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THE CRYSTAL STRUCTURE OF CESIUM AQUOPENTACHLORORUTHENATE

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Submitted to Inorganic Chemistry

UCRL-16759

UNIVERSITY OF CALIFORNIA

Lawrence Radiation Laboratory
Berkeley, California

AEC Contract No. W-7405-eng-48

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March 1966

UCRL-16759

CONTRIBUTION FROM THE LAWRENCE RADIATION LABORATORY AND DEPARTMENT OF CHEMISTRY,
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The Crystal Structure of Cesium Aquopentachlororuthenate [1]

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

X-ray diffraction study of a single crystal of $Cs_2RuCl_5H_2O$ showed that it is orthorhombic with a = 7.986 Å, b = 17.289 Å, and c = 7.400 Å, 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 Å and 2.34 Å.

(1)* Work done under the auspices of the U. S. Atomic Energy Commission.

INTRODUCTION

Aqueous ruthenium species have been studied extensively by Connick, et al, [1], 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

Small well-shaped crystals of $Cs_2RuCl_5H_2O$ were obtained by adding sufficient 1M 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°C for 48 hours, crystals were separated from the mother liquor on a Buchner funnel, quickly washed twice with ice-cold water and partially dried by suction. Final traces of moisture were removed by heating at 105°C for 30 minutes. Analyses for Ru, Cl and H_2O confirmed the formula $Cs_2RuCl_5H_2O$.

The preliminary determination of the space group and cell dimensions was made using the Weissenberg technique and copper radiation. A single crystal in the form of a prism 0.14 x 0.021 x 0.02 mm was used. The sides

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

(3) T. S. Khodashova, Zhur. Struckt. Khim., 1, 333 (1960).

parallel to the long axis comprised the form {120}. The measured dihedral angle was ~ 85°, while the calculated angle is 85°28'. The crystal was mounted with the c - axis as the axis of rotation in phi. The cell dimensions and intensities were measured with a General Electric XRD-5 goniostat equipped with a scintillation counter, using MoKα radiation (λ = 0.70926 Å for $K\alpha_1$).

There are 525 independent reflections permitted by the space group in the sphere of reflection with $\sin\theta/\lambda < 0.596$ ($2\theta < 50°$). These were measured with counting times of 10 seconds each and 95 were recorded as zero intensity. No correction was made for either absorption or extinction. The calculated linear absorption coefficient is $\mu = 100.0\ cm^{-1}$ for molybdenum radiation. For the crystal used, μR was less than 0.7.

Calculations were made using an IBM 7044 computer and our version (unpublished) of the Gantzel-Sparks-Trueblood full-matrix least squares program which minimizes $\sum w(F_o - |F_c|)^2 / (\sum wF_o^2)$, where F_o and F_c are the observed and calculated structure factors, and the weights w, were all taken as unity. The Fourier and distance programs were written by Zalkin. The atomic scattering factors were chosen as follows: Cs^+, Ru^{+++} from Thomas and Umeda [4], Cl^-, O values taken from the International Tables [5]. Corrections for anomalous dispersion ($\Delta f'$) were made for cesium, ruthenium and chlorine by adding -0.5, -1.3 and +0.1, respectively to their atomic scattering functions.

UNIT CELL AND SPACE GROUP

The A-centered cell contains four formula units of $Cs_2RuCl_5H_2O$ and is orthorhombic with dimensions:

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(4) L. H. Thomas, and K. Umeda, J. Chem. Phys., 26, 293 (1957).

(5) "International Tables for X-ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962.

$\underline{a}$ = 7.986 ± 0.005, $\underline{b}$ = 17.289 ± 0.008, $\underline{c}$ = 7.400 ± 0.004 Å.

The calculated density is 3.65 g cm^{-3}. The crystals sank in methylene iodide (density 3.3 g cm^{-3}).

The Weissenberg photographs showed the following reflection restrictions: $\underline{hk\ell}$: $\underline{k} + \underline{\ell} = 2\underline{n}$, $\underline{h0\ell}$: $\underline{h} = 2\underline{n}$. Measurements with the goniostat confirmed these observations. The axes were chosen according to the c < a < b convention. The extinctions correspond to the space groups $\underline{A2am}$, $\underline{Ama2}$ and $\underline{Amam}$, for the particular choice of axes for the crystal. The success of the structure determination confirmed the choice of the centro-symetric space group $\underline{Amam}$. The observed and calculated structure factor magnitudes are given in Table I.

