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<u>CHAPTER</u> III

PHOTOLUMINESCENCE

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CHAPTER III : PHOTCLUMINESCENCE

STUDY OF PHOSPHORESCENCE :

3.1 INTRODUCTION :-

According to the theory of Phosphorescence this is electronic process involved in the luminescent material. These electronic processes are significently imfluenced by trapping levels in the phosphor. Phosphorescence decay is a tool to investigate trapping levels in the phosphor and help to understand nature of the trapping states, energy levels and type of kinetics involved in the luminescent process.

In present study Photoluminescence of CaS: Sm : Nd and Gas:Dy:Tb are studied at room temp. (300° k) so as to get information of the following aspects.

- 1) The nature of decay curve.
- 2) Energy distribution of trap levels.
- 3) Decay time constant.
- 4) Kinetics involved in decay mechanism and
- 5) Effect of activetors on decay behaviour.

3.2 THEORETICAL BACKGROUND

3.2.1 DECAY LAWS :

While studing the decay of emission from organic molecules in rigid media Jablonskii (1). Proposed an energy level scheme for emitting centre as shown in fig (1:1). The fluorescence is given by the transitions between the excited state F and the ground state G, but the excited molecules or centre may relax to metastable state M. transition $M \leftrightarrow G$ being forbidden. To return to normal state transition $M \rightarrow F$ must be effected by thermal or optical activation. Thus phospherescence emission due to $F \rightarrow G$ transition is conditioned in rate by optical process $M \rightarrow F$. The energy required for the latter is E. Then the probability of transition occurs per second is given by

 $P = S \exp(-E/KT), ----(3.1)$

Where S is escape frequency factor, E is activation energy (trap depth) and T is the abolute temperature.

According to E.Becquerel (2) phosphorescence decay can be either exponential or hyperbolic as per the kinetic involved in the luminescent process.

A) EXPONENTIAL DECAY :

The electron traps and luminescent centre are situated close to each other in the phosphor. Nature of decay in such phospors is exponential decay or monomolecular decay and kinetics involved in these material is first order kinetics.

for If P is the transition probability \angle return of the centre to ground state when n centres are excited at any instant then $\frac{dn}{dt} = J - Pn$, ------ (3.2).

Where J is the number of excited centres per second by the incident radiation. According to Randall and Wilkins (3,4) The transition probability is given by equation (3.1)

 $P = S \exp (-E/KT)$

and the luminescent intensity I = Pn = solution equation (3.2) $I = I_0 \begin{bmatrix} 1 - exp.(-pt) \end{bmatrix}$ (3.3)

Where I_0 is emission intensity at t = 0. Under steady excitation I = 0 so luminescent decay from equation (3.2) $\frac{dn}{dt} = -pn$ $\frac{dn}{n} = -pdt. by integrating$ $n = n_0 exp. (-pt.) (3.4)$ Here n_0 are number of excited electrons at t = 0The luminescent intensity is given by $I = -\frac{dn}{dt} = -(-pn).$ $I = Pn_0 exp (-Pt).$ $I = I_0 exp (-Pt). (3.4)$ Where $I_0 = Pn_0$ is the intensity (when t = 0) and $P = \frac{1}{7}$,

where T is the life time of the phosphors.

In above equation rate of decay depends on transition probability P. Which is usefull to determine trap depth.

B) HYPERBOLIC DECAY :

This is known as the bimolecular decay of luminescence, exist when electron traps are located away from the luminescence centre and kinetics involved in it is second order.

Let n be the impurity centres with which only n number of electrons combine at a time. If J centres are emptied per second by excitation, the process is given by

 $\frac{dn}{dt} = J - An^2 \qquad ----- (3.6)$ Where A is a constant known as recombination coefficient.
If P is the probability with which an electron combines

with any of the centre in time dt , then

If all electrons return to ground states of the centres through the conduction band with the above said probability P then with J = 0, the rate of decay will be

$$\frac{dn}{dt} = -An^{2}, \text{ on integration.}$$

$$n = \frac{n_{0}}{(1 + n_{0}At)} \qquad -----(3.8)$$
Luminescence intensity I = $-\frac{dn}{dt}$

$$= -(-An^{2})$$

$$= \frac{\frac{n_{0}^{2}A}{(1 + n_{0}At)^{2}}$$

$$= \frac{I_{0}}{(1 + \infty_{0}t)^{2}} \qquad (3.9)$$

Where $\infty = n_0^{\circ} A$ an another constant for large value of t (t>1) above equation becomes.

$$I = \frac{IO}{cc^2 t^2}$$
 (3.10)

Above equation shows decay depends on initial intensity of luminescence, and coefficient A, & temperature.

