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The Crystal and Molecular Structure of Ruthenium-Sulfur Dioxide Coordination Compounds. I. Chlorotetraammine(sulfur dioxide)-ruthenium(II) Chloride

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The Ru-SO_2 complex, $[Ru^{II}(NH_3)_4(SO_2)Cl]Cl$, has an orthorhombic unit cell, a=13.962, b=9.308, c=7.312 Å. The space group is Pnam with four formula weights per unit cell. A three-dimensional crystal structure analysis of the complex yielded the positions of all of the atoms but the hydrogens, with a discrepancy factor of 0.047 for 1054 independent reflections. The SO_2 is a monodentate ligand, coordinated through the sulfur. The bond distances and bond angle in the coordinated SO_2 are approximately the same as in free, solid SO_2 and the Ru-N, Ru-S and Ru-Cl bond lengths are comparable to those observed in other platinum group complexes.

[redacted]

Introduction

The only metal complexes reported in the literature to contain sulfur dioxide as a ligand are those of the ruthenium-ammine series described by Gleu[2,3] and possibly the products of the reactions of iron carbonyls with

(2) K. Gleu and K. Rehm, Z. anorg. u. allgem. Chem., 227, 237 (1936).

(3) K. Gleu, W. Breuel and K. Rehm, Ibid., 235, 201,211 (1938).

SO_2.[4] Vaska[5] has prepared several platinum group complexes containing

(4) E. H. Braye and W. Hübel, Angew. Chem., 75, 345B (1963).

(5) L. Vaska, private communication, 1964.

SO_2 ligands as well as carbonyl and substituted phosphine ligands.

Since the ruthenium-ammines are the only well characterized SO_2 complexes, it would be informative to know more about the bonding involved, viz., (1) whether the SO_2 is coordinated through the sulfur or oxygen, (2) how the bond parameters and infrared frequencies of coordinated SO_2 compare with those of free SO_2, and (3) the nature of the N, Cl and S bonds to the ruthenium. A three-dimensional crystal structure analysis was performed on a representative comples, $[Ru^{II}(NH_3)_4(SO_2)Cl]Cl$. The nature of the bonding of the SO_2 ligand in several other Ru-SO_2 complexes is discussed elsewhere.[6]

(6) L. H. Vogt, Jr., J. L. Katz and S. E. Wiberley, Inorg. Chem., __, (1965).

[redacted]

Experimental

Preparation.—The Ru-SO_2 complex was prepared according to the procedures briefly outlined by Gleu,[2,3] with the exception of the $[Ru^{III}(NH_3)_6]Cl_3$ which was prepared by (and purchased from) Johnson, Matthey and Co., Ltd., Hatton Garden, London E.C. 1, England, using Lever's method.[7] A detailed account of the preparation of the complex

(7) F. M. Lever, in "International Conference on Co-ordination Chemistry," Spec. Publ. No. 13, The Chemical Society, Burlington House, London W.1, England.

follows.

$[Ru^{III}(NH_3)_6]Cl_3$, (7.0 g., 0.023 moles) was dissolved in 75 ml. water with warming and 75 ml. conc. HCl was added. The solution was refluxed for 3.5 hrs. During this time a yellow crystalline precipitate formed which was subsequently filtered off, washed first with 1:1 HCl, then with methanol, and dried under vacuum at room temperature. A yield of 6.42 g. (97%) of bright yellow crystals of $[Ru^{III}(NH_3)_5 Cl]Cl_2$ was obtained.

$[Ru^{III}(NH_3)_5 Cl]Cl_2$ (4.00 g., o.014 moles) was dissolved in 160 ml. water at 75-85°. To this solution was added 5.66 g. (0.056 moles) of solid $NaHSO_3$. Sulfur dioxide was slowly bubbled through the solution which was kept at 75° on a water bath. After about 15 min. at 75°, small, clear, faintly yellow crystals started to form. These conditions were maintained for 1 hr., after which the system was allowed to cool to room temperature but with continued saturation with SO_2. Thereafter, the crystals were filtered off, washed first with water, then with methanol, and dried under vacuum at room temperature. A yield of 2.98 g. (70%) of

$[Ru^{II}(NH_3)_4(HSO_3)_2]$ was obtained. This slightly soluble complex could not be recrystallized since it reacts in aqueous solution giving a pale blue color. Use of the SO_2, which was not mentioned in the literature, was found necessary in order to prevent the occurrence of side reactions which greatly reduced the yield of the desired complex. Presumably, the $NaHSO_3$ alone is too weak an acid in solution to prevent some OH^- from coordinating with the ruthenium and resulting in the formation of highly colored, insoluble products.

$[Ru^{II}(NH_3)_4(HSO_3)_2]$, (2.77 g., 0.001 moles) was dissolved in 325 ml. of 1:1 HCl by heating at the boiling point for about 15 min. The $[Ru^{II}(NH_3)_4(HSO_3)_2]$ turned a rust color when treated with the acid and went into solution slowly, producing a deep red solution. The solution was filtered hot, then reheated to redissolve any crystals that had formed and allowed to cool slowly overnight. Deep red-orange, needle shaped crystals of $[Ru^{II}(NH_3)_4(SO_2)Cl]Cl$ formed which were filtered off, washed first with 1:1 HCl, then with methanol, and dried under vacuum at room temperature.

Anal. Calcd. for $[Ru^{II}(NH_3)_4(SO_2)Cl]Cl$: Ru, 33.23; N, 18.41; H, 3.98; S, 10.54; Cl, 23.31; mol. wt., 304.16. Found: Ru, 33.12; N, 18.27; H, 4.00; S, 10.44; Cl, 23.56.

The complex is slightly soluble in water (in which an equilibrium exists between the chloro and aquo forms)[3] and ethanol, but is insoluble in acetonitrile, dimethyl sulfoxide and dimethyl formamide.

X-Ray Diffraction.—Preliminary information on the crystal system, cell constants, space group and atom positions were obtained using limited precession and Weissenberg film data. A microphotodensitometer designed for reading spectroscopic plates was employed to measure the intensities. It was then decided to collect extensive three-dimensional intensity data using a

G.E. XRD-6 diffractometer equipped with a Goniostat, pulse-height discriminator and scintillation counter. Nickel filtered Cu Kα radiation (λ=1.54051 Å) was employed. A red-orange, needle shaped crystal with a rhombohedral cross-section (0.042 x 0.042 x 0.275 mm.) was used. The long crystal axis corresponds to the [001] direction in Pnam.

The crystal is orthorhombic with cell constants $\underline{a}$=13.962 ± 0.007, $\underline{b}$=9.308 ± 0.003, $\underline{c}$=7.312 ± 0.003 Å, as calculated from the single crystal diffractometer data. The density, measured by floatation is 2.15 ± 0.03 g./cc. at 24° (d_c=2.127 g./cc.) and corresponds to a calculated value of 4.0 formula weights per unit cell. Extinctions were observed for: $0k\ell$, $k+\ell \neq 2n$; $hk0$, $h \neq 2n$; $h00$, $h \neq 2n$; $0k0$, $k \neq 2n$; 00ℓ, $\ell \neq 2n$, which are consistent with two space groups, *viz.*, $Pna2_1$ (No. 33) and Pnam (equivalent to Pnma-No. 62-by exchanging b and c).

