Crystal Structures of Complexes of Nickel Perchlorate with Substituted Pyridines. I. Bisperchloratotetrakis-(3,5-dimethylpyridine)nickel(II)*

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X-ray structure analysis shows that the blue solid Ni(3.5-dimethylpyridine)₄(ClO₄)₂ is an octahedral complex compound in which the perchlorate ions are coordinated to the nickel ion, in agreement with the interpretation of spectroscopic and magnetic studies (Moore, Gayhart, & Bull, J. Inorg. and Nuclear Chem. (1964), 26, 896; Buffagni, Vallarino & Quagliano, Inorg. Chem. (1964), 3, 671). Crystals of the compound have the space-group symmetry $I4_1/acd$; a=b=15.8759 (17), c=26.7581 (25) Å, Z=8. Intensities of 1712 independent reflections (Cu Ka radiation, $\sin \theta/\lambda \le 0.639$) were recorded with the Oak Ridge computer-controlled diffractometer and were corrected for absorption. The structure was solved essentially from Patterson and Fourier syntheses as a heavy-atom problem. Disorder in the arrangement of the perchlorate group made the location of the three perchlorate oxygen atoms not coordinated to nickel rather difficult; these atoms were located eventually by use of the method of least-squares, the occupancy factors of closely spaced possible positions being adjusted to find the actual positions. Although the parameters of the remainder of the structure are fairly precisely determined, those of most of the atoms of the perchlorate group are not well determined because of the disorder; and the final value of the usual discrepancy index R(F) has the rather high value 0.101. The nickel atom of the asymmetric unit is located at the point $0, \frac{1}{4}, \frac{1}{8}$ of the Wyckoff set (b) (point-group symmetry 222); the perchlorate group is in a disordered arrangement about the twofold axis $x, \frac{1}{4} + x, \frac{1}{8}$, which is perpendicular to the average plane of the nickel and nitrogen atoms defined by the twofold axes $x, \frac{1}{4} - x, \frac{1}{8}$ and $0, \frac{1}{4}, z$. The angle between the Ni–O bond and the twofold axis is 9.2⁻; the Ni–O bond length is 2.187 (4)°. The four organic ligands are in a propeller-like arrangement (pitch $\sim 47^{\circ}$) around the axis through the perchlorate. The Ni-N bond length is 2.093 (2) Å. The nitrogen atoms are alternately 0.065 Å above and 0.065 Å below their average plane. The structure is very similar to that of Ni(pyridine)₄Cl₂, which has the same space-group symmetry and nearly the same translations a and b (Porai-Koshits, Structure Reports (1954), 18, 749).

Introduction

There has been considerable interest recently in the perchlorate ion with respect to its ability to act as a ligand in forming coordination compounds with metallic ions. Conductance, spectroscopic, and magnetic studies have been interpreted as indicating coordination of perchlorate to a variety of transition-metal ions. In two complex compounds of cobalt(II) the coordination of perchlorate has been authenticated by X-ray crystalstructure analysis: Co(CH₃SCH₂CH₂SCH₃)₂(ClO₄)₂ (Cotton & Weaver, 1965); Co(diphenylmethylarsine oxide)₄(ClO₄)₂ (Pauling, Robertson & Rodley, 1965). One interesting series of compounds whose magnetic and spectral properties have been studied is the series $Ni(py)_4(ClO_4)_2$, where py stands for pyridine and various substituted pyridines. The compounds studied fall into two classes:

(a) Blue compounds with magnetic moments about 3.25 Bohr magnetons: py = pyridine (Rosenthal &

Drago, 1965), 3,5-dimethylpyridine (Moore, Gayhart & Bull, 1964; Buffagni, Vallarino & Quagliano, 1964), 3-bromopyridine (Moore *et al.*, 1964), 4-isopropylpyridine (Moore *et al.*, 1964).

(b) Yellow diamagnetic compounds: py = 3,4-dimethylpyridine (Buffagni *et al.*, 1964), 4-methylpyridine (Moore *et al.*, 1964), 4-aminopyridine (Moore *et al.*, 1964).

Magnetic and spectral data have been interpreted as showing that the blue compounds are octahedral complexes with the perchlorate ions coordinated to nickel through oxygen and that the yellow compounds are square-planar complexes in which the perchlorate ions are not coordinated.

We have determined the crystal structure of Ni(3,5dimethylpyridine)₄(ClO₄)₂ and (with Dr W. R. Busing) that of Ni(3,4-dimethylpyridine)₄(ClO₄)₂ to obtain direct evidence as to the role of the perchlorate ion and to discover the manner of arrangement of the substituted pyridine ligands in each case. Although the structure analysis for each compound was complicated by problems of disorder, our results show unambiguously that perchlorate ions are coordinated to nickel in the 3,5-dimethylpyridine compound and that they are not coordinated in the 3,4-dimethyl compound, in agree-

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ment with the conclusions from magnetic and spectral data.

In this paper we report the structure of the 3,5dimethylpyridine complex compound. Because our mode of handling the problems caused by disorder of the perchlorate ions may be of interest to others, we describe the procedure of structure analysis in some detail. In the following article (Madaule-Aubry, Busing & Brown, 1968) the structure of the 3,4-dimethylpyridine complex compound is presented.

Data

A sample of Ni(3,5-dimethylpyridine)₄(ClO₄)₂ was given to us by Professor W. E. Bull of the University of Tennessee. Crystals were grown by evaporation from dichloromethane solution. The characteristic form of an isolated crystal is that of a tetragonal bipyramid. Usually the crystals are rather imperfect and aggregated. An apparently suitable specimen, roughly a cube 0.5 mm on an edge, was cut from a larger crystal and sealed inside a thin-walled glass tube for protection from the atmosphere.

From X-ray precession films approximate cell parameters were obtained; the space group $I4_1/acd$ was indicated by the systematic absences (*International Tables for X-ray Crystallography*, 1952, pp.247–248). The cell parameters, together with the orientational parameters of the crystal, were refined by the method of least-squares from angle data of 8 reflections (Cu K α_1 radiation, wavelength 1.54051 Å assumed) determined with the Oak Ridge computer-controlled X-ray diffractometer (Busing, Ellison, Levy, King & Roseberry, 1968). The cell parameters established are a=b=15.8759(17) Å and c=26.7581(25) Å.*

The density of the compound was not determined accurately but was found to lie between that of chloroform, 1·498 g.cm⁻³, and dichloromethane, 1·336 g.cm⁻³. For 8 units Ni(C_7 NH₉)₄(ClO₄)₂ of formula weight 686·2 in the cell volume of 6744·2 Å³ the calculated density is 1·351 g.cm⁻³. It was therefore assumed that there are 8 formula units per cell.

Intensity data were recorded with the automatic diffractometer using the θ -2 θ step-scan method and Cu $K\alpha$ radiation. The 2 θ step width was 0.05°. The total width of scan for each reflection was 2° plus the width of the α_1 - α_2 doublet separation. For the 2 θ range 0° to 95° the count time for each point in a scan was 2 seconds, and the time for the background counts taken at the beginning and end of each scan was 10 seconds; these count times were doubled for the 2 θ range 95 to 159.6°. A nickel filter was used for reflections with 2 θ below 95°; no filter was used for reflections at higher angles. The bisecting position of the χ circle (ω =0°) was used for the 2θ range up to 133.5° and the parallel position ($\chi = 90^{\circ}$) for the 2θ range between 133.5° and 159.6° . A reference reflection (004 for the bisecting position and 0,0,32 for the parallel position) was recorded after every 20 reflections as a check on the stability of the instrument and the crystal. Exclusive of the measurements of the reference reflections, a total of 1956 observations were made of the intensities of 1712 independent reflections.

The quality of the intensity data is not so high as is normally attainable diffractometrically because the crystal specimen evidently contained one or more small volume elements misoriented with respect to the main body of material. This fact became apparent through the occasional appearance of a small satellite peak near the principal peak of a reflection. We estimate that the maximum error in peak intensity caused by a satellite is about 5%. Since no more promising crystal specimen was available, we decided to complete the data collection with the crystal in hand.

The raw data on punched paper tape were converted by calculations using the CDC 1604 computer to a set of structure-factor squares F_o^2 and standard errors $\sigma(F^2)$ on cards. The slight variations of intensity of each reference reflection were assumed to represent slowly varying instrumental conditions, and the reference intensities were used to normalize the data to a fixed standard value for each reference reflection. Absorption corrections calculated by the method of Busing & Levy (1957) were applied. The value of the absorption coefficient used was 26.9 cm^{-1} . The data for the parallel position were scaled to those of the bisecting position by use of the ratio of intensities of reflections 004 and 0,0,32 after correction for absorption. Different scalefactor identifiers were assigned to the groups of reflections in three ranges of 2θ as follows: (1) 0 to 95° (filtered radiation); (2) 95 to 133.5° (unfiltered radiation); (3) 133.5 to 159.6° (unfiltered radiation, parallel position). The value 5.6 Å² for the overall isotropic temperature factor and an approximate scale factor for the observations were obtained by the method of Wilson (1942).

Solution and refinement

Since there appeared to be 8 formula units Ni(3,5dimethylpyridine)₄(ClO₄)₂ in the cell, for which the general positions are 32-fold, it was clear that the nickel atoms must lie in one of the 2 sets of eightfold positions (point-group symmetry either 222 or $\overline{4}$) and that the chlorine atoms must lie on one of the 3 sets of twofold axes – if the structure is ordered with respect to the arrangement of these atoms.

