wherein the selenocyanates are noncoordinated, emphasizing the effects of the steric hindrance due to the ethyl groups of the $Et_4$dien in determining the overall geometry of the complexes.

As can be seen from Table I, all of the $Cu^{II}$–$Et_4$dien and –dien complexes generally function as 1:1 electrolytes in acetonitrile. The $[Cu(dien)NCO][B(C_6H_5)_4]\cdot CH_3OH$ complex retains the methanol molecule even on pumping under vacuum for several days, in contrast to $[Ni(Et_4dien)NCO][B(C_6H_5)_4]\cdot CH_3OH$, indicating that the methanol is probably coordinated to the copper(II). The $\nu_{CN}$ doublet in the solid-state infrared spectrum (Table II) of $[Cu(Et_4dien)NCO][B(C_6H_5)_4]$ must be due to different crystal environments, since the extra band does not appear in the solution spectrum. Heating the solid compound at 40° for 2 weeks did not change the spectrum, indicating that linkage isomerism is most likely not involved. The visible spectra of these complexes (Table III) are typical of copper(II) complexes, consisting of broad bands which are, in some cases, resolved into a doublet.

Other complexes whose preparations were attempted included $[Cu(dien)NCO]NCO$, $[Cu(dien)Cl][B(C_6H_5)_4]$, $[Cu(dien)NCS][B(C_6H_5)_4]$, and $[Cu(Et_4dien)NCO]NCO$. In all cases a product was isolated and the infrared spectrum exhibited the appropriate absorption bands, but the analyses were unsatisfactory.

**Acknowledgments.**—The authors wish to express their sincere appreciation to the National Science Foundation (Grant No. GP-8327) for the support of this research.

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# The Crystal and Molecular Structure of 4-Methylpyridinium Triphenylphosphine Tribromozincate

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*Received September 20, 1970*

The structure of 4-methylpyridinium triphenylphosphine tribromozincate, $[4\text{-}CH_3C_5H_4NH][ZnBr_3((C_6H_5)_3P)]$, has been determined from three-dimensional X-ray data collected by counter methods. The structure has been refined to a final unweighted $R$ factor of 0.063 and a weighted $R$ factor of 0.074 for 1533 independent nonzero reflections. The compound crystallizes in the space group $C_{2h}^5\text{-}P2_1/c$ of the monoclinic system containing four molecules per unit cell of dimensions $a$ = 10.021 (12), $b$ = 9.827 (15), $c$ = 26.674 (40) Å, and $\beta$ = 101.30 (7)°. The measured and calculated densities are 1.706 ± 0.015 and 1.700 g/cm³, respectively. The coordination geometry about the zinc atom is a distorted tetrahedron with one bromine atom strongly hydrogen bonded to the N–H proton of the 4-methylpyridinium cation, resulting in significant deviation from the expected $C_{3v}$ symmetry. Phenyl groups of the triphenylphosphine molecule were refined as rigid groups with angles of 120° and carbon–carbon distances of 1.392 Å. Coordination geometry about the phosphorus atom is that of a slightly elongated tetrahedron. The distortion of the 4-methylpyridinium cation due to the hydrogen-bonding interaction with the bromine atom appears to be less than can be determined from the data.

With the intention of studying the magnetic properties of transition metal ions with fourfold coordination and $C_{3v}$ symmetry, we have determined the crystal and molecular structure of 4-methylpyridinium triphenylphosphine tribromozincate, which could serve as a diamagnetic host lattice for other divalent paramagnetic ions. In this connection, the effect of lattice–anion interactions as well as the details of the anion geometry were of interest. In addition, we note that there is little structural data on four-coordination complexes of zinc(II).[1–4]

## Experimental Section

4-Methylpyridinium bromide was prepared by bubbling HBr through a solution of previously distilled 4-methylpyridine in diethyl ether. The white, hygroscopic product which immediately formed was filtered and washed under $N_2$ with dry ether.

$[4\text{-}CH_3C_5H_4NH][ZnBr_3(C_6H_5)_3P]$ was prepared by combining ethanol solutions, each containing 0.01 mol of triphenylphosphine, 4-methylpyridinium bromide, and anhydrous zinc bromide. The resulting solution was boiled for about 5 min to ensure complete reaction and was then filtered. Upon cooling, crystals of the desired complex precipitated in good yield. These were washed with ethanol and ether and dried *in vacuo*. *Anal.* Calcd for $C_{24}H_{23}Br_3NPZn$: C, 43.58; H, 3.50; Br, 36.24; Zn, 9.88. Found: C, 43.85; H, 3.49; Br, 36.29; Zn, 9.87. Well-formed crystals were grown from ethanol–acetonitrile mixtures by slow evaporation under vacuum. The compound exhibited three crystal habits depending on the composition of the solution from which crystals were grown. X-Ray investigation showed all forms to be crystallographically equivalent.

## Collection and Reduction of Intensity Data

Optical investigation immediately showed the crystals to be biaxial. Precession photographs of the $hk0$, $hk1$, $0kl$, and $1kl$ reciprocal lattice nets using Mo K$\bar{\alpha}$ radiation (λ 0.71069 Å) indicated a monoclinic space group. The systematic absences, $l = 2n$ for $h0l$ and $k = 2n$ for $0k0$ uniquely determined the space group to be $C_{2h}^5\text{-}P2_1/c$.[5]

For collection of intensity data, a small crystal approximating a hexagonal cylinder of dimensions ~0.3

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(1) R. S. Sager and W. H. Watson, *Inorg. Chem.*, **7**, 1358 (1968).

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mm in height and 0.25 mm in diameter was mounted in a 0.3-mm glass capillary and aligned on a Picker four-circle automatic diffractometer with the $a^*$ axis roughly along the spindle axis. Lattice constants (22°, λ 0.71069 Å, Mo K$\bar{\alpha}$) and standard deviations obtained by least-squares refinement[6] of the angular settings of 12 moderately intense hand-centered reflections were determined to be $a$ = 10.021 (12) Å, $b$ = 9.827 (15) Å, $c$ = 26.674 (40) Å, and $\beta$ = 101.30 (7)°, giving a unit cell volume of 2585.7 Å$^3$. The density calculated for 4 molecules/unit cell is 1.700 g/cm$^3$ and agrees well with the measured density of 1.706 ± 0.015 g/cm$^3$ measured by flotation in a chloroform–bromoform mixture.

Intensities were measured with a scintillation counter mounted 29 cm from the crystal which was positioned 21 cm from the source. The diffractometer was equipped with a highly oriented graphite single-crystal monochromator and the detector aperture was 4 mm × 4 mm; no attenuation of the diffracted beam was found to be necessary. Data were collected using the $\theta$–$2\theta$ scan technique to 50° in $2\theta$ with a scan width of 1.50° and a takeoff angle of 0.94°. Stationary background counts of 10-sec duration were taken at the beginning and end of the scan. One form ($hkl$ and $\bar{h}kl$) was measured giving a total of 4675 reflections. The small number of symmetry-related reflections collected was averaged.