DETERMINATION OF THE STRUCTURE

A three dimensional Patterson function was calculated from the observed intensities after correction for Lorentz and polarization effects. The cesium-ruthenium and ruthenium chlorine vectors were identified and coordinates for the trial structure were chosen.

Except for one set of chlorines, all of the atoms are in special positions:

4(c): (1/4, $\underline{y}$, 0; 3/4, -$\underline{y}$, 0) + (0, 0, 0; 0, 1/2, 1/2).

A set of four chlorines are in the general positions:

16(h): ± ($\underline{x}$, $\underline{y}$, $\underline{z}$; -$\underline{x}$, -$\underline{y}$, $\underline{z}$; 1/2 - $\underline{x}$, $\underline{y}$, $\underline{z}$; 1/2 + $\underline{x}$, -$\underline{y}$, $\underline{z}$)
+ (0, 0, 0; 0, 1/2, 1/2).

Three cycles of least squares refinement resulted in an $\underline{R}$ value of 0.09 with anisotropic temperature factors for cesium and ruthenium and isotropic temperature factors for the chlorines and oxygen. A three dimensional Fourier was run on the basis of the refined structure. This did not

Table I. Observed and Calculated Structure Factors (X 1.0) for $Cs_2RuCl_5H_2O$.

H,K= 0, 0
L FOB FCA
2 186 190
4 237 255
6 110 111
8 85 83

H,K= 0, 1
L FOB FCA
1 3 -8
3 44 -50
5 22 20
7 28 -25

H,K= 0, 2
L FOB FCA
0 10 7
2 30 -30
4 0 -1
6 12 -10
8 12 -5

H,K= 0, 3
L FOB FCA
1 125-128
3 68 -74
5 78 -83
7 25 -28

H,K= 0, 4
L FOB FCA
0 32 27
2 199 200
4 32 33
6 62 62
8 17 18

H,K= 0, 5
L FOB FCA
1 131-110
3 42 -43
5 82 -89
7 15 -12

H,K= 0, 6
L FOB FCA
0 135-132
2 37 -37
4 76 -75
6 33 -32
8 24 -26

H,K= 0, 7
L FOB FCA
1 0 -4
3 6 -8
5 7 1
7 12 -5

H,K= 0, 8
L FOB FCA
0 220 222
2 93 98
4 136 139
6 57 62
8 53 51

H,K= 0, 9
L FOB FCA
1 86 86
3 35 37
5 72 75
7 8 9

H,K= 0,10
L FOB FCA
0 8 -10
2 86 -89
4 9 -15
6 34 -34

H,K= 0,11
L FOB FCA
1 13 7
3 6 -2
5 7 7
7 0 -4

H,K= 0,12
L FOB FCA
0 22 -15
2 33 31
4 10 -9
6 8 10

H,K= 0,13
L FOB FCA
1 0 -1
3 21 20
5 15 -20

H,K= 0,14
L FOB FCA
0 148-148
2 70 -70
4 101-103
6 47 -50

H,K= 0,15
L FOB FCA
1 14 10
3 20 20
5 0 -2

H,K= 0,16
L FOB FCA
0 41 41
2 26 27
4 28 33

H,K= 0,17
L FOB FCA
1 70 70
3 48 50

H,K= 0,18
L FOB FCA
0 22 -8
2 54 -54
4 9 -9

H,K= 0,19
L FOB FCA
1 20 21
3 0 7

H,K= 0,20
L FOB FCA
0 16 10
2 9 3

H,K= 1, 1