Intensities of 1099 independent reflections (all those for which 160°>2θ>0°) were obtained using the full 2θ scan method. Corrections for Lp, absorption[8] (μ=208.3 $cm.^{-1}$) and spectral dispersion[9] were applied to the data. The atomic scattering factors given in the International Tables[10] were used

(8) Using a modification of an absorption program for a crystal bounded by $\underline{n}$ plane faces ($n \leq 20$) written by B. M. Craven, Crystallographic Laboratory, Univ. of Pittsburgh, Pittsburgh, Pa.

(9) L. E. Alexander and C. G. Smith, "Single Crystal Intensity Measurements with the Three Circle Counter Diffractometer," Mellon Institute, Pittsburgh, Pa., 1961.

(10) J. A. Ibers in "International Tables for X-Ray Crystallography." Vol. III, Table 3.31A, the Kynoch Press, Birmingham, 1962.

for Cl^-, Cl^o, N^o, S^o, O^o, and those given by Thomas and Umeda[11] were

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

used for Ru^{+2}. Least-square calculations were made with a block-diagonal program[12] using an IBM 1620 except for the final full-matrix[13] refine-

(12) Program written by D. Van der Helm, Physics Dept., The Institute for Cancer Research, Philadelphia, Pa.

(13) A. Zalkin's modification of an unpublished program by P. K. Gantzel, R. A. Sparks and K. N. Trueblood, U.C.L.A., Los Angeles, Calif.

ments which were made using an IBM 7044.

Infrared Spectra.--The infrared spectra of the complexes (in KBr) were recorded with a Perkin-Elmer #421 Infrared Spectrophotometer in the rock salt region. There was no evidence for reaction between the complexes and the KBr.

Solution of the Crystal Structure

The intensities of 70 independent zero level reflections from the preliminary precession and Weissenberg films were used to obtain first estimates of the atom positions. The zero level precession photographs show regions of very weak or absent reflections, which is characteristic of crystals having high symmetry; therefore the initial refinement was based on Pnam rather that on $Pna2_1$. Of the three sets of equivalent positions in Pnam for four asymmetric units per unit cell, only the set for which the point symmetry is m $(x,y,1/4;\bar{x},\bar{y},3/4;\ 1/2-x,\ 1/2+y,3/4;\ 1/2+x,\ 1/2-y,1/4)$ need be considered, since the complex cannot have a center.

of symmetry. Thus, the ruthenium, two chlorines and sulfur must have their $\underline{z}$ coordinates in the mirror plane at $\underline{z}=1/4$; both of the oxygens must be either in the mirror plane or symmetrically arranged above and below $\underline{z}=1/4$. The four nitrogens could be symmetrically arranged two above and two below the mirror plane or two in the mirror plane with the other two symmetrically placed above and below $\underline{z}=1/4$.

Possible positions of the Ru were determined from Patterson projections. The conventional R factor ($R=\sum \Big| |Fo| - |Fc| \Big| / \sum |Fo|$), using isotropic temperature factors, was then computed for each of the possible Ru positions and the coordinates giving the lowest R, (0.41), were selected. Positions of the coordinated chlorine and sulfur atoms were estimated by using Harker-Patterson maps and Bragg-Lipson contours combined with chemical considerations. An R factor of 0.28 was computed using the coordinates of the Ru, Cl and S thus far determined. The Cl anion was located from full and difference Fourier projections synthesized with the aid of a Von Eller optical analog computer and using the phases determined by the other three heavy atoms. The nitrogen and oxygen atoms were very poorly resolved in the Fourier. Block-diagonal least-squares refinements were performed using the four heavy atom positions and possible positions for the nitrogen and oxygen atoms. The R factor differed by less than 0.01 for the various light atom arrangements but showed a slight preference for placing the nitrogen atoms two above and two below the mirror plane and for the two oxygen atoms in the mirror plane. This arrangement of atoms (all with isotropic thermal parameters) gave a value of R=0.234.

A series of block-diagonal least-squares refinements and Fourier syntheses followed, using the intensities of the 1099 reflections measured on the diffractometer and the coordinates of the atoms determined from the film data. The atom positions refined in this way gave an R factor of 0.105 using isotropic thermal parameters. Isotropic refinement based on $Pna2_1$ gave an R factor lower by only 0.003. Anisotropic refinement of all atoms based on $Pna2_1$ resulted in an R factor of 0.071 after six cycles. After deletion of 43 very weak or zero intensity reflections plus 12 reflections which were apparently recorded incorrectly the R factor dropped to 0.050. The standard deviations of the z parameters of the four heavy atoms indicated that the departure of this parameter from $z=1/4$ was small but significant. However, block-diagonal refinement does not always correctly estimate the standard deviations. When the structure was refined anisotropically on the basis of Pnam with a full-matrix least-squares program, the R factor leveled off at 0.047 after 3 cycles. Attempts to refine on $Pna2_1$ with the full-matrix program gave several meaningless anisotropic temperature factors. These results indicate that Pnam is very likely the correct space group and illustrate one of the shortcomings of the block-diagonal least-squares method. An attempt to locate the hydrogens from the difference Fourier sections down [001] was not successful.

The final positional and anisotropic temperature parameters and their standard deviations are given in Tables I and II. Table III contains a list of the observed and calculated structure factors.

Table I

Final Positional Parameters (Based on Pnam) and Their Standard Deviations for $[Ru^{II}(NH_3)_4(SO_2)Cl]Cl$

Atom	x/a	y/b	z/c	σ (x)	σ (y)	σ (z)
Ru	0.0857	0.2189	0.2500	0.0001	0.0001	0[b]
Cl_1[a]	0.0028	-0.0087	0.2500	0.0002	0.0003	0[b]
Cl_2[a,c]	0.1503	0.4324	-0.2500	0.0002	0.0003	0[b]
S	0.1653	0.4068	0.2500	0.0002	0.0003	0[b]
N_1	-0.0126	0.2899	0.4529	0.0005	0.0007	0.0008
N_2	0.1748	0.1300	0.4568	0.0004	0.0007	0.0008
O_1	0.2699	0.3986	0.2500	0.0006	0.0010	0[b]
O_2	0.1299	0.5469	0.2500	0.0007	0.0010	0[b]

a. Cl_1 is the coordinated chlorine and Cl_2 the anion chlorine.

b. z parameter is fixed by symmetry.

c. This set of coordinates places the Cl anion closer to the positive region of the complex than the symmetry related coordinates in which z/c = +0.2500.