Intensities were corrected for Lorentz polarization[7] and absorption[8] ($\mu$ = 59.8 cm$^{-1}$; transmission factors varied from 0.20 to 0.28). Reflections were considered to be observed if $I_o \leq 3\sigma(I_o)$. The resulting set of 1533 unique observed reflections was used in the subsequent refinement. Scattering factors for nonhydrogen atoms were taken from the compilation of Hanson, *et al.*[9] Hydrogen scattering factors were those of Stewart, *et al.*,[10] and anomalous dispersion corrections ($\Delta f'$ and $\Delta f''$) for Zn and Br were taken from the compilation of Cromer.[11]

## Solution and Refinement of the Structure

The structure was refined by full-matrix least-squares and Fourier techniques.[12] A three-dimensional Patterson function revealed the positions of the zinc and bromine atoms. The phosphorus atom was located by a subsequent Fourier synthesis. A structure factor calculation including the three bromine, the zinc, and the phosphorus atoms resulted in an unweighted $R$ factor $R_1 = \Sigma||F_o| - |F_c||/\Sigma F_o$ = 0.244 and a weighted $R$ factor $R_2 = (\Sigma w(F_o - F_c)^2/\Sigma wF_o^2)^{1/2}$ = 0.302. A Fourier synthesis based on this calculation revealed the nitrogen atom and all 24 carbon atoms. The phenyl groups, with hydrogen atoms, were included as rigid bodies giving $R_1$ = 0.155 and $R_2$ = 0.165. Two cycles of least-squares refinement on the scale factor, group, and positional parameters reduced $R_1$ to 0.102 and $R_2$ to 0.110. Two further cycles of refinement including isotropic thermal parameters reduced $R_1$ to 0.112 and $R_2$ to 0.089. Introduction of anisotropic thermal parameters for zinc, bromine, and phosphorus along with the anomalous dispersion correction reduced $R_1$ to 0.079 and $R_2$ to 0.072. At this point, it was found that the values of $w(F_o - F_c)$ displayed a strong systematic variation with both $(\sin\theta)/\lambda$ and the magnitude of $F_o$, indicating that the counting statistics weighting scheme was unsatisfactory. Assignment of equal weights to all reflections eliminated this difficulty and it was decided to continue with this weighting since no other weighting scheme seemed justifiable. Two cycles of least-squares refinement based on "unit weights" with a total of 110 parameters yielded $R_1$ = 0.065 and $R_2$ = 0.076. The N–H proton of the cation was located from a difference Fourier map and the ring protons of the cation were put in their "calculated positions" 1.0 Å from their respective carbon atoms. In an attempt to improve the reliability of the cation ring parameters, anisotropic thermal parameters were assigned to the nitrogen atom and to the six carbon atoms of the cation. Two cycles of least-squares refinement of the 63 cation parameters (hydrogen parameters were not varied) and the scale factor resulted in final values of $R_1$ = 0.063 and $R_2$ = 0.074. No peaks larger than 0.5 e$^-$/Å$^3$ were observed in the final difference Fourier map. The final positional group and thermal parameters are tabulated in Tables I and II. A partial listing of bond lengths and angles and their errors appears in Table III. The observed and calculated structure factors for the observed reflections are given in Table IV. Figure 1 shows the coordination sphere about the zinc atom, Figure 2 the anion, Figure 3 the entire molecule looking approximately down the zinc–phosphorus bond, and Figure 4 is a projection of the contents of the unit cell onto the $ac$ plane.

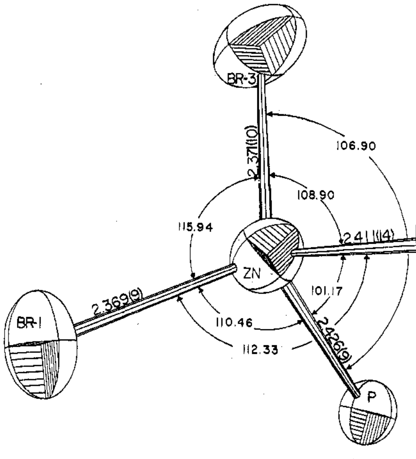

Figure 1. Inner coordination sphere about the zinc atom. Thermal ellipsoids are drawn to encompass 50% probability.

## Description of the Structure

**Anion Geometry.**—One of the assumptions made before the determination of this structure was that the anion would approximate closely enough to $C_{3v}$ symmetry that the spectral and magnetic properties could be interpreted on the basis of an axial model. The

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(6) W. R. Busing and H. A. Levy, *Acta Crystallogr.*, **22**, 457 (1967).

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(8) ORABS-II, a local modification of the program ORABS by D. J. Wehe, W. R. Busing, and H. A. Levy.

(9) H. P. Hanson, F. Herman, J. O. Lea, and S. Skillman, *Acta Crystallogr.*, **17**, 1040 (1964).

(10) R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, **42**, 3175 (1965).

(11) D. T. Cromer, *Acta Crystallogr.*, **17**, 1040 (1964).

(12) Local versions of several standard computer programs were used. Fourier syntheses were carried out with J. Gvildey's "Two- and Three-Dimensional Fourier Summation Program." The least-squares program was W. R. Busing, K. O. Martin, and H. A. Levy's ORFLS. Interatomic distances and angles and their standard deviations were calculated by Busing, Martin, and Levy's ORFFE. C. K. Johnson's ORTEP was used to draw the structural illustrations.

TABLE I

POSITIONAL, THERMAL, AND GROUP PARAMETERS FOR $[ZnBr_3((C_6H_5)_3P)][4\text{-}CH_3C_5H_4NH]$[a]

