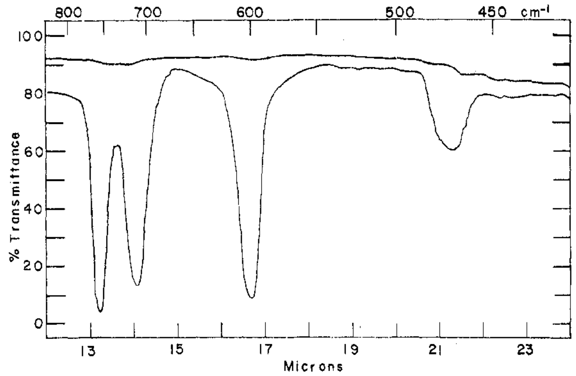

Figure 2.—Infrared spectrum of $SF_5N{=}SF_2$ at 4 mm.

3:2:2. The chemical shift values expressed in ppm relative to CF_3COOH were −108.2, −137.5, and −140.9, respectively. The first resonance, −108.2 ppm, was in agreement with those reported for the iminosulfur difluorides.[3,5] The other two resonances, −137.5 and −140.9 ppm, were also in the reported ranges[20,21] for pentafluorosulfanyl groups. The fine structure was not detected, for the solution was too dilute.

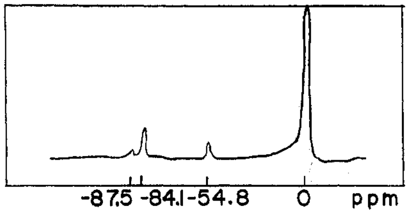

Figure 3.—F^{19} nmr spectrum of $SF_5N{=}SF_2$ 0.2 *M* in reference solvent CCl_3F.

(20) J. A. Pople, W. G. Schneider, and H. J. Bernstein, "High-resolution Nuclear Magnetic Resonance," McGraw-Hill Book Co., Inc., New York, N. Y., 1959, p 340.

(21) C. I. Merrill, S. M. Williamson, G. H. Cady, and D. F. Eggers, Jr., *Inorg. Chem.*, **1**, 215 (1962).

CONTRIBUTION FROM THE LAWRENCE RADIATION LABORATORY AND DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF CALIFORNIA, BERKELEY, CALIFORNIA

The Crystal and Molecular Structure of Tetraphenylarsonium *cis*-Diaquotetrachlororuthenate Monohydrate[1]

BY TED E. HOPKINS, ALLAN ZALKIN, DAVID H. TEMPLETON, AND MARTYN G. ADAMSON

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X-Ray diffraction study of a single crystal of $(C_6H_5)_4AsRuCl_4(H_2O)_2\cdot H_2O$ showed that it is monoclinic with a = 15.059, b = 16.711, c = 10.996 A, β = 99.88°, D_x = 1.619 g cm^{-3}. The space group is $P2_1/n$ and there are four formula units per unit cell. The ruthenium is surrounded by an octahedron composed of the four chlorine atoms and two water molecules. A *cis* configuration was found for the waters in the octahedron. The average Ru–O and Ru–Cl distances are 2.12 and 2.34 A. An unsymmetrical configuration was found for the tetraphenylarsonium ion, with an average As–C bond length of 1.91 A.

Introduction

In the course of a series of studies of aqueous ruthenium species by Professor Connick and others at this laboratory, a number of ruthenium salts have been prepared. We have determined the crystal structure of a cesium aquopentachlororuthenate[2a] and of a hexachlororuthenate.[2b] The diaquochloro complex is interesting because of the possibility for *cis–trans* isomers. When it was found that single crystals of the complex could be obtained using the tetraphenylarsonium cation, the structure determination was undertaken to determine the configuration of the ruthenium complex.

Experimental Section

A solution of chlororuthenate species containing a high proportion of the complex ion $RuCl_4(H_2O)_2^-$ was prepared by shaking a solution of 0.1 *M* ruthenium(III) in 2.5 *M* hydrochloric acid with clean mercury and allowing the Ru(III) solution to stand over the mercury for several hours. After the solution was separated from the mercury, it was filtered and sufficient aqueous tetraphenylarsonium chloride solution (0.3 *M*) was added, dropwise with stirring, to give equimolar amounts of $As(C_6H_5)_4^+$ and Ru(III). On standing at 0° for 48 hr, the solution yielded a number of fairly large ruby-red prisms which were collected on a filter pad, washed well with ice-cold water, and finally dried over Anhydrone in an evacuated desiccator.

The nature of the anion in this salt was shown to be exclusively one isomer of the complex. This was done by carrying out ion-exchange and spectral analysis on a freshly prepared solution of the salt in 1 *M* hydrochloric acid at 0°. A study of the aqueous complexes will be reported elsewhere.[3,4]

The Weissenberg technique was used for preliminary determination of the space group and cell dimensions. For taking the data, a single crystal, roughly cubic, 0.15 × 0.17 × 0.18 mm, was mounted for rotation around the b axis. Cell dimensions and intensities were measured with a General Electric XRD-5 diffractometer equipped with a scintillation counter, a pulse-height discriminator, and a quarter-circle Eulerian cradle goniostat. A molybdenum tube was used, operated at 50 kv and 20 ma, with a Zr filter at the receiving slit. For Mo Kα_1, λ is 0.70926 A.

A graph of the background was prepared for various values of χ and ϕ as a function of 2θ. Typical values were 30, 15, 11, and 4 counts/sec for 2θ = 4, 6, 11, and 21°. For $2\theta > 22°$, the back-

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[1] Work done under the auspices of the U. S. Atomic Energy Commission.

[2] (a) T. E. Hopkins, A. Zalkin, D. H. Templeton, and M. G. Adamson, *Inorg. Chem.*, **5**, 1431 (1966); (b) to be published.

[3] D. A. Fine, Ph.D. Thesis, University of California at Berkeley, 1958.

[4] M. G. Adamson and R. E. Connick, to be published.