Table II

Final Anistropic Temperature Factors and Their Standard Deviations in $Å^2$

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ru	1.8	1.5	0.9	0.1	0^a	0^a
Cl_1	3.4	2.2	1.8	-0.6	0^a	0^a
Cl_2	2.9	2.8	2.7	0.7	0^a	0^a
S	3.0	2.5	1.5	-0.7	0^a	0^a
N_1	3.1	3.0	1.7	0.9	0.7	-0.3
N_2	3.0	3.6	1.4	0.9	-0.3	0.4
O_1	3.3	4.2	5.4	-2.0	0^a	0^a
O_2	6.0	3.4	2.6	-1.2	0^a	0^a

	$\sigma(B_{11})$	$\sigma(B_{22})$	$\sigma(B_{33})$	$\sigma(B_{12})$	$\sigma(B_{13})$	$\sigma(B_{23})$
Ru	0.0	0.0	0.0	0.0	0^a	0^a
Cl_1	0.1	0.1	0.1	0.1	0^a	0^a
Cl_2	0.1	0.1	0.1	0.1	0^a	0^a
S	0.1	0.1	0.1	0.1	0^a	0^a
N_1	0.3	0.3	0.2	0.2	0.2	0.2
N_2	0.3	0.3	0.2	0.2	0.2	0.2
O_1	0.4	0.4	0.5	0.3	0^a	0^a
O_2	0.5	0.4	0.4	0.4	0^a	0^a

a. Zero by symmetry.

Table III

Observed and Calculated Structure Factors[a,b] for $[Ru^{II}(NH_3)_4(SO_2)Cl]Cl$

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a. An asterisk, *, denotes reflections assigned zero weight.

b. Both F_c and F_o have been multiplied by 3.4.

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H,K= 0, 0
L FOB FCA
2 897-979
4 939 908
6 627-586
8 367 360