| Atom | *X* | *Y* | *Z* | $\beta_{11}$[b] | $\beta_{22}$ | $\beta_{33}$ | $\beta_{12}$ | $\beta_{13}$ | $\beta_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| Br(1) | 0.0430 (5) | 0.0888 (6) | 0.1142 (2) | 0.0100 (7) | 0.0125 (8) | 0.0030 (1) | −0.0021 (7) | 0.0012 (3) | 0.0002 (3) |
| Br(2) | 0.4354 (6) | 0.0091 (6) | 0.1526 (2) | 0.0115 (7) | 0.0103 (7) | 0.0024 (1) | 0.0001 (7) | 0.0002 (2) | 0.0000 (3) |
| Br(3) | 0.2968 (7) | 0.2851 (7) | 0.2263 (2) | 0.0204 (11) | 0.0190 (11) | 0.0020 (1) | −0.0044 (9) | 0.0029 (3) | −0.0018 (3) |
| Zn | 0.2659 (6) | 0.1693 (6) | 0.1469 (2) | 0.0112 (8) | 0.0100 (8) | 0.0016 (1) | −0.0010 (7) | 0.0013 (2) | 0.0002 (3) |
| P | 0.3386 (12) | 0.3273 (13) | 0.0869 (5) | 0.0077 (15) | 0.0072 (16) | 0.0013 (2) | −0.0006 (13) | 0.0009 (5) | 0.0002 (3) |
| N | 0.682 (6) | 0.139 (5) | 0.229 (2) | 0.026 (10) | 0.014 (7) | 0.001 (1) | 0.000 (6) | −0.000 (2) | 0.001 (2) |
| C(19) | 0.799 (8) | 0.117 (7) | 0.215 (3) | 0.024 (12) | 0.019 (11) | 0.003 (2) | 0.004 (10) | 0.005 (4) | −0.002 (3) |
| C(20) | 0.920 (5) | 0.155 (7) | 0.245 (2) | 0.008 (7) | 0.017 (9) | 0.002 (1) | 0.002 (7) | 0.001 (2) | 0.001 (3) |
| C(21) | 0.921 (6) | 0.224 (5) | 0.290 (2) | 0.016 (8) | 0.006 (6) | 0.002 (1) | 0.002 (6) | 0.002 (2) | −0.001 (2) |
| C(22) | 0.801 (6) | 0.247 (6) | 0.302 (2) | 0.014 (8) | 0.016 (9) | 0.002 (1) | 0.007 (7) | 0.002 (2) | −0.001 (2) |
| C(23) | 0.675 (6) | 0.204 (8) | 0.271 (3) | 0.011 (9) | 0.029 (14) | 0.003 (2) | 0.004 (9) | 0.001 (3) | 0.006 (4) |
| C(24) | 1.059 (5) | 0.268 (6) | 0.321 (2) | 0.009 (7) | 0.018 (9) | 0.003 (1) | −0.002 (7) | 0.000 (2) | −0.002 (3) |
| H(16[c]) | 0.587 | 0.107 | 0.230 | 7.0[d] | | | | | |
| H(17[c]) | 0.798 | 0.072 | 0.181 | 7.0[d] | | | | | |
| H(18[c]) | 1.007 | 0.131 | 0.235 | 7.0[d] | | | | | |
| H(19[c]) | 0.586 | 0.226 | 0.283 | 7.0[d] | | | | | |
| H(20[c]) | 0.800 | 0.297 | 0.335 | 7.0[d] | | | | | |

| Group | | | | $B$[e] | $\rho$[f] | $\theta$[f] | $\varphi$[f] |
|---|---|---|---|---|---|---|---|
| Ring 1 | 0.5192 (25) | 0.3660 (23) | 0.1091 (10) | 0.0 | 3.790 (32) | −0.881 (21) | 3.863 (32) |
| Ring 2 | 0.2451 (24) | 0.4831 (27) | 0.0758 (9) | 0.0 | 2.912 (22) | −0.078 (22) | −1.043 (22) |
| Ring 3 | 0.3290 (20) | 0.2503 (27) | 0.0234 (10) | 0.0 | 1.808 (22) | 0.385 (22) | 2.839 (24) |

[a] Numbers in parentheses are estimated uncertainties in last decimal places. [b] The form of the anisotropic ellipsoid is $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$. [c] Positional and thermal parameters of the hydrogen atoms were not refined. [d] Hydrogen atoms were given isotropic temperature factors of the form $\exp[-B(\sin^2\theta)/\lambda^2]$ which were set numerically equal to 7.0. [e] Group *B* set equal to zero. Atomic *B*'s varied in refinement. [f] In radians.

TABLE II

PARAMETERS OF GROUP ATOMS

| Atom | *X* | *Y* | *Z* | $B$[a] |
|---|---|---|---|---|
| C(1) | 0.5193 | 0.3660 | 0.1092 | 3.72 (114) |
| C(2) | 0.6196 | 0.3199 | 0.0839 | 5.61 (133) |
| H(1) | 0.5930 | 0.2678 | 0.0504 | 7.0 (. . .) |
| C(3) | 0.7561 | 0.3449 | 0.1044 | 5.57 (137) |
| H(2) | 0.8296 | 0.3111 | 0.0858 | 7.0 (. . .) |
| C(4) | 0.7924 | 0.4160 | 0.1501 | 5.75 (145) |
| H(3) | 0.8924 | 0.4343 | 0.1651 | 7.0 (. . .) |
| C(5) | 0.6921 | 0.4621 | 0.1754 | 5.46 (141) |
| H(4) | 0.7186 | 0.5142 | 0.2090 | 7.0 (. . .) |
| C(6) | 0.5555 | 0.4371 | 0.1550 | 4.66 (132) |
| H(5) | 0.4820 | 0.4709 | 0.1735 | 7.0 (. . .) |
| C(7) | 0.2452 | 0.4833 | 0.0758 | 3.62 (111) |
| C(8) | 0.3086 | 0.6038 | 0.0662 | 6.07 (153) |
| H(6) | 0.4111 | 0.6058 | 0.0679 | 7.0 (. . .) |
| C(9) | 0.2320 | 0.7217 | 0.0541 | 7.62 (179) |
| H(7) | 0.2784 | 0.8100 | 0.0470 | 7.0 (. . .) |
| C(10) | 0.0920 | 0.7190 | 0.0516 | 5.98 (153) |
| H(8) | 0.0359 | 0.8054 | 0.0428 | 7.0 (. . .) |
| C(11) | 0.0286 | 0.5985 | 0.0613 | 5.55 (138) |
| H(9) | −0.0739 | 0.5965 | 0.0595 | 7.0 (. . .) |
| C(12) | 0.1052 | 0.4806 | 0.0734 | 5.22 (130) |
| H(10) | 0.0588 | 0.3923 | 0.0804 | 7.0 (. . .) |
| C(13) | 0.3290 | 0.2504 | 0.0234 | 3.53 (113) |
| C(14) | 0.3003 | 0.1123 | 0.0167 | 6.21 (159) |
| H(11) | 0.2858 | 0.0545 | 0.0470 | 7.0 (. . .) |
| C(15) | 0.2914 | 0.0530 | −0.0312 | 7.88 (191) |
| H(12) | 0.2704 | −0.0482 | −0.0361 | 7.0 (. . .) |
| C(16) | 0.3111 | 0.1319 | −0.0725 | 7.34 (180) |
| H(13) | 0.3047 | 0.0884 | −0.1076 | 7.0 (. . .) |
| C(17) | 0.3399 | 0.2700 | −0.0658 | 5.44 (140) |
| H(14) | 0.3543 | 0.3278 | −0.0960 | 7.0 (. . .) |
| C(18) | 0.3488 | 0.3292 | −0.0179 | 5.17 (131) |
| H(15) | 0.3698 | 0.4305 | −0.0130 | 7.0 (. . .) |

[a] Isotropic temperature factor of the form $\exp[-B(\sin^2\theta)/\lambda^2]$.