Table I

Observed and Calculated Structure Factors for $(C_6H_5)_4AsRuCl_4(H_2O)_2 \cdot H_2O$

```
H,K= 0, 0
  L FOB FCA
  2 336-344
  4  89  88
  6 140-147
  8  76  79
 10  45 -48

H,K= 0, 1
  L FOB FCA
  1  37 -35
  2  20  16
  3 106 111
  4  88 -81
  5  92 -85
  6  21  14
  7  37  40
  8  27  27
  9  76 -82
 10  31 -28

H,K= 0, 2
  L FOB FCA
 -0 309 304
  1 180 180
  2 286-285
  3  42 -50
  4 130 128
  5  41  37
  6 118-120
  7  66 -62
  8  70  68
  9  32  30
 10  53 -52

H,K= 0, 3
  L FOB FCA
  1   6  13
  2   0  -1
  3  48 -45
  4   8   9
  5  48 -46
  6  10 -14
  7  29  29
  8  55  54
  9  21 -21
 10  45 -46

H,K= 0, 4
  L FOB FCA
 -0  69 -75
  1 216 211
  2  38  35
  3  99 -97
  4 115 117
  5  85  85
  6 114-118
  7  77 -76
  8  23  24
  9  54  50
 10  13 -14

H,K= 0, 5
  L FOB FCA
  1 125 131
  2 109-115
  3  47 -50
  4  57  57
  5  13 -14
  6  35 -39
  7  14   6
  8  31  27
  9  10  -3
 10   8  -6

H,K= 0, 6
  L FOB FCA
 -0  41 -33
  1   0   8
  2  32  26
  3  76 -84
  4   0   3
  5  46  48
  6  61 -60
  7  34 -28
  8  12 -16
  9  43  39

H,K= 0, 7
  L FOB FCA
  1  51  54
  2  44 -52
  3  48 -45
  4  18 -13
  5  26  21
  6  11   8
  7  12 -14
  8   7  -5
  9  17  17

H,K= 0, 8
  L FOB FCA
 -0  56  57
  1  15 -10
  2  15   6
  3  10  -2
  4  15 -16
  5   9   6
  6   6 -10
  7   7   6
  8   0   6
  9   8   6

H,K= 0, 9
  L FOB FCA
  1  20 -17
  2  18  17
  3  45  50
  4  58 -58
  5  21 -19
  6   0   5
  7  24 -29
  8   0   1

H,K= 0,10
  L FOB FCA
 -0  82  83
  1  14 -10
  2  42 -42
  3   6  -4
  4  16  20
  5  15  18
  6  55 -56
  7  19 -19
  8  30  31

H,K= 0,11
  L FOB FCA
  1   9 -11
  2   0   3
  3  55  56
  4  15 -16
  5  71 -72
  6  10  -4
  7   7  -2

H,K= 0,12
  L FOB FCA
 -0  52  49
  1   0  -1
  2  57 -57
  3  48 -47
  4  82  83
  5  66  68
  6  34 -41
  7  36 -37

H,K= 0,13
  L FOB FCA
  1  10   8
  2  37 -33
  3  14   8
  4  31  29
  5  13 -20
  6  20 -19

H,K= 0,14
  L FOB FCA
 -0  48  49
  1  66  71
  2  34 -41
  3  90 -90
  4  25  28
  5  42  47

H,K= 0,15
  L FOB FCA
  1  10   6
  2  34 -33
  3  11 -15
  4  15  17

H,K= 0,16
  L FOB FCA
 -0  11   3
  1  97 100
  2  13   9

H,K= 1, 0
  L FOB FCA
 -9  23  30
 -7   0  -9
 -5   0 -10
 -3  33  24
 -1 101-107
  1 129 128
  3 135-116
  5  99  95
  7 111-114
  9  39  40

H,K= 1, 1
  L FOB FCA
-10  13 -14
 -9  20  15
 -8  21  17
 -7  22 -21
 -6  28  25
 -5   9  -9
 -4 164-164
 -3  45  38
 -2   6  -9
 -1  37  37
 -0 136 126
  1  34 -25
  2  52 -60
  3  44 -40
  4  76  80
  5  11 -12
  6  40 -47
  7  49  51
  8   0  -2
  9   0  -1
 10  50 -49

H,K= 1, 2
  L FOB FCA
-10   0   3
 -9  14 -18
 -8  33 -30
 -7  14  15
 -6   8   4
 -5  47 -50
 -4  83  83
 -3  40  38
 -2 104-109
 -1  45 -46
 -0  78 -67
  1  75  79
  2  84 -69
  3  58  63
  4  47 -51
  5  40  39
  6  55  53
  7  51 -52
  8  30 -26
  9   0   5
 10   0   9

H,K= 1, 3
  L FOB FCA
-10   0  -7
 -9  14  10
 -8  65  65
 -7  11  -7
 -6 150-153
 -5  35  31
 -4  44  50
 -3  49 -51
 -2  62 -72
 -1 102  97
 -0 223 204
  1  79 -68
  2  35 -30
  3   9  10
  4 120 122
  5   7  -4
  6 108-114
  7   0   7
  8  43  40
  9   0  -5
 10  45 -40

H,K= 1, 4
  L FOB FCA
-10   0  -4
 -9  34 -37
 -8   7   4
 -7  61  63
 -6  34 -31
 -5  65 -70
 -4  43 -40
 -3   6   8
 -2 104 107
 -1   8  11
 -0  23  18
  1  18  -9
  2  12  14
  3  17  19
  4  48 -42
  5  48 -51
  6  14  11
  7  44  44
  8  45 -45
  9  49 -45
 10  34  33

H,K= 1, 5
  L FOB FCA
-10  13 -14
 -9  14 -16
 -8  76  77
 -7  47  54
 -6 154-158
 -5  13 -13
 -4 161 164
 -3  65 -62
 -2 150-165
 -1  61  54
 -0 187 176
  1  64  55
  2 119-113
  3  41  34
  4 134 131
  5  43  41
  6 151-157
  7  30 -30
  8  64  67
  9  41  38
 10  22 -24

H,K= 1, 6
  L FOB FCA
-10   0  -4
 -9  34 -37
 -8   0  -7
 -7  49  49
 -6   6   5
 -5  64 -68
 -4  63 -66
 -3  55 -64
 -2 149 148
 -1  84  83
 -0  28 -10
  1  44  33
  2 110-103
  3  10  -8
  4  55  59
  5  49 -49
  6  31 -28
  7  67  67
  8   0  -4
  9  78 -80

H,K= 1, 7
  L FOB FCA
 -9  48 -44
 -8  45  40
 -7  52  55
 -6  82 -82
 -5  78 -84
 -4 151 152
 -3 100 102
 -2 225-234
 -1 136-137
 -0 226 225
  1 106 104
  2  97 -95
  3 113-112
  4 135 133
  5  30  36
  6  91 -93
  7  47 -51
  8  10  10
  9  30  30

H,K= 1, 8
  L FOB FCA
 -9  13 -13
 -8  41 -38
 -7  22  20
 -6   6  10
 -5  39 -39
 -4  15 -17
 -3  26 -31
 -2  53  46
 -1  46  35
 -0  76  74
  1   0  -6
  2  52 -52
  3  56  58
  4  12  10
  5  53 -52
  6  61 -64
  7  29  34
  8  50  51
  9  51 -53

H,K= 1, 9
  L FOB FCA
 -9  46 -43
 -8  31  30
 -7  46  44
 -6  33 -28
 -5 120-125
 -4  64  62
 -3 152 160
 -2 120-126
 -1 155-158
 -0  94  84
  1 123 124
  2  51 -55
  3  99-105
  4  47  48
  5  89  91
  6  20 -19
  7  58 -59
  8   8   7

H,K= 1,10
  L FOB FCA
 -8  46 -43
 -7  16 -21
 -6  31  34
 -5  27 -22
 -4  13  14
 -3  26  25
 -2  59 -53
 -1  30 -29
 -0  41  36
  1  18  16
  2  10  11
  3  51  53
  4  16  19
  5  28 -29
  6  41 -46
  7   0  -4
  8  69  68

H,K= 1,11
  L FOB FCA
 -8  19  23
 -7  59  56
 -6  28 -28
 -5 116-114
 -4  40 -42
 -3 135 138
 -2  39  33
 -1  75 -78
 -0   0  -0
  1  52  54
  2  31  31
  3  87 -89
  4  16 -17
  5  73  74
  6   0  -3
  7  65 -60

H,K= 1,12
  L FOB FCA
 -7  11 -16
 -6  24  24
 -5   7   6
 -4   0  -0
 -3  24  16
 -2  31  30
 -1  37 -32
 -0  17  15
  1  45  42
  2  25 -21
  3  13   0
  4   0  -0
  5  16   9
  6  42 -45
  7  36 -42

H,K= 1,13
  L FOB FCA
 -6   8   5
 -5  35 -38
 -4  32 -32
 -3  53  53
 -2  49  49
 -1  32 -32
 -0  34 -35
  1  43  35
  2  14  12
  3  35 -37
  4  16 -21
  5  39  37
  6   0   1

H,K= 1,14
  L FOB FCA
 -5   0   3
 -4  30 -29
 -3  13  -9
 -2  40  38
 -1  16  -8
 -0  21 -20
  1  38  35
  2   0   2
  3  28 -29
  4   7  -8
  5  26  30

H,K= 1,15
  L FOB FCA
 -4   0  -4
 -3   8 -12
 -2   7   7
 -1  20 -22
 -0  13 -12
  1  56  54
  2   7   5
  3  24 -23
  4   8  11

H,K= 1,16
  L FOB FCA
 -2  26  25
 -1   8   6
 -0  23 -25
  1  19   6
  2  11  15

H,K= 2, 0
  L FOB FCA
-10  70 -68
 -8 135 137
 -6 169-167
 -4  51  53
 -2 271-275
 -0 281 286
  2 266-280
  4 152 157
  6  14  11
  8  44 -46
 10  51 -49

H,K= 2, 1
  L FOB FCA
-10  29  27
 -9  39 -40
 -8  30 -29
 -7  15  14
 -6  39 -39
 -5  40  38
 -4  64  68
 -3  92 -90
 -2  71  58
 -1  52  44
 -0  87  79
  1  59 -47
  2  79  78
  3 146 151
  4  13 -11
  5 111-115
  6  28 -30
  7  59  56
  8  27  25
  9  24 -26
 10  23 -22

H,K= 2, 2
  L FOB FCA
-10  37 -34
 -9  33 -28
 -8 125 120
 -7  61  58