Coefficients for a sharpened and modified threedimensional Patterson synthesis (space group I4/mmm) were prepared by the method described by Donohue & Trueblood (1952). The most prominent peaks in the Patterson map are listed in Table 1. The principal interactions which were inferred to contribute to each

^{*} The numbers in parentheses corresponding to the least significant digits of the parameters are the standard errors from the least-squares refinement. We use this convention for indicating standard errors throughout the paper.

peak are shown in the last column of the table. All of these peaks are explained satisfactorily if one assumes that the nickel atoms are located on the 8 points of symmetry 222 of Wyckoff set (b) and that the chlorine atoms are located at 16 points of symmetry 2 of the Wyckoff set (f). The nickel atom and the chlorine atom of the asymmetric unit were taken to be at 0, 0.250, 0.125 and 0.155, 0.405, 0.125, respectively.* We presumed at this point that the perchlorate group occurs in the structure in twofold disorder, because on chemical grounds it seemed highly probable that one of the oxygen atoms would lie between Ni and Cl on the twofold axis, which could not then be a symmetry element for the perchlorate tetrahedron. The Patterson peaks representing the Ni-Cl interactions were elongated in the plane z=0.25 perpendicular to the twofold axis, suggesting that the chlorine atom might not be exactly on the axis.

Table 1. The most prominent peaks in the three-dimensional Patterson map of Ni(3,5-dimethylpyridine)₄(ClO₄)₂

The peak heights are on an arbitrary scale such that the peak at the origin has the height 999.

	Co	oordinat	es	
Height	Ū	V	Ŵ	Vectors
628	0.5	0	0.25	Ni-Ni and Cl-Cl
519	0.5	0.5	0	Ni-Ni
188	0.155	0.155	0	Ni-Cl
90	0.188	0.2	0	CI-CI
	Height 628 519 188 90	Height U 628 0.5 519 0.5 188 0.155 90 0.188	$\begin{array}{c} \text{Coordinat} \\ \text{Height} & U & V \\ 628 & 0.5 & 0 \\ 519 & 0.5 & 0.5 \\ 188 & 0.155 & 0.155 \\ 90 & 0.188 & 0.5 \end{array}$	U V W 628 0.5 0 0.25 519 0.5 0.5 0 188 0.155 0.155 0 90 0.188 0.5 0

A three-dimensional Fourier synthesis was calculated for which the coefficients $|F_o|$ were phased by the signs of the structure-factor contributions of the nickel and chlorine atoms and weighted by the factor

$$w = \tanh \frac{|F_o| |F_c|}{\sum_i f_i^2 \exp(-2B \sin^2\theta/\lambda^2)},$$

where f_i is the scattering factor for atom *j* at rest, and the summation is taken over all atoms in the cell except nickel and chlorine atoms (Woolfson, 1956). The nickel atoms in the Wyckoff positions (b) contribute only to one-fourth of the general reflections hkl, those for which l = 2n and 2k + l = 4n; and the chlorine atoms contribute only to one-half the general reflections, those for which 2k+l=2n+1 or 4n. Consequently, many of the observed structure factors could not be entered into the Fourier synthesis, and the map contained much false detail. Nevertheless, it was possible to pick out eight atoms in a configuration which roughly described the 3,5-dimethylpyridine ligand. Another peak located on the twofold axis between the nickel atom and the chlorine atom was tentatively identified as an oxygen atom. This peak was somewhat low in

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height for an oxygen atom and somewhat elongated in the z direction.

A structure-factor calculation based on the nickel, chlorine, and carbon atoms gave a discrepancy index R(F) = 0.34, to be compared with the value 0.52 that had been obtained when only the nickel and chlorine atoms were included. In the following Fourier synthesis the peak for the oxygen atom, O(1), on the twofold axis again appeared; but the six half-weight oxygen atoms representing in the disordered model the other three perchlorate oxygen atoms, O(2), O(3), and O(4), could not be placed in the smear of electron density found near the chlorine atom.

The problems of locating approximately the oxygen atoms O(2), O(3), and O(4) and of locating precisely the atoms O(1) and Cl originally placed on the twofold axis proved to be difficult. Several Fourier and difference syntheses, many cycles of least-squares refinement, and a number of bond-length and angle calculations were required to bring the structure determination to a reasonably satisfactory conclusion. A synopsis of the successive operations follows.

A series of least-squares refinement cycles was performed into which the atoms O(2), O(3), and O(4) were entered as 12 quarter-weight oxygen atoms equally spaced around a circle and in which the weights (occupancy factors) of the fractional oxygen atoms and the coordinates and isotropic temperature factors of the other atoms were adjusted. R(F) dropped to 0.24, and the structure of the dimethylpyridine ligand became much more satisfactory. When anisotropic thermal parameters were used for all atoms except O(2), O(3), and O(4), the value of R(F) fell to 0.20. Approximate positions for 6 half-oxygen atoms were deduced from the occupancy factors of the 12 quarter-weight positions. Atom O(1) was moved slightly from the twofold axis and represented as two half-atoms 180° apart around the axis, as suggested by analysis of its anisotropic thermal parameters. Least-squares refinement brought R(F) down to 0.14. The three hydrogen atoms attached to the ring carbon atoms were located in a difference map, and the three hydrogen atoms of each methyl group were represented by 12 quarterweight atoms on a circle. The chlorine atom was moved slightly from the twofold axis and represented by twohalf atoms. Adjustment of the occupancy factors of the fractional hydrogen atoms, the coordinates of the ring hydrogen atoms, and the coordinates and the anisotropic thermal parameters of the other atoms lowered R(F) to 0.122. The hydrogen atoms of each methyl group were individually located from a difference map, and the coordinates and isotropic thermal parameters of all the hydrogen atoms were subsequently included among the parameters refined. The scattering factor curve for nickel was corrected for anomalous dispersion at this stage (through oversight this correction had not been made earlier). R(F) became 0.099; $R(F^2)$, 0.087; R_w , 0.1089; σ_1 , 1.70. Finally the six reflections of highest intensity, judged as a group to be affected

^{*} All coordinates quoted in this paper are referred to the origin at \overline{I} (*International Tables*, 1952, p. 248).

Table 2. Observed and calculated structure factors for $Ni(3,5-dimethylpyridine)_4(ClO_4)_2$

For each reflection, identified by the indices h and k of a subheading and the running index l, the values of $|F_o| \times 10$ and $F_c \times 10$ are given. The standard error $\times 10$ of $|F_o|$ (see text) is given under the heading SG, except that for each reflection marked W, for which $|F_o|^2 < \sigma(F^2)$, the standard error $\times 1$ of $|F_o|^2$ is given instead. The 6 reflections marked X are the reflections of highest intensity, which were excluded from the final refinement as possibly affected slightly by extinction.