TABLE III

SELECTED BOND LENGTHS (Å) AND ANGLES (DEG)[a]

| | | | |
|---|---|---|---|
| Zn–Br(1) | 2.369 (9) | Br(1)–Zn–Br(3) | 115.9 (3) |
| Zn–Br(2) | 2.411 (14) | Br(1)–Zn–P | 110.5 (4) |
| Zn–Br(3) | 2.371 (10) | Br(1)–Zn–Br(2) | 112.3 (3) |
| Zn–P | 2.425 (9) | Br(3)–Zn–P | 106.9 (4) |
| | | Br(3)–Zn–Br(2) | 108.9 (4) |
| | | Br(2)–Zn–P | 101.2 (4) |
| P–C(1) | 1.84 (3) | C(13)–P–C(7) | 104.7 (12) |
| P–C(7) | 1.83 (3) | C(13)–P–C(1) | 105.0 (12) |
| P–C(13) | 1.82 (3) | C(13)–P–Zn | 114.1 (11) |
| | | C(7)–P–C(1) | 106.9 (12) |
| | | C(7)–P–Zn | 115.7 (10) |
| | | C(1)–P–Zn | 109.7 (9) |
| C(19)–N | 1.32 (7) | C(19)–N–C(23) | 121.7 (50) |
| C(20)–C(19) | 1.37 (7) | C(20)–C(19)–N | 121.1 (50) |
| C(21)–C(20) | 1.36 (7) | C(21)–C(20)–C(19) | 120.5 (50) |
| C(22)–C(21) | 1.33 (7) | C(22)–C(21)–C(20) | 116.5 (50) |
| C(23)–C(22) | 1.42 (7) | C(21)–C(22)–C(23) | 123.5 (50) |
| C(24)–C(21) | 1.54 (7) | C(22)–C(22)–C(23) | 123.5 (50) |
| C(23)–N | 1.32 (7) | Av ring | 119.9 (50) |
| N–Br(2) | 3.22 | N–H(16)–Br(2) | 120.5 |
| N–H(16) | 1.01 | C(21)–N–Br(2) | 169.2 |
| H(16)–Br(2) | 2.57 | C(21)–N–H(16) | 142.5 |

[a] Errors in lattice parameters are included in the estimated standard deviations.

Zn–Br distances in two of the bonds are ~2.37 Å while the third is ~2.41 Å, a considerable and significant elongation due, it appears, to a strong hydrogen-bonding interaction with the cation.[13] Angles around the zinc atom range from ~101.2 to ~115.9° but are within the range noted by Taylor, *et al.*,[14] in Ni((C6-

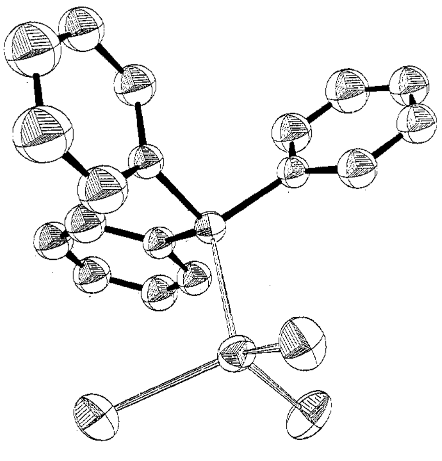

Figure 2.—Structure of the anion $ZnBr_3(C_6H_5)_3P^-$.

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[13] F. K. Ross and G. D. Stucky, *J. Amer. Chem. Soc.*, **92**, 4538 (1970).

[14] R. P. Taylor, D. H. Templeton, A. Zalkin, and W. D. Horrocks, *Inorg. Chem.*, **7**, 2629 (1968).