 -6 113-119
 -5  93 -89
 -4  86  89
 -3  36  37
 -2 173-178
 -1  51 -46
 -0 222 210
  1 134 130
  2 141-134
  3  92 -98
  4 152 149
  5  38  38
  6  68 -73
  7   0  -5
  8  22  23
  9   0   9
 10  41 -41

H,K= 2, 3
  L FOB FCA
-10  32  32
 -9   7  -9
 -8  34 -36
 -7  69  67
 -6  42 -39
 -5  24 -30
 -4  90  88
 -3  19  15
 -2  30 -37
 -1 114-113
 -0  12  -2
  1   0   3
  2  26 -18
  3  87  84
  4  61  56
  5  19  14
  6  85 -84
  7  18  18
  8  54  52
  9  44 -47
 10  21 -21

H,K= 2, 4
  L FOB FCA
-10   0  -5
 -9  43 -39
 -8  46  47
 -7 104 103
 -6  74 -74
 -5 147-151
 -4  63  67
 -3  95  93
 -2 186-183
 -1 100 -92
 -0  56  46
  1  28  38
  2 109  96
  3  69 -67
  4 132 139
  5  16  17
  6 131-136
  7  25 -28
  8  62  58
  9  22  24
 10  44 -39

H,K= 2, 5
  L FOB FCA
-10  11  11
 -9   7  -8
 -8   0   8
 -7  59  61
 -6  10 -13
 -5 128-130
 -4   5 -10
 -3  12  10
 -2 108 102
 -1  39  36
 -0  15  -8
  1  60  56
  2  38 -39
  3  74 -73
  4 100  98
  5  75  79
  6 101-101
  7  37 -37
  8  16  17
  9  24 -22

H,K= 2, 6
  L FOB FCA
-10  13  14
 -9  27 -27
 -8  14  -9
 -7  38  34
 -6  11   5
 -5  60 -63
 -4   0   6
 -3  85  89
 -2  65 -76
 -1  83 -80
 -0  81  77
  1  40  46
  2   7   5
  3  15 -15
  4  74  77
  5  57  58
  6  78 -81
  7  54 -58
  8  44  41
  9  25  27

H,K= 2, 7
  L FOB FCA
 -9   7 -11
 -8   7  -9
 -7  48  49
 -6   6  -6
 -5  70 -73
 -4  86 -87
 -3  21  21
 -2 105 106
 -1  59 -54
 -0  76 -67
  1  53  47
  2  19  17
  3 107-106
  4   6  -2
  5  97  97
  6   6  -7
  7  53 -54
  8   0  -3
  9   0  -2

H,K= 2, 8
  L FOB FCA
 -9   0   9
 -8   0   1
 -7  19 -18
 -6  29  31
 -5   6   5
 -4  39 -40
 -3  40  42
 -2  46 -53
 -1  80 -82
 -0  54  57
  1  73  70
  2  87 -86
  3  44 -43
  4  83  87
  5  51  53
  6  18 -17
  7  35 -36
  8  25  20

H,K= 2, 9
  L FOB FCA
 -9   0   2
 -8  32 -33
 -7  12  15
 -6  38  36
 -5  35 -38
 -4  60 -66
 -3  39  39
 -2  50  49
 -1  66 -66
 -0  15  -5
  1  61  55
  2  79  80
  3  17  21
  4 105-103
  5  15  15
  6  34  32
  7  10 -11
  8  15 -17

H,K= 2,10
  L FOB FCA
 -8  49  45
 -7   0  -7
 -6  36 -35
 -5   9  -7
 -4  34  39
 -3  32  34
 -2  39 -39
 -1  35 -31
 -0  62  58
  1  62  63
  2  52 -50
  3  32 -36
  4  50  48
  5  41  37
  6  21 -22
  7  57 -57
  8  11  14

H,K= 2,11
  L FOB FCA
 -8  41 -40
 -7   0   5
 -6  21  24
 -5   7  -6
 -4  59 -60
 -3  11  10
 -2  47  46
 -1  21 -23
 -0  19  -8
  1  14  -5
  2  58  56
  3  77  80
  4  78 -78
  5  63 -62
  6  10  15
  7  22  21

H,K= 2,12
  L FOB FCA
 -7  19  18
 -6  73 -73
 -5  53 -55
 -4  57  55
 -3  34  38
 -2  55 -54
 -1  13 -12
 -0  41  39
  1  11  16
  2  39 -35
  3  38 -39
  4  12  16
  5  48  50
  6  29 -30

H,K= 2,13
  L FOB FCA
 -6  15  18
 -5   7  13
 -4  12 -11
 -3  47 -53
 -2  32  27
 -1  17  18
 -0  15 -10
  1  15 -16
  2   7  -1
  3  53  52
  4  13  11
  5  20 -23
  6   0 -11

H,K= 2,14
  L FOB FCA
 -5  54 -55
 -4  30  31
 -3  51  52
 -2  39 -41
 -1  41 -41
 -0  12   9
  1  62  63
  2   7  -6
  3  53 -55
  4  23  25
  5  30  32

H,K= 2,15
  L FOB FCA
 -4   0   3
 -3  29 -31
 -2   7  -7
 -1  28  29
 -0  26  28
  1   0   0
  2  23 -21
  3  11  -3

H,K= 2,16
  L FOB FCA
 -2  11  11
 -1  69 -67
 -0   0  -3
  1  88  87

H,K= 3, 0
  L FOB FCA
 -9  87 -85
 -7 121 123
 -5  83 -85
 -3  62  61
 -1 244-255
  1 172 172
  3 115-101
  5  69  66
  7  40 -36
  9   7   8

H,K= 3, 1
  L FOB FCA
-10   0  -0
 -9  20  21
 -8  55  54
 -7   9  -6
 -6  10 -11
 -5  40  41
 -4  42 -36
 -3   7  -2
 -2  75 -85
 -1  96 -90
 -0 264 252
  1  75 -66
  2 228-234
  3  22 -22
  4 242 243
  5  36  35
  6  77 -85
  7  22 -22
  8  50  48
  9   0  -1
 10  63 -63

H,K= 3, 2
  L FOB FCA
-10  37  36
 -9  52 -51
 -8  78 -77
 -7  93  94
 -6  64  63
 -5  82 -78
 -4  11  16
 -3  66  65
 -2  15  -9
 -1 120-131
 -0  38 -36
  1  82  83
  2 157 152
  3  56 -47
  4 143-142
  5  56  53
  6  56  59
  7   0  -6
  8   0  -9
  9   0   1

H,K= 3, 3
  L FOB FCA
-10  21 -18
 -9  48  48
 -8  82  79
 -7  18 -15
 -6  56 -60
 -5   0 -10
 -4 122 120
 -3   0   7
 -2 202-209
 -1   5   8
 -0  29 -38
  1  95  87
  2  57 -57
  3  25 -30
  4 178 179
  5  77  78
  6  68 -73
  7  55 -54
  8  48  48
  9  17  17

H,K= 3, 4
  L FOB FCA
-10  34  31
 -9   0  -5
 -8  55 -54
 -7  58  57
 -6  35  36
 -5  51 -58
 -4 107-102
 -3  27  29
 -2  59  60
 -1   9 -14
 -0 104 -99
  1  84 -74
  2 233 216
  3  24  24
  4  41 -43
  5   6  -5
  6   9  -8
  7  25  26
  8  26 -26
  9  40 -37

H,K= 3, 5
  L FOB FCA
-10  33 -29
 -9  10   5
 -8  73  71
 -7  21  23
 -6  90 -91
 -5  70 -69
 -4 125 121
 -3   5   3
 -2 192-193
 -1  34 -32
 -0  52  51
  1  17  18
  2  45 -47
  3  88 -84
  4  75  80
  5  30  32
  6  53 -53
  7  35 -35
  8  45  43
  9  41  42

H,K= 3, 6
  L FOB FCA
-10  13  15
 -9   7   9
 -8   0   1
 -7  23  23
 -6  16 -17
 -5   0   2
 -4  37 -35
 -3  34 -34
 -2  90  91
 -1  92  84
 -0  69 -70
  1 103 -97
  2   7  -3
  3 106 100
  4  10  12
  5  16  15
  6  29 -33
  7  45  44
  8   0  -3
  9  39 -42

H,K= 3, 7
  L FOB FCA
 -9  36 -37
 -8  62  61
 -7  47  50
 -6  28 -29
 -5  21 -25
 -4  51  48
 -3  84  84
 -2 168-166
 -1  63 -67
 -0 108 105
  1  67  67
  2  39 -41
  3  40 -40
  4  46  46
  5  18  16
  6   9 -10
  7   0   1
  8  13   8
  9  27  25

H,K= 3, 8
  L FOB FCA
 -9   0   4
 -8  13  14
 -7   0   2
 -6  28 -28
 -5  27  30
 -4  10  12
 -3  32 -29
 -2   5 -12
 -1  12  -2
 -0  74  69
  1  72 -66
  2  26 -26
  3  81  76
  4  41  41
  5  13 -11
  6  34 -33
  7   0   7
  8   0   1

H,K= 3, 9
  L FOB FCA
 -9  42 -43
 -8  40  40
 -7  32  33
 -6  20 -22
 -5  52 -52
 -4  21  19
 -3  58  59
 -2  45 -50
 -1 120-123
 -0  72  68
  1 102 100
  2  44 -46
  3   9  -7
  4  17  16
  5  15  15
  6  21 -20
  7  31 -31
  8   0   7

H,K= 3,10
  L FOB FCA
 -8   0   1
 -7   0   3
 -6  23 -26
 -5  21 -20
 -4   0  -2
 -3  41  36
 -2  51 -51
 -1 103 -96
 -0  97  93
  1  54  50
  2  37 -39
  3  14  14
  4   0   3
  5   7 -11
  6  31 -30
  7   8   4

H,K= 3,11
  L FOB FCA
 -8   0   7
 -7  50  51
 -6  12 -14
 -5  71 -74
 -4  19  19
 -3  82  81
 -2  11   4
 -1  78 -77
 -0  34 -26
  1  32  28
  2  19 -17
  3   7   7
  4  58  53
  5  40  37
  6   7 -15
  7  45 -45

H,K= 3,12
  L FOB FCA
 -7  27  24
 -6   7   9
 -5  34 -34
 -4  24 -27
 -3  25  23
 -2   0  -1
 -1  77 -72
 -0  26  26
  1  79  77
  2   7  -8
  3  59 -55
  4  22  22
  5  27  26
  6  28 -28

H,K= 3,13
  L FOB FCA
 -6   0   8
 -5  73 -79
 -4  16  18
 -3  59  57
 -2  10   5
 -1  31 -28
