
| 7 | 25 | 18 |
| 8 | 34 | -37 |
| 9* | 0 | -4 |
| 10 | 23 | 37 |
| 11 | 17 | -11 |
| 12 | 42 | -44 |
| 13 | 20 | 27 |
| 14* | 0 | 14 |
| 15 | 38 | -46 |
| 16* | 0 | 14 |
| 17* | 0 | 8 |
| **-1, 5, L** | | |
| 1 | 29 | -39 |
| 2 | 64 | -66 |
| 3 | 49 | 55 |
| 4 | 116 | 81 |
| 5 | 72 | -71 |
| 6 | 22 | -32 |
| 7 | 28 | -33 |
| 8* | 0 | -6 |
| 9* | 0 | -1 |
| 10 | 44 | 49 |
| 11* | 0 | 11 |
| 12 | 48 | -55 |
| 13 | 22 | 30 |
| 14 | 21 | 36 |
| 15* | 0 | -27 |
| 16 | 32 | -33 |
| 17 | 44 | 51 |

| | | |
|---|---|---|
| **0, 5, L** | | |
| 1 | 27 | 25 |
| 2 | 46 | -32 |
| 3 | 53 | 57 |
| 4* | 0 | 0 |
| 5 | 35 | -46 |
| 6 | 37 | -27 |
| 7* | 0 | 17 |
| 8 | 72 | 69 |
| 9 | 25 | -2 |
| 10 | 24 | -31 |
| 11 | 23 | -13 |
| 12 | 28 | -19 |
| 13 | 21 | -27 |
| 14 | 62 | 53 |
| 15 | 17 | -17 |
| 16 | 31 | -23 |
| 17* | 0 | 11 |
| 18 | 30 | 25 |
| **1, 5, L** | | |
| 0 | 34 | -32 |
| 1* | 0 | 1 |
| 2* | 0 | -5 |
| 3* | 0 | -12 |
| 4 | 71 | 81 |
| 5 | 67 | -69 |
| 6 | 91 | -91 |
| 7 | 124 | 125 |
| 8* | 0 | 13 |
| 9 | 43 | 37 |
| 10* | 0 | 14 |
| 11* | 0 | -16 |
| 12* | 0 | 4 |
| 13* | 0 | 19 |
| 14* | 0 | 23 |
| 15 | 27 | -24 |
| 16 | 41 | -38 |
| **2, 5, L** | | |
| 0 | 64 | -77 |
| 1* | 0 | 14 |
| 2* | 0 | 29 |
| 3 | 22 | -36 |
| 4 | 23 | -34 |
| 5 | 23 | 22 |
| 6* | 0 | 8 |
| 7* | 0 | 23 |
| 8 | 45 | 53 |
| 9 | 95 | -122 |
| 10 | 76 | -96 |
| 11 | 19 | 20 |
| 12 | 22 | 37 |
| 13 | 21 | 27 |
| 14* | 0 | 4 |
| 15* | 0 | 15 |
| 16* | 0 | -13 |
| **3, 5, L** | | |
| 0 | 29 | -35 |
| 1* | 0 | 6 |
| 2 | 43 | 57 |
| 3* | 0 | 15 |
| 4* | 0 | -5 |
| 5* | 0 | 17 |
| 6 | 17 | -30 |
| 7* | 0 | -12 |
| 8* | 0 | 10 |
| 9* | 0 | -10 |
| 10* | 0 | 3 |
| 11* | 0 | 29 |
| 12* | 0 | 10 |
| **4, 5, L** | | |
| 0* | 0 | -14 |
| 1 | 34 | 23 |
| 2 | 58 | 39 |
| 3* | 0 | -14 |
| 4 | 56 | -46 |
| 5* | 0 | -10 |
| 6 | 46 | 32 |
| 7* | 0 | -8 |
| **-1, 6, L** | | |
| 1 | 24 | -16 |
| 0 | 40 | 38 |
| **0, 6, L** | | |
| 1 | 34 | -36 |
| 2* | 0 | -2 |
| 3 | 19 | 22 |
| 4* | 0 | 4 |
| **1, 6, L** | | |
| 0* | 0 | -32 |
| 1 | 46 | -51 |