L FOB FCA
1 15 -3
3 9 6
5 9 -4
7 0 2

H,K= 1, 2
L FOB FCA
0 75 -67
2 14 -9
4 43 -40
6 17 -15
8 16 -19

H,K= 1, 3
L FOB FCA
1 178-173
3 131-130
5 103-103
7 57 -56

H,K= 1, 4
L FOB FCA
0 31 29
2 41 39
4 20 18
6 15 14
8 8 5

H,K= 1, 5
L FOB FCA
1 33 28
3 26 23
5 20 22
7 8 12

H,K= 1, 6
L FOB FCA
0 163 164
2 108 113
4 105 106
6 58 60
8 47 43

H,K= 1, 7
L FOB FCA
1 55 -60
3 53 -56
5 30 -26
7 22 -22

H,K= 1, 8
L FOB FCA
0 99 98
2 68 69
4 67 65
6 36 37
8 27 25

H,K= 1, 9
L FOB FCA
1 21 -26
3 19 -22
5 16 -15
7 8 -10

H,K= 1,10
L FOB FCA
0 18 19
2 33 33
4 9 11
6 11 12

H,K= 1,11
L FOB FCA
1 127-130
3 104-105
5 89 -85
7 51 -49

H,K= 1,12
L FOB FCA
0 9 -10
2 9 8
4 8 -8
6 0 -1

H,K= 1,13
L FOB FCA
1 3 1
3 3 3
5 2 -1

H,K= 1,14
L FOB FCA
0 61 63
2 53 52
4 45 47
6 32 30

H,K= 1,15
L FOB FCA
1 0 -2
3 0 -4
5 0 4

H,K= 1,16
L FOB FCA
0 31 33
2 17 17
4 28 25

H,K= 1,17
L FOB FCA
1 30 24
3 23 18

H,K= 1,18
L FOB FCA
0 0 -4
2 0 -4
4 9 -4

H,K= 1,19
L FOB FCA
1 31 -30
3 24 -25

H,K= 1,20
L FOB FCA
0 42 -40

H,K= 2, 0
L FOB FCA
0 209-198
2 332-338
4 146-149
6 142-137
8 73 -72

H,K= 2, 1
L FOB FCA
1 34 35
3 14 -11
5 39 34
7 15 -16

H,K= 2, 2
L FOB FCA
0 30 27
2 14 -4
4 14 13
6 9 4
8 10 4

H,K= 2, 3
L FOB FCA
1 98 98
3 103 102
5 43 46
7 52 51

H,K= 2, 4
L FOB FCA
0 207-199
2 39 -37
4 108-102
6 30 -27
8 24 -22

H,K= 2, 5
L FOB FCA
1 72 73
3 99 100
5 23 24
7 61 57

H,K= 2, 6
L FOB FCA
0 39 37
2 94 95
4 37 36
6 43 41
8 20 24

H,K= 2, 7
L FOB FCA
1 5 6
3 0 1
5 0 6
7 0 -3

H,K= 2, 8
L FOB FCA
0 101-101
2 173-171
4 77 -79
6 84 -81

H,K= 2, 9
L FOB FCA
1 55 -60
3 81 -81
5 17 -19
7 51 -49

H,K= 2,10
L FOB FCA
0 88 87
2 16 14
4 51 53
6 16 15

H,K= 2,11
L FOB FCA
1 0 -3
3 7 -8
5 0 3
7 0 -4

H,K= 2,12
L FOB FCA
0 29 -28
2 9 11
4 16 -16
6 8 5

H,K= 2,13
L FOB FCA
1 11 -10
3 12 13
5 11 -15

H,K= 2,14
L FOB FCA
0 71 72
2 121 123
4 61 61
6 64 62

H,K= 2,15
L FOB FCA
1 16 -17
3 0 -3
5 20 -16

H,K= 2,16
L FOB FCA
0 28 -28
2 34 -35
4 23 -22

H,K= 2,18
L FOB FCA
0 56 53
2 8 10

H,K= 2,19
L FOB FCA
1 14 -14

H,K= 2,20
L FOB FCA
0 0 -2

H,K= 3, 1
L FOB FCA
1 9 -4
3 15 13
5 9 -11
7 13 13

H,K= 3, 2
L FOB FCA
0 29 -28
2 80 78
4 0 -4
6 35 35
8 8 9

H,K= 3, 3
L FOB FCA
1 136 132
3 131 131
5 65 67
7 69 68

H,K= 3, 4
L FOB FCA
0 46 -48
2 13 -11
4 29 -28
6 0 -7