 -0  17 -14
  1  35  33
  2  10 -10
  3  49 -45
  4  31  35
  5  36  37

H,K= 3,14
  L FOB FCA
 -5  11 -11
 -4  45 -44
 -3  19 -13
 -2  39  40
 -1  19 -22
 -0  40 -38
  1  39  37
  2  38  36
  3  33 -34
  4   0   6

H,K= 3,15
  L FOB FCA
 -4   0  -4
 -3   8   8
 -2  17 -15
 -1  28 -24
 -0  18  16
  1  79  74
  2  15 -11
  3  65 -64

H,K= 3,16
  L FOB FCA
 -1   0   0
 -0  44 -44

H,K= 4, 0
  L FOB FCA
-10  62 -60
 -8  79  78
 -6  45 -47
 -4  82  89
 -2  27 -31
 -0  23   8
  2 194-200
  4 164 166
  6  21  21
  8   0  -7

H,K= 4, 1
  L FOB FCA
-10   7 -10
 -9  19 -21
 -8  11 -11
 -7  42  42
 -6  16  16
 -5  30  31
 -4  50  42
 -3  17 -24
 -2 194-198
 -1  43  34
 -0  27  34
  1  68 -69
  2  22  17
  3  38  31
  4  12  12
  5  20 -19
  6   6  -2
  7  25  27
  8   7  -4
  9   0  -0

H,K= 4, 2
  L FOB FCA
-10  37 -34
 -9  12  12
 -8  82  82
 -7   6  -3
 -6  58 -58
 -5  57 -55
 -4  80  81
 -3  74  82
 -2 157-155
 -1  70  68
 -0  22 -20
  1  82 -72
  2  82 -79
  3  39 -39
  4 140 144
  5  21  20
  6  37 -38
  7  38  41
  8  17  15
  9  30 -31

H,K= 4, 3
  L FOB FCA
-10   0  -9
 -9  17 -18
 -8   7   2
 -7  54  53
 -6  16 -13
 -5  48 -50
 -4  81  80
 -3  50  52
 -2 187-192
 -1  87 -88
 -0  92  94
  1  16 -15
  2  17 -10
  3   0   2
  4  19 -17
  5  10  14
  6  33 -33
  7  24  23
  8  17  18
  9   0  -1

H,K= 4, 4
  L FOB FCA
-10  20 -18
 -9   0  -3
 -8  53  49
 -7  61  59
 -6  44 -46
 -5  59 -62
 -4  97 104
 -3  57  57
 -2 125-133
 -1   0   3
 -0  16  18
  1  41 -41
  2  37 -38
  3  39  32
  4  86  85
  5  13  12
  6  70 -71
  7   7  10
  8 105 104
  9   0 -11

H,K= 4, 5
  L FOB FCA
-10   0  -2
 -9  51 -46
 -8  17  14
 -7  73  70
 -6  35 -35
 -5  67 -69
 -4  14 -15
 -3 135 133
 -2  14  -2
 -1 101-103
 -0  17  12
  1  11   7
  2  25  21
  3  76 -76
  4  58  58
  5  71  72
  6  60 -60
  7   7 -11
  8  15  11
  9   0   3

H,K= 4, 6
  L FOB FCA
-10  16 -16
 -9  30 -26
 -8  27  27
 -7  46  47
 -6  16  14
 -5  41 -42
 -4  73  71
 -3  47  51
 -2 101-104
 -1  91 -88
 -0 122 119
  1  75  76
  2 134-139
  3   0  -6
  4  81  83
  5  35  36
  6  79 -80
  7  30 -28
  8  85  82
  9   8  12

H,K= 4, 7
  L FOB FCA
 -9  53 -49
 -8   7  -9
 -7  70  70
 -6   0  -0
 -5  66 -69
 -4  66 -65
 -3  95  95
 -2  99 102
 -1  93 -97
 -0  52 -54
  1  44  40
  2  40  38
  3  38 -41
  4  26  22
  5  70  67
  6  13 -19
  7  21 -22
  8  11 -10

H,K= 4, 8
  L FOB FCA
 -9  27 -22
 -8  25  25
 -7  15  16
 -6  11  14
 -5   0  -5
 -4  18  18
 -3  26  29
 -2 102-105
 -1 143-144
 -0 102 104
  1 120 118
  2 130-129
  3 100-101
  4 121 116
  5  53  49
  6  27 -23
  7  54 -55
  8  40  40

H,K= 4, 9
  L FOB FCA
 -8  41 -43
 -7  27  26
 -6  51  52
 -5   9 -10
 -4  86 -87
 -3  21  20
 -2  61  62
 -1 105-106
 -0  49 -44
  1  25  30
  2  67  66
  3  12  12
  4  43 -41
  5  14  13
  6  36  34
  7  13  16

H,K= 4,10
  L FOB FCA
 -8  26  30
 -7  13  14
 -6  36 -36
 -5  45 -47
 -4  40  38
 -3  72  73
 -2  29 -32
 -1 102-106
 -0  24  20
  1 113 114
  2  44 -44
  3  88 -89
  4  13  13
  5  70  70
  6   0  -5
  7  59 -59

H,K= 4,11
  L FOB FCA
 -7   0   2
 -6  74  75
 -5  37  33
 -4  83 -79
 -3  16 -19
 -2  41  42
 -1   9   6
 -0  46 -42
  1  17 -10
  2  64  67
  3  18  14
  4  14 -11
  5  31 -35
  6  17  17

H,K= 4,12
  L FOB FCA
 -7  33  33
 -6  36 -37
 -5  63 -58
 -4  48  47
 -3  69  70
 -2  20 -20
 -1  83 -80
 -0  19 -14
  1  54  51
  2  30  29
  3  36 -37
  4   7 -13
  5  66  64
  6  11  10

H,K= 4,13
  L FOB FCA
 -6  17  18
 -5  25  26
 -4  39 -38
 -3  61 -61
 -2  32  32
 -1  48  46
 -0  36 -31
  1  38 -35
  2  19  19
  3  10  13
  4   0  -5
  5   0   0

H,K= 4,14
  L FOB FCA
 -5  30 -30
 -4  19  17
 -3  21  19
 -2   7  -5
 -1  34 -37
 -0  19 -21
  1  40  39
  2   0   2
  3  15 -15
  4   0  -6

H,K= 4,15
  L FOB FCA
 -3  15 -22
 -2  26 -27
 -1  15  21
 -0   8   9
  1  11 -15
  2   0   0

H,K= 5, 0
  L FOB FCA
 -9  87 -78
 -7 104  99
 -5  98-104
 -3 165 162
 -1  10   6
  1  35  33
  3 114-118
  5  13  17
  7  57  53
  9   0   1

H,K= 5, 1
  L FOB FCA
-10  50 -48
 -9   0   1
 -8  61  64
 -7   0   4
 -6  38 -41
 -5  23 -25
 -4 123 119
 -3  65  64
 -2 164-166
 -1  93 -86
 -0  62  70
  1  19  17
  2 246-241
  3  60 -56
  4 197 198
  5  41  45
  6 110-110
  7  25 -23
  8  84  82
  9  13  17

H,K= 5, 2
  L FOB FCA
-10  34  32
 -9  70 -67
 -8  80 -76
 -7  87  85
 -6 107 109
 -5  73 -73
 -4  39 -38
 -3 111 109
 -2  84 -87
 -1 102-106
 -0  71  72
  1  24  20
  2  37  36
  3  49 -47
  4  52 -54
  5   0   3
  6  14  12
  7  30  28
  8  18  19
  9   0  -0

H,K= 5, 3
  L FOB FCA
-10  30 -30
 -9   0  -2
 -8  35  36
 -7  13  15
 -6  66 -70
 -5  86 -87
 -4 190 192
 -3 143 144
 -2  98-105
 -1 151-145
 -0  42  41
  1  74  70
  2 107-105
  3  78 -75
  4  76  79
  5  86  89
  6  59 -57
  7  68 -66
  8  90  87
  9  35  34

H,K= 5, 4
  L FOB FCA
-10  34  34
 -9  23 -24
 -8  47 -48
 -7  36  41
 -6  89  86
 -5  25 -23
 -4  76 -77
 -3  88  93
 -2  33  30
 -1  43 -44
 -0   8   1
  1   5  -4
  2  87  82
  3  27  25
  4  20 -23
  5   9   6
  6  17 -18
  7   0  -0
  8  25  25
  9   0  -1

H,K= 5, 5
  L FOB FCA
-10  22 -20
 -9   0  -4
 -8  32  31
 -7  28  25
 -6  26 -31
 -5  64 -66
 -4  78  82
 -3 110 115
 -2  69 -74
 -1  87 -83
 -0  37  34
  1  60  54
  2  24 -27
  3  74 -74
  4  26  21
  5  74  70
  6   7   2
  7  58 -57
  8  39  38

H,K= 5, 6
  L FOB FCA
 -9  18 -19
 -8   0   6
 -7   9  -8
 -6  11  14
 -5   8   2
 -4  53 -52
 -3   8   4
 -2  59  57
 -1  28 -28
 -0 122-123
  1  65 -69
  2  71  67
  3  26 -23
  4  22  22
  5  18  15
  6  19 -16
  7  16 -16
  8   0   0

H,K= 5, 7
  L FOB FCA
 -9  21 -22
 -8  24  24
 -7  26  28
 -6  18  17
 -5   6   9
 -4  20 -18
 -3   0   6
 -2  16  15
 -1  65 -70
 -0  41  34
  1  46  49
  2  93 -88
  3  51 -46
  4   6   4
  5  37  37
  6  23  25
  7   0   1
  8  13  11

H,K= 5, 8
  L FOB FCA
 -9  15 -13
 -8   0   2
 -7   0   2
 -6  21 -22
 -5  48  47
 -4   0  -1
 -3  18  14
 -2  18 -17
 -1  13 -12
 -0  37 -37
  1  16 -16
  2  24  25
  3  15 -17
  4  18  19
  5  10  10
  6  26  24
  7   0  -2

H,K= 5, 9
  L FOB FCA
 -8  36  36
 -7  22  22
 -6   0   1
 -5  30  32
 -4  20  19
 -3  31 -29
 -2  24 -24
 -1  36 -34
 -0  32  32
  1  62  61
  2  44 -44
  3   9 -12
  4  33  31
  5   7  -8
  6   0   1
  7  13  12

H,K= 5,10
  L FOB FCA
 -8   0   2
 -7  13  11
 -6  10 -13
 -5   7  -3
 -4   0  -1
 -3  33  32
 -2  28 -25
 -1  50 -50
 -0  27  18
  1  15  14
  2  13  16
  3   7  -8
  4  21 -20
```

