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APPENDIX A.

Calculation of the Number and Activity of Vibrations of a Molecule XY_4 Belonging to the Point Group Td.

a) Determination of the Infrared Activity.

Consider the rotation of a line joining the origin to a point (x, y, z) through an angle θ , in a clockwise sense, the new coordinates (x', y', z') are

$$x' = x \cos\theta + y \sin\theta$$

$$y' = -x\sin\theta + y\cos\theta$$

$$z' = z$$

or in the matrix form

$$\begin{pmatrix} x' \\ y' \\ z' \end{pmatrix} = \begin{pmatrix} \cos\theta & \sin\theta & 0 \\ -\sin\theta & \cos\theta & 0 \\ 0 & 0 & 1 \end{pmatrix} \begin{pmatrix} x \\ y \\ z \end{pmatrix}$$

The character for this rotation $\chi(C) = 1+2\cos\theta$.

If the rotation is combined with a reflection, the new coordinates are :

$$x'' = x\cos\theta + y\sin\theta$$

$$y'' = -x\sin\theta + y\cos\theta$$

$$z'' = -z$$

The character for the improper rotation $\chi(S) = -1+2\cos\theta$.

For the inversion, $x''' = -x$, $y''' = -y$ and $z''' = -z$, the character $\chi(i)$ is -3 (by putting $\theta = 180°$ in $-1+2\cos\theta$)

For the reflection, $x^{+} = -x$, $y^{+} = y$, $z^{+} = z$, the character $\chi(\sigma)$ is 1 (by putting $\theta = 0°$ in $-1+2\cos\theta$).

The character $\chi(\mu)$ of the reducible representation of the dipole moment $\Gamma(\mu)$ is constructed as in Table 50. which shows the characters for each operation in T_d. + sign in $\chi(\mu)$ refers to the proper rotations (C_n^k). - sign refers to the improper rotations(S_n^k), reflections (σ) and inversion (i).

Table 50. Calculation of $\chi(\mu)$ for T_d

	E	8 C_3	3 C_2	6 S_4	6 σ_d
$\theta°$	0	120	180	90	0
$\chi(\mu) = \pm 1+2\cos\theta$	3	0	-1	-1	1

Table 51. Character table of T_d

T_d (h=24)	E	8 C_3	3 C_2	6 S_4	6 σ_d
A_1	1	1	1	1	1
A_2	1	1	1	-1	-1
E	2	-1	2	0	0
T_1	3	0	-1	1	-1
T_2	3	0	-1	-1	1

To determine how many times each of the irreducible representations Γ_j of T_d, occurs in the reducible representation of the dipole moment $\Gamma(\mu)$, the following formula is applied.

$$a_j = \frac{1}{h} \sum_R n \, \chi(R) \, \chi_j(R)$$

a_j is the number of times Γ_j appears in $\Gamma(\mu)$.

h is the number of operations in the point group.

n is the number of lelements in the class of operation.

$\chi(R)$ is the character of the reducible representation $\Gamma(\mu)$.

$\chi_j(R)$ is the character of the irreducible representation Γ_j.

a_j can be calculated by using the character of the reducible representations in Table 50 and the character of the irreducible representations in Table 51. Since there are 24 operations in T_d, h = 24.

$$a(A_1) = \frac{1}{24}\left[(1)(1)(3)+(8)(1)(0)+(3)(1)(-1)+(6)(1)(-1)+(6)(1)(1)\right]=0$$

$$a(A_2) = \frac{1}{24}\left[(1)(1)(3)+(8)(1)(0)+(3)(1)(-1)+(6)(1)(-1)+(6)(1)(-1)\right]=0$$

$$a(E) = \frac{1}{24}\left[(1)(2)(3)+(8)(-1)(0)+(3)(2)(-1)+(6)(-1)(0)+(6)(1)(0)\right]=0$$

$$a(T) = \frac{1}{24}\left[(1)(3)(3)+(8)(0)(0)+(3)(-1)(-1)+(6)(1)(-1)+(6)(1)(-1)\right]=0$$

$$a(T) = \frac{1}{24}\left[(1)(3)(3)+(8)(0)(0)+(3)(-1)(-1)+(6)(-1)(-1)+(6)(1)(1)\right]=1$$

If vibrations of an irreducible representation are infrared active, a_j will be equal to 1, if they are inactive, a_j will be zero.