8 0 -7

H,K= 3, 5
L FOB FCA
1 22 -20
3 26 -25
5 10 -11
7 19 -17

H,K= 3, 6
L FOB FCA
0 83 -79
2 143-144
4 62 -61
6 74 -70

H,K= 3, 7
L FOB FCA
1 53 56
3 26 26
5 41 42
7 0 -1

H,K= 3, 8
L FOB FCA
0 50 -49
2 89 -89
4 40 -38
6 45 -43

H,K= 3, 9
L FOB FCA
1 20 25
3 19 19
5 16 15
7 8 9

H,K= 3,10
L FOB FCA
0 46 -45
2 0 -1
4 30 -28
6 0 2

H,K= 3,11
L FOB FCA
1 107 106
3 105 105
5 59 58

H,K= 3,12
L FOB FCA
0 25 -24
2 23 22
4 13 -12
6 12 11

H,K= 3,13
L FOB FCA
1 0 -2
3 0 4
5 0 -5

H,K= 3,14
L FOB FCA
0 45 -43
2 58 -56
4 33 -33

H,K= 3,15
L FOB FCA
1 0 3
3 0 -7
5 0 5

H,K= 3,16
L FOB FCA
0 0 -4
2 40 -39
4 12 -7

H,K= 3,17
L FOB FCA
1 19 -18
3 25 -22

H,K= 3,18
L FOB FCA
0 8 4
2 0 5

H,K= 3,19
L FOB FCA
1 27 25

H,K= 4, 0
L FOB FCA
0 256 255
2 160 163
4 167 163
6 98 96

H,K= 4, 1
L FOB FCA
1 10 -8
3 25 -26
5 0 9
7 11 -13

H,K= 4, 2
L FOB FCA
0 7 6
2 8 -6
4 0 1
6 0 -3

H,K= 4, 3
L FOB FCA
1 88 -86
3 62 -60
5 59 -60
7 26 -26

H,K= 4, 4
L FOB FCA
0 43 45
2 98 96
4 38 36
6 32 33

H,K= 4, 5
L FOB FCA
1 77 -76
3 43 -43
5 63 -62
7 12 -18

H,K= 4, 6
L FOB FCA
0 69 -69
2 38 -35
4 43 -46
6 24 -27

H,K= 4, 7
L FOB FCA
1 0 -2
3 0 -5
5 0 1
7 0 -2

H,K= 4, 8
L FOB FCA
0 135 134
2 84 86
4 96 92
6 56 55

H,K= 4, 9
L FOB FCA
1 64 62
3 36 34
5 53 52

H,K= 4,10
L FOB FCA
0 16 -18
2 49 -49
4 18 -17
6 18 -18

H,K= 4,11
L FOB FCA
1 0 5
3 0 0
5 0 5

H,K= 4,12
L FOB FCA
0 10 -7
2 11 10
4 8 -5
6 0 1

H,K= 4,13
L FOB FCA
1 0 -2
3 11 9
5 12 -13

H,K= 4,14
L FOB FCA
0 101-100
2 66 -65
4 73 -74

H,K= 4,15
L FOB FCA
1 0 9
3 14 15

H,K= 4,16
L FOB FCA
0 30 28
2 23 23
4 24 23

H,K= 4,17
L FOB FCA
1 60 55
3 46 42

H,K= 4,18
L FOB FCA
0 8 -12
2 30 -31

H,K= 5, 1
L FOB FCA
1 8 -10
3 0 8
5 18 -20
7 8 7

H,K= 5, 2
L FOB FCA
0 77 -77
2 23 21
4 53 -50
6 8 -2

H,K= 5, 3
L FOB FCA
1 107-107
3 71 -68
5 85 -83
7 24 -26

H,K= 5, 4
L FOB FCA
0 0 5
2 33 33
4 0 5
6 11 13

H,K= 5, 5
L FOB FCA
1 15 17
3 0 8
5 21 18
7 0 3

H,K= 5, 6
L FOB FCA
0 132 129
2 47 48
4 93 89
6 36 34

H,K= 5, 7
L FOB FCA
1 27 -26
3 43 -43
5 0 3

H,K= 5, 8
L FOB FCA
0 79 83
2 32 30
4 57 56
6 20 22

H,K= 5, 9
L FOB FCA
1 22 -21
3 19 -19
5 14 -13

H,K= 5,10
L FOB FCA
0 28 -8
2 35 34
4 0 -6
6 15 12

H,K= 5,11
L FOB FCA
1 89 -89
3 61 -60
5 75 -71

H,K= 5,12
L FOB FCA
0 25 -27
2 21 22
4 16 -18