H atoms on the benzene ring as fixed atoms, followed by anisotropic variation of thermal parameters for the heavier atoms gave, after one cycle, the final $R = 0{\cdot}116$, $R_{F^2} = \Sigma|F_o^2 - F_c^2|/\Sigma F_o^2 = 0{\cdot}20$ and $R_{wF^2} = \sqrt{[\Sigma w|F_o^2 - F_c^2|^2/\Sigma w|F_o|^4]} = 0{\cdot}30$ for the 1238 observed diffraction maxima. When the 338 unobserved reflections were included the value of $R$ became 0·151. The full matrix was used in all least-squares refinements, and all positional shifts were less than their standard deviations in the final refinement. For example, in the final refinement of '*syn*'-*p*-chlorobenzaldoxime the largest parameter shift was 0·0010 Å, and the average absolute shift was 0·0004 Å.

The observed and calculated structure factors are given in Table 7, where unobserved values are indicated by an asterisk. A summary of the disagreement index $R$ (observed reflections only) is given in Table 8, and the final atomic parameters and their standard deviations are given in Tables 9 and 10. The bond distances and angles and the corresponding standard deviations were calculated using the full variance–covariance matrix of the least-squares refinement (Table 11). The numbering diagram for '*syn*'-*p*-chlorobenzaldoxime is shown in Fig. 1(*b*). The distances and angles obtained from the refined structure are, as would be expected, very close to the values reported earlier. The average C–C distance in the benzene ring is 1·384 Å and the average angle not involving H in the ring is 119·99°.

Table 8. *Final values of* $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ *for 'syn'-p-chlorobenzaldoxime*

| *hkl* | $R$ | Range of sin $\theta$ | $R$ |
|---|---|---|---|
| All orders | 0·118 | 0·00 to 0·40 | 0·112 |
| $h$ even | 0·124 | 0·40 to 0·50 | 0·106 |
| $h$ odd | 0·114 | 0·50 to 0·60 | 0·119 |
| $k$ even | 0·123 | 0·60 to 0·65 | 0·096 |
| $k$ odd | 0·115 | 0·65 to 0·70 | 0·109 |
| $l$ even | 0·124 | 0·70 to 0·75 | 0·114 |
| $l$ odd | 0·114 | 0·75 to 0·80 | 0·109 |
| $k+l$ even | 0·120 | 0·80 to 0·85 | 0·109 |
| $k+l$ odd | 0·117 | 0·85 to 0·90 | 0·119 |
| $h+l$ even | 0·112 | 0·90 to 1·00 | 0·163 |
| $h+l$ odd | 0·125 | | |
| $h+k$ even | 0·116 | | |
| $h+k$ odd | 0·121 | | |
| $h+k+l$ even | 0·122 | | |
| $h+k+l$ odd | 0·115 | | |

Overall disagreement

$R_F = 0{\cdot}116$
$R_{F^2} = 0{\cdot}202$
$R_{wF^2} = 0{\cdot}298$

The two sets of two-dimensional data for the '*anti*'-*p*-chlorobenzaldoxime (Jerslev, 1957) were refined by least-squares with individual isotropic thermal parameters. As can be seen from the resulting parameters given in Table 12 the $z$ coordinates