ground was constant at about 3 counts/sec. Individual backgrounds were determined for those reflections which were higher orders of strong reflections.

Of the 2731 independent reflections measured ($2\theta < 41°$), 255 were recorded as zero intensity. Ten-second counts were taken, with the crystal stationary. The strongest reflection (020) gave 15,320 counts/sec. No correction was made for either absorption or extinction. The calculated linear absorption coefficient is $\mu = 22.4$ cm^{-1}, with $\mu R \sim 0.2$ for molybdenum radiation. The crystal did not show any sign of deterioration during the taking of the data.

Calculations were made using IBM 7044 and CDC 6600 computers. The Fourier and distance programs used were written by Zalkin, and our modified (unpublished) version of the Gantzel–Sparks–Trueblood full-matrix, least-squares program which minimizes $\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2$. F_o and F_c are the observed and calculated structure factors, and the weights, w, were all taken as unity. The atomic scattering factors were chosen as follows: neutral As, Ru +3, values taken from Thomas and Umeda,[5] Cl −1 and neutral O, C, and H, from the "International Tables."[6] Corrections for the real part of the anomalous dispersion ($\Delta f'$) were made for arsenic, ruthenium, and chlorine by adding +0.1, −1.3 and +0.1, respectively, to their atomic scattering functions.[7]

Unit Cell and Space Group

The primitive cell contains four formula units of $(C_6H_5)_4AsRuCl_4(H_2O)_2 \cdot H_2O$. It is monoclinic with dimensions $a = 15.059 \pm 0.005$, $b = 16.711 \pm 0.005$, $c = 10.996 \pm 0.005$ A, $\beta = 99.88 \pm 0.01°$. The calculated density is 1.619 g cm^{-3}, and a rough measurement gave 1.7 g cm^{-3}.

The Weissenberg photographs showed the following reflection restrictions: $h0l$: $h + l = 2n$. Measurements with the goniostat confirmed this observation and the restriction $0k0$: $k = 2n$ was observed. These conditions correspond to space group $P2_1/n$. The observed and calculated structure factor magnitudes are given in Table I.

---

[5] L. H. Thomas and K. Umeda, *J. Chem. Phys.*, **26**, 293 (1957).

[6] "International Tables for X-ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962.