H,K= 5,13
L FOB FCA
1 0 -4
3 0 3
5 9 -7

H,K= 5,14
L FOB FCA
0 53 50
2 32 30
4 40 38

H,K= 5,15
L FOB FCA
1 8 6
3 0 -4

H,K= 5,16
L FOB FCA
0 41 41
2 0 0

H,K= 5,17
L FOB FCA
1 0 17

H,K= 6, 0
L FOB FCA
0 137-138
2 133-133
4 108-106
6 69 -67

H,K= 6, 1
L FOB FCA
1 9 12
3 0 2
5 0 8

H,K= 6, 2
L FOB FCA
0 6 -4
2 0 -5
4 0 -3
6 0 0

H,K= 6, 3
L FOB FCA
1 57 57
3 58 53
5 36 35

H,K= 6, 4
L FOB FCA
0 50 -49
2 41 -40
4 28 -28
6 23 -23

H,K= 6, 5
L FOB FCA
1 49 49
3 46 51
5 25 29

H,K= 6, 6
L FOB FCA
0 30 31
2 31 33
4 26 27
6 17 17

H,K= 6, 7
L FOB FCA
1 0 2
3 0 -0
5 0 1

H,K= 6, 8
L FOB FCA
0 72 -76
2 72 -73
4 62 -60

H,K= 6, 9
L FOB FCA
1 37 -39
3 43 -42
5 20 -23

H,K= 6,10
L FOB FCA
0 21 24
2 19 18
4 11 14

H,K= 6,11
L FOB FCA
1 0 -3
3 0 -4

H,K= 6,12
L FOB FCA
0 12 2
2 0 3
4 8 4

H,K= 6,13
L FOB FCA
1 0 0
3 0 6

H,K= 6,14
L FOB FCA
0 58 59
2 55 58

H,K= 6,15
L FOB FCA
1 12 -10

H,K= 7, 1
L FOB FCA
1 0 4
3 18 18
5 8 -5

H,K= 7, 2
L FOB FCA
0 14 -16
2 50 50
4 0 -4

H,K= 7, 3
L FOB FCA
1 60 60
3 72 71
5 31 27

H,K= 7, 4
L FOB FCA
0 27 -26
2 0 -2
4 16 -16

H,K= 7, 5
L FOB FCA
1 0 -6
3 0 -12
5 0 -1

H,K= 7, 6
L FOB FCA
0 39 -34
2 75 -82
4 32 -31

H,K= 7, 7
L FOB FCA
1 21 23
3 0 -1
5 27 25

H,K= 7, 8
L FOB FCA
0 0 -21
2 55 -54
4 20 -20

H,K= 7, 9
L FOB FCA
1 16 18
3 12 15

H,K= 7,10
L FOB FCA
0 25 -25
2 0 8
4 17 -14

H,K= 7,11
L FOB FCA
1 51 54
3 62 62

H,K= 7,12
L FOB FCA
0 18 -19
0 16 -19

H,K= 7,13
L FOB FCA
1 8 2

H,K= 8, 0
L FOB FCA
0 82 77
2 98 95
4 54 56

H,K= 8, 1
L FOB FCA
1 0 -4
3 8 -2

H,K= 8, 2
L FOB FCA
0 0 5
2 0 8
4 0 3

H,K= 8, 3
L FOB FCA
1 40 -39
3 34 -34

H,K= 8, 4
L FOB FCA
0 33 34
2 11 10
4 21 25

H,K= 8, 5
L FOB FCA
1 37 -35
3 29 -32

H,K= 8, 6
L FOB FCA
0 0 -14
2 20 -23

H,K= 8, 7
L FOB FCA
1 0 0
3 0 1

H,K= 8, 8
L FOB FCA
0 42 42
2 50 54

H,K= 8, 9
L FOB FCA
1 29 29

H,K= 8,10
L FOB FCA
0 17 -16
2 0 -3

H,K= 9, 1
L FOB FCA
1 0 -12

H,K= 9, 2
L FOB FCA
0 31 -33
2 0 1

H,K= 9, 3
L FOB FCA
1 44 -43

H,K= 9, 4
L FOB FCA
0 0 2
2 12 12

H,K= 9, 5
L FOB FCA
1 0 5

H,K= 9, 6
L FOB FCA
0 55 54

show any unexpected features. Some reflections were then remeasured and all of the atoms were allowed anisotropic temperature factors. Several more cycles of least squares were run, and the final value for R was 0.069.