L OBS CALC SG	L OBS CALC SG	L OBS CALC SG	L OBS CALC SG	L OBS CALC SG	L OBS CALC SG	L OBS CALC SG	L OBS CALC SG	L OBS CALC SG	L OBS CALC SG	L OBS CALC SG L OBS CALC SG
*** 0 0 L ***	8 824 762 14	17 508 -539 9	19 88 18 13	4 2196 2187 33	4 979 973 11	*** 15 7 1 ***	174 56 68102	h 111 -107 27	14 159 176 15	2214 14 68 42 8 129 -25 30
4 1648 -1448 25	10 600 -637 12	19 340 -318 12	211 34 99 62	8 2016 -1993 30	6 156 170 17	1 144 153 21	19 275 319 13	6w 0 91 64	16 341 371 17	10 806 -807 14
8 1733 1722 26	12 299 -279 13	21 277 309 12	23 103 28 24	12 680 693 12	8 1195 -1206 13	3 100 -69 20	21W 76 2 60	8 97 -3 30	184 32 -4 61	1 107 -10 16 15 767 750 15
16 3030 3179 46	16 757 755 9	25 152 124 20	274 0 -53 37	20 1049 1048 17	12 480 482 10	7 137 123 20	259 18 -2 41	12 104 -14 27	22W 48 0 61	3 272 258 17 16 161 -208 20
20 1748 -1740 27	18 101 -152 31	27 150 -125 19		24 256 -269 14	144 65 -102 72	9 189 194 15	27¥ 33 -43 49	14 125 115 24	24W 57 56 57	5W 0 55 62 18 612 -623 12
28 125 138 21	20 425 -436 10	31 92 -58 19	2 517 -516 9	28 196 181 15	16 349 -364 12 18w 0 57 62	11 227 -185 13 13W 0 -54 51	29 163 158 13	16 159 -126 18	264 69 32 50	7 79 12 37 20W 45 -122 64 9 115 -49 26 22 419 371 11
32 355 345 8	24 149 200 14	33 108 -95 11	4 222 -221 15		20 300 330 12	15 81 43 28		204 49 -64 45		11W 63 17 59 24 93 -61 29
			6 547 550 10	*** 3 2 L ***	22 96 70 31	171 0 100 44	*** 7 3 1 ***	22 74 -42 25	*** 7 % [***	13 96 102 29 26 194 -196 13
0 1718 1777 18	0 728 698 13	2 393 -393 6	10 610 -622 10	3 1810 -1785 19	26 266 -267 12	199 60 -97 38	4 313 305 12	*** 14 3 1 ***	3 1100 -1032 17	15 105
2 520 517 6	2W 0 -94 61	4 504 490 7	12 236 -247 15	5 165 143 10	28 226 246 10	*** 16 2 L ***	6 611 -574 12	1W 0 -92 61	5 204 131 18	19₩ 0 71 48
6 519 -516 6	6W 0 77 61	8 747 729 9	14 228 237 16	7 83 -39 22	*** 9 7 1 ***	0 306 -272 11	10 258 265 17	3 299 -314 11 5W 74 144 61	7 229 -237 17	21W 0 24 40 40 8 5 L 400
8 234 -218 8	8 219 164 14	10 1452 -1446 16	18 316 -277 11	11 1173 -1171 13	1 329 341 14	4 291 280 11	12W 0 -19 93	7 141 159 21	11 264 -195 17	*** 14 4 L *** 3 515 -472 12
10W 0 -55 36	10 188 209 12	12 529 522 8	2014 0 -58 59	13 819 -825 10	3 853 -868 14	6W 31 35 52	144 0 -49102	94 0 -29 41	13 115 -129 43	OW 0 76 62 5 802 760 14
14 222 -169 11	14 125 113 23	16 112 60 27	24 91 34 22	17 129 -146 25	7 402 -375 13	10 68 4 34	18w 32 -32 65	13 267 269 12	17 236 213 13	4 326 -342 12 9 226 -240 22
16 738 718 9	16 530 522 11	18 627 -640 10		19 200 206 18	9 146 -182 34	12 226 203 11	20 142 95 21	15 91 49 28	19W 0 -33 63	6 92 -1 31 11 145 -144 36
20 982 -955 12	20 282 -295 9	22 354 368 11	1 136 -166 26	23 181 -174 17	11 246 -264 22	16 204 -220 11	24 93 -19 29	19 77 -38 26	23 148 118 19	8 287 306 9 13 326 308 18 10w 27 -11 57 15 274 298 14
22 84 22 36		24 152 -207 22	3 163 -206 23	25 206 211 11	15 475 479 11		26 177 169 16		25 85 -5 31	12 202 -203 15 17 251 -241 13
24W 6Z -5 60 26 298 306 11	0 131 -131 20	26 535 -530 11	5 111 59 32	27 380 -374 10	17 189 -198 17	1 187 -172 13	28 69 85 30	*** 15 3 L ***	274 60 67 46	14 93 -19 27 19 423 -442 11
28 133 -121 20	2 161 157 16	30 214 185 10	9 446 -461 11	31 90 -89 15	21 245 -266 15	3 114 76 20	*** 8 3 1 ***	4w 73 -59 56	314 0 27 20	18W 32 24 42 23W 74 84 57
30% 57 73 42	4 100 -72 18		11 429 -434 11	33 67 45 17	23W 50 18 55	5W 50 58 47	1 568 529 11	6 382 -350 11	*** 8 4 1 ***	20 129 -108 15 25 138 -128 17
,	8 93 -67 27	1 1226 -1254 11	15W 0 79 65	*** 4 2 1 ***	27 0 -64 40	9 96 -79 22	5 205 -201 19	10 335 321 11	2 565 -520 12	*** 15 & L *** 29W 0 10 25
01477 -1174 L	10W 64 37 48	3 90 -121 20	17W 65 73 61	0 2406 -2365 26		11 78 -70 24	7₩ 59 111 81	12W 0 13 50	4 486 -548 12	1 171 164 17
2×3527 -3673 37	14 131 148 16	7 607 618 8	194 58 31 58	2 295 -290 6	0 758 -797 14	13 75 11 23	9W 17 135 89 11W 0 -48 96	14 252 -246 11	6 745 -710 13 8 385 390 14	3 217 -207 14
4 1455 -1494 16	16 148 168 13	9 355 360 8	23W 0 48 42	6 435 424 7	2 99 85 42	*** 18 2 L ***	13 105 13 46	18 170 165 12	10 114 -58 40	7 103 77 27 4 126 -113 36
8 383 -360 7	*** 18 0 L ***	13 347 345 10	*** 13 1 1 ***	0 1975 -2007 21 10 319 324 R	- 345 877 15 6w 4818 et	2 128 -98 16	17 268 -267 13	*** 16 3 L ***	12 613 -610 13 14W 55 -57105	9W 61 58 54 6 162 145 29 11W 35 21 50 8 179
10 838 840 10	0 98 69 21	15W 0 -106 66	2 970 -965 16	12 1704 1741 18	8 1076 -1125 18	4 185 164 11	19W 31 -96 64	1W 60 129 57	16 423 447 11	13 105 -112 23 10 368 -363 15
12 1189 -1185 13	2 105 101 20	17 595 -587 10	4 153 128 20	14 95 -40 28	10 197 -156 28	6W 32 4 36	21 98 -139 32	3 94 67 20	18w 36 -109 65	15 85 -22 24 12 149 -186 36
16 510 -478 9	6 74 -48 26	218 55 -51 60	8 218 -223 15	18w 70 -109 70	14 102 -103 30		25W 0 -40 51	7 92 40 27	22 263 -236 12	164 0 60 62
18w 80 18 67	8 67 82 28	234 41 -50 60	10 158 -110 20	20 580 582 11	16 408 -411 11	*** 19 2 L ***	27W 0 -69 31	9W 34 3 49	24 341 312 11	*** 16 & L *** 18 427 -440 11
22 124 -130 25	*** 20 0 L ***	27 72 -54 35	12 276 -272 13	22 112 -126 28	18 189 176 17	1W 23 -47 29	31W 0 37 18	11W 0 546	26 75 26 29	0 357 355 11 20% 0 -14 62
24 90 98 34	0 163 173 8	294 45 -51 42	16 95 -53 29	26 115 -154 26	22 137 123 20	*** 20 2 L ***	*** 9 3 L ***	15W 39 -41 38	30W 0 -10 22	4 311 -316 11 24 82 5 27
28 368 -367 10	4 118 -142 9		18 513 -483 10	28 299 259 11	24 255 -252 11	0 57 -55 18	2 1304 1322 20			6w 49 -60 49 26 76 -81 26
30 74 -62 29		2 566 590 7	22 251 258 10	32 235 -215 7		4 64 57 14	6 1015 -997 17	2 261 231 11	1 238 227 17	10W 0 20 45 *** 10 5 L ***
*** 6 0 1 ***	2X4478 -5482 67	4 895 877 10			*** 11 2 L ***		8w 0 28 95	48 48 1 44	3 348 -378 14	12 230 -259 12 1 124 182 39
0 1552 1563 17	6 2063 2045 31	8 71 76 34	1 164 146 19	1 863 -848 10	3 399 371 14	214460 4694 67	12 142 -87 36	8w 0 -31 42	7 245 224 19	5 475 -521 14
2 1527 -1480 16	10 1316 -1354 20	10 1379 -1392 19	3 366 340 11	3 1278 1281 14	5 108 -117 47	6 2568 -2584 39	14 899 -873 16	10 217 204 11	9 216 183 24	*** 17 & L *** 7 308 322 18