Table 9. *Atomic parameters for 'syn'-p-chlorobenzaldoxime**

| Atom | $x$ | $y$ | $z$ | $\sigma(x)$ | $\sigma(y)$ | $\sigma(z)$ |
|---|---|---|---|---|---|---|
| Cl(1) | 0·0921 | −0·1305 | 0·2014 | 0·0002 | 0·0004 | 0·0001 |
| C(2) | 0·2524 | 0·1014 | 0·1671 | 0·0009 | 0·0012 | 0·0002 |
| C(3) | 0·4584 | 0·1679 | 0·1886 | 0·0010 | 0·0011 | 0·0002 |
| C(4) | 0·5900 | 0·3542 | 0·1612 | 0·0010 | 0·0013 | 0·0002 |
| C(5) | 0·5082 | 0·4681 | 0·1126 | 0·0009 | 0·0013 | 0·0002 |
| C(6) | 0·3042 | 0·3938 | 0·0909 | 0·0010 | 0·0014 | 0·0002 |
| C(7) | 0·1737 | 0·2076 | 0·1176 | 0·0010 | 0·0013 | 0·0002 |
| C(8) | 0·6544 | 0·6700 | 0·0861 | 0·0011 | 0·0015 | 0·0003 |
| N(9) | 0·5839 | 0·8095 | 0·0458 | 0·0009 | 0·0012 | 0·0002 |
| O(10) | 0·7505 | 0·9864 | 0·0276 | 0·0008 | 0·0013 | 0·0002 |

Scale factor: $s = 0{\cdot}904 \pm 0{\cdot}004$ (as previously defined)

* Unit-cell parameters are $a = 6{\cdot}06$, $b = 4{\cdot}73$, $c = 25{\cdot}06$ Å and $\beta = 93{\cdot}4°$. The space group is $P2_1/c$, and there are four molecules in the unit cell (Jerslev, 1958).

Table 10. *Anisotropic thermal parameters for 'syn'-p-chlorobenzaldoxime*

The anisotropic thermal parameters are in the form:

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

| Atom | $\beta_{11}$ | $\beta_{22}$ | $\beta_{33}$ | $\beta_{12}$ | $\beta_{13}$ | $\beta_{23}$ |
|---|---|---|---|---|---|---|
| Cl(1) | 0·0306 | 0·0522 | 0·0017 | −0·0094 | 0·0007 | 0·0009 |
| C(2) | 0·0272 | 0·0324 | 0·0016 | 0·0024 | 0·0004 | −0·0004 |
| C(3) | 0·0302 | 0·0308 | 0·0016 | −0·0034 | −0·0005 | 0·0005 |
| C(4) | 0·0296 | 0·0484 | 0·0016 | −0·0022 | −0·0006 | −0·0003 |
| C(5) | 0·0252 | 0·0467 | 0·0013 | −0·0006 | −0·0004 | −0·0001 |
| C(6) | 0·0319 | 0·0507 | 0·0012 | −0·0049 | −0·0006 | 0·0008 |
| C(7) | 0·0290 | 0·0442 | 0·0016 | −0·0020 | −0·0004 | −0·0002 |
| C(8) | 0·0313 | 0·0550 | 0·0017 | −0·0054 | −0·0002 | 0·0003 |
| N(9) | 0·0313 | 0·0508 | 0·0015 | −0·0092 | −0·0005 | 0·0005 |
| O(10) | 0·0364 | 0·0750 | 0·0019 | −0·0213 | −0·0005 | 0·0028 |

Table 11. *Intramolecular bonds and angles for 'syn'-p-chlorobenzaldoxime*

| Atoms | Distance | Standard deviation* |
|---|---|---|
| Cl(1)–C(2) | 1·728 Å | ±0·006 Å |
| C(2)–C(3) | 1·367 | 0·008 |
| C(3)–C(4) | 1·396 | 0·008 |
| C(4)–C(5) | 1·396 | 0·008 |
| C(5)–C(6) | 1·367 | 0·008 |
| C(6)–C(7) | 1·382 | 0·008 |
| C(7)–C(2) | 1·396 | 0·008 |
| C(5)–C(8) | 1·486 | 0·009 |
| C(8)–N(9) | 1·260 | 0·008 |
| N(9)–O(10) | 1·408 | 0·007 |

| Atoms | Angle | Standard deviation* |
|---|---|---|
| Cl(1)–C(2)–C(3) | 118·5° | ±0·4 |
| C(2)–C(3)–C(4) | 119·1 | 0·5 |
| C(3)–C(4)–C(5) | 119·3 | 0·5 |
| C(4)–C(5)–C(6) | 121·0 | 0·5 |
| C(5)–C(6)–C(7) | 120·1 | 0·5 |
| C(6)–C(7)–C(2) | 119·0 | 0·5 |
| C(7)–C(2)–C(3) | 121·6 | 0·5 |
| C(7)–C(2)–Cl(1) | 119·9 | 0·4 |
| C(4)–C(5)–C(8) | 116·6 | 0·5 |
| C(6)–C(5)–C(8) | 122·5 | 0·5 |
| C(5)–C(8)–N(9) | 120·8 | 0·5 |
| C(8)–N(9)–O(10) | 110·8 | 0·4 |