[7] See ref 6, p 216.

TABLE I (*Continued*)

H,K	L	FOB	FCA
	5	7	-1
	6	20	18
	7	27	29
5,11	-7	21	20
	-6	42	-42
	-5	17	-8
	-4	72	72
	-3	16	17
	-2	47	-44
	-1	24	-22
	-0	27	23
	1	0	6
	2	54	-55
	3	0	5
	4	32	32
	5	7	3
	6	25	-36
5,12	-6	24	21
	-5	29	-28
	-4	10	-12
	-3	34	35
	-2	10	6
	-1	42	-42
	-0	26	-25
	1	31	31
	2	17	12
	3	20	-23
	4	0	1
	5	17	14
5,13	-6	0	-10
	-5	54	-50
	-4	45	48
	-3	50	53
	-2	38	-43
	-1	44	-46
	-0	33	31
	1	10	10
	2	40	-42
	3	7	-14
	4	38	37
5,14	-4	33	-37
	-3	8	6
	-2	17	21
	-1	7	7
	-0	29	-26
	1	7	4
	2	13	19
	3	35	-33
5,15	-3	39	37
	-2	19	-20
	-1	62	-57
	-0	19	21
	1	69	66
6, 0	-10	44	-37
	-8	21	-18
	-6	10	11
	-4	50	50
	-2	92	86
	-0	43	-38
	2	80	-89
	4	16	15
	6	26	-31
	8	38	39
6, 1	-10	22	-19
	-9	30	-30
	-8	15	-17
	-7	0	7
	-6	25	25
	-5	34	-39
	-4	24	22
	-3	71	74
	-2	27	27
	-1	24	-19
	-0	47	47
	1	22	24
	2	17	15
	3	92	-93
	4	47	-44
	5	36	34
	6	72	72
	7	32	-33
	8	7	-9
6, 2	-10	24	-21
	-9	34	38
	-8	15	14
	-7	72	-72
	-6	13	-8
	-5	76	78
	-4	93	91
	-3	35	-32
	-2	21	-26
	-1	29	21
	-0	0	4
	1	51	-50
	2	32	-38
	3	73	68
	4	45	44
	5	6	1
	6	35	-37
	7	18	-18
	8	59	56
6, 3	-10	22	-25
	-9	28	-23
	-8	14	15
	-7	28	24
	-6	39	36
	-5	65	-70
	-4	49	-53
	-3	119	125
	-2	46	-47
	-1	65	-63
	-0	20	17
	1	49	48
	2	64	63
	3	45	-46
	4	88	-88
	5	0	1
	6	75	73
	7	0	5
	8	28	-30
6, 4	-10	17	-18
	-9	22	27
	-8	33	31
	-7	48	-48
	-6	71	-71
	-5	18	22
	-4	135	139
	-3	11	7
	-2	133	-137
	-1	19	18
	-0	78	77
	1	101	-90
	2	121	-119
	3	27	31
	4	52	52
	5	23	-19
	6	66	-70
	7	0	-4
	8	88	85
6, 5	-9	68	-63
	-8	25	29
	-7	58	54
	-6	6	2
	-5	58	-59
	-4	25	-27
	-3	122	128
	-2	7	2
	-1	38	-37
	-0	39	-38
	1	0	0
	2	63	62
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11, 1	-8	30	28
	-7	0	4
	-6	25	-27
	-5	10	15
	-4	21	-24
	-3	29	-28
	-2	15	14
	-1	30	35
	-0	20	-10
	1	18	-16
	2	24	-21
	3	0	9
	4	52	48
	5	0	-1
11, 2	-8	8	5
	-7	50	49
	-6	14	-16
	-5	12	-12
	-4	18	18
	-3	0	11
	-2	11	-13
	-1	66	-69
	-0	36	27
	1	49	53
	2	38	-36
	3	20	-24
	4	18	19
	5	44	45
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	-7	18	18
	-6	0	6
	-5	0	-8
	-4	10	8
	-3	46	-49
	-2	56	-55
	-1	44	40
	-0	46	38
	1	21	-23
	2	18	-20
	3	0	8
	4	31	28
	5	20	-25
11, 4	-8	0	5
	-7	34	31
	-6	10	-9
	-5	76	-79
	-4	30	32
	-3	51	48
	-2	9	-6
	-1	65	-68
	-0	15	3
	1	51	52
	2	24	-23
	3	37	-38
	4	11	12
	5	51	48
11, 5	-7	13	16
	-6	0	5
	-5	20	-25
	-4	34	36
	-3	0	2
	-2	90	-94
	-1	24	25
	-0	83	82
	1	12	13
	2	26	-28
	3	0	12
	4	47	47
	5	8	-11
11, 6	-7	69	64
	-6	0	1
	-5	81	-82
	-4	20	-21
	-3	73	75
	-2	24	23
	-1	64	-62
	-0	17	-9
	1	32	28
	2	35	37
	3	22	-22
	4	23	-22
11, 7	-6	0	-3
	-5	39	-38
	-4	24	25
	-3	7	5
	-2	60	-60
	-1	19	-19
	-0	55	52
	1	29	28
	2	42	-43
	3	21	-21
	4	66	67
11, 8	-6	50	51
	-5	42	-44
	-4	66	-64
	-3	28	30
	-2	43	42
	-1	57	-60
	-0	32	-29
	1	30	28
	2	21	20
	3	13	-14
11, 9	-5	26	-29
	-4	13	11
	-3	37	41
	-2	35	-34
	-1	42	-42
	-0	21	15
	1	48	51
	2	22	-22
11,10	-4	66	-69
	-3	8	-7
	-2	69	66
	-1	17	-17
	-0	22	-16
	1	32	30
11,11	-2	0	-7
	-1	54	-55
12, 0	-6	54	-53
	-4	53	50
	-2	38	-35
	-0	27	31
	2	68	-76
	4	80	82
12, 1	-7	76	74
	-6	29	28
	-5	34	-33
	-4	19	-21
	-3	26	28
	-2	17	12
	-1	66	-68
	-0	0	-0
	1	27	23
	2	24	-26
	3	29	-29
	4	17	13
12, 2	-7	29	30
	-6	25	-27
	-5	44	-51
	-4	48	51
	-3	26	24
	-2	57	-62
	-1	32	-34
	-0	40	37
	1	50	50
	2	41	-43
	3	40	-46
	4	61	62
12, 3	-7	51	-46
	-6	32	36
	-5	24	-25
	-4	41	-43
	-3	30	22
	-2	43	49
	-1	53	-49
	-0	17	-9
	1	43	41
	2	0	7
	3	25	-25
	4	8	23
12, 4	-7	23	20
	-6	27	25
	-5	56	-55
	-4	7	6
	-3	44	43
	-2	54	-53
	-1	47	-47
	-0	34	30
	1	51	57
	2	13	-12
	3	41	-46
	4	16	11
12, 5	-6	23	24
	-5	25	-18
	-4	19	-21
	-3	42	44
	-2	31	33
	-1	61	-62
	-0	52	-49
	1	16	17
	2	45	40
	3	22	-21
12, 6	-6	39	39
	-5	26	-26
	-4	36	-42
	-3	27	30
	-2	0	-3
	-1	32	-35
	-0	19	-16
	1	0	8
	2	0	4
	3	13	-15
12, 7	-5	29	-31
	-4	25	-23
	-3	36	34
	-2	22	23
	-1	34	-34
	-0	31	-27
	1	0	1
	2	38	34
12, 8	-5	0	-1
	-4	19	-18
	-3	20	-18
	-2	18	21
	-1	13	15
	-0	20	-20
	1	0	1
	2	0	-0
12, 9	-3	23	22
	-2	34	33
	-1	23	-21
	-0	13	-12
13, 0	-5	56	-54
	-3	32	27
	-1	30	-35
	1	53	51
	3	90	-85
13, 1	-6	17	17
	-5	13	13
	-4	0	3
	-3	23	-28
	-2	7	12
	-1	31	30
	-0	30	-34
	1	7	-8
	2	8	-8
	3	13	9
13, 2	-6	20	23
	-5	32	-32
	-4	33	-34
	-3	48	50
	-2	7	12
	-1	56	-55
	-0	27	-25
	1	41	39
	2	47	42
	3	66	-65
13, 3	-6	11	13
	-5	32	42
	-4	13	11
	-3	33	-34
	-2	28	-32
	-1	36	37
	-0	35	37
	1	39	-41
	2	47	-46
	3	17	21
13, 4	-6	8	7
	-5	8	-9
	-4	13	-11
	-3	57	55
	-2	10	13
	-1	65	-66
	-0	43	-38
	1	0	-1
	2	50	45
13, 5	-5	0	1
	-4	26	28
	-3	13	-15
	-2	53	-54
	-1	7	6
	-0	55	52
	1	8	-15
	2	56	-58
13, 6	-5	14	-16
	-4	0	-10
	-3	50	49
	-2	20	20
	-1	28	-28
	-0	30	-28
	1	13	-17
13, 7	-4	30	32
	-3	13	14
	-2	33	-32
	-1	26	-25
	-0	22	26
14, 0	-4	47	50
	-2	23	-26
	-0	0	3
14, 1	-5	22	-23
	-4	0	-1
	-3	40	37
	-2	0	3
	-1	0	2
	-0	17	19
	1	8	-15
14, 2	-5	19	-19
	-4	32	28
	-3	25	26
	-2	17	-20
	-1	23	-24
	-0	11	7
	1	0	-3
14, 3	-4	0	-7
	-3	44	42
	-2	8	-12
	-1	30	-30
	-0	27	31
	1	21	19
14, 4	-4	8	-2
	-3	44	43
	-2	0	-0
	-1	39	-40
	-0	25	23
14, 5	-3	65	65
	-2	11	-12
	-1	59	-60