6 420 412 7	18 1370 -1394 22	14 1024 1015 12	7 182 -201 18	5 1758 1722 19	7 249 -247 21 9 933 -947 16	10 1509 1553 23	18 612 635 12	17W 0 -6 35	11 259 -250 20	1 W 31 -12 43 9 159 -172 34 3 80 21 26 11 276 300 13
8 1677 1658 18	22 1391 1375 22	16 410 427 10	9 85 69 34	9 151 74 13	11 788 791 13	18 1278 1291 20	20 160 155 14	*** 18 3 L ***	15 136 135 24	5 71 68 31 13 88 10 35
12 1088 -1056 12	30 247 233 11	20 242 252 14	11 193 148 15	11 756 754 9	13 184 190 19 159 79 -81 64	22 743 -755 13	22 525 -514 11	1W 0 -3 39	17 166 172 20	7 0 -26 42 15 165 -170 20
14 198 203 16	34 177 -179 7	22 509 523 11	15¥ 39 -51 55	15 443 -430 9	17W 0 -3 63	30 436 -412 9	26 276 274 10	5W 39 15 37	21₩ 0 -21 61	11 85 37 21 19 246 250 14
18 493 469 10	*** 2 1 1 ***	26 487 -467 10	17¥ 70 56 50	17 310 -308 13	19W 0 -30 62	*** 1 1 ***	30 204 -213 6	7W 0 -3 36	234 41 54 55	21 166 -167 17
20 487 -474 11	1 466 436 5	28W 0 40 43	21 76 -85 25	21 694 726 12	23W 47 -97 47	1 1120 1095 17	*** 10 3 L ***	*** 19 3 L ***	27 68 -11 26	0 192 168 11 25 68 -32 27
22 73 128 63	3X2451 2405 26 5X4169 -4290 44	32W 14 33 18		23 97 -123 31	25 96 -168 21	3 390 374 9	1 350 -291 14	2 68 38 20		2 83 -52 23
26 204 -212 14	7 2144 -2114 23	*** 8 1 L ***	2 181 -148 17	27 124 -68 20	*** 12 2	7 410 429 10	5 167 -171 29	10 69 38 14	0 1298 1284 21	6w 43 17 34 2 380 -391 16
28 488 -506 10 30 99 -107 14	9 657 661 8	1W 28 113 45 3 939 958 11	4W 0 -31 40 6 131 116 77	29₩ 0 48 31	0 540 -599 14	9 897 -893 14	7 212 -233 25	*** 30 5 1 ***	2 409 431 14	14W 28 57 18 4 118 106 43
	13 858 -873 10	5 279 263 10	8w 47 50 55	*** 6 2 1 ***	4 491 533 14	13 107 -147 36	11 283 308 20	1 53 26 16	6 157 90 30	*** 19 & L *** 8W 0 -2 60
*** 8 0 L***	15 84 -101 26	7 233 240 12	10 169 174 17	0 550 -529 7	6 186 -171 16	15 160 -116 26	13 375 -392 11		8 897 885 16	1W 32 -23 26 10 297 -310 13
2 1407 1323 15	19 744 755 10	11 220 175 15	14 184 197 15	4 568 571 7	10 86 -23 36	19 536 516 14	17 319 -368 12	0 1287 1296 20	10 211 -219 26	3W 0 35 27 12W 26 23 62 5 48 -7 24 14 128 191 26
4 1078 -1064 12	21 101 55 29	13 354 -303 11	16- 0 -8 45	6 284 290 8	12 204 249 17	21 169 -149 19	194 0 -33 64	4 1554 -1574 24	14 186 -206 18	7 54 -4 20 16 100 36 29
8 2247 2281 24	25 .232 246 14	17 184 189 18	10 154 -105 8	10 401 387 8	16 303 -305 13	23 74 55 59	21W 0 -13 58 23W 0 -8 51	8 2296 2305 35	16 669 683 12 18w 80	9 90 -73 11 18 204 -186 15
10 234 234 13	27 274 266 12	19 219 205 15	*** 16 1 L ***	12 847 868 10	18w 75 34 56	27 101 -5 25	25W 52 -11 42	16 1518 1590 24	20 587 -550 11	*** 5 5 L *** 22 247 225 10
149 71 -69 71	31W 0 -68 40	23 190 -200 16	3 99 45 75	16 436 -425 10	20 246 238 12 27 79 -31 28	31W 0 -20 31	*** 11 3 L ***	20 512 -533 11 24 498 509 8	22 130 81 20	2 T453 -1445 22 24W 52 53 38 6 2022 2025 31
16 418 382 11	33w 25 31 26	25 126 118 21	5W 64 51 52	18 317 -314 13	24 91 -89 20	33 86 73 11	2 762 823 14	28 303 -289 10	26 106 95 16	10 138 -201 30 *** 12 5 L ***
20 170 -172 20	*** 3 1 L ***	2/4 23 111 4/	9 130 68 13	22 137 -160 23		*** 5 3 1 ***	6 701 -726 14	32 137 132 10		14 951 948 16 1 500 529 11 18 542
22 278 272 9	2 208 -223 6	*** 9 1 L ***	11W 15 69 47	24 437 -394 10	1 454 458 10	2 789 818 13	8 151 27 32	*** 5 % L ***	1 523 -493 14	22 185 217 17 5 517 -496 11
26 45 64 34	6 807 -760 9	4 209 -228 13	13W 0 -0 43	28 269 252 11	5W 61 84 60	4 535 -541 10 6 1415 -1444 27	10 654 648 12	1 929 -977 15	3 436 448 15	26 224 -211 13 7W 0 -32 61
28 307 -337 9	8 599 620 7	6 800 837 10		30W 54 40 36	7₩ 0 -4 62	8 377 -399 11	14 376 -396 11	5 1168 1179 18	7 348 -369 17	11 205 206 16
*** 10 0 L ***	10 1047 -998 12	10 1240 -1273 14	2 151 -117 16	32 92 -71 12	9 168 154 17	10 587 559 11	16W 0 34 63	7 1012 -989 16	9 273 277 13	*** 6 5 L *** 13 105 -138 31
OW 66 47 55	14 286 311 10	12 88 29 40	4W 60 -67 48	*** 7 2 L ***	13 66 59 32	14 987 -999 16	20# 64 -52 58	11 427 471 12	13 239 213 14	3W 38 24 66 17 188 179 16
7 169 175 17 4w 75 158 50	16 111 -160 27 18 476 -504 10	14 384 422 12	6 164 150 15	1 134 -30 15	15 316 305 11	16 286 305 19	22 306 -283 11	13 391 349 13	15₩ 0 -40 64	5 144 -164 24 19 186 197 15
6 109 -125 28	20 285 -275 13	18 166 165 19	10W 40 -14 41	5W 0 49 45	19W 69 8 49	20 103 -38 29	244 51 -29 40	15 244 -243 20	17 511 -465 11	7 96 83 37 21 144 -130 15
8 348 341 11	22 108 -18 27	20 110 144 28	12W 23 29 38	7 633 621 8	21 106 -70 20	22 388 -401 11	*** 12 3 L ***	19 212 237 16	21 110 62 23	11 340 283 14
12 931 -976 12	26 373 -330 11	244 44 92 55		11 400 397 9	*** 14 2 1 ***	26 264 261 12	3 213 -240 25	21 168 177 18	23 73 -107 31 25W 54 47 14	131 98 -163101 *** 13 5 L *** 15 215 -187 24 2 *08 -208 **
14 160 -162 20	28 131 -130 20	26 247 -254 11	1 85 -57 24	13 352 -331 11	0 384 -375 11	28W 0 62 33	5W 65 50 57	254 74 -110 57		17 167 191 19 4w 43 25 60
18 126 -160 24	32 59 -72 25	20 6/ 101 26	3 104 -92 19 5 77 60 26	15 128 78 26 17 190 135 19	2W 0 15 58	30 142 -153 14 32 87 106 14	7 327 340 12 9 260 254 11	27 232 213 12 29 86 81 17	0 310 940 19	19W 0 -68 65 6 296 283 12
20 145 166 22		*** 10 1 L ***	7W 35 108 40	19 206 -238 16	6 123 -134 25		11 182 147 18	31W 35 50 27	2W 0 26 60	23 127 -135 24 10 493 -481 11
24 136 -139 23 24 198 192 13	1 2834 -2749 30	3W 69 41 58	9W 55 19 35	21W 0 -19 64 23W 30 -31 61	8W 0 11 61 10W 0 -5 47	1 709 700 17	13 106 -125 31	*** 6 1 1 ***	4₩ 0 22 62	258 61 73 53 12 206 179 15
264 0 -2 40	3 2080 -2072 22	5 290 262 12	*** 20 1 L ***	25 245 265 13	12 192 147 15	3 218 238 13	17 138 -115 21	0 234 216 12	8 96 -89 31	29W 29
*** 12 0 L ***	5 637 -649 8 7 726 -674 8	9 126 44 27	1W 8 -12 21 3W 0 -20 10	27 98 -94 25	14 146 88 19	5 925 937 15	19W 0 -26 55 21W 38 97 40	2 98 89 29	10 339 349 12	31W 45 -28 20 18 168 -164 15
0 999 942 12	9 451 418 7	11 406 -402 11	5 43 -3 20		18W 0 4 46	94 48 -15 69	23W 52 1 39	6 369 -354 12	14 214 248 15	*** 7 5 L ***
z 91 -17 38	11 86 -44 25	13 290 303 12 15W 0 -1 41		0 124 -0 -0	20 137 140 16	118 69 -11 76		8 614 587 12	16 101 -86 30	2 1454 -1498 22 *** 14 5 L ***
6 199 -126 19	15 371 364 9	17 253 268 14	0 1876 -1827 28	2 98 37 23		15 350 343 16	2 90 -36 33	12 249 -772 19	20 131 -127 20	6 1178 1766 19 3 183 -725 18