It is seen that T_2 occurs once in the reducible representation of the dipole moment $\Gamma(\mu)$, therefore only T_2 is infrared active.

b) Determination of the Raman Activity

Consider the polarizability referred to two sets of axes ox, oy, oz; ox$'$, oy$'$, oz$'$. If a rotation by an angle θ about the z-axis causes the components of the polarizability to undergo the changes $\alpha_{xx} \longrightarrow \alpha_{xx}''$, $\alpha_{yy} \longrightarrow \alpha_{yy}''$, ...etc. The new six components of the polarizability are :

$$\alpha_{xx}'' = \alpha_{xx}\cos^2\theta + \alpha_{yy}\sin^2\theta + 2\alpha_{xy}\sin\theta\cos\theta$$

$$\alpha_{yy}'' = \alpha_{xx}\sin^2\theta + \alpha_{yy}\cos^2\theta - 2\alpha_{xy}\sin\theta\cos\theta$$

$$\alpha_{zz}'' = \alpha_{zz}$$

$$\alpha_{yz}'' = \pm\alpha_{yz}\cos\theta \mp \alpha_{zx}\sin\theta$$

$$\alpha_{zx}'' = \pm\alpha_{yz}\sin\theta \pm \alpha_{zx}\cos\theta$$

$$\alpha_{xy}'' = -\alpha_{xx}\sin\theta\cos\theta + \alpha_{yz}\sin\theta\cos\theta + \alpha_{xy}(\cos^2\theta - \sin^2\theta)$$

or in the matrix form

$$\begin{pmatrix} \alpha_{xx}'' \\ \alpha_{yy}'' \\ \alpha_{zz}'' \\ \alpha_{xy}'' \\ \alpha_{yz}'' \\ \alpha_{zx}'' \end{pmatrix} = \begin{pmatrix} \cos^2\theta & \sin^2\theta & 0 & 2\sin\theta\cos\theta & 0 & 0 \\ \sin^2\theta & \cos^2\theta & 0 & -2\sin\theta\cos\theta & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 \\ -\sin\theta\cos\theta & 0 & 0 & 2\cos^2\theta - 1 & \sin\theta\cos\theta & 0 \\ 0 & 0 & 0 & 0 & \pm\cos\theta & \mp\sin\theta \\ 0 & 0 & 0 & 0 & \pm\sin\theta & \pm\cos\theta \end{pmatrix} \begin{pmatrix} \alpha_{xx} \\ \alpha_{yy} \\ \alpha_{zz} \\ \alpha_{xy} \\ \alpha_{yz} \\ \alpha_{zx} \end{pmatrix}$$

The character of the transformation matrix is

$$4\cos^2 \pm 2\cos\theta, \text{ or } 2\cos\theta\,(\pm 1 + 2\cos\theta)$$

+ sign refers to the proper rotations (C_n^k).

- sign refers to the improper rotations (S_n^k), reflection (σ) and inversion (i).

The character $\chi(\alpha)$ of the reducible representation for the polarizability $\Gamma(\alpha)$ is constructed in Table 52. which shows the characters for each operation in T_d .