Hydrogen bond distance

O–H · · · N 2·825 ± 0·006 Å

* The standard deviations are calculated using the full variance–covariance matrix from the least-squares refinement. The values should be multiplied by 2 or 3 for significant uncertainty.

Table 12. *Atomic parameters for 'anti'-p-chlorobenzaldoxime**

*0kl* Refinement

| Atom | $y$ | $z$ | $B$ | $\sigma(y)$ | $\sigma(z)$ | $\sigma(B)$ |
|---|---|---|---|---|---|---|
| Cl(1) | 0·3207 | 0·0528 | 4·89 | 0·0018 | 0·0004 | 0·23 |
| C(2) | 0·4466 | 0·0898 | 3·03 | 0·0049 | 0·0009 | 0·49 |
| C(3) | 0·6506 | 0·0627 | 3·37 | 0·0053 | 0·0009 | 0·59 |
| C(4) | 0·7493 | 0·0905 | 2·75 | 0·0047 | 0·0009 | 0·48 |
| C(5) | 0·6398 | 0·1457 | 2·43 | 0·0046 | 0·0007 | 0·46 |
| C(6) | 0·4286 | 0·1692 | 2·74 | 0·0052 | 0·0008 | 0·48 |
| C(7) | 0·3304 | 0·1434 | 3·29 | 0·0057 | 0·0009 | 0·60 |
| C(8) | 0·7594 | 0·1723 | 2·80 | 0·0046 | 0·0009 | 0·45 |
| N(9) | 0·6570 | 0·2111 | 3·32 | 0·0042 | 0·0009 | 0·44 |
| O(10) | 0·3948 | 0·2338 | 3·49 | 0·0037 | 0·0007 | 0·38 |

Scale factor $s = 0{\cdot}895 \pm 0{\cdot}030$

*h0l* Refinement

| Atom | $x$ | $z$ | $B$ | $\sigma(x)$ | $\sigma(z)$ | $\sigma(B)$ |
|---|---|---|---|---|---|---|
| Cl(1) | 0·0002 | 0·0527 | 5·24 | 0·0012 | 0·0003 | 0·23 |
| C(2) | 0·7782 | 0·0872 | 3·41 | 0·0036 | 0·0009 | 0·56 |
| C(3) | 0·6730 | 0·0630 | 6·61 | 0·0057 | 0·0013 | 0·91 |
| C(4) | 0·4924 | 0·0917 | 4·62 | 0·0045 | 0·0009 | 0·59 |
| C(5) | 0·4471 | 0·1436 | 2·35 | 0·0031 | 0·0007 | 0·39 |
| C(6) | 0·5555 | 0·1678 | 3·57 | 0·0041 | 0·0008 | 0·54 |
| C(7) | 0·7497 | 0·1427 | 6·00 | 0·0059 | 0·0011 | 0·80 |
| C(8) | 0·2412 | 0·1698 | 5·63 | 0·0048 | 0·0010 | 0·71 |
| N(9) | 0·1432 | 0·2130 | 3·88 | 0·0034 | 0·0010 | 0·52 |
| O(10) | 0·2027 | 0·2357 | 5·73 | 0·0034 | 0·0009 | 0·61 |

Scale factor $s = 0{\cdot}930 \pm 0{\cdot}040$

* Unit-cell parameters for *'anti'*-p-chlorobenzaldoxime are $a = 6{\cdot}60$, $b = 4{\cdot}67$ and $c = 23{\cdot}52$ Å. The space group is $P2_12_12_1$, and there are four molecules in the unit cell (Jerslev, 1958).