Table 2 (cont.)

Ľ	08 S	CALC S	e	L	085	CALC SG	L	08 S	CALC S	١٦	ιc	BS CALC SC	5 L	. 04	BS CALC SG	۱L	08 S	CALC SG	۱۰	08 S	CALC SG	۱	08 S	CALC SG	L	08S (ALC SG	۲Ľ.	08 S	CALC SG	ויי	085 (CALC SG	LO	85 CA	LC 56
***	14 9		•	,, ,	185 -	-211 28	21	114	-71 1	۹Ì,	εv	0 5 10	, I ,,	11	KR 115 16		96	-101 23		• 11	8 6 ***	4	157	-148 14	159	67	18 46		11 10	1 ***	,	7 10	L ====	16	57	57 19
5	199	243 1	7 I	1 3W	0	58110	2 3 W	46	62 3	8			23	w .	0 8 39	Ś	72	53 30	1	159	115 19	6	96	29 20	178	38	-84 42	•	123	-70 24	1₩	31	71 26			
7₩	0	-27 5	8 ·	15 3	206 ·	-175 16				•	** 1	8 6 L ***	25	w	0 23 27	7	68	70 31	33	4 71	-127 64	8	125	100 16	19	69	-58 26	3	93	142 34	3	66	28 17	*** 1	3 12	L
9₩	32	-78 5	9	17 4	612	-444 11	***	12	6 L **	•	0 1	54 -149 11	27		69 33 13	94	56	-120 43	5	341	-367 11	101	• •	-15 36	21W	41	-5 24	5	190	-144 15	5W		3 27	1 1	15	73 20
11	214	-213 1	1	19	85	-2 35	0	661	-680 1	2	2₩	42 48 29	3	• •		11	. 79	-102 23	1 7	394	413 11	12	138	-139 11	2 3W	0	-23 16	12	234	212 14	7	66	45 14	3	17 -	57 30
15₩	53	36 4	å	21 · ·	67	9 55	1	244	260 1	2	6W	53 62 20 44 19 26		. 10	RA 410 11	17		+1 18	11	107	-91 28	16	47	26 18	***	13 9		hí	117	-104 25	1	1 11	ر	7₩	53	-6 39
17W	ő	64	3	25	101	7 22	6	191	177 1	6	8	88 -104 1	5 4	1	53 -132 19	1			13	219	-209 16	1			2	119	112 23	13W	71	-66 54	2 1	33	71 21	9W	56 -	75 38
19	118	-113 1	5	27	119	90 16	8	450	-449 1	1	0₩	0 -14 22	2 6	i 11	68 -189 19	•••	• 17	7 L***	15	103	-43 27		• 17	8 L ***	4	145 -	128 20	15	84	62 30	63	166 ·	-359 11	11₩	0	57 36
2 3 W	0	30 1	8	29	94	88 14	104	, 0	-21 6	5 1	2	82 111 11	ו ו	1	30 -1 32 25	2	73	103 27	17	204	196 15	11	w 29	49 36	6	218	211 14	17₩	0	-40 44	10 1	66	169 16	13	56	27 19
					a 4		12	475	460	8			. 10	1	61 156 20	4		-51 37	19	168	-159 14	1.	w 0	-2 35	8	91 161 .	-67 29	19	62	92 30	14 1	27 · 80	-116 18 68 20	15W	9	13 21
,	366	-148 1		0 10	059-	1072 17	16	251	-218 1	; -	14	0 4 21			45 -10 01 90 -253 12	8	. 49	-4,28	2 3		13 27	1 7	w 0	-22 29	124	22	21 48	230	0	20 18		•,	,0 ,0	*** 1	12	
4	114	86 2	4	2 :	215	176 21	18	51	-44 5	2	3W	39 43 21	1 10	w :	34 13 57	10	133	110 10	25	46	51 20	9	w 0	3 27	14	126	99 18				*** 1	2 11	۱ ***	03	1 3	039
6	349	345 1	٥í	4	600	585 13	20	244	235 1	1	5W	43 -44 19	9 18	2	65 264 12	121	31	-6 24				į m	w o	1 23	16w	51	-47 37	***	12 10	L	1 1	19	91 23	2 1	7	94 17
8	1 39	101 1	8	6	176	-210 26	22	123	92 1	5			20	1	06 54 21	14	67	-23 14	1.	12	8 . ***				18	70	76 20	0	303	243 11	3₩	•	-44 57	4 1	55 - 1	57 12
10	340	-322 1	<u>°</u>	8	342	-324 16				. '		776	2	1	84 -178 12				1 0	486	493 11		- 18 RL	3 1 14	70W	0	8 22		175	-14/ 1/	, ,	144	318 11	8 1	(9 (1 1	41 11
14	208	206 1	;	12	613	660 14	1	186	-197 1	8	6 8	319 -798 14	211	,	50 57 10	1	74	42 17	14	107	-278 12	21	w 0	5 23	***	14 9		6	99	-34 27	9 1	98 -	-187 14	104	o -	73 76
16	96	-47 1	9	149	0	-110 64	31	1 0	49 6	1 1	0	98 988 1	, ••		2 7 L ***	31	23	4 25	6	160	-1 39 20	4	114	-126 10	19	0	63 53	8	215	-211 14	11 2	107 •	-204 12	12 1	19 -1	37 9
				16	503	-493 11	5W	r o	-14 6	2 1	14 8	391 -901 1	5 1	1	23 95 25	51	• 0	-15 25	8	607	649 12	61	w 7	23 20	3	129	124 20	10W	0	47 53	13 1	28	126 18	149	39 -	30 17
***	16	5 L **	*	184	0	-7 64	1	109	-45 2	7 1	8 (695 1	2	2	43 258 14	71	• •	-23 23	10	118	-103 25	8	1 35	150 7	5	179 .	-175 15	12	108	94 23	15	65	41 29			
1.	61	-27 4	9	20	394	376 11	9	87	-80 2	5 2		357 -353 11		; 1 	39 -105 22	19	• •	-40 20	12	214	-207 14	i	• •		1	41	-40 49	140	44	39 45	174	0	-60 29		, יי , _	41 17
54	0	24 5		22 91. ·	104 208	-135 17		198	150 1			239 10	12	2	59 51 04 07 186 17			7	16	149	118 10	,	960	-1008 16	11	143	143 16	184	21	6 14			-,		7	-0 27
,	120	-70 1	9	26	72	-3 25	154	1 37	40 5	2 .		87L+++		2	27 218 15	2	78	78 11	181	- 53	-73 43	6	526	544 11	13	129 -	119 16	20¥	47	-21 23	*** 1	3 11	ι	5	53 -	24 19
94	0	45 4	5	28	63	73 21	17	81	77 2	9	1.6	571 -681 1	3 1 1	1.1	18 -23 24	· ·			20	120	-91 16	10	253	-294 14	15W	20	-53 36				2 1	12	64 22	7W	0 -	33 73
119	49	-7 4	1				19	14	15 4	2	3 (68 -717 14	1 19	; 1	81 -124 35	***	• 8	8 L ***	2 21	26	-43 25	14	406	402 8	17₩	34	11 25	***	13 10	L ***	49	0	-26 50	9	+8	50 21
134	0	-55 3	19		96						5 1	48 112 33	2 13	W 1	69 50 51	0	302	323 18	24	140	137 7	18	571	-515 11	19	91	72 10	11	109	-134 26	6 1	82 -	-177 14		-	48 12
194	21	6 1	1		116	167 40		14	6 L		7	11 54 4	3 19	*	0 -16 39	4				. 12		22	288	281 10		16 9	,	12	182	-173 16	10 1		150 16	*** 1	\$ 12	
***	17			ŝ	.,,,	21 41	25	, 56	-13 4	å,		158 -109 1	7 2	w	34	12	293	-251 12	· 1	104	59 19	1.0			214	6	-21 45	17	106	92 24	12W	0	13 40	0	98	98 11
2	138	-100 1	5	7w	0	96105	4	445	412 1	ī ,	3	249 278 14				16	315	327 12	31	. 0	-116 64		• 10	9 L ***	4.	0	18 47	9	163	143 16	14 1	06	-83 14	, ,	31	84 13
44	0	76 4	2	9₩	0	-64106	6	184	189 1	6 1	15 1	79 170 18	8 **	* 1	37 L***	20	386	-373 10	; 5	88	92 35	1	129	157 26	6	227	194 11	11	130	-163 19	16W	45	-10 24	4w	0	3 19
6¥	0	50 4	٩í	11	104	-70 27	8	610	-580 1	, ,	17 1	36 -126 2	3	1	22 448 11	24	73	5 26	: 7	79	-31 35	3	286	-261 13	8w	36	-17 47	1 3W	.64	-87 42	18	81	85 12			
8	69	45 2	2	1 3W		-76 65	10	4.40	29 5	4	9 1	96 -203 1			70 180 18	28	64	-62 15	1 94		27 56	1	255	269 14	10	207 4	-195 11	174	20	20 30		4 11		, ,	, , , , , _ ,	12 9
164	126	-13/ 1		17	101	57 30	14	134	-109.1	8 2		62 54 44			5/ -5/0 0 85 -85 16		• •	8 1 ***	13	- 0 - 0	-41 52	l 'a	W 67	67 63	144	50	48 26	19	67	-23 15	1₩	0	45 46	6 1	50 1	34 13
	•	• •	1	19	150	-137 21	16	208	-207 1	2 2	25W	0 17 4	1 10	5	55 512 11	1	478	-471 14	15	w 0	-6 46	11	93	-64 33	16	49	25 21				3 1	16	86 19	10 1	.7 -1	46 10
***	18	5 L **	-	21W	60	35 57	18	91	-25 1	9 2	27W	44 37 28	8 1:	2	96 -77 28	3	405	374 15	171	w 14	-45 41	13	115	48 25					14 10	L ***	5	99	59 20	14 1	55 1	497
1	105	89 1	7	73	81	-33 29	224	34	-1 1	9 2	9₩	0 3 18	B 14	• 3	95 -382 10	5	360	342 16	211	w 0	-11 23	15	1 38	81 22	***	16 9	L ***	ow	72	147 52	7W	19	-30 40			
3W	42	82 3	15	25W	0	12 41							. 1	5 1	35 118 17	17	273	-271 12		• • •		17	¥ 0	-37 57	11	ŝ	-7 38	2	86	79 28	9₩	53	5 36		• 13	
5	62	-23 2	6	•••						<u> </u>	· ·	9 / L		5 1 5 1	36 134 16 90199 8	1.	222	216 15	1 0	,	28 53	1,,	71	35 27	, 54	0	-76 39	64	15	-19 45	13	91	-44 13	3.	0	10 28
	59	76 1	9	0	121	47 41		85	50 1	1	4	237 -208 2	2		<i>,</i> ,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	113	114	147 29	21	w 56	38 52	23	107	-83 13	7.	0	-9 35	8	173	-141 13	15W	27	-37 21	5	54	-2 74
1 3W	41	-36 1	8	2	149	-171 34	ŚW	, ō	-28 5	4	6w	78 7810	2 4		4 7 L ***	15	108	-110 28	41	w o	-63 56	25	w 10	-41 20	9 w	0	-15 30	104	0	-54 43				7₩	0 -	28 25
			. 1	4	102	-7 47	7	112	-26 2	"	8 1	64 121 31	1	w.	0 -23 57	17	190	-211 17	6	127	-98 21				119	0	14 26	12	142	133 14		5 11	1 ***	9	•6	31 23
	19 1	5 L **		6	123	-123 42	9	88	30 2	7 1	0	99 -93 30			93 11 29	19	330	312 11		212	207 14		- 11	9 1	1 314	27	23 21	144	17	-13 28		79 8a	0 18		-	79 17
Ĺ	47	-13 2		10	166	145 19	1 35		-7 4	2 1	4	80 -23 1		, .	84 112 34	23	. °,	-16 40	12	280	-257 10	4	w 0	-77 67	***	17 9	1 ***	184	0	4 17	6 1	61 .	-162 11	*** 1	5 13	
6	49	63 2	· 1	12₩	64	-102 63	15	94	78 2	0 1	16W	0 -110 6	7 9		34 -5 54	25	w 52	22 29	141	w 44	30 41	6	521	511 11	2	136	-127 11				8	53	47 75	2	- 37	91 15
8w	0	51	8	14	172	177 19				1	8	353 325 1	1 11	1 1	03 10 23	27	w 25	-23 19	16	88	-46 20	8	W 58	-19 59	44	36	25 28	***	15 10	1 ***	10 1	33	142 10	4	61 -	40 16
				16₩	55	-25 61	***	16	6 L **	* 2	20¥	24 -45 5	8 1	3 1	04 24 21				181	w 0	-26 25	10	385	-357 11	6	209	207 8	1.	0	-69 42	12₩	32	25 20	6₩	20	17 17
	6	6 L **		18	105	-77 30	0	104	-57 2	2 2	22	00 -8 24	11	5	55 66 47		10	8 [20	w o	2 20	12	4 0 9	29 58	10	1 20	-120 24	3	108	-54 41		6 11	,		. 11	
	1118	2227 9		20	243	-91 20	1	/ 0 81	44.7	9 2	74 26	55 22 2			92 -49 20 34 -11 19	1,	217	/8/ 13		• 15	8 L ***	16	420	36 32	1.0	130	-129 0	7.	21	11 38	1	61	-36 22	0 1	30 -1	50 10
8 1	468	-1511 2		24	64	1 30	6	, °o	11.4	, ,	28w	0 -21 1	- i		, -ii ii		468	467 11	1	109	-108 23	18	196	-195 13	***	18 9		9₩	0	-14 36	3	65	-45 21	4	55	59 18
12 1	202	1182 1	9				8	90	-83 2	5			•	•• 1	57 L***	6	123	99 25	3	188	173 14	20	w 24	9 39	1	57	-38 16	119	0	28 29	5₩	0	-9 26			
16 1	232	-1237 1	9	***	11 6	5 L ***	10	66	163	• 0	•••	10 7 L **	•	2 1	07 78 24	8	213	195 15	5	125	115 19	22	214	216 8	3	64	23 13	1 3¥	0	22 26	7₩	0	-53 25			
20	557	605 1	1	1	350	396 18	12	. 44	67 3	9	1.1	139 -178 3	8	₩.	0 46 52	10	165	180 19	1	76	-73 30	24	W 32	10 18				15	61	-60 15	9₩	19	-2 20	i i		
24	385 238	-379 1		3	116	128 27	1.9	/0	-411	1	34	90 -128110 16 169 1		5 1 Ru	32	12	195	-160 17	1,1	129	-12/ 27		• 12	9 1 ***	1.0	10 10 1139 -	1178 18	***	16.10			17 11	1			
20	4.30	1.57	1	,	134	-123 24		17	6 L ••	-	,	80 -27 1	8 10)" 2	19 196 11	16	424	394 11	113	112	120 16	1	w 69	-112 60	4	471	512 11	0	159	-119 12	2₩	0	35 19	•		
***	7	6 L ••	•	9	191	206 17	1	95	111 2	2	9w	46 105 6	4 1	7W .	0 -33 43	18	117	89 24	15	wo	-44 27	,	W 25	-34 61	8	386	-425 11	2	90	-0 18	1			i		
1₩	0	31 7	18	11	179	-163 18	31	4 46	-36 4	u 1	11 - I	104 -82 2	9 11	•	72 -53 26	20	306	5 -312 10	17	49	-70 23	5	195	199 11	12	563	530 11	4	181	182 10		12 17	۲			
3	122	59 3	12	13	88	-92 34	5		-56 4	10	3	163 102 1	8 18	3	50 5 22	22	83	-12 23	-	• • •		17	W 47	-59 63	16	423		6	90	-56 16	• •	84	-51 29			
5	370	367 1		15	169	149 18	1 .	(45 (14	42 3	12 I	15	34 -51 2	¦ _			24	147	112 10		- 16	8 L ***	1,2	130	106 22	20	194	191 11 -200 4	1 OF	11	-/08 8	. 8	357 · 107	-329 10			
9	336	313 1	6	19	127	-102 21	113	· 30	-69 2	5 1	19	196190 1	;	. 1	11 -89 20	10		·, 20	2	w 0	-12 43	1.	- 0 w 68	-17 57	11	-04		12	110	108 9	112	208	-192 11			
-				-							·					×																				