TABLE IV

OBSERVED AND CALCULATED STRUCTURE FACTORS IN ELECTRONS (×10) FOR $[4\text{-}CH_3C_5H_4NH][ZnBr_3(P(C_6H_5)_3)]$

| H | K | OBS | CAL |
|---|---|---|---|
| ******L= 0****** | | | |
| 0 | 2 | 1155 | 956 |
| 0 | 4 | 432 | 396 |
| 0 | 6 | 567 | 571 |
| 1 | 0 | 913 | 951 |
| 1 | 1 | 1865 | 1850 |
| 1 | 2 | 360 | 248 |
| 1 | 3 | 759 | 685 |
| 1 | 4 | 1192 | 1142 |
| 1 | 5 | 334 | 346 |
| 1 | 6 | 1332 | 1350 |
| 1 | 7 | 535 | 494 |
| 1 | 8 | 309 | 285 |
| 2 | 0 | 1718 | 1660 |
| 2 | 1 | 1504 | 1476 |
| 2 | 2 | 2498 | 2530 |
| 2 | 3 | 1503 | 1433 |
| 2 | 5 | 344 | 324 |
| 2 | 6 | 643 | 649 |
| 2 | 8 | 460 | 453 |
| 3 | 0 | 2683 | 2971 |
| 3 | 1 | 1079 | 1088 |
| 3 | 2 | 1458 | 1378 |
| 3 | 3 | 844 | 775 |
| 3 | 4 | 512 | 519 |
| 3 | 5 | 413 | 391 |
| 3 | 6 | 322 | 300 |
| 3 | 7 | 795 | 795 |
| 3 | 8 | 352 | 338 |
| 3 | 9 | 333 | 219 |
| 4 | 0 | 1327 | 1352 |
| 4 | 1 | 2149 | 2086 |
| 4 | 3 | 289 | 322 |
| 4 | 4 | 598 | 561 |
| 4 | 5 | 665 | 434 |
| 5 | 0 | 1090 | 1116 |
| -5 | 1 | 301 | 329 |
| 5 | 2 | 1078 | 1047 |
| 5 | 3 | 568 | 581 |
| 6 | 0 | 624 | 597 |
| 6 | 1 | 408 | 407 |
| 6 | 2 | 620 | 583 |
| 6 | 3 | 655 | 641 |
| 6 | 4 | 320 | 340 |
| 6 | 5 | 305 | 281 |
| 6 | 6 | 437 | 486 |
| 7 | 0 | 435 | 442 |
| 7 | 1 | 399 | 357 |
| 7 | 2 | 304 | 381 |
| 7 | 4 | 361 | 373 |
| 7 | 5 | 286 | 240 |
| 7 | 6 | 380 | 441 |
| 7 | 7 | 343 | 332 |
| 8 | 0 | 783 | 806 |
| 8 | 1 | 431 | 497 |
| 8 | 2 | 326 | 375 |
| 8 | 3 | 291 | 285 |
| 8 | 4 | 396 | 377 |
| -9 | 3 | 377 | 238 |
| 9 | 4 | 418 | 394 |
| 10 | 0 | 467 | 451 |
| -10 | 2 | 316 | 188 |
| 11 | 4 | 560 | 81 |
| ******L= 1****** | | | |
| 0 | 1 | 735 | 651 |
| 0 | 2 | 927 | 804 |
| 0 | 3 | 821 | 748 |
| 0 | 5 | 482 | 451 |
| 0 | 6 | 863 | 868 |
| 0 | 7 | 312 | 233 |
| 0 | 10 | 345 | 353 |
| 1 | 1 | 366 | 363 |
| -1 | 1 | 396 | 378 |
| 1 | 2 | 2402 | 2410 |
| 1 | 3 | 1762 | 1688 |
| -1 | 3 | 338 | 344 |
| 1 | 4 | 517 | 531 |
| -1 | 4 | 360 | 341 |
| 1 | 5 | 494 | 507 |
| -1 | 5 | 952 | 939 |
| -1 | 6 | 512 | 462 |
| 1 | 7 | 977 | 997 |
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| -3 | 3 | 591 | 470 |
| 3 | 4 | 1047 | 1085 |
| -3 | 4 | 1146 | 1163 |
| -3 | 5 | 281 | 317 |
| 3 | 6 | 503 | 502 |
| 3 | 8 | 331 | 358 |
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| 4 | 3 | 402 | 447 |
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| 4 | 4 | 468 | 465 |
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| -4 | 7 | 631 | 625 |
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| -5 | 0 | 575 | 628 |
| 5 | 1 | 559 | 564 |
| -5 | 1 | 393 | 361 |
| 5 | 2 | 338 | 332 |
| -5 | 2 | 526 | 462 |
| -5 | 3 | 782 | 810 |
| -5 | 5 | 256 | 250 |
| 5 | 6 | 848 | 852 |
| -5 | 6 | 904 | 955 |
| 6 | 0 | 343 | 325 |
| -6 | 0 | 787 | 738 |
| 6 | 1 | 363 | 327 |
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| -6 | 2 | 614 | 592 |
| 6 | 3 | 517 | 518 |
| -6 | 3 | 431 | 421 |
| -6 | 4 | 337 | 386 |
| 6 | 5 | 387 | 446 |
| -6 | 5 | 359 | 347 |
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| 7 | 2 | 526 | 507 |
| 7 | 3 | 426 | 455 |
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| -7 | 5 | 658 | 685 |
| -7 | 7 | 384 | 376 |
| 8 | 1 | 481 | 465 |
| -8 | 6 | 475 | 508 |
| 9 | 2 | 310 | 250 |
| -9 | 2 | 405 | 373 |
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| -11 | 0 | 318 | 275 |
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| 0 | 4 | 1150 | 1096 |
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| 0 | 7 | 406 | 371 |
| 0 | 8 | 397 | 368 |
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| -1 | 2 | 896 | 799 |
| 1 | 3 | 832 | 703 |
| -1 | 3 | 654 | 530 |
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| 1 | 7 | 368 | 302 |
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| 2 | 3 | 1114 | 1080 |
| -2 | 3 | 1046 | 1013 |
| 2 | 4 | 545 | 514 |
| -2 | 4 | 618 | 653 |
| -2 | 5 | 463 | 496 |
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| 2 | 8 | 569 | 579 |
| -2 | 8 | 301 | 275 |
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| 3 | 2 | 1485 | 1474 |
| 3 | 3 | 592 | 595 |
| -3 | 3 | 1593 | 1659 |
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| -4 | 6 | 297 | 290 |
| -4 | 7 | 311 | 213 |
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| 5 | 4 | 547 | 562 |
| -5 | 4 | 504 | 484 |
| 5 | 5 | 289 | 234 |
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| 5 | 8 | 310 | 245 |
| 6 | 1 | 467 | 477 |
| 6 | 2 | 320 | 393 |
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| 6 | 5 | 415 | 376 |
| 6 | 7 | 420 | 375 |
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| -7 | 1 | 426 | 428 |
| -7 | 2 | 365 | 355 |
| -7 | 4 | 332 | 244 |
| -7 | 5 | 446 | 452 |
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| 0 | 2 | 516 | 439 |
| 0 | 3 | 298 | 54 |
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| 0 | 7 | 603 | 596 |
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| 1 | 4 | 260 | 203 |
| -1 | 4 | 992 | 998 |
| 1 | 5 | 299 | 344 |
| -1 | 5 | 365 | 371 |
| 1 | 6 | 338 | 374 |
| -1 | 6 | 368 | 381 |
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| 2 | 1 | 289 | 374 |
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| 2 | 7 | 264 | 252 |
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| -3 | 3 | 453 | 391 |
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| -3 | 5 | 552 | 525 |
| 3 | 6 | 373 | 413 |
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| -4 | 0 | 987 | 939 |
| 4 | 1 | 725 | 754 |
| -4 | 1 | 674 | 583 |
| 4 | 2 | 637 | 615 |
| 4 | 3 | 268 | 270 |
| -4 | 4 | 474 | 474 |
| 4 | 5 | 341 | 313 |
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| -4 | 8 | 452 | 389 |
| 4 | 9 | 323 | 316 |
| 5 | 0 | 332 | 313 |
| -5 | 0 | 781 | 774 |
| 5 | 1 | 547 | 607 |
| -5 | 1 | 256 | 297 |
| 5 | 2 | 307 | 291 |
| -5 | 2 | 585 | 580 |
| 5 | 3 | 671 | 644 |
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| 5 | 5 | 493 | 542 |
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| -5 | 7 | 377 | 417 |
| -5 | 8 | 323 | 331 |