The anisotropic parameters were introduced in the form $\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}\ell^2 - 2\beta_{12}hk - 2\beta_{13}h\ell - 2\beta_{23}k\ell)$, with $4\beta_{ij} = a_i^* a_j^* B_{ij}$, where a_i^* is the length of the i th reciprocal axis. With this notation, the anisotropic thermal parameters B_{ij} are in units of ($Å^2$) which are used for isotropic thermal parameters B in the temperature factor of the form $\exp(-B\lambda^{-2}\sin^2\theta)$. The atoms in special positions lie on perpendicular mirror planes, thus requiring that the axes of the ellipsoids of thermal vibration be parallel to the reciprocal axes. Therefore, for these atoms, $B_{12} = B_{13} = B_{23} = 0$.

The atomic coordinates are listed in Table II, and the anisotropic temperature factors in Table III. An electron density difference function was calculated following the last cycle of least squares refinement. The largest peak corresponded to 0.79 electrons/$Å^3$.

Table II. Atomic coordinates from least squares refinement. Standard deviations are $\sigma(y) = 0.0001$ for Cs and Ru, 0.0004 for Cl(1) and 0.002 for O. For Cl(2), $\sigma(x) = \sigma(y) = 0.0006$. $Cs_2RuCl_5H_2O$.

Atom	x	y	z
Cs(1)	(0.2500)[a]	0.4715	(0.0)
Cs(2)	(0.2500)	0.7534	(0.0)
Ru	(0.2500)	0.1153	(0.0)
Cl(1)	(0.2500)	0.2490	(0.0)
Cl(2)	0.4605	0.1112	0.2222
O	(0.2500)	0.9936	(0.0)

(a) Parantheses indicate parameters which were subject to constraints.

Table III. Anisotropic thermal parameters for $Cs_2RuCl_5H_2O$.

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cs(1)	2.69	3.36	3.21	(0)[a]	(0)	(0)
Cs(2)	4.05	2.63	2.86	(0)	(0)	(0)
Ru	1.60	1.63	1.66	(0)	(0)	(0)
Cl(1)	3.54	1.26	2.90	(0)	(0)	(0)
Cl(2)	2.73	2.95	3.03	-0.13	-1.07	0.37
O	1.79	4.44	3.70	(0)	(0)	(0)

(a) Parentheses indicate parameters which were subject to constraints.

DESCRIPTION OF THE STRUCTURE

Figure 1 is a drawing of the unit cell in a clinographic projection. The octahedral environment of the ruthenium is shown in Figure 2. A list of the distances and angles including the estimated standard deviation is given in Tables IV and V.

Table IV. Distances in $Cs_2RuCl_5H_2O$.

Arom 1 to Atom 2	Distance (Å)	Atom 1 to Atom 2	Distance (Å)
Ru - O	2.104 ± 0.028	Cs(2) - 4 Cl(2)	3.619 ± 0.004
- Cl(1)	2.311 ± 0.008	- 2 Cl(1)	3.701 ± 0.002
- 4 Cl(2)	2.353 ± 0.004	- O	4.153 ± 0.028
- Cs(2)	4.404 ± 0.002	Cl(1) - 4 Cl(2)	3.347 ± 0.007
- Cs(1)	4.458 ± 0.003	- O	4.415 ± 0.028
Cs(1) - 4 Cl(2)	3.408 ± 0.004	Cl(2) - O	3.109 ± 0.018
- 4 Cl(2)	3.590 ± 0.004	- Cl(2)	3.289 ± 0.008
- 2 O	3.720 ± 0.003	- Cl(2)	3.363 ± 0.008
- Cl(1)	3.848 ± 0.008	O - 2 O	3.999 ± 0.004
- Cs(1)	4.113 ± 0.003		

Figure 1. Unit cell of $Cs_2RuCl_5H_2O$.