by extinction, were omitted, yielding values of the goodness-of-fit parameters* as follows: R(F)=0.101; $R(F^2)=0.065$; $R_w(F^2)=0.103$; $\sigma_1(F^2)=1.58$. The final parameter shifts were all less than or equal to the corresponding standard errors σ except that the shift of β_{33} for the chlorine atom was 1.5σ .

In the full-matrix least-squares refinement the quantity minimized was $\sum w(|F_o|^2 - S^2|F_c|^2)^2$, where w is the weight of a particular observation, S is the factor scaling the $|F_c|$ values to the $|F_o|$ values for a given group of reflections, and the summation is taken over all symmetrically independent reflections. Three dif-

* $R(F^m) \equiv \Sigma ||F_o|^m - |F_c|^m |\Sigma|F_o|^m$; for m = 1, $R(F^m)$ is the discrepancy index usually reported.

$$R_w(F^2) \equiv [\Sigma w(|F_o|^2 - |F_c|^2)^2 / \Sigma w|F_o|^4]^{1/2}.$$

$$\sigma_1(F^2) \equiv [\Sigma w(|F_o|^2 - |F_c|^2)^2 / (n-p)]^{1/2}.$$

In these equations w is the weight of the observation $|F_o|^2$ and p is the number of parameters fitted to the n observations; the weights and the structure factors are on the correct absolute scale established by the refinement. ferent scale factors for the three groups of data given different scale-factor identifiers were adjusted in the refinement. The weight of each observation $|F_o|^2$ was taken to be the reciprocal of the variance $\sigma^2(F^2)$, after first empirically correcting the purely statistical variance by addition of the term $(0.03F^2)^2$, as is customary in this laboratory. The values $|F_o|^2$ for 1243 of the 1712 independent reflections were equal to or greater than their corrected standard errors $\sigma(F^2)$. The scattering factors used for the hydrogen atoms were from Table 2 of Stewart, Davidson & Simpson (1965); those used for the other atoms were the neutral-atom factors from self-consistent wave functions as tabulated in International Tables (1962), page 202 et seq. The constant $\Delta f' = -3.1$ (page 214, International Tables, 1962) was used to correct the scattering factor of nickel for dispersion.

Although the agreement between the calculated and observed F^2 values was not entirely satisfactory and although the coordinates for the atoms in the per-

chlorate ion do not describe a regular tetrahedral ion, the refinement was terminated. We could see no further hope of improving the model for the disordered perchlorate ion. Our Fourier and difference maps suggest that the twofold disorder allowed for in our structurefactor calculations is only a good first approximation to the true situation. The disorder can be of a much more general nature so long as the twofold symmetry is maintained. For example, there may be disorder about the axis O(1)-Cl superposed on the disorder about the twofold axis.

The observed structure-factor magnitudes $|F_o|$ and the calculated structure factors F_c are listed in Table 2. Each entry $|F_o|$ has been scaled by the factor 1/S, where S is the appropriate scale factor on F_c established in the least-squares refinement. For each reflection for which $|F_o|^2$ is equal to or greater than $\sigma(F^2)$, the standard error $\sigma(F)$, computed as $\sigma(F^2)/2|F_o|$, is

Table 3. Parameters of the structure of $Ni(3,5-dimethylpyridine)_4(ClO_4)_2$

The elements β_{ij} form the matrix β in the anisotropic temperature factor exp $(-h^T\beta h)$, where h is the vector of reflection indices. The parameter B given for each hydrogen atom is the parameter of the isotropic temperature factor exp $(-B \sin^2 \theta/\lambda^2)$. The least-squares standard errors are indicated in parentheses. Each hydrogen atom is given the same identifying number as the carbon atom to which it is attached and when necessary an alphabetic identifier.