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| -6 | 0 | 912 | 897 |
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| 6 | 2 | 345 | 295 |
| -6 | 2 | 682 | 633 |
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| -6 | 5 | 454 | 371 |
| 6 | 6 | 521 | 587 |
| -6 | 6 | 434 | 396 |
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| -7 | 0 | 496 | 500 |
| 7 | 1 | 456 | 530 |
| -7 | 3 | 613 | 636 |
| 7 | 4 | 332 | 276 |
| -7 | 6 | 674 | 729 |
| -8 | 0 | 609 | 660 |
| -8 | 2 | 347 | 296 |
| 8 | 4 | 386 | 381 |
| -8 | 5 | 459 | 428 |
| 9 | 2 | 422 | 401 |
| -9 | 4 | 409 | 424 |
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| 0 | 7 | 719 | 748 |
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| -1 | 6 | 386 | 337 |
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| -1 | 10 | 352 | 394 |
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| 4 | 7 | 296 | 217 |
| 4 | 8 | 370 | 410 |
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| 5 | 7 | 464 | 458 |
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| -7 | 6 | 312 | 105 |
| -7 | 7 | 337 | 320 |
| -8 | 2 | 418 | 425 |
| -8 | 3 | 563 | 494 |
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| -9 | 5 | 319 | 229 |
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| 0 | 5 | 309 | 373 |
| 0 | 6 | 749 | 733 |
| 0 | 7 | 405 | 408 |
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| 1 | 2 | 874 | 864 |
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| -1 | 3 | 586 | 595 |
| 1 | 4 | 418 | 354 |
| -1 | 4 | 522 | 487 |
| 1 | 6 | 540 | 635 |
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| 2 | 1 | 987 | 1033 |
| -2 | 1 | 1455 | 1455 |
| 2 | 2 | 269 | 267 |
| -2 | 2 | 302 | 271 |
| -2 | 3 | 380 | 394 |
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| -2 | 5 | 447 | 386 |
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| 3 | 2 | 419 | 378 |
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| 3 | 8 | 317 | 333 |
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| -4 | 8 | 345 | 333 |
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| -5 | 1 | 903 | 898 |
| -5 | 2 | 268 | 256 |
| 5 | 3 | 397 | 377 |
| 5 | 4 | 455 | 468 |
| 5 | 6 | 620 | 616 |
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| -5 | 8 | 302 | 119 |
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| -6 | 0 | 1371 | 1383 |
| -6 | 1 | 434 | 387 |
| 6 | 2 | 321 | 336 |
| -6 | 2 | 425 | 417 |
| -6 | 3 | 299 | 317 |
| -6 | 5 | 300 | 345 |
| -7 | 0 | 577 | 652 |
| -7 | 1 | 567 | 529 |
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| -7 | 3 | 485 | 499 |
| 7 | 4 | 364 | 321 |
| -8 | 0 | 364 | 319 |
| -8 | 6 | 395 | 383 |
| -9 | 0 | 489 | 516 |
| ******L=13****** | | | |
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| 0 | 4 | 301 | 300 |
| 0 | 5 | 516 | 488 |
| 0 | 7 | 900 | 958 |
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| 1 | 3 | 657 | 651 |
| -1 | 3 | 620 | 551 |
| 1 | 6 | 438 | 382 |
| -1 | 6 | 328 | 308 |
| -1 | 7 | 424 | 440 |
| 1 | 9 | 421 | 413 |
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| 2 | 3 | 652 | 609 |
| -2 | 3 | 716 | 695 |
| 2 | 4 | 264 | 358 |
| 2 | 5 | 519 | 552 |
| -2 | 5 | 874 | 859 |
| 2 | 6 | 313 | 193 |
| -2 | 6 | 307 | 291 |
| 2 | 9 | 351 | 313 |
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| 3 | 2 | 640 | 645 |
| -3 | 2 | 669 | 619 |
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| 4 | 2 | 499 | 540 |
| -4 | 2 | 308 | 337 |
| 4 | 3 | 489 | 534 |
| -4 | 3 | 387 | 329 |
| -4 | 8 | 548 | 552 |
| 4 | 9 | 357 | 301 |
| 5 | 3 | 286 | 267 |
| 5 | 4 | 430 | 472 |
| -5 | 4 | 651 | 699 |
| 5 | 5 | 348 | 353 |
| -5 | 5 | 393 | 423 |
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| -6 | 3 | 284 | 282 |
| 6 | 5 | 321 | 309 |
| -6 | 5 | 507 | 538 |
| -7 | 1 | 600 | 594 |
| 7 | 2 | 353 | 333 |
| -7 | 2 | 625 | 640 |
| 8 | 3 | 318 | 313 |
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| -8 | 7 | 343 | 350 |
| -9 | 3 | 385 | 403 |
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| 0 | 2 | 368 | 287 |
| 0 | 3 | 805 | 796 |
| 0 | 4 | 417 | 387 |
| 0 | 6 | 833 | 811 |
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| 1 | 3 | 380 | 402 |
| 1 | 4 | 459 | 483 |
| -1 | 4 | 398 | 374 |
| 1 | 5 | 393 | 416 |
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| 1 | 7 | 509 | 521 |
| -1 | 7 | 371 | 370 |
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| -2 | 2 | 1022 | 1016 |
| 2 | 4 | 924 | 955 |
| 2 | 8 | 299 | 294 |
| 3 | 0 | 296 | 310 |
| -3 | 0 | 457 | 434 |
| -3 | 1 | 1614 | 1672 |
| 3 | 2 | 1040 | 1031 |
| 3 | 3 | 427 | 402 |
| -3 | 3 | 364 | 399 |
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| -3 | 4 | 278 | 232 |
| -3 | 6 | 474 | 509 |
| -3 | 7 | 457 | 462 |
| 3 | 8 | 337 | 288 |
| -4 | 0 | 484 | 492 |
| 4 | 2 | 566 | 577 |
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| 4 | 3 | 296 | 244 |
| 4 | 6 | 421 | 363 |
| -4 | 6 | 281 | 233 |
| -4 | 8 | 321 | 332 |
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| -5 | 4 | 373 | 365 |
| 5 | 5 | 342 | 352 |
| -5 | 5 | 420 | 457 |
| -5 | 6 | 375 | 348 |
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| -6 | 1 | 763 | 800 |
| -6 | 4 | 352 | 263 |
| 6 | 6 | 318 | 224 |
| -6 | 6 | 299 | 270 |
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| -7 | 3 | 315 | 290 |
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| -9 | 2 | 370 | 285 |
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| 0 | 3 | 614 | 589 |
| 0 | 4 | 435 | 446 |
| 0 | 6 | 548 | 541 |
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| 1 | 4 | 708 | 706 |
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| 3 | 2 | 455 | 447 |
| 3 | 3 | 665 | 665 |
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| 4 | 4 | 667 | 615 |
| 4 | 5 | 318 | 338 |
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| 5 | 1 | 323 | 313 |
| 5 | 5 | 613 | 613 |
| -5 | 5 | 492 | 483 |
| 6 | 1 | 485 | 447 |
| -6 | 1 | 439 | 440 |
| 6 | 4 | 390 | 367 |
| -6 | 4 | 329 | 299 |