Determination of the Structure

A three-dimensional Patterson function was calculated from which the Ru–Ru, Ru–Cl, and Ru–As vectors were identified. After several series of least-squares refinements and adjustments of the structure, the R factor was 27%. A three-dimensional Fourier was then calculated from which all of the carbon atoms were located, as well as an additional molecule of water. All of the atoms are in general positions: 4(e): $\pm(x, y, z;\ {}^1/_2 - x,\ {}^1/_2 + y,\ {}^1/_2 - z)$.

The isotropic thermal parameters had the form exp $(-B\lambda^{-2} \sin^2 \theta)$. The anisotropic thermal parameters were introduced in the form $\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$, with $4\beta_{ij} = a^*_i a^*_j B_{ij}$, where a^*_i is the length of the ith reciprocal axis. The carbon atoms were assigned isotropic temperature factors while the Ru, As, Cl, and O atoms were given anisotropic temperature factors. With a full-matrix treatment of all 178 parameters, the R factor was reduced to 0.067. The final values of the parameters are listed in Table II. The unit cell is shown in Figure 1.

Discussion of the Structure.—Several structures involving tetraphenylarsonium and tetraphenylphosphonium cations have been reported: $(C_6H_5)_4AsI$,[8] $(C_6H_5)_4AsI_3$,[9] $(C_6H_5)_4AsFeCl_4$,[10] and $(C_6H_5)_4PI$.[11] In the first two examples, the point symmetry at the arsenic is $\bar{4}2m$, with the planes of the phenyl rings perpendicular to the mirror planes. In the second two examples, the point symmetry is only $\bar{4}2$. The phenyl rings in $(C_6H_5)_4AsFeCl_4$ point to the corners of a regular tetrahedron, but the planes of the rings are rotated 28°, destroying the mirror planes. In $(C_6H_5)_4PI$, the phenyl rings point to the corners of a distorted tetrahedron, forming angles about the phosphorus of 103 and 113°, and the planes are rotated 52° in this case. No simple description is possible for the configuration of the phenyl rings in the present example of the ruthenium salt. In this case, the arsenic is situated at a point

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[8] R. C. L. Mooney, *J. Am. Chem. Soc.*, **62**, 2955 (1940).

[9] R. C. L. Mooney-Slater, *Acta Cryst.*, **12**, 187 (1959).

[10] B. Zaslow and R. E. Rundle, *J. Phys. Chem.*, **61**, 490 (1957).

[11] T. L. Khostyanova and U. T. Struchkov, *Kristallografiya*, **1**, 669 (1956).

TABLE II

POSITIONAL AND THERMAL PARAMETERS IN $(C_6H_5)_4AsRuCl_4(H_2O)_2 \cdot H_2O$[a]

Atom	*x*	*y*	*z*	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ru	0.4872	0.5164	0.7585	3.28	3.72	3.32	0.11	0.41	0.00
As	0.4169	0.0785	0.7332	2.56	2.75	3.38	0.23	0.12	−0.01
Cl(1)	0.3829	0.5818	0.8566	3.96	5.36	4.29	0.66	0.84	−0.43
Cl(2)	0.3813	0.4884	0.5838	3.52	6.15	3.51	−0.26	0.09	−0.45
Cl(3)	0.5308	0.6382	0.6796	4.75	3.46	5.04	0.14	0.89	0.39
Cl(4)	0.4614	0.3895	0.8391	5.75	4.02	4.39	−0.55	1.05	0.61
O(1)	0.5865	0.5358	0.9173	3.89	5.89	3.41	−0.20	0.40	0.04
O(2)	0.5884	0.4623	0.6748	3.24	4.46	3.74	0.72	0.40	−0.02
O(3)	0.0442	0.1822	0.0983	12.4	4.47	7.63	−0.66	0.01	0.17
C(I1)	0.3493	0.1309	0.5926	3.0[b]					
C(I2)	0.3189	0.0852	0.4865	4.9					
C(I3)	0.2656	0.1249	0.3866	5.9					
C(I4)	0.2489	0.2063	0.3914	6.3					
C(I5)	0.2840	0.2497	0.4922	7.3					
C(I6)	0.3338	0.2124	0.5995	5.6					
C(II1)	0.3914	0.1309	0.8793	3.6					
C(II2)	0.3130	0.1121	0.9194	5.3					
C(II3)	0.2920	0.1577	1.0226	6.2					
C(II4)	0.3530	0.2161	1.0785	5.2					
C(II5)	0.4301	0.2313	1.0359	5.1					
C(II6)	0.4502	0.1898	0.9339	4.6					
C(III1)	0.5421	0.0904	0.7312	3.6					
C(III2)	0.6046	0.0611	0.8340	5.0					
C(III3)	0.6969	0.0656	0.8253	5.4					
C(III4)	0.7249	0.0968	0.7267	6.0					
C(III5)	0.6651	0.1247	0.6252	5.4					
C(III6)	0.5707	0.1204	0.6283	4.3					
C(IV1)	0.3893	0.9670	0.7260	3.3					
C(IV2)	0.3014	−0.0579	0.7264	5.0					
C(IV3)	0.2828	−0.1419	0.7187	6.2					
C(IV4)	0.3528	−0.1957	0.7106	6.1					
C(IV5)	0.4380	−0.1698	0.7054	5.8					
C(IV6)	0.4599	−0.0871	0.7150	5.0					

[a] Standard deviations of the positional parameters of Ru and As are 0.0001 or less; Cl, 0.0003 or less; and O and C, 0.001 or less. Standard deviations of the thermal parameters are 0.05 or less for Ru and As, 0.2 or less for Cl, 0.4 or less for O and C, except for O(3) which was 0.8 or less. [b] Only isotropic temperature factors were used for the carbon atoms.