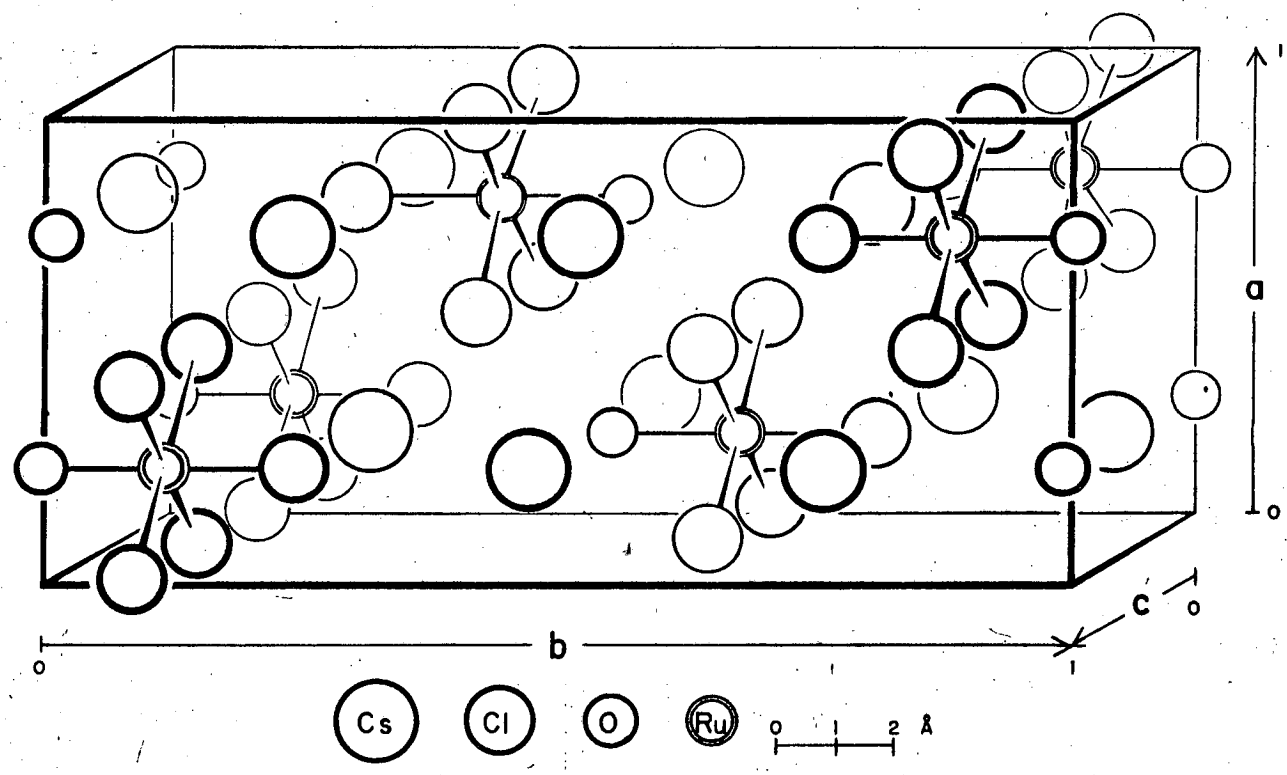

Figure 2. Ruthenium environment in $Cs_2RuCl_5H_2O$.