| -6 | 5 | 309 | 303 |
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| -8 | 2 | 311 | 322 |
| -10 | 3 | 360 | 361 |
| ******L=16****** | | | |
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| -1 | 7 | 340 | 318 |
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| 2 | 2 | 322 | 304 |
| -2 | 2 | 1075 | 1106 |
| 2 | 3 | 505 | 517 |
| -2 | 3 | 489 | 482 |
| 2 | 4 | 575 | 610 |
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| 2 | 8 | 359 | 295 |
| -3 | 0 | 625 | 601 |
| 3 | 1 | 317 | 392 |
| -3 | 1 | 858 | 856 |
| 3 | 2 | 482 | 492 |
| -3 | 2 | 684 | 701 |
| 3 | 4 | 451 | 433 |
| -3 | 4 | 360 | 398 |
| -3 | 5 | 381 | 419 |
| -3 | 6 | 317 | 311 |
| 3 | 7 | 339 | 332 |
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| -3 | 9 | 309 | 87 |
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| -4 | 4 | 305 | 261 |
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| -5 | 0 | 508 | 507 |
| -5 | 2 | 716 | 731 |
| -5 | 3 | 497 | 392 |
| -5 | 6 | 333 | 407 |
| 6 | 0 | 463 | 483 |
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| -6 | 4 | 508 | 467 |
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| 1 | 2 | 489 | 471 |
| 1 | 3 | 682 | 690 |
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| -1 | 7 | 305 | 207 |
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| -2 | 2 | 376 | 367 |
| 2 | 3 | 784 | 829 |
| -2 | 3 | 392 | 391 |
| 2 | 7 | 360 | 357 |
| -2 | 8 | 413 | 371 |
| 3 | 1 | 516 | 543 |
| -3 | 1 | 250 | 183 |
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| -3 | 2 | 640 | 661 |
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| -4 | 4 | 475 | 521 |
| 4 | 5 | 507 | 539 |
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| 5 | 1 | 343 | 312 |
| -5 | 3 | 496 | 461 |
| 5 | 4 | 482 | 490 |
| -5 | 4 | 460 | 440 |
| -5 | 8 | 350 | 339 |
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| -6 | 2 | 573 | 531 |
| -6 | 7 | 391 | 406 |
| -7 | 1 | 508 | 554 |
| 7 | 2 | 381 | 335 |
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| -8 | 4 | 379 | 416 |
| -8 | 5 | 315 | 238 |
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| 0 | 6 | 528 | 507 |
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| 1 | 4 | 288 | 247 |
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| 1 | 6 | 353 | 345 |
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| -2 | 2 | 641 | 719 |
| 2 | 3 | 438 | 392 |
| 2 | 4 | 346 | 312 |
| -2 | 4 | 396 | 379 |
| 2 | 5 | 358 | 373 |
| -2 | 6 | 426 | 387 |
| 3 | 0 | 366 | 332 |
| -3 | 0 | 254 | 68 |
| 3 | 2 | 303 | 296 |
| -3 | 2 | 355 | 252 |
| 3 | 3 | 292 | 303 |
| -3 | 3 | 598 | 411 |
| 3 | 4 | 699 | 506 |
| -3 | 4 | 354 | 338 |
| -3 | 5 | 322 | 397 |
| -3 | 6 | 365 | 328 |
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| -4 | 1 | 290 | 351 |
| 4 | 2 | 353 | 355 |
| -4 | 2 | 551 | 373 |
| 4 | 5 | 322 | 234 |
| -5 | 0 | 473 | 473 |
| -5 | 4 | 559 | 519 |
| -5 | 5 | 250 | 353 |
| -6 | 0 | 530 | 318 |
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| -6 | 3 | 467 | 474 |
| -7 | 0 | 558 | 572 |
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| -9 | 0 | 586 | [illegible] |
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| -2 | 1 | 455 | 426 |
| 2 | 2 | 410 | 454 |
| -2 | 2 | 554 | 563 |
| 2 | 3 | 276 | 233 |
| -2 | 3 | 348 | 274 |
| -2 | 4 | 474 | 463 |
| -3 | 1 | 285 | 328 |
| 3 | 3 | 554 | 546 |
| -3 | 3 | 979 | 1047 |
| -4 | 2 | 591 | 606 |
| -4 | 3 | 416 | 433 |
| -4 | 4 | 656 | 653 |
| -5 | 4 | 287 | 317 |
| -5 | 5 | 552 | 552 |
| 6 | 1 | 519 | 543 |
| -6 | 1 | 289 | 247 |
| -6 | 2 | 363 | 317 |
| -6 | 3 | 474 | 542 |
| -6 | 4 | 575 | 538 |
| -7 | 1 | 345 | 357 |
| -7 | 2 | 389 | 451 |
| -7 | 3 | 407 | 351 |
| ******L=20****** | | | |
| 0 | 2 | 359 | 344 |
| 0 | 3 | 402 | 376 |
| 0 | 6 | 381 | 394 |
| -1 | 0 | 293 | 362 |
| -1 | 1 | 372 | 498 |
| 1 | 2 | 705 | 724 |
| 1 | 3 | 327 | 321 |
| -1 | 5 | 433 | 436 |
| -2 | 0 | 692 | 663 |
| 2 | 1 | 308 | 327 |
| 2 | 2 | 401 | 382 |
| 2 | 4 | 430 | 414 |
| -2 | 4 | 367 | 410 |
| 3 | 0 | 820 | 841 |
| 3 | 1 | 419 | 357 |
| -3 | 3 | 423 | 416 |
| -4 | 1 | 519 | 469 |
| -4 | 4 | 554 | 431 |
| -5 | 0 | 832 | 858 |
| -5 | 4 | 352 | 378 |
| 6 | 0 | 432 | 429 |
| -6 | 0 | 319 | 351 |
| -6 | 6 | 373 | 352 |
| -7 | 1 | 406 | 353 |
| -7 | 2 | 494 | 436 |
| -8 | 0 | 438 | 463 |
| -8 | 2 | 399 | 341 |
| -9 | 1 | 375 | 346 |
| ******L=21****** | | | |
| 0 | 2 | 475 | 521 |
| 0 | 4 | 451 | 444 |
| 1 | 1 | 505 | 601 |
| -1 | 3 | 438 | 429 |
| -1 | 4 | 520 | 546 |
| -1 | 5 | 470 | 494 |
| -2 | 1 | 567 | 523 |
| 2 | 2 | 303 | 223 |
| -2 | 3 | 495 | 475 |
| -2 | 4 | 357 | 351 |
| -2 | 5 | 419 | 429 |
| -3 | 2 | 911 | 966 |
| 3 | 3 | 369 | 457 |
| -3 | 3 | 470 | 449 |
| -3 | 4 | 384 | 396 |
| -4 | 2 | 285 | 280 |
| -4 | 3 | 812 | 858 |
| -4 | 5 | 331 | 313 |
| -5 | 2 | 392 | 368 |
| -5 | 3 | 324 | 343 |
| -5 | 4 | 575 | 563 |
| -5 | 6 | 338 | 272 |
| -6 | 1 | 383 | 348 |
| -6 | 5 | 480 | 485 |
| -7 | 1 | 344 | 315 |
| -7 | 2 | 377 | 410 |
| -8 | 4 | 331 | 247 |
| ******L=22****** | | | |
| 0 | 2 | 557 | 605 |
| 1 | 0 | 446 | 399 |
| 2 | 0 | 341 | 348 |
| -2 | 0 | 720 | 805 |
| 2 | 1 | 325 | 269 |
| 3 | 0 | 303 | 285 |
| 3 | 2 | 342 | 393 |
| -3 | 4 | 457 | 431 |
| -4 | 2 | 305 | 190 |
| -4 | 3 | 322 | 400 |
| 5 | 0 | 462 | 391 |
| ******L=23****** | | | |
| 0 | 1 | 496 | 440 |
| 0 | 5 | 423 | 385 |
| 1 | 2 | 463 | 479 |
| -1 | 2 | 321 | 263 |
| -1 | 4 | 361 | 348 |
| 2 | 3 | 302 | 333 |
| -2 | 3 | 425 | 467 |
| -3 | 3 | 413 | 402 |
| -3 | 4 | 283 | 313 |
| -3 | 6 | 314 | 224 |
| -4 | 2 | 373 | 304 |
| -5 | 3 | 353 | 392 |
| -7 | 1 | 354 | 355 |
| -7 | 2 | 328 | 164 |
| ******L=24****** | | | |
| 0 | 0 | 600 | 637 |
| 1 | 1 | 465 | 485 |
| -1 | 3 | 382 | 339 |
| 2 | 0 | 363 | 332 |
| -2 | 0 | 327 | 350 |
| 2 | 2 | 446 | 416 |
| -3 | 0 | 521 | 495 |
| 4 | 0 | 388 | 346 |
| -4 | 1 | 375 | 289 |
| 5 | 0 | 378 | 279 |
| -5 | 2 | 418 | 437 |
| -6 | 6 | 349 | 375 |
| -7 | 0 | 504 | 429 |
| ******L=26****** | | | |
| 0 | 0 | 452 | 454 |
| -1 | 0 | 319 | 384 |