H,K= 0, 1
L FOB FCA
1 686-651
3 349 326
5 487-459
7 219 208
9 203-222

H,K= 0, 2
L FOB FCA
0 158-127
2 121 -73
4 120-114
6 119 101
8 72 -72

H,K= 0, 3
L FOB FCA
1 432 423
3 367-344
5 289 274
7 193-201

H,K= 0, 4
L FOB FCA
0 267 268
2 342-324
4 248 231
6 189-195
8 148 157

H,K= 0, 5
L FOB FCA
1 60 -53
3 100 90
5 72 -53
7 84 82

H,K= 0, 6
L FOB FCA
0 228-226
2 226 219
4 173-172
6 132 132
8 96 -96

H,K= 0, 7
L FOB FCA
1 218 213
3 165-174
5 146 152
7 90 -97

H,K= 0, 8
L FOB FCA
0 0 11*
2 22 18
4 16 7
6 0 -0*

H,K= 0, 9
L FOB FCA
1 93 96
3 85 -88
5 69 71

H,K= 0,10
L FOB FCA
0 159 145
2 126-120
4 121 114

H,K= 0,11
L FOB FCA
1 126-115
3 91 84

H,K= 1, 1
L FOB FCA
0 71 55
1 26 38
2 54 -38
3 100 -89
4 68 53
5 38 -25
6 50 -41
7 27 -26
8 42 37
9 24 -10

H,K= 1, 2
L FOB FCA
0 27 75*
1 190-190
2 291-263
3 169 157
4 71 58
5 115-106
6 47 -47
7 75 73
8 46 43

H,K= 1, 3
L FOB FCA
0 47 -73
1 112 103
2 145 141
3 34 -29
4 94 -92
5 108 101
6 64 58
7 33 -27
8 65 -57

H,K= 1, 4
L FOB FCA
0 340 358
1 182 192
2 298-290
3 243-242
4 253 241
5 132 131
6 182-176
7 147-157
8 122 125

H,K= 1, 5
L FOB FCA
0 357 368
1 228-233
2 495-495
3 230 225
4 344 331
5 138-145
6 255-268
7 125 135
8 212 226

H,K= 1, 6
L FOB FCA
0 148-147
1 279-273
2 212 212
3 316 313
4 135-133
5 162-167
6 108 115
7 185 198
8 90 -97

H,K= 1, 7
L FOB FCA
0 41 -33
1 143 146
2 128 138
3 153-154
4 69 -68
5 95 98
6 74 76
7 102-104

H,K= 1, 8
L FOB FCA
0 33 31
1 193 204
2 40 -41
3 214-217
4 42 36
5 137 145
6 46 -42

H,K= 1, 9
L FOB FCA
0 244 245
1 58 -56
2 255-260
3 46 42
4 209 211
5 60 -58
6 168-176

H,K= 1,10
L FOB FCA
0 157-154
1 111-112
2 116 117
3 110 109
4 119-121
5 93 -92

H,K= 1,11
L FOB FCA
0 91 -91
1 101 102
2 91 90
3 89 -83

H,K= 2, 0
L FOB FCA
0 293 272
1 549-543
2 172-151
3 417 382
4 208 180
5 380-373
6 164-149
7 232 227
8 115 103
9 201-203

H,K= 2, 1
L FOB FCA
0 631-672
1 190-185
2 487 479
3 109 103
4 449-427
5 161-155
6 314 306
7 92 84
8 222-223
9 62 -79

H,K= 2, 2
L FOB FCA
0 267-259
1 378 389
2 66 49
3 348-340
4 134-130
5 254 252
6 97 90
7 191-199
8 49 -46

H,K= 2, 3
L FOB FCA
0 62 65
1 316 323
2 212-200
3 158-163
4 109 112
5 230 226
6 102 -99
7 92 -93
8 94 97

H,K= 2, 4
L FOB FCA
0 459 474
1 220-217
2 264-250
3 255 250
4 287 278
5 122-118
6 188-190
7 149 147
8 108 116

H,K= 2, 5
L FOB FCA
0 201-198
1 106-103
2 275 272
3 58 46
4 193-179
5 92 -93
6 151 155
7 34 27
8 122-124

H,K= 2, 6
L FOB FCA
0 116 107
1 117 114
2 114-115
3 113-111
4 79 74
5 89 88
6 59 -59
7 80 -82

H,K= 2, 7
L FOB FCA
0 124 130
1 18 -6
2 92 -92
3 26 -18
4 95 96
5 0 -5*
6 74 -75
7 21 -16

H,K= 2, 8
L FOB FCA
0 39 41
1 50 -47
2 94 -94
3 13 20
4 57 51
5 48 -46
6 41 -41

H,K= 2, 9
L FOB FCA
0 96 98
1 36 40
2 119-123
3 21 -9
4 85 87
5 51 46
6 17 -73*

H,K= 2,10
L FOB FCA
0 74 73
1 103-100
2 37 -34
3 98 97
4 52 53

H,K= 2,11
L FOB FCA
0 62 -48
1 10 -4*
2 53 41
3 0 -6*

H,K= 3, 1
L FOB FCA
0 358 405
1 169-167
2 567-545
3 140 130
4 290 270
5 94 -84
6 204-201
7 66 63
8 152 155

H,K= 3, 2
L FOB FCA
0 217-236
1 119 114
2 223 220
3 69 63
4 155-154
5 86 88
6 110 111
7 42 50
8 71 -80

H,K= 3, 3
L FOB FCA
0 403 382
1 265 268
2 103 -73
3 216-223
4 183 170
5 174 173
6 102 -96
7 125-132
8 40 36

H,K= 3, 4
L FOB FCA
0 265 285
1 69 67
2 256-252
3 112-109
4 220 224
5 24 -5
6 198-186
7 38 -42
8 127 139

H,K= 3, 5
L FOB FCA
0 67 -66
1 248-260
2 24 21
3 216 222
4 43 -41
5 205-211
6 34 32
7 150 159
8 19 -16

H,K= 3, 6
L FOB FCA
0 287-293
1 132 132
2 253 246
3 117-109
4 233-231
5 84 89
6 196 192
7 56 -65

H,K= 3, 7
L FOB FCA
0 37 -27
1 281 289
2 44 36
3 239-246
4 32 -24
5 219 228
6 10 10*
7 160-166

H,K= 3, 8
L FOB FCA
0 247 267
1 60 63
2 226-235
3 81 -78
4 210 217
5 36 37
6 171-178

H,K= 3, 9
L FOB FCA
0 0 -12*
1 216-215
2 44 -42
3 195 192
4 14 9
5 169-172

H,K= 3,10
L FOB FCA
0 182-182
1 21 28
2 173 170
3 16 -1*
4 157-155

H,K= 3,11
L FOB FCA
0 92 85
1 126 125
2 55 -51
3 121-117

H,K= 4, 0
L FOB FCA
0 222-250
1 356-367
2 386 364
3 451 428
4 238-225
5 193-184
6 176 174
7 230 227
8 151-148

H,K= 4, 1
L FOB FCA
0 129-168
1 165 170
2 393 376
3 240-232
4 206-205
5 122 119
6 176 174
7 150-148
8 155-158

H,K= 4, 2