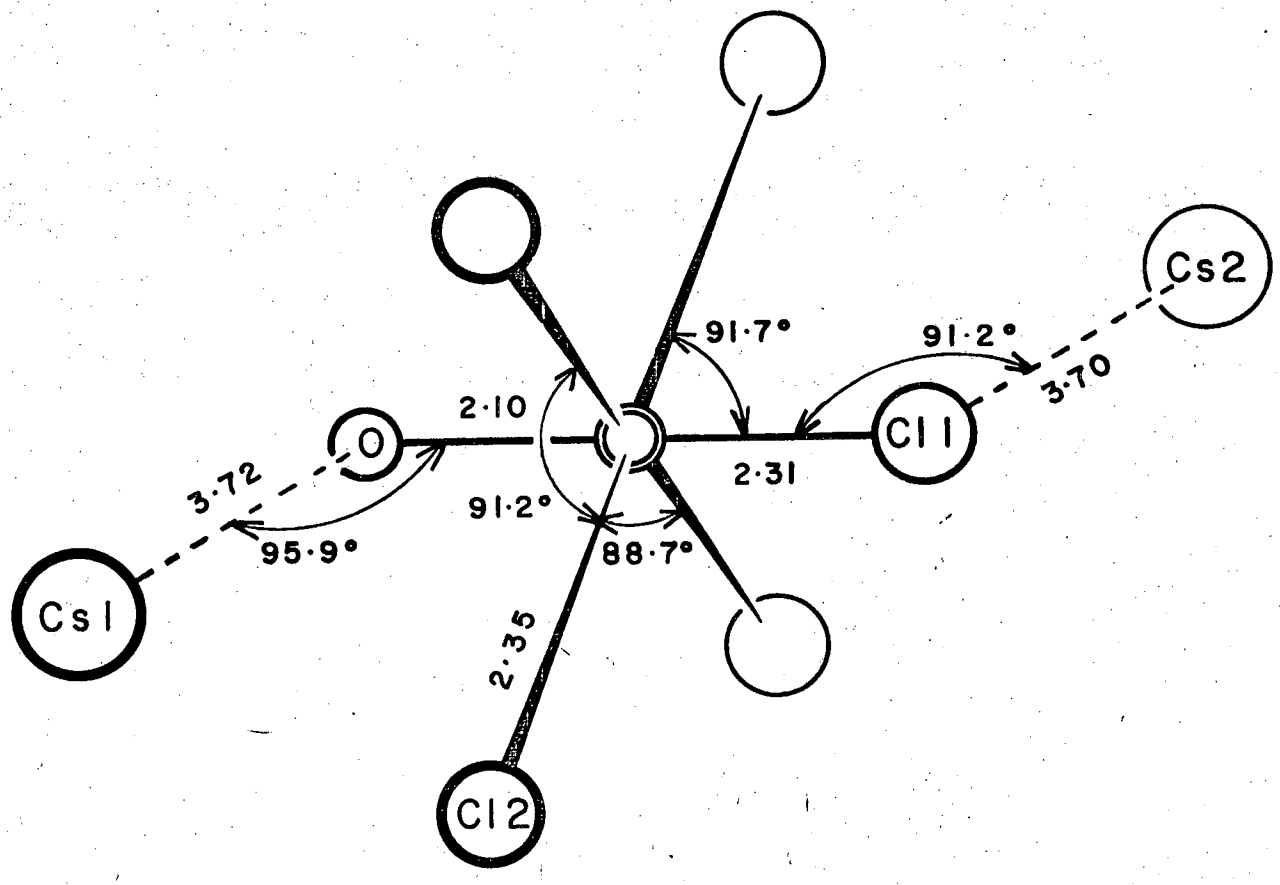

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Table V. Bond Angles in $Cs_2RuCl_5H_2O$.

Atom 1 - Atom 2 - Atom 3	Angle (degrees)
Cl(2) - Ru - O	88.3 ± 0.1
Cl(2) - Ru - Cl(2)	91.2 ± 0.2
Cl(2) - Ru - Cl(2)	88.7 ± 0.2
Cl(2) - Ru - Cl(1)	91.7 ± 0.1
Cs(1) - O - Cs(2)	84.1 ± 0.4
Cs(1) - O - Ru	95.9 ± 0.4

The ruthenium atom lies out of the plane of the four Cl(2) atoms a distance of 0.07 Å, and the resulting Cl(1) - Ru - Cl(2) angles differ from 90° by several times the estimated standard deviation. In addition, the Cl(2) atoms lie on the corners of a rectangle instead of a square. Thus, the point group at the ruthenium is 2mm instead of the more symmetrical group 4mm. In the potassium salt [3], the point group at the ruthenium is 1. The four chlorines do not lie in a plane, and the ruthenium lies on a line through two of the chlorines, rather than on the chlorine - oxygen line. The space group of the potassium salt is Pnma with Z = 4.

A review of Ru - Cl and Ru - O distances is given in the paper by Khodashova on the potassium salt. We may note here, however, that in the cesium salt the average Ru - Cl and Ru - O distances are the same as those found for the potassium salt, within the limits of the reported experimental errors: 2.10 ± 0.03 Å compared with 2.12 ± 0.05 Å, and 2.34 ± 0.005 Å compared with 2.35 ± 0.02 Å. In both cases, the Ru - Cl bond opposite the Ru - O bond was found to be shorter by about 0.05 Å compared to the remaining Ru - Cl bonds.

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