$H_5)_3P)I_3^-$ where all Ni–I distances are within ∼0.01 Å of one another. The entire "tetrahedron" is slightly flattened, and the Br atoms are rotationally staggered with respect to the phenyl rings, as expected. The pitch of the phenyl rings also noted by Taylor, *et al.*, appears to be different than in $Ni((C_6H_5)_3P)I_3^-$ and presumably reflects the difference in the cation and in the crystal packing.

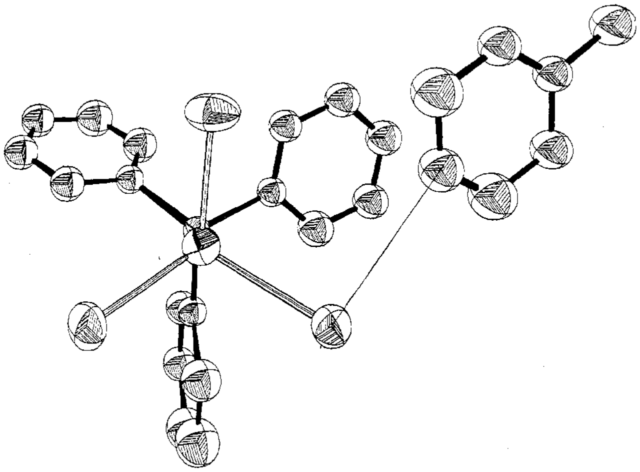

Figure 3.—Structure of the molecule looking approximately along the zinc–phosphorus bond.

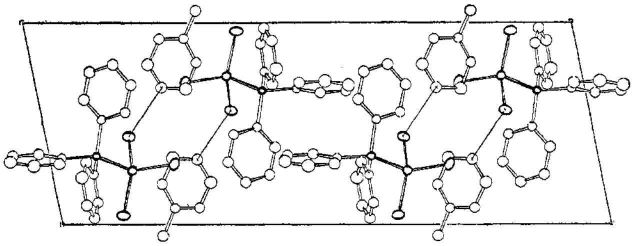

Figure 4.—Projection of the contents of the unit cell onto the *ac* plane.

**Cation Geometry.**—Unlike the phenyl groups which were refined as rigid bodies, atoms of the 4-methylpyridinium cation were located individually and refined with anisotropic temperature factors. Probably as a result of the domination of the scattering by the five heavy atoms, the positions of the light atoms are not well defined. Comparison of the cation positional parameters before and after introduction of the anisotropic thermal parameters shows little change and no significant improvement in precision. The average bond length in the ring is 1.35 Å, somewhat shorter than might be expected, and the average bond angle within the ring is 119.9°. The single bond to the methyl carbon is of the length expected (1.54 Å).

It seems reasonable that the hydrogen-bonding interaction should disturb the cation and anion equally, but the effect on the cation is too small to detect in view of the large standard deviations in the light-atom bond lengths.

As mentioned above, the N–H proton was located from a difference Fourier map and the cation ring protons were put in their predicted positions. Proton parameters were not varied since it was not expected that they could be sufficiently well defined. The N–H distance of 1.01 Å is slightly longer than expected for a normal N–H proton but perhaps reasonable considering the hydrogen bonding.

**Overall Structure.**—The most notable aspect of the structure is the strong hydrogen bonding which presumably is the force holding the molecule together in the lattice. The N–Br(2) distance of 3.2 Å is quite short and has some interesting implications for nmr contact shift studies of ion pairing in solution (involving, for example, the analogous $Co^{2+}$ or $Ni^{2+}$ complexes). While solid-state and solution structures may not be very similar, especially with respect to distortion of the anion, the fact that the cation can approach the anion so closely along what appears to be a direction of no symmetry clearly points up the difficulty of trying to work from a simple model (*i.e.*, trigonal or tetragonal) for a quantitative treatment of all but the most favorable cases. We also note that 3.2 Å is certainly a short enough distance for direct overlap of nitrogen and bromine orbitals containing unpaired spin and that any dipolar shifts on the cation are likely to be complicated by a direct contact interaction.[15]

**Electron Spin Resonance.**—Examination of the single-crystal electron spin resonance spectra of $Co^{2+}$ doped into the zinc compound indicates immediately that there is a rather strong rhombic component to the electronic structure. The ratio of the zero-field splitting parameters $D$ and $E$ is such that off-diagonal terms coupling the $\pm ^1/_2$ ground spin doublet to the excited $\pm ^3/_2$ doublet[16] are of sufficient magnitude that they cannot be neglected and thus transitions only within the $\pm ^1/_2$ doublet observed in the esr experiment do not by themselves serve to determine either $g$ values or zero-field parameters. Powder spectra support this view and indicate that both $D$ and $E$ terms are too large to apply the method of Hempel, Morgan, and Lewis[17] for the determination of spin Hamiltonian parameters. No quantitative data are thus reported here.

**Acknowledgment.**—The authors wish to express their appreciation to the Advanced Research Projects Agency for financial and equipment support under Contract SD-131 and to Professor R. S. Drago for helpful discussions.

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