L FOB FCA
0 111 136
1 307 318
2 302-290
3 311-311
4 161 162
5 215 214
6 139-141
7 183-191
8 126 130

H,K= 4, 3
L FOB FCA
0 471 497
1 289-277
2 399-396
3 287 288
4 358 352
5 128-127
6 261-266
7 133 144
8 182 193

H,K= 4, 4
L FOB FCA
0 38 23
1 281-275
2 94 91
3 203 202
4 23 -13
5 224-224
6 28 23
7 130 134
8 41 -30

H,K= 4, 5
L FOB FCA
0 129-131
1 36 37
2 65 59
3 74 -65
4 93 -95
5 30 32
6 70 69
7 64 -59
8 54 -53

H,K= 4, 6
L FOB FCA
0 237 230
1 79 83
2 222-220
3 64 -69
4 185 183
5 56 59
6 142-150
7 39 -40

H,K= 4, 7
L FOB FCA
0 0 -10*
1 54 -53
2 19 -23
3 41 32
4 23 0*
5 51 -49
6 22 -3
7 25 23

H,K= 4, 8
L FOB FCA
0 102 100
1 40 32
2 134-132
3 0 -8*
4 93 88
5 39 36
6 69 -67

H,K= 4, 9
L FOB FCA
0 0 -3*
1 23 11
2 35 31
3 16 11
4 0 -5*
5 22 14

H,K= 4,10
L FOB FCA
0 43 35
1 21 -22
2 13 -6*
3 19 21
4 20 16

H,K= 4,11
L FOB FCA
0 72 -76
1 91 86
2 88 86

H,K= 5, 1
L FOB FCA
0 112 111
1 22 -10*
2 46 -43
3 50 53
4 42 38
5 25 -13
6 15 -11
7 53 46
8 11 -4

H,K= 5, 2
L FOB FCA
0 141-151
1 181 192
2 239 233
3 188-184
4 20-136*
5 127 128
6 132 127
7 113-118
8 93 -97

H,K= 5, 3
L FOB FCA
0 336 352
1 97 98
2 306-311
3 93-103
4 254 255
5 62 66
6 194-202
7 66 -73
8 138 145

H,K= 5, 4
L FOB FCA
0 19 20
1 173-179
2 26 9
3 112 119
4 13 3
5 148-151
6 0 -10*
7 82 86
8 0 0*

H,K= 5, 5
L FOB FCA
0 193-206
1 143-143
2 75 70
3 86 78
4 120-126
5 109-109
6 96 96
7 44 42
8 51 -54

H,K= 5, 6
L FOB FCA
0 178-173
1 303 303
2 92 79
3 203-206
4 119-114
5 245 253
6 89 85
7 124-131

H,K= 5, 7
L FOB FCA
0 344 362
1 89 87
2 245-248
3 58 -53
4 263 274
5 97 91
6 203-212
7 43 -45

H,K= 5, 8
L FOB FCA
0 180 184
1 189-197
2 147-151
3 160 157
4 141 148
5 10-175*
6 119-118

H,K= 5, 9
L FOB FCA
0 164-161
1 129-128
2 145 136
3 123 117
4 141-137
5 99-100

H,K= 5,10
L FOB FCA
0 21 -4*
1 161 161
2 31 14
3 156-152
4 24 -10

H,K= 5,11
L FOB FCA
0 117 117
1 30 31
2 121-120

H,K= 6, 0
L FOB FCA
0 116 101
1 57 52
2 245 234
3 24 -23
4 50 -49
5 27 24
6 86 79
7 0 -7*
8 111-105

H,K= 6, 1
L FOB FCA
0 100 105
1 378 374
2 66 -60
3 431-434
4 59 55
5 215 206
6 43 -43
7 248-255
8 33 21

H,K= 6, 2
L FOB FCA
0 498 525
1 63 -55
2 595-602
3 25 20
4 428 425
5 57 -51
6 340-343
7 30 21
8 257 267

H,K= 6, 3
L FOB FCA
0 19 -2*
1 256-272
2 53 -60
3 253 252
4 14 13
5 215-219
6 14 -4
7 175 180
8 15 14

H,K= 6, 4
L FOB FCA
0 229-235
1 24 18
2 172 171
3 25 15
4 181-184
5 36 33
6 143 146
7 0 2*
8 109-109

H,K= 6, 5
L FOB FCA
0 65 72
1 245 248
2 23 -22
3 201-198
4 54 47
5 179 189
6 26 -35
7 118-120

H,K= 6, 6
L FOB FCA
0 84 86
1 0 -12*
2 66 -71
3 16 -1
4 66 67
5 19 -13
6 53 -53
7 10 -2*

H,K= 6, 7
L FOB FCA
0 26 -16
1 32 26
2 19 13
3 23 -16
4 13 -11
5 25 25
6 0 2*

H,K= 6, 8
L FOB FCA
0 26 20
1 20 20
2 16 1
3 23 -26
4 25 15
5 0 8*
6 22 -7

H,K= 6, 9
L FOB FCA
0 21 -25
1 16 -3
2 16 6
3 19 3*
4 10 -13
5 17 -3

H,K= 6,10
L FOB FCA
0 13 1*
1 13 14
2 16 16
3 14 -3*

H,K= 6,11
L FOB FCA
0 21 -1*
1 69 65

H,K= 7, 1
L FOB FCA
0 179-176
1 14 6
2 146 142
3 70 -67
4 115-107
5 17 1
6 84 75
7 56 -54
8 45 -44

H,K= 7, 2
L FOB FCA
0 88 90
1 120 125
2 234-238
3 89 -90
4 124 120
5 94 95
6 106-108
7 63 -62
8 100 103

H,K= 7, 3
L FOB FCA
0 248 261
1 124-128
2 172-185
3 174 173
4 182 185
5 68 -67
6 137-147
7 106 108
8 97 99

H,K= 7, 4
L FOB FCA
0 24 -18
1 226-234
2 66 70
3 175 166
4 36 -35
5 171-184
6 33 33
7 96 102

H,K= 7, 5
L FOB FCA
0 88 -77
1 105 108
2 24 -5*
3 90 -94
4 58 -53
5 79 79
6 47 43
7 56 -55

H,K= 7, 6
L FOB FCA
0 164 163
1 158 153
2 126-126
3 109-111
4 127 129
5 156 149
6 96 -95
7 94 -86

H,K= 7, 7
L FOB FCA
0 354 355
1 133-131
2 285-289
3 119 120
4 277 281
5 14-113*
6 218-216

H,K= 7, 8
L FOB FCA
0 147-149
1 181-183
2 157 155
3 146 155
4 139-136
5 151-151

H,K= 7, 9
L FOB FCA
0 92 -88
1 130 127
2 76 72
3 150-137
4 87 -76

H,K= 7,10
L FOB FCA
0 56 53
1 162 152
2 95 -89
3 148-137

H,K= 8, 0
L FOB FCA
0 25 4*
1 282 281
2 17 5
3 246-246
4 37 -21
5 210 210
6 27 30
7 174-171
8 15 -30

H,K= 8, 1
L FOB FCA
0 228 225
1 163 168
2 203-200
3 158-159
4 182 180
5 119 119
6 159-164
7 94-101
8 112 119

H,K= 8, 2
L FOB FCA
0 213 221
1 30-300*
2 254-269
3 242 235
4 181 183
5 232-235
6 137-145
7 166 161
8 109 115.