Table 52. Calculation of $\chi(\alpha)$ for the point group T_d

	E	8 C_3	3C_2	6 S_4	6 σ_d
θ	0	120	180	90	0
$\chi(\alpha) = 2\cos\theta(\pm 1 + 2\cos\theta)$	6	0	2	0	2

To determine how many times each of the irreducible representations Γ_j of T_d occurs in the reducible representations of the polarizability $\Gamma(\alpha)$, the formula $a_j = \frac{1}{h}\sum_R n\, \chi(R)\, \chi_j(R)$ is used as in the procedure previously described.

a_j can be calculated by using the character of the reducible representations in Table 52. and the character of the irreducible representations in Table 51 .

$$a(A_1) = \frac{1}{24}\left[(1)(1)(6)+(8)(1)(0)+(3)(1)(2)+(6)(1)(0)+(6)(1)(2)\right] = 1$$

$$a(A_2) = \frac{1}{24}\left[(1)(1)(6)+(8)(1)(0)+(3)(1)(2)+(6)(-1)(0)+(6)(-1)(2)\right] = 0$$

$$a(E) = \frac{1}{24}\left[(1)(2)(6)+(8)(-1)(0)+(3)(2)(2)+(6)(0)(0)+(6)(0)(2)\right] = 1$$

$$a(T_1) = \frac{1}{24}\left[(1)(3)(6)+(8)(0)(0)+(3)(-1)(2)+(6)(1)(0)+(6)(-1)(2)\right] = 0$$

$$a(T_2) = \frac{1}{24}\left[(1)(3)(6)+(8)(0)(0)+(3)(-1)(2)+(6)(-1)(0)+(6)(1)(2)\right] = 1$$

It is seen that the irreducible representations A_1 ,E and T_2 occur in the reducible representation of the polarizability $\Gamma(\alpha)$, therefore, A_1 ,E and T_2 are Raman active.

APPENDIX B.

The Determination of Unit Cell Parameters by X-ray Powder Diffraction Method.

From Bragg law,

$$n\lambda = 2\, d \sin\theta$$

$$\left(\frac{1}{d}\right)^2 = \left(\frac{2\sin\theta}{\lambda}\right)^2 \qquad (n=1)$$

$$= \frac{4\sin^2\theta}{\lambda^2}$$

In orthorhombic system, the interplanar spacing d_{hkl}, is a function both of the plane indices (hkl), and the lattice constants (a,b,c) as in the following :

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}$$

$$\frac{4\sin^2\theta}{\lambda^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}$$

$$\left(\frac{\sin\theta}{\lambda}\right)^2 = \frac{1}{4}\left(\frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}\right)$$

$$\left(\frac{\sin\theta}{\lambda}\right)^2 = A\, h^2 + B\, k^2 + C\, l^2$$

where

$$A = \frac{1}{4\, a^2}$$

$$B = \frac{1}{4\, b^2}$$

$$C = \frac{1}{4\, c^2}$$

Least Square Method.

$$A h^2 + B k^2 + C l^2 = \left(\frac{\sin\theta}{\lambda}\right)^2 \qquad (1)$$

$$A\,\Sigma h^2 + B\,\Sigma k^2 + C\,\Sigma l^2 = \Sigma\left(\frac{\sin\theta}{\lambda}\right)^2 \qquad (2)$$

(1) x h^2

$$A h^4 + B h^2k^2 + C h^2 l^2 = \left(\frac{\sin\theta\ h}{\lambda}\right)^2$$

$$A\,\Sigma h^4 + B\,\Sigma(h^2k^2) + C\,\Sigma(h^2 l^2) = \Sigma\left(\frac{\sin\theta\ h}{\lambda}\right)^2 \qquad (3)$$

(1) x k^2

$$A h^2 k^2 + B k^4 + C k^2 l^2 = \left(\frac{\sin\theta\ k}{\lambda}\right)^2$$

$$A\,\Sigma(h^2 k^2) + B\,\Sigma k^4 + C\,\Sigma(k^2 l^2) = \Sigma\left(\frac{\sin\theta\ k}{\lambda}\right)^2 \qquad (4)$$

(2) x $\Sigma(h^2 k^2)$

$$A\,\Sigma h^2\,\Sigma(h^2k^2) + B\,\Sigma k^2\,\Sigma(h^2k^2) + C\,\Sigma l^2\,\Sigma(h^2k^2) = \Sigma\left(\frac{\sin\theta}{\lambda}\right)^2\Sigma(h^2k^2) \qquad (5)$$