	Fract	ional coordinat	tes $\times 10^5$		Elements $\beta_{ij} \times 10^5$							
	x	y	Z	β_{11}	β ₂₂	β ₃₃	β ₁₂	β ₁₃	β ₂₃			
Ni	0*	1 *	1 *	509 (4)	509 †	121 (2)	102 (5)	0 *	0 *			
Ν	6891 (17)	18684 (18)	18015 (8)	469 (14)	630 (1	7) $128(3)$	125 (1)	-19(6)	20 (6)			
C(1)	15268 (22)	18167 (25)	17657 (12) 468 (17)	628 (2	0) 148 (5)	110 (1:	5) - 10(8)	-13(8)			
C(2)	20402 (24)	14720 (24)	21210 (13) 528 (19)	614 (2	0) 183 (6)	154 (15	5) - 55(8)	- 66 (9)			
C(3)	16473 (27)	11558 (27)	25425 (15) 685 (22)	772 (2	5) 179 (6)	167 (20	$\dot{)} - 115(10)$	7 (10)			
C(4)	7922 (25)	11890 (25)	25942 (12	686 (22)	763 (2	3) 151 (5)	128 (18	() - 26(9)	53 (9)			
C(5)	3257 (26)	15413 (28)	22125 (13) 484 (17)	745 (2	2) 149 (5)	72 (10	5) - 4(7)	62 (9)			
C(6)	29766 (35)	14458 (55)	20556 (28) 549 (25)	1008 (4	1) 325 (11)	184 (26	-87(14)	- 89 (19)			
C(7)	3321 (56)	8374 (71)	30505 (23) 1000 (44)	1542 (6	2) 203 (8)	217 (49	ý 68 (17)	226 (20)			
Cl	-16249 (63)	10248 (65)	12733 (95	613 (33)	547 (2	2) 197 (10)	- 91 (20	5) 57 (22)	- 85 (14)			
O (1)	15507 (59)	-9734 (57)	13803 (15	620 (33)	640 (3	4) 185 (11)	-127 (18	3) 78 (20)	-108(21)			
O(2)	-23808(68)	13868 (80)	13452 (37) 488 (37)	1953 (8	6) 381 (24)	-18(4)	5) 99 (23)	- 359 (34)			
O(3)	9408 (53)	-15906 (62)	6913 (22) 1505 (67)	1314 (5	9) 201 (10)	- 470 (50) 34(23)	-221(22)			
O(4)	2967 (82)	-15925 (127)) 14366 (68) 3194 (212)	821 (8	3) 701 (48)	635 (10	9) – 394 (69)	439 (49)			
	Fraction	al coordinates	× 104			Fractional	coordinate	es × 104				
		y		B (Ų)		x	у	z	B (Ų)			
H(1)	1726 (22)	2100 (21)	1479 (12)	7.3 (1.1)	H(6c)	3123 (39)	1050 (40)	1895 (22)	15.4 (3.0)			
H(3)	1995 (24)	1000 (23)	2785 (13)	7·7 (1·2)	H(7a)	44 (31)	1268 (23)	3237 (14)	8.5 (1.5)			
H(5)	-269 (19)	1543 (19)	2235 (99)	4.4 (0.8)	H(7b)	- 79 (46)	443 (44)	2993 (21)	17.8 (4.0)			
H(6a)	3237 (42)	1969 (44)	1952 (23)	19.2 (3.4)	H(7c)	734 (44)	956 (44)	3278 (25)	17.8 (3.1)			

* Parameter fixed by symmetry.

3286 (36)

H(6b)

 $\dagger \beta_{22}$ of Ni is constrained equal to β_{11} by symmetry.

2365 (21)

14.4 (2.3)

1361 (34)



Fig. 1. Stereoscopic drawing of the octahedral complex Ni(3,5-dimethylpyridine)₄(ClO₄)₂. The axes labelled 2B, 2C, and 2D are the three twofold axes of the point group 222. See text for explanation of other labelling in the Figure. Each disordered perchlorate ion is shown in only one of its two orientations about the axis 2D. Hydrogen atoms are not shown. given; for the other reflections, marked W in the table, $\sigma(F^2)$ is given. The reflections marked X were in the group of reflections thought to be subject to extinction error and omitted in the final refinement. The final atomic parameters and their standard errors appear in Table 3.

Discussion

A main feature of interest in our results is the confirmation that the nickel atom is coordinated to two perchlorate ligands as well as to the four organic ligands. The structure of the octahedral complex is represented in the stereoscopic drawing of Fig.1 and is described in some detail by the bond-length and angle data of Table 4 and the non-bonded distances of Table 5. In Fig. 1 the atoms of the dimethylpyridine group and the half-weight perchlorate group of the asymmetric unit are labelled individually, and the two groups are labelled A. The groups labelled B, C, and D are related to A by rotations about the twofold axes 2B, 2C, and 2D shown in the Figure. These are the axes $0, \frac{1}{4}, z, x, \frac{1}{4} - x, \frac{1}{8}$, and $x, \frac{1}{4} + x, \frac{1}{8}$. In Tables 4 and 5 the letters A, B, C, and D are included in the atom designations when necessary to distinguish atoms related by symmetry. An atom designation without a letter is equivalent to one with the letter A.

We emphasize that the problem of disorder of the arrangement of the perchlorate ions has not led to any

Table 5. Some intramolecular non-bonded distances in $Ni(3,5-dimethylpyridine)_4(ClO_4)_2$

N(A) - N(C)	2·954 (4) Å	C(1A)-C(1C)	3·347 (7) Å
N(A) - N(B)	2.968 (5)	C(1A)-O(1B)	2.925 (11)
N(A)-C(1C)	3.190 (4)	C(1A) - O(1C)	3.278 (10)
N(A)-C(5B)	3.191 (5)	C(1A)-O(2B)	3.352 (12)
N(A) - O(1A)	2.914 (8)	C(5A)-C(5B)	3.215 (9)
N(A)-O(1B)	2.788 (7)	C(5A)-O(1A)	3.035 (9)
N(A)-O(1C)	3.161 (6)	C(5A)-O(1D)	3.557 (8)
N(A)-O(1D)	3.223 (8)	C(5A)-O(3D)	3.336 (9)

ambiguity in regard to the coordination of perchlorate. Although the perchlorate ion described by our parameters is somewhat distorted (see the bond lengths and angles in Table 4), it is still a fair approximation to the tetrahedral ion expected. Furthermore, the oxygen atom of most interest, O(1), is positioned more precisely than any other atom of the perchlorate group except O(3) and about as precisely as atoms C(6) and C(7). The root-mean-square principal-axis vibrational amplitudes of O(1) are comparable to those of the atoms of the organic ligands and are quite reasonable (Table 6).

The atom O(1) is at a distance of 0.350(4) Å from the nearby twofold axis (axis 2D in Fig. 1). Therefore, the complex – even that part of it consisting of the nickel atom and the atoms immediately attached to it – does not, except in the statistical sense, have the expected symmetry consistent with the location of the

Table 4. Bond lengths and angles in the complex molecule $Ni(3,5-dimethylpyridine)_4(ClO_4)_2$

Each angle specified by four atoms, as a-b-c-d, is a torsion angle, defined to be the angle measured clockwise from the projection of ba to the projection of cd viewed in the direction of bc. The indicated standard errors were calculated from the least-squares covariance matrix. For the key to the numbering of the atoms see Fig. 1 and the text.

NiO(1)	2·187 (4) Å	Ni - N - C(2)	120.0 (2)
Ni—–N	2.093 (2)	Ni - N - C(5)	122.5 (2)
	x ,	C(1) - N - C(5)	117.5 (2)
N(A) - Ni - N(B)	90·3 (1)°	N - C(1) - C(2)	124.7(3)
N(A) - Ni - N(C)	89.8 (1)	C(1)-C(2)-C(3)	116.3 (4)
N(A) - Ni - N(D)	176.5(2)	C(1) - C(2) - C(6)	121.5 (5)
N(A) - Ni - O(1A)	87.8 (3)	C(3) - C(2) - C(6)	122.2(4)
N(A) - Ni - O(1B)	81.3 (2)	C(2) - C(3) - C(4)	121.1 (4)
N(A) - Ni - O(1C)	95.2 (2)	C(3) - C(4) - C(5)	118·3 (4)
N(A) - Ni - O(1D)	97.7 (3)	C(3) - C(4) - C(7)	122.8 (4)
	.,	C(5) - C(4) - C(7)	118.9 (5)
$C_{1} = O(1)$	1·40 (1) Å	C(4) - C(5) - N	122.1 (4)
$C_{1} = O(2)$	1.34(2)		
ClO(3)	1.45 (3)	N C(1) - C(2) - C(3)	0.3 (6)
$C_{1} O(4)$	1.24(2)	N - C(1) - C(2) - C(6)	-179.3(5)
		C(1)-C(2)-C(3)-C(4)	0.2 (6)
O(1) - Cl - O(2)	113 (1)°	C(6)-C(2)-C(3)-C(4)	179.8 (5)
O(1) - C1 - O(3)	104 (1)	C(2)-C(3)-C(4)-C(5)	0.5 (6)
O(1) - C1 - O(4)	119 (2)	C(2)-C(3)-C(4)-C(7)	179-2 (6)
O(2) - C1 - O(3)	102 (1)	C(3)-C(4)-C(5)-N	1.7 (6)
O(2) - Cl - O(4)	113 (1)	C(7)-C(4)-C(5)-N	179.5 (6)
O(3) - Cl - O(4)	106 (1)	C(4)-C(5)-N-C(1)	2.2 (6)
		C(4)-C(5)-NN	-174.4 (3)
N = -C(1)	1·336 (4) Å	C(5)-N-C(1)-C(2)	- 1.5 (6)
N = -C(5)	1.346 (4)	Ni - N - C(1) - C(2)	175-2 (3)
C(1) - C(2)	1.367 (4)		
C(2) - C(3)	1.383 (5)		
C(3) - C(4)	1.366 (5)		
C(4)C(5)	1.380 (4)		
C(2) - C(6)	1.497 (6)		
C(4) - C(7)	1.528 (7)		