Table 13. *Disagreement indices for 'anti'-p-chlorobenzaldoxime*

*0kl* Refinement

| Class | $R_F$ | sin $\theta$ | $R_F$ |
|---|---|---|---|
| All orders | 0·134 | 0·00 to 0·40 | 0·176 |
| *k* even | 0·142 | 0·40 to 0·50 | 0·114 |
| *k* odd | 0·129 | 0·50 to 0·60 | 0·101 |
| *l* even | 0·129 | 0·60 to 0·65 | 0·151 |
| *l* odd | 0·139 | 0·65 to 0·70 | 0·130 |
| *k*+*l* even | 0·140 | 0·70 to 0·75 | 0·121 |
| *k*+*l* odd | 0·129 | 0·75 to 0·80 | 0·081 |
| | | 0·80 to 0·85 | 0·193 |
| | | 0·85 to 0·90 | 0·109 |
| | | 0·90 to 1·00 | 0·166 |

Overall disagreement

$R_F = 0{\cdot}145$ $R_{F2} = 0{\cdot}28$ $R_{wF2} = 0{\cdot}33$

*h0l* Refinement

| Class | $R_F$ | sin $\theta$ | $R_F$ |
|---|---|---|---|
| All orders | 0·197 | 0·00 to 0·40 | 0·145 |
| *h* even | 0·201 | 0·40 to 0·50 | 0·172 |
| *h* odd | 0·195 | 0·50 to 0·60 | 0·187 |
| *l* even | 0·207 | 0·60 to 0·65 | 0·237 |
| *l* odd | 0·188 | 0·65 to 0·70 | 0·260 |
| *h*+*l* even | 0·160 | 0·70 to 0·75 | 0·230 |
| *h*+*l* odd | 0·254 | 0·75 to 0·80 | 0·240 |
| | | 0·80 to 0·85 | 0·235 |
| | | 0·85 to 0·90 | 0·236 |
| | | 0·90 to 1·00 | 0·242 |

Overall disagreement

$R_F = 0{\cdot}182$ $R_{F2} = 0{\cdot}26$ $R_{wF2} = 0{\cdot}42$

suggest that, although the basic structure is correct, the revised bond distances are not of sufficient accuracy to justify comparison with the other two structures. Accordingly, bond distances and angles will not be reported until a revised structure is obtained from three-dimensional data. The final disagreement indices $R$ for the two sets of data are given, however, in Table 13. For the *h0l* data $R = 0{\cdot}182$, $R_{F2} = 0{\cdot}26$, $R_{wF2} = 0{\cdot}42$ and for the *0kl* data $R = 0{\cdot}144$, $R_{F2} = 0{\cdot}28$ and $R_{wF2} = 0{\cdot}33$.

## Comparison of the *N*-methyl oxime with the '*syn*' and '*anti*' oximes

The Cl–C distances of 1·768 Å in the *N*-methyl oxime and of 1·728 Å in the '*syn*' oxime differ by 0·04 Å, which is some six times the standard deviations based upon the assumption of random errors. Nevertheless, there may be some systematic problems near the relatively heavy Cl atoms and hence we are reluctant to press a claim for this difference, which we do not understand. Also, the C–C bond distances in the benzene rings vary from extremes of 1·363 Å to 1·411 Å in the *N*-methyl oxime, and from 1·367 Å to 1·396 Å in the '*syn*' oxime, and hence we have suggested a factor of 2 or 3 times standard deviations as a requirement for significant uncertainty. In the oxime part of the molecule the N → O distance of 1·284 Å in the *N*-methyl oxime and the N–O distance of 1·408 Å in the '*syn*' oxime are, of course, quite significantly different in the expected direction. Moreover, the C=N distance of 1·309 Å in the *N*-methyl oxime is 0·05 Å longer than the C=N distance of 1·260 Å in the '*syn*' oxime, and we believe that this difference is significant partly because it is far removed from the Cl atom, and partly because it seems understandable in view of the reverse trend of the N–O distances in these two compounds.