(3) x Σk^2

$$A\,\Sigma h^4\,\Sigma k^2 + B\,\Sigma k^2\,\Sigma(h^2k^2) + C\,\Sigma k^2\,\Sigma(h^2l^2) = \Sigma\left(\frac{\sin\theta\ h}{\lambda}\right)^2 \Sigma k^2 \qquad (6)$$

(5)-(6)

$$A\left[\Sigma h^2\,\Sigma(h^2k^2) - \Sigma h^4\,\Sigma k^2\right] + C\left[\Sigma l^2\,\Sigma(h^2k^2) - \Sigma k^2\,\Sigma(h^2l^2)\right] = \Sigma\left(\frac{\sin\theta}{\lambda}\right)^2\Sigma(h^2k^2) - \Sigma\left(\frac{\sin\theta\ h}{\lambda}\right)^2\Sigma k^2 \qquad (7)$$

(3) x $\Sigma(k^2 l^2)$

$$A\,\Sigma h^4\,\Sigma(k^2l^2) + B\,\Sigma(h^2k^2)\,\Sigma(k^2l^2) + C\,\Sigma(h^2l^2)\,\Sigma(k^2l^2) = \Sigma\left(\frac{\sin\theta\ h}{\lambda}\right)^2\Sigma(k^2l^2) \qquad (8)$$

(4) x $\Sigma(h^2 l^2)$

$$A\,\Sigma(h^2k^2)\,\Sigma(h^2l^2) + B\,\Sigma k^4\,\Sigma(h^2l^2) + C\,\Sigma(h^2l^2)\,\Sigma(k^2l^2) = \Sigma\left(\frac{\sin\theta\ k}{\lambda}\right)^2\Sigma(h^2l^2) \qquad (9)$$

(8)-(9)

$$A\left[\Sigma h^4\,\Sigma(k^2l^2) - \Sigma(h^2k^2)\,\Sigma(h^2l^2)\right] + B\left[\Sigma(h^2k^2)\,\Sigma(k^2l^2) - \Sigma k^4\,\Sigma(h^2l^2)\right] = \Sigma\left(\frac{\sin\theta h}{\lambda}\right)^2\Sigma(k^2l^2) - \Sigma\left(\frac{\sin\theta k}{\lambda}\right)^2\Sigma(h^2l^2) \qquad (10)$$

$(2) \times \sum(h^2k^2)$

$$A \sum h^2 \sum(h^2k^2) + B \sum k^2 \sum(h^2k^2) + C \sum l^2 \sum(h^2k^2) = \sum\left(\frac{\sin\theta}{\lambda}\right)^2 \sum(h^2k^2) \qquad (11)$$

$(4) \times \sum h^2$

$$A \sum h^2 \sum(h^2k^2) + B \sum k^4 \sum h^2 + C \sum(k^2l^2) \sum h^2 = \sum\left(\frac{\sin\theta\, k}{\lambda}\right)^2 \sum h^2 \qquad (12)$$

$(11)-(12)$

$$B\left[\sum k^2 \sum(h^2k^2) - \sum k^4 \sum h^2\right] + C\left[\sum l^2 \sum(h^2k^2) - \sum(k^2l^2) \sum h^2\right] = \sum\left(\frac{\sin\theta}{\lambda}\right)^2 \sum(h^2k^2) - \sum\left(\frac{\sin\theta\, k}{\lambda}\right)^2 \sum h^2 \qquad (13)$$

For convenience, the various terms are replaced by the following letters;

$\sum h^2$	=	P
$\sum h^4$	=	Q
$\sum k^2$	=	V
$\sum k^4$	=	R
$\sum l^2$	=	T
$\sum (h^2k^2)$	=	U
$\sum (h^2l^2)$	=	W
$\sum (k^2l^2)$	=	X
$\sum \left(\frac{\sin\theta}{\lambda}\right)^2$	=	Y
$\sum \left(\frac{\sin\theta \cdot h}{\lambda}\right)^2$	=	Z
$\sum \left(\frac{\sin\theta\, k}{\lambda}\right)^2$	=	S