H,K= 8, 3
L FOB FCA
0 472-476
1 19 -13
2 361 355
3 63 62
4 342-342
5 0 10*
6 248 260
7 48 46

H,K= 8, 4
L FOB FCA
0 78 84
1 243 250
2 27 21
3 183-182
4 37 38
5 196 203
6 22 -14
7 110-116

H,K= 8, 5
L FOB FCA
0 115 116
1 109 101
2 59 -61
3 127-119
4 83 87
5 67 57
6 62 -66
7 67 -77

H,K= 8, 6
L FOB FCA
0 78 84
1 86 -86
2 90 -94
3 73 76
4 71 73
5 64 -69
6 66 -69

H,K= 8, 7
L FOB FCA
0 26 27
1 19 6
2 26 -6
3 21 -20
4 21 11*
5 21 -9
6 20 -5

H,K= 8, 8
L FOB FCA
0 21 -9*
1 52 -49
2 16 -39
3 30 27
4 16 9
5 44 -39

H,K= 8, 9
L FOB FCA
0 25 32
1 16 5
2 55 -50
3 25 20
4 27 29

H,K= 8,10
L FOB FCA
0 13 -6
1 13 8
2 44 36

H,K= 9, 1
L FOB FCA
0 179 175
1 23 28
2 242-242
3 20 -18
4 151 150
5 17 23
6 126-122
7 17 -12
8 85 95

H,K= 9, 2
L FOB FCA
0 183 187
1 55 -58
2 157-158
3 101 105
4 147 143
6 105-108
7 70 64

H,K= 9, 3
L FOB FCA
0 94 93
1 93-175*
2 31 15
3 163 161
4 46 49
5 134-134
6 22 -24
7 105 104

H,K= 9, 4
L FOB FCA
0 115-109
1 16 2
2 129 130
3 36 -29
4 111-109
5 0 -10*
6 85 90
7 22 -22

H,K= 9, 5
L FOB FCA
0 34 -25
1 205 203
2 19 10
3 214-215
4 23 -11
5 143 148
6 0 2*
7 141-144

H,K= 9, 6
L FOB FCA
0 214 212
1 36 31
2 255-258
3 30 -28
4 201 200
5 23 16
6 159-163

H,K= 9, 7
L FOB FCA
0 34 -32
1 176-176
2 41 33
3 191 192
4 25 -28
5 126-128

H,K= 9, 8
L FOB FCA
0 170-172
1 74 76
2 197 201
3 90 -79
4 157-158
5 55 52

H,K= 9, 9
L FOB FCA
0 52 59
1 172 167
2 98 -86
3 169-165

H,K= 9,10
L FOB FCA
0 113 101
1 9 -7

H,K=10, 0
L FOB FCA
0 140 154
1 299 308
2 91 -91
3 205-200
4 117 118
5 59 261*
6 92 -98
7 128-130

H,K=10, 1
L FOB FCA
0 350 357
1 181-190
2 224-219
3 148 143
4 248 250
5 150-157
6 184-192
7 92 93

H,K=10, 2
L FOB FCA
0 210-207
1 205-209
2 147 142
3 159 160
4 156-153
5 163-173
6 109 113
7 105 110

H,K=10, 3
L FOB FCA
0 66 -73
1 138 135
2 78 79
3 110-107
4 68 -71
5 123 118
6 68 71
7 75 -75

H,K=10, 4
L FOB FCA
0 220 216
1 71 69
2 176-183
3 85 -83
4 169 175
5 56 53
6 129-132
7 69 -68

H,K=10, 5
L FOB FCA
0 104 106
1 110-104
2 132-128
3 85 96
4 103 95
5 80 -79
6 83 -84

H,K=10, 6
L FOB FCA
0 79 81
1 103-107
2 0 -62*
3 97 96
4 60 55
5 81 -81
6 42 -39

H,K=10, 7
L FOB FCA
0 80 -77
1 19 10
2 49 51
3 24 -20
4 55 -57
5 0 4*

H,K=10, 8
L FOB FCA
0 16 81*
1 9 10
2 100 -95
3 13 4*
4 63 68

H,K=10, 9
L FOB FCA
0 39 -37
1 35 24
2 54 51
3 16 -15

H,K=11, 1
L FOB FCA
0 138 137
1 23 9
2 103-102
3 16 -3
4 99 104
5 14 7
6 79 -80
7 14 4

H,K=11, 2
L FOB FCA
0 0 -58*
1 19 -11
2 72 65
3 0 -3*
4 45 -41
5 30 -31
6 41 38
7 19 12

H,K=11, 3
L FOB FCA
0 39 -36
1 37 -38
2 28 23
3 43 45
4 39 -34
5 25 -28
6 39 33

H,K=11, 4
L FOB FCA
0 132-130
1 222 223
2 157 159
3 216-212
4 113-118
5 157 159
6 89 87

H,K=11, 5
L FOB FCA
0 211 215
1 72 68
2 259-258
3 97 -92
4 189 192
5 46 40
6 161-163

H,K=11, 6
L FOB FCA
0 34 33
1 139-138
2 91 -84
3 163 160
4 47 46
5 89 -94

H,K=11, 7
L FOB FCA
0 111-115
1 72 -67
2 163 161
3 89 82
4 114-111

H,K=11, 8
L FOB FCA
0 9 13
1 143 141
2 0 8*
3 151-143

H,K=11, 9
L FOB FCA
0 121 115
1 16 13
2 118-115

H,K=12, 0
L FOB FCA
0 401 412
1 115-119
2 305-306
3 109 112
4 318 323
5 74 -77
6 243-245

H,K=12, 1
L FOB FCA
0 26 -17
1 269-261
2 35 35
3 205 208
4 25 -22
5 212-223
6 13 15

H,K=12, 2
L FOB FCA
0 146-148
1 19 -11
2 114 114
3 23 -14
4 115-124
5 21 -12
6 95 97

H,K=12, 3
L FOB FCA
0 58 50
1 136 142
2 91 -98
3 137-139
4 63 61
5 109 111
6 49 -49

H,K=12, 4
L FOB FCA
0 135 132
1 9 -9
2 158-156
3 40 39
4 120 121
5 10 2*
6 106-105

H,K=12, 5
L FOB FCA
0 60 52
1 22-105*
2 19 -10
3 105 108
4 32 29
5 78 -75

H,K=12, 6
L FOB FCA
0 80 -82
1 44 44
2 80 85
3 43 -46
4 70 -71
5 31 23

H,K=12, 7
L FOB FCA
0 49 39
1 74 63
2 42 -34
3 57 -55
4 38 27

H,K=12, 8
L FOB FCA
0 35 25
1 16 6
2 16 -15

H,K=13, 1
L FOB FCA
0 13 17
1 9 -4
2 31 -27
3 24 -19
4 26 17
5 13 -17
6 20 -14

H,K=13, 2
L FOB FCA
0 48 -54
1 78 -76
2 26 -14
3 78 79
4 31 -27
5 48 -47
6 22 17

H,K=13, 3
L FOB FCA
0 48 -41
1 74 69
2 64 67
3 38 -33
4 46 -43
5 73 71

H,K=13, 4
L FOB FCA
0 136 131
1 89 81
2 105 -94
3 95 -91
4 108 100
5 68 62

H,K=13, 5
L FOB FCA
0 170 168
1 140-137
2 190-187
3 125 121
4 153 148
5 108-110

H,K=13, 6
L FOB FCA
0 105-105
1 113-115
2 109 106
3 116 112
4 89 -90

H,K=13, 7
L FOB FCA
0 35 -34
1 70 68
2 46 41
3 65 -61

H,K=13, 8
L FOB FCA
0 83 78
1 117 109

H,K=14, 0
L FOB FCA
0 114 113
1 183-182
2 130-128
3 183 188
4 101 100
5 151-146

H,K=14, 1
L FOB FCA
0 193-193
1 33 -30
2 207 212
3 38 37
4 175-177
5 24 -22

H,K=14, 2
L FOB FCA
0 9 3*
1 164 157
2 20 -13
3 163-165
4 13 -0
5 113 113

H,K=14, 3
L FOB FCA
0 74 70
1 104 101
2 109-107
3 77 -76
4 76 73
5 90 86

H,K=14, 4
L FOB FCA
0 111 102
1 111-111
2 69 -61
3 122 117
4 86 78

H,K=14, 5
L FOB FCA
0 93-100
1 18 12
2 115 113
3 22 -20
4 90 -88

H,K=14, 6
L FOB FCA
0 16 9