Comparison of the bond angles in these two compounds is also interesting. In the *N*-methyl oxime, ∠ C(6)C(5)C(8) = 127·8°, ∠ C(5)C(8)N(9) = 124·7° and ∠ C(8)N(9)O(10) = 125·2° are all increased above 120° in such a way that the H atom attached to C(6) is 2·2 Å away from O(10). Even this H · · · O distance is about 0·4 Å less than the van der Waals contact of 2·6 Å which is found from intermolecular, rather than intramolecular, contacts. It is clear that if the $CH_3$ and O are reversed by rotation about the C=N bond, there are then H · · · H contacts between the H atom attached to C(6) and two H atoms of the $CH_3$ group of only 1·6 Å, which is 0·8 Å less than the usual van der Waals contact. Certainly, then, it is no surprise that a planar '*anti*' configuration is more stable than the planar '*syn*' configuration for the *N*-methyl oxime; but, since in the non-methylated '*anti*' oxime a plane through C(8), N(9) and O(10) deviates considerably (19°) (Jerslev, 1958) from the plane of the benzene ring, it is surprising that the *N*-methylated '*anti*' form is so nearly coplanar, except, of course, for some H atoms of the $CH_3$ group. In view of these short contacts, it is probable that if the as yet unknown '*syn*' form of the *N*-methyl oxime were discovered it would not have the C, N and O atoms in coplanar arrangement. The external C–C bond is displaced from the C(2)C(5) bisector of the benzene ring in both the '*syn*' and '*anti*' oximes in the direction expected from steric considerations, and, also as expected, this displacement is comparatively smaller in the '*syn*' oxime. The C(6)C(5)C(8) angle in the '*syn*' oxime is only 122·5° (only 2·5° above 120°), and the distance from N(9) to the H atom attached to C(6) is a reasonable 2·6 Å.

In Table 14 we summarize bond distances, which are in common among a number of oximes and hydrochlorides of amine oxides. In trimethylamine oxide the N → O distances of 1·36 ± 0·03 Å (Lister & Sutton, 1939) and 1·44 ± 0·04 Å (Rundle, 1950) have been reported. In the present study, where this N → O bond is much more highly conjugated it is remarkably short, much shorter in the *N*-methyl oxime than the N–O distances found in hydrochlorides of amine oxides.

Table 14. *Comparison of distances in some oximes and amine oxides*

| Compound | N–O | C=N | Reference |
|---|---|---|---|
| Acetoxime | 1·36 Å | 1·29 Å | Bierlein & Lingafelter (1951) |
| Dimethylglyoxime* | 1·32 | 1·25 | Hamilton (1961) |
| 5-Chlorosalicylaldoxime | 1·385 | 1·237 | Simonsen, Pfluger & Thompson (1961) |
| '*Syn*'-*p*-chlorobenzaldoxime | 1·408 | 1·260 | This study |
| '*Anti*'-*p*-chlorobenzaldoxime | 1·39 | 1·26 | Jerslev (1957) |
| *N*-Methyl-*p*-chlorobenzaldoxime | 1·284 | 1·309 | This study |
| Trimethylamine oxide† | 1·36<br>1·44 | —<br>— | Allen & Sutton (1950) |
| Trimethylamine oxide HCl | 1·425 | — | Caron & Donohue (1962) |
| 4-Nitropyridine-*N*-oxide | 1·260 | — | Eichhorn (1956) |
| 4,4-*Trans*-azopyridine-*N*-oxide | 1·283 | — | Eichhorn (1959) |
| Pyridinoxide HCl | 1·375 | — | Tsoucaris (1961) |
| Formaldoxime‡ | 1·408 | 1·276 | Levine (1963) |
| Trimethylamine oxide† | 1·36<br>1·44 | —<br>— | Lister & Sutton (1939)<br>Rundle (1950) |

* Neutron diffraction study. † Electron diffraction studies.
‡ Microwave study.

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