So,

(7) is written as:

$$A\left[P U - Q V\right] + C\left[T U - V W\right] = Y U - Z V \qquad (14)$$

(10) is written as:

$$A\left[Q X - U W\right] + B\left[U X - R W\right] = Z X - S W \qquad (15)$$

(13) is written as :

$$B\left[V U - R P\right] + C\left[T U - X P\right] = Y U - S P \qquad (16)$$

Three unknown A, B, C can be found from three equations; 14, 15, 16.

APPENDIX C

Crystal Structures of Potassium Chromate and Potassium Sulphate. (38)

Potassium Chromate

Potassium chromate has an orthorhombic system, space group Pnma (D_{2h}^{16}), four formula units per unit cell (Z= 4).

The unit cell dimensions are :

$\alpha = \beta = \gamma = 90^{o}$

a = 7.61 Å

b = 5.92 Å

c = 10.10 Å

Table 53. Atomic positions and parameters of potassium chromate.

Atom	Position	x	y	z
K (1)	(4c)	0.644	1/4	0.417
K (2)	(4c)	0.000	1/4	-0.305
Cr	(4c)	0.230	1/4	0.417
O (1)	(4c)	0.019	1/4	0.417
O (2)	(4c)	0.300	1/4	0.561
O (3)	(8d)	0.300	0.028	0.345

Potassium Sulphate .

Potassium sulphate has an orthorhombic system, space group Pnma (D_{2h}^{16}), four formula units per unit cell (Z = 4).

The unit cell dimensions are :

$\alpha = \beta = \gamma = 90^{o}$

a = 7.483 Å

b = 5.772 Å

c = 10.072 Å

Table 54. Atomic positions and parameters of potassium sulphate .

Atom	Position	x	y	z
K (1)	(4c)	0.6768	1/4	0.4182
K (2)	(4c)	- 0.0115	1/4	- 0.2954
S	(4c)	0.2358	1/4	0.4155
O (1)	(4c)	0.0315	1/4	0.4032
O (2)	(4c)	0.2970	1/4	0.5579
O (3)	(8d)	0.2997	0.0410	0.3484

Table 55. Structural data and sulphate ion sites in potassium sulphate.

Site	x	y	z
S (1)	0.2358	0.4155	1/4
S (2)	-0.2358	-0.4155	- 1/4
S (3)	0.7358	0.0845	1/4
S (4)	-0.7358	-0.0845	- 1/4

The nearest potassium ions to the sulphur atom at (0.2358, 0.4155, 1/4) in the x, y and z directions are at (0.6768, 0.4182, 1/4), (0.1768, 0.0818, 1/4), and (0.3232, 0.5818, -1/4) with the distance of 3.300 Å, 3.390 Å, and 3.400 Å, respectively. (See Figure 37)

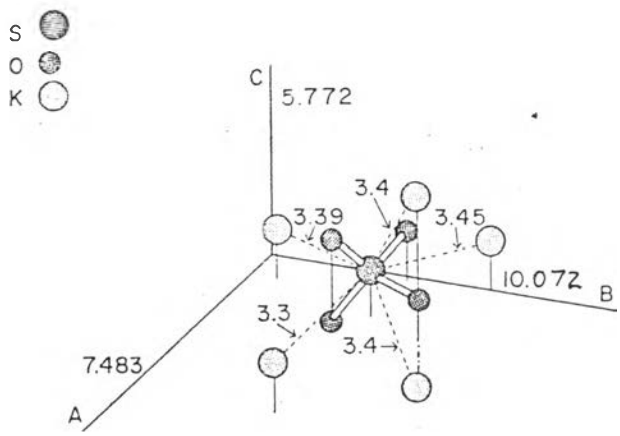

Figure 37 Potassium-sulphur distances of potassium sulphate. (18)

Vita

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