1 49 50
2 13 -10
3 53 -47

H,K=14, 7
L FOB FCA
0 59 54
1 13 9

H,K=15, 1
L FOB FCA
0 33 33
1 70 -71
2 61 -56
3 71 69
4 44 34
5 51 -50

H,K=15, 2
L FOB FCA
0 27 -16
1 0 -4*
2 33 27
3 22 21
4 24 -21

H,K=15, 3
L FOB FCA
0 65 54
1 71 68
2 34 -22
3 69 -63
4 47 40

H,K=15, 4
L FOB FCA
0 171 162
1 43 -38*
2 143-141
3 30 31

H,K=15, 5
L FOB FCA
0 13 -24
1 120-115
2 18 26
3 91 89

H,K=15, 6
L FOB FCA
0 118-118
1 33 30

H,K=16, 0
L FOB FCA
0 113-118
1 74 -74
2 126 132
3 100 101
4 101-104

H,K=16, 1
L FOB FCA
0 80 -75
1 107 105
2 122 122
3 108-105
4 80 -79

H,K=16, 2
L FOB FCA
0 100 96
1 107 104
2 106-103
3 115-111

H,K=16, 3
L FOB FCA
0 94 88
1 81 -83
2 94 -88
3 75 76

H,K=16, 4
L FOB FCA
0 25 -27
1 91 -87
2 25 21

H,K=16, 5
L FOB FCA
0 64 -63
1 49 48

H,K=17, 1
L FOB FCA
0 32 30
1 30 -29
2 19 -13

H,K=17, 2
L FOB FCA
0 56 -54
1 73 70
2 48 45

H,K=17, 3
L FOB FCA
0 106 96
1 28 25
```

Discussion of the Structure

The complex is in the form of a slightly distorted octahedron with the four ammine groups at the corners of a square whose plane is essentially perpendicular to the line joining the S, Ru, and coordinated Cl, and in which the latter three atoms, the two oxygen atoms and the chlorine anion lie in a plane of mirror symmetry. A schematic diagram of the structure projected down [001] is shown in Fig. 1. Insert Fig. 1. Fig. 2 shows a perspective drawing of $[Ru^{II}(NH_3)_4(SO_2)Cl]^+$. Insert Fig. 2 A list of the most important bond distances and angles are given in Table IV. The most significant results of the structure analysis are (1) that the SO_2 ligand has been shown to be coordinated through the sulfur and (2) that the S-O bond lengths and the O-S-O bond angle are approximately the same as in free, solid SO_2 (S-O, 1.430 ± 0.015 Å; O-S-O, 119 ± 2°).[14]

(14) B. Post, R. S. Schwartz and I. Fankuchen, Acta Cryst., 5, 372 (1952).

The significance of the difference between the individual S-O bond lengths and the average of these values (1.428 ± 0.010 Å), which amounts to three standard deviations, is open to question. On chemical grounds there is no reason to expect that the two S-O bonds should differ. If the observed deviation is real, it is most likely the result of packing effects. The same argument can also be applied to the deviation of the Cl_1-Ru-S bond angle from 180°. Coordination of the SO_2 through the sulfur might have been anticipated since ligands containing both sulfur and oxygen generally perfer to coordinate through the sulfur when the metal involved contains electrons in low lying d orbitals which can pi bond with the empty d orbitals of sulfur.[15]

(15) K. Suzuki and K. Yamasaki, J. Inorg. Nucl. Chem., 24, 1093 (1962).

Table IV

Interatomic Distances (in Å) and Angles and Their Standard Deviations

Ru-N_1	2.127 ± 0.006
Ru-N_2	2.126 ± 0.006
Ru-Cl_1	2.415 ± 0.003
Ru-Cl_2	4.258 ± 0.002
Ru-S	2.072 ± 0.003
S-O_1	1.462 ± 0.010
S-O_2	1.394 ± 0.010
O_1-O_2	2.393 ± 0.014
∠ O-S-O	113.8 ± 0.6
∠ Cl_1-Ru-S	176.3 ± 0.1
∠ Cl_1-Ru-N_1	87.9 ± 0.2
∠ Cl_1-Ru-N_2	86.5 ± 0.2
∠ S-Ru-N_1	94.8 ± 0.2
∠ S-Ru-N_2	90.9 ± 0.2
∠ N_1-Ru-N_1^*	88.5 ± 0.3
∠ N_1-Ru-N_2	90.1 ± 0.2
∠ N_1^*-Ru-N_2^*	90.1 ± 0.2
∠ N_2-Ru-N_2^*	90.7 ± 0.3

* Designates the atom related by the mirror symmetry.

The Ru-N bond distance is comparable to the value of 2.10 Å found in $[Ru^{III}(NH_3)_5Cl]Cl_2$[16] and is within the range of 2.00-2.35 Å found in

(16) C. K. Prout and H. M. Powell, J. Chem. Soc., 1962, 137.

other platinum group ammines.[17] The $Ru-Cl_1$ distance appears to be typical

(17) The Chemical Society (London), "Tables of Interatomic Distances and Configurations in Molecules and Ions," Spec. Publ. No. 11, Burlington House, London W.1, 1958.

of chlorine coordinated to platinum group metals.[16,17] Transition-metal sulfur bond lengths of 2.1-2.5 have been reported,[17,18] the lower limit of

(18) C. K. Jorgensen, "Inorganic Complexes," Academic Press, N. Y., 1963, p. 142.

which is slightly greater than the distance observed in the $Ru-SO_2$ complex.

Realizing that no firm conclusions can be drawn from utilizing the sums of ionic or covalent radii to determine the ionic or covalent character of bonds, except in some organic compounds where sufficient statistical data are available, the following observations are presented: (1) the sum of the octahedral covalent radius of Ru(II)[19] and single bond covalent radius of

(19) L. Pauling, "The Nature of the Chemical Bond," 3rd ed., Cornell University Press, Ithaca, N. Y., 1960.

N[19] is 2.03 Å, which is about 0.1 Å smaller than the observed Ru-N distance, indicating partial ionic character in the Ru-N bond (infrared evidence is also consistent with ionic character in $M-NH_3$ bonds- see Nakamoto,[20] p. 145);