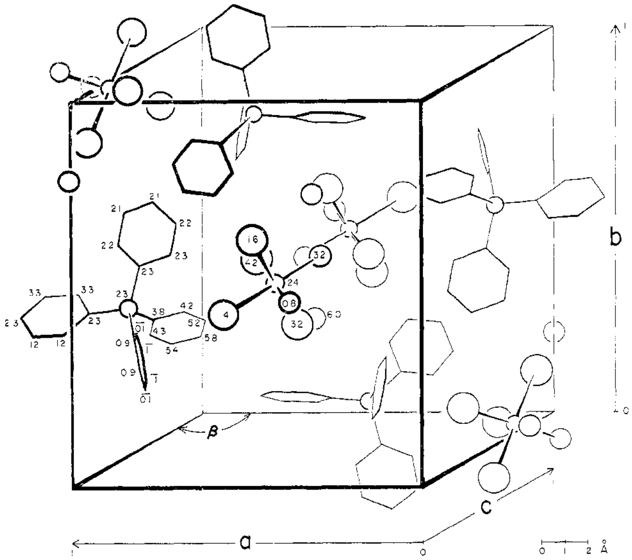

Figure 1.—Unit cell of $(C_6H_5)_4AsRuCl_4(H_2O)_2 \cdot H_2O$. Numbers are coordinates along the *c* axis.

of symmetry 1, and the phenyl groups have taken correspondingly free positions around the arsenic. The details of the configuration are given in Table III, where I, II, III, and IV refer to the four phenyl rings.

TABLE III

CONFIGURATION OF TETRAPHENYLARSONIUM ION IN $(C_6H_5)_4AsRuCl_4(H_2O)_2 \cdot H_2O$

Bond angles around arsenic, deg (±1)		Dihedral angles between planes of phenyl rings, deg (±3)	
C(I1)-As-C(II1)	109	I-II	70
C(I1)-As-C(III1)	108	I-III	89
C(I1)-As-C(IV1)	110	I-IV	70
C(II1)-As-C(III1)	107	II-III	36
C(II1)-As-C(IV1)	114	II-IV	70

Since the carbon atoms in the phenyl rings were not restrained in the least-squares refinement, the individual rings departed slightly from 6/mm symmetry. The average of the 24 C–C bond lengths is 1.396 A, with a spread in bond lengths of 1.35 to 1.45 A and a standard deviation of about 0.03 A. This bond length does not differ significantly from the benzene C–C bond of 1.397 A. The C(1)–C(4) diameters of all four rings, pointing to the central arsenic, are shorter by about 0.02 A compared to the average diameter of 2.79 A. This apparent shortening may be the result of torsional thermal motion.

The octahedral environment of the ruthenium is detailed in Table IV. The most interesting aspect of the configuration is the *cis* arrangement of the two water molecules in the octahedron. The average

TABLE IV

DISTANCES AND ANGLES IN $(C_6H_5)_4AsRuCl_4(H_2O)_2 \cdot H_2O$

Atom 1–atom 2	Bond, A	Bond angles	Deg (±1)
Ru–O(1)	2.12 ± 0.01	Cl(1)–Ru–O(2)	177
Ru–O(2)	2.11 ± 0.01	Cl(2)–Ru–O(1)	178
Ru–O(3)	3.92 ± 0.01	Cl(2)–Ru–O(2)	90
Ru–Cl(1)	2.33 ± 0.01	Cl(2)–Ru–Cl(1)	93
Ru–Cl(2)	2.32 ± 0.01	Cl(2)–Ru–Cl(3)	94
Ru–Cl(3)	2.35 ± 0.01	Cl(2)–Ru–Cl(4)	90
Ru–Cl(4)	2.36 ± 0.01		
As–C(1)	1.91 ± 0.01 (av of 4)		
C–C	1.396 ± 0.03 (av of 24)		

Ru–Cl and Ru–O distances of 2.34 and 2.12 A are in agreement with the values we have found for the cesium salt[2a] and those reported by Khodashova for the potassium salt.[12] The Ru–Cl bonds opposite the oxygens in the octahedron were again found to be several hundreths of an angstrom shorter than those for which the opposite atoms were both chlorines.

The possibilities for hydrogen bonding are shown in Figure 2. The water of crystallization, designated O(3) in the figure, is probably involved in two hydrogen bonds: to water molecule O(2) and to chlorine Cl(3). The distances of 2.61 and 3.16 A and the angle of 97° are acceptable evidence for bonding. In the case of the two water molecules in the octahedron surrounding the ruthenium, reasonable hydrogen bonds can only be postulated for one of the molecules, O(2). In addition to the bond to O(3), it appears to be bonded to chlorine, Cl(2), at a distance of 3.07 A, giving an angle of about 91° between the bonds. The other water molecule in the octahedron, O(1), is not too distant from chlorines Cl(1) and Cl(4) of the neighboring octahedron, but the acute 66° angle is too small to indicate a pair of hydrogen bonds.

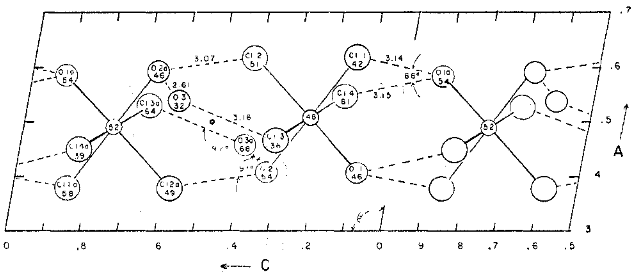

Figure 2.—Possibilities for hydrogen bonding in $(C_6H_5)_4AsRuCl_4$-$(H_2O)_2 \cdot H_2O$.

Water molecule O(3) appears to be vibrating quite anisotropically, with $B_{11} \sim 12$, $B_{22} \sim 4$, $B_{33} \sim 8$. This would indicate that most of the vibration is occurring in the *ac* plane, or, alternatively, some disorder concerning the location of the oxygen atom.

It is interesting to note that the structure of this complex salt can be visualized as a series of alternating layers of ruthenium octahedra and tetraphenylarsonium ions. This is shown in Figure 1, where the alternating planes run roughly parallel to the (110) planes.

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[12] T. S. Khodashova, *Zh. Strukt. Khim.*, **1**, 333 (1960).

CONTRIBUTION FROM THE LAWRENCE RADIATION LABORATORY AND DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF CALIFORNIA, BERKELEY, CALIFORNIA

The Crystal Structure of Cesium Aquopentachlororuthenate[1]

BY TED E. HOPKINS, ALLAN ZALKIN, DAVID H. TEMPLETON, AND MARTYN G. ADAMSON

Received March 14, 1966

X-Ray diffraction study of a single crystal of $Cs_2RuCl_5H_2O$ showed that it is orthorhombic with $a = 7.986$ A, $b = 17.289$ A, and $c = 7.400$ A, $Z = 4$, $d_x = 3.65$ g cm^{-3}. The space group is Amam. The ruthenium is surrounded by an octahedron composed of the five chlorine atoms and one water molecule. The Ru–O and average Ru–Cl distances are 2.10 and 2.34 A.

Introduction

Aqueous ruthenium species have been studied extensively by Connick, *et al.*, with particular emphasis on ruthenium(III).[2] In the course of their work, a number of aquochlororuthenates with various cations have been prepared, and it seemed of interest to determine the crystal structures of some of them. Aquopentachlororuthenates with cations of potassium, rubidium, and cesium have been obtained. As far as we know, the crystal structure of only the potassium salt has been reported.[3] None of the salts in this series is isostructural with either of the others. This paper is a report on the cesium salt.

Experimental Section

Small well-shaped crystals of $Cs_2RuCl_5H_2O$ were obtained by adding sufficient 1 *M* cesium chloride solution to a ruthenium(III) solution in hydrochloric acid to give the following concentrations: $[Ru(III)] = 0.05$ *M*, $[Cs^+] = 0.25$ *M*, and $[Cl^-] \cong 2$ *M*. Under these conditions, the chloride ion concentration is low enough to assure that crystal growth is slow. After standing at 0° for 48 hr, crystals were separated from the mother liquor on a Buchner funnel, quickly washed twice with ice-cold water,

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[1] Work done under the auspices of the U. S. Atomic Energy Commission.

[2] H. H. Cady and R. E. Connick, *J. Am. Chem. Soc.*, **80**, 2646 (1958); R. E. Connick and D. A. Fine, *ibid.*, **83**, 3414 (1961).

[3] T. S. Khodashova, *Zh. Strucht. Khim.*, **1**, 333 (1960).