Table 6. Atomic root-mean-square displacements in the directions of the principal axes of the thermal ellipsoids

	R.m.s.d.	(A) in directio	on of axis
	1	2	3
Ni	0.210 (2)	0.228(2)	0.279(2)
N	0.207 (3)	0.234(3)	0·299 (4)
C(1)	0.229 (4)	0.231(4)	0.296(5)
C(2)	0.229(5)	0.239 (4)	0.321(5)
C(3)	0.216 (5)	0.289 (5)	0.347 (6)
C(4)	0.222(4)	0.283(5)	0.332(5)
C(5)	0.221(4)	0.247(4)	0.318(5)
C(6)	0.238 (6)	0.344 (7)	0.473 (10)
C(7)	0.244(6)	0.322(7)	0·392 (8)
Cl	0.229 (10)	0.257 (6)	0.318 (9)
O (1)	0.221(11)	0.255(5)	0.338 (9)
O(2)	0.233 (10)	0·325 (11)	0.538 (12)
O(3)	0.225 (7)	0.365 (9)	0.499 (10)
O(4)	0.234 (12)	0.482 (15)	0·693 (21)

nickel atom. The chlorine atom is 0.179(12) Å from axis 2D; and the angle Ni–O(1)–Cl is $158 \cdot 0(1 \cdot 1)^\circ$. The twofold disorder of the perchlorate arrangement is quite similar to the fourfold disorder of the one perchlorate ion coordinated to cobalt in Co(diphenylmethylarsine $oxide_4(ClO_4)_2$ (Pauling *et al.*, 1965). In the latter compound the oxygen corresponding to our O(1) is 0.72 Å from the fourfold axis, the chlorine atom is either on or very near the axis, and the Co-O-Cl angle is 130°. The disorder in our compound is also similar to that reported from the structure analysis of $[Ni(ethylenediamine)_2NO_2]BF_4$ (Drew, Goodgame, Hitchman & Rogers, 1965), in which a half-oxygen atom and a half-nitrogen atom correspond to our two half-oxygen sites (1A) and (1D) (though the pointgroup symmetry of the disordered complex molecule is I and the symmetry element involved in the disorder is a mirror). In this case the N-Ni-O angle* is 24.2° , to be compared with our O(1A)-Ni-O(1D) angle* of 18.5(2)°.

Our Ni–O(1) distance of 2·187(4) Å is not significantly different from the Ni–O distance 2·215(17) Å reported in [Ni(ethylenediamine)₂NO₂]BF₄. Somewhat smaller distances have been reported in other octahedral complexes of nickel; for example, 2·085(12), 2·083(12), and 2·036(12) Å in nickel ammonium sulfate hexahydrate (Montgomery & Lingafelter, 1964) and 2·107(8) Å in dinitritobis-(*N*,*N*-dimethylethylenediamine)nickel(II) (Drew, Goodgame, Hitchman & Rogers, 1964).

The distance Ni-N of 2.093(2) Å found in the present work is in the middle range of values reported in other octahedral complexes of nickel: 2.00(5) Å in bischlorotetrakispyridine-nickel(II) (Porai-Koshits, 1954; *Structure Reports*, 1954, p.749); 2.120(13) Å in tris(ethylenediamine)nickel(II) nitrate (Swink & Atoji, 1960); 2.089(9) and 2.219(12) Å in dinitritobis-(*N*,*N*dimethylethylenediamine)nickel(II) (Drew *et al.*, 1964); 2.097(12), 2.117(11), and 2.155(19) Å in [Ni(ethylenediamine)₂NO₂]BF₄ (Drew *et al.*, 1965).

The C-N bond lengths in the dimethylpyridine ligand average 1.341 Å, very close to the value 1.3402(10) Å reported from a microwave study (Bak, Hansen-Nygaard & Rastrup-Andersen, 1958) of pyridine. The ring C-C bond lengths average 1.370 Å, somewhat lower than the average value of 1.3945(10) Å reported from the microwave study. The two external C-C bond lengths average 1.513 Å, close to the average value of 1.505(5) Å for a number of bonds between tetrahedral carbon atoms and aromatic rings (Tables of Interatomic Distances, 1965, page S15s). The atoms of the organic ligand are not quite coplanar, as indicated by the deviations of the torsion angles about the ring bonds from the values 0° and 180° characteristic of a plane structure (see Table 4). The apparent departure from the symmetry mm2 expected for an isolated molecule of 3,5-dimethylpyridine probably is not of much significance and is partly the result of 'noise' resulting from the incomplete model for the perchlorate disorder.

The 3,5-dimethylpyridine ligands form the blades of a four-bladed propeller. The angle between the average plane of each of the four organic groups and the plane perpendicular to the twofold axis through the perchlorate ions is 46.6°. The configuration of the nickel atom and the four nitrogen atoms in each molecule is not quite plane; the nitrogen atoms are alternately 0.065 Å above and 0.065 Å below their average plane through the nickel atom. The torsion angles C(4)-C(5)-N-Ni and Ni-N-C(1)-C(2), -174.4(3)° and 175.2(3)° respectively, and the angle Ni-N-C(3) of 175.6(2)° show that the N-Ni bond is bent significantly out of the average plane of the organic ligand.

The geometry of the complex molecule and the packing of the molecules in the crystal (see the stereoscopic drawing of Fig. 2) are very similar to the geometry and packing reported for Ni(pyridine)₄Cl₂ (Porai-Koshits, 1954; *Structure Reports*, 1954, p. 749). Crystals of this compound also have the space-group symmetry $I4_1/acd$, with cell translations *a* and *b* identical within experimental error with those of our compound and with a translation *c* of 16.90(5) Å. The nickel and chlorine atoms are in the same kinds of special positions as the nickel atom and the centroid of the disordered perchlorate in our structure.

As is usually the case in X-ray structure analysis, the apparent C-H bond lengths are short, varying from 0.80 Å to 0.97 Å. Otherwise the approximate positions established for the hydrogen atoms make good chemical sense, except that one of the hydrogen atoms of the methyl carbon C(7) is rather grossly misplaced, by 25° to 30° , in its orientation about the axis C(4)-C(7).

The contacts between the adjacent organic ligands within the complex molecule are of the types $N(1A)\cdots$ C(1C)H, $N(1A)\cdots C(5B)H$, $C(1A)H\cdots C(1C)H$, and $C(5A)H\cdots C(5B)H$. The distances corresponding to these and other non-bonded contacts within the molecule seem reasonable (see Table 5). The $N(1A)\cdots$

^{*} These angles are, of course, not valence angles but angles defined by the nickel atoms and possible sites for the atoms in disorder.



Fig. 2. Packing of the complex molecules Ni(3,5-dimethylpyridine)₄(ClO₄)₂ in the crystal structure. The origin of the axial system in this Figure is at the lower left rear corner, a site of point-group symmetry $\overline{4}$ which has coordinates $0, \frac{1}{4}, \frac{1}{8}$ with reference to the origin at $\overline{1}$ to which our atomic coordinates are referred. A screw axis 4₁ parallel to **c** runs through the center of the parallelepiped, which has dimensions a/2, b/2, c. Each disordered perchlorate group is represented by a circle corresponding to its centroid.

C(5B) and C(5A) \cdots C(5B) distances of about 3·2 Å are slightly less than the aromatic ring thickness (~3·40 Å) but are consistent with the relative orientation of the two ligands A and B (see Fig. 1). The closest intermolecular contacts of various kinds are as follows: CH₃ \cdots O, 3·241(10), 3·296(14), 3·480(16) Å; CH \cdots O, 3·358(9), 3·372(11) Å; CH \cdots CH, 3·677(9) Å; CH₃ \cdots CH₃, 3·873(10), 3·969(21), 4·226(10) Å. Of these the first two distances are slightly but not impossibly short.

Computations for this work were carried out on CDC 1604, IBM 7090, and IBM 360/75 computers. Programs used, with accession numbers in the *World List of Crystallographic Computer Programs* (Shoemaker, 1966), are as follows:

Absorption correction	ORABS ¹	-
Statistical analysis of data	ORSTAT	496
Fourier synthesis	XFOUR ²	391
Least-squares refinement	XFLS ³	389
Distances and angles	ORFFE ³	363
Structure drawings	ORTEP	387
Structure-factor table	EDIT ⁴	393

[(1) Wehe, Busing & Levy, 1962, revised for the CDC 1604 by R.D.Ellison & H.A.Levy. (2) Modified for the IBM 360/75 by G.Brunton. (3) Modified for the IBM 360/75 by C.K.Johnson. (4) New version with variable format for output.]

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