(20) K. Nakamoto, "Infrared Spectra of Inorganic and Coordination Compounds," John Wiley and Sons, Inc., N. Y., 1963.

(2) the sum of the octahedral covalent radius of Ru(II) and the radius of sulfur (calculated from the S-O bond distance in solid SO_2 and the double bond covalent radius of oxygen[19]) is longer by 0.14 Å than the observed Ru-S distance, indicating partial double bond character in the Ru-S bond; (3) the sum of the octahedral covalent radius of Ru(II) and the single bond covalent radius of Cl[19] is 2.32 Å which is 0.1 Å smaller than the observed Ru-Cl distance and considerably smaller than the sum obtained using the chloride anion (3.13 Å), thus the Ru-Cl bond is predominantly covalent.

Like other ammine complexes[20] containing very electronegative atoms, the Ru-SO_2 complex is hydrogen bonded, as is evidenced by inter- and intramolecular spacings of 2.9-3.3 Å between the ammine nitrogens and the sulfur, two chlorines and two oxygens. These inter- and intramolecular spacings of 2.9-3.3 Å fall into the range of values reported in Table 4.1.12 of the International Tables[10] for hydrogen bonds between N-N, N-Cl and N-O atoms.

The Infrared Study

Coordinated ammines exhibit five fundamental modes in their vibrational spectra (plus the M-N mode, whose position is still a subject of controversy).[20] Table V gives the positions and empirical assignments of the bands in the Ru-SO_2 complex and in three of its precursors, that are considered to arise from the ammine ligand. The remaining medium and strong bands in the spectrum of $[Ru^{II}(NH_3)_4(SO_2)Cl]Cl$ are attributed to the SO_2 ligand and are presented in Table VI along with the band assignments for uncoordinated, solid SO_2.[21]

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(21) R. N. Wilson and E. R. Nixon, J. Chem. Phys., 25, 175 (1956).

Table V

Infrared Frequencies (in $cm.^{-1}$) of the NH_3 Ligand in Ruthenium-Ammine Complexes

Complex	Sym. Str.[a]	Asy. Str.	Sym. Def.	Asy. Def.	Rock
$[Ru^{III}(NH_3)_6]Cl_3$	3218(s)	3405(M)	1312(m)	1608(m)	775(m)
$[Ru^{III}(NH_3)_5Cl]Cl_2$	3208(s)	3405(m)	1290(s)	1610(m)	790(m)
$[Ru^{II}(NH_3)_4(HSO_3)_2]$	3260(s)	3430(M)	1299(s)	1633(m)	782(m)
$[Ru^{II}(NH_3)_4(SO_2)Cl]Cl$	3225(s)	3420(ms)	1245(s)	1625(m)	779(m)

a. A broad irregularly shaped band.

Table VI

Infrared Frequencies (in $cm.^{-1}$) of Free and Coordinated SO_2

	Sym. Str.	Asy. Str.	Bend
$[Ru^{II}(NH_3)_4(SO_2)Cl]Cl$	1100(s)	1301, 1278 (s)	552(m)
SO_2 (Solid)[21]	1147(m)	1330, 1308 (s)	521(m)

In the Ru-So_2 comples, the band at 1245 $cm.^{-1}$ was assigned to the symmetric deformation mode of the NH_3 ligand rather than the band at 1301 $cm.^{-1}$, on the basis of its intensity relative to the asymmetric deformation band in the SO_2- free complexes. The 1301 $cm.^{-1}$ band is considered to arise from the SO_2 group. Additional support for this assignment is the appearance of a band at 1302 $cm.^{-1}$ in two of Vaska's[5] SO_2 complexes which do not contain ammine or other ligands having absorptions in this region (aside from the SO_2).

The lower values of the stretching modes of the SO_2 in the complexes, compared to those in uncoordinated, solid SO_2 may arise from the same factors that are responsible for the C-O stretching frequencies in metal carbonyls being lower than those in free CO.[22] However, the effect in the metal carbonyls is of much greater magnitude than in the SO_2 complex. It is suggested therefore that the S-O bond order may be lowered and the Ru-S bond order raised by overlap of empty antibonding *pi* orbitals on the SO_2 ligand with the filled non-bonding *d* orbitals of the ruthenium. A molecular orbital treatment of the complex would be helpful in deciding the validity of this suggestion.

(22) E. W. Abel, Quart. Revs., 1963, 133.

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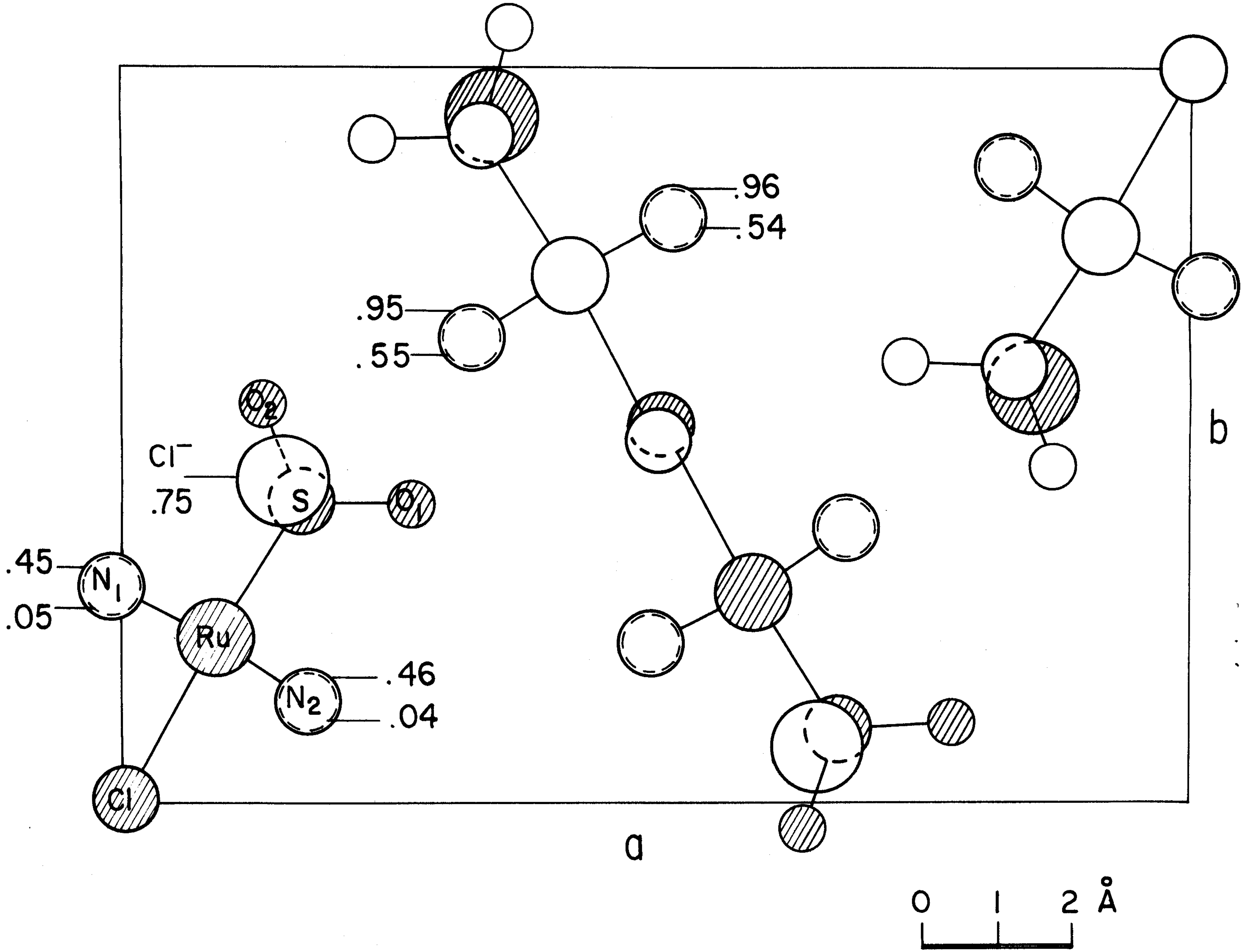
research on SO_2 complexes. Chemical analyses for N, H, and S were performed by Galbraith Laboratories and Schwarzkopf Microanalytical Laboratory.

Figure 1

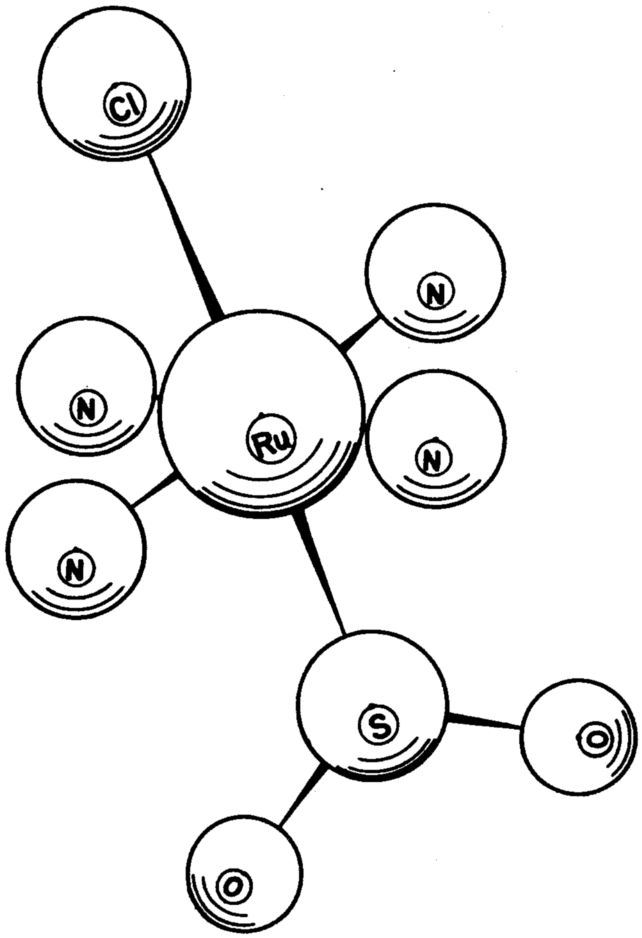
Projection of the structure of $[Ru^{II}(NH_3)_4(SO_2)Cl]Cl$ (excluding hydrogen atoms) down the c axis (the origin is at the bottom left). The shaded atoms are at $z = 1/4$ and the unshaded atoms are at $z = 3/4$ except for the nitrogen atoms whose z coordinates are indicated.

Figure 2

Perspective view of $[Ru^{II}(NH_3)_4(SO_2)Cl]^+$ (excluding hydrogen atoms).



.96
.54
.95
.55
O2
Cl⁻
.75
S
O1
.45
N1
.05
Ru
N2
.46
.04
Cl
b
a
0 1 2 Å



Dr. for Dr. Katz
by: G.K.
Dec. 14. 64.