Treating the process more generally in the light of different capture cross sections for electrons, empty centres and traps, Adirowitch (5,6) arrived at an approximate relation which is of the form given by Becquerel.

$$I = \frac{Constant}{(t + to)} b \qquad (4.11)$$

Where b depends on the ratio of the cepture cross sections of traps and empty centres. However, this equation fails in cases where well separated group of traps exist.

3.2.2 EFFECT OF STEADY EXCITATION OF PHOSPHOR

When the phosphor is excited at a fixed temperature by steady excitation, its luminescent intensity reaches a constant value and the state of the phosphor is referred to as the equilibrium state, and inithis condition the concentration of filled electron traps is in equilibrium. If only a single trap occurs in the phosphor then we have following two cases

A) First Order Kinetics :

Let J be the intensity of excitation. Then the rate of raising of electrons to the excited state is proportional to J, and is given by CJ where C is constant of proportionality. If there are n electrons in N traps, then the rate of capture at excited electrons in traps is CJa (N-n), 'a being the probability of capture per trap. The rate of escape of electrons from traps is given by ns exp (-E / KT). Under the equilibrium condition, the rate of capture of excited electrons in traps and rate of escape of electrons from traps will be equal and thus we get

> CJa $(N - n) = ns \exp(-E/KT) -- (3.12)$ which gives

$$n = \frac{N}{1 + s \exp(-E / KT)} ----- (3.13)$$

Where A = CJa is a factor directly proportional to the intensity of excitation.

B) Second Order Kinetics

Let n be the number of trapped electrons and m be the number in conduction band. Then (m + n) is the number of empty luminescence centre and rate of electrons returning from the conduction band is given by

 $\frac{dm}{dt} = o = cJ - B m (m + n) + ns exp. (-E/KT)$ - mb ((N - m) ---- (3.15))and the rate of the electrons escaping from the traps is $\frac{dn}{dt} = o = ns exp. (-E/KT) - mb (N-n) ---- (3.16)$

Where B and b are constants and CJ is the rate of electrons raising to the excited state. If the excitation is not of an appreciably Low intensity the number of electrons in the conduction band is much greater than the number of trapped ones and thus m + n + n

Hence

$$\beta m^{1} = CJ \text{ and thus}$$

 $n = \frac{N}{1 + S \exp(-E/KT)}$ ------ (3.17)
B

Where $B = b \sqrt{CJ/\beta}$ and thus is proportional to the square root of the excitation intensity.

C) When the traps exist with complex distribution

The equation (3.13) is the rate of capture of electrons excited in traps is equal to rate of escape of electrons from the traps. This show trap depth is nearly constant. Suppose there is variation of the trap depth. Let N(r) is total number of filled traps. So from a given excitation intensity.

$$M_{T} = \int_{0}^{E_{max}} ndE -----(3.18)$$

$$= \int_{1+S}^{E_{max}} dE (3.19)$$

$$= \int_{1+S}^{0} \frac{dE}{A} (3.19)$$

For all the practical excitation intensities $A \leq C$ and thus above equation becomes after integratiion.

 $n_{T} = NKT \log (A^{1} + 1)$ ----- (3.20)

Where $A^{1} = A\overline{S}^{1} \exp(-Emax + Emax \neq KT)$ is proportional to the excitation intensity. At high excitation intensities when, $A^{\frac{1}{2}}$ 1. The number of filled traps will be a linear function of logarithm of the excitation intensities provided that $A \ll S$. When A appreaches S a saturation state will be reached. A similar logarithmic relation can be obtained by using equation. (3.17) which includes consideration of retrapping.

3.3 PHOSPHORESCENCE DECAY FOR DIFFERENT TRAP DISTRIBUTIONS :

(Considering retrapping negligible).

Generally a phosphor centains trap of various depths. If there be N number of traps with activition energies lying between E and E+dE then if we assume that retrapping is negligible, the phosphorescence intensity I at time t and after cessation of excitation is given by

Emax

$$I = \int_{E_{mm}} Ns \exp(-E/KT) \exp(-ST \exp -E/KT) dE ---- (3.21)$$
Emax

$$I = \frac{NS}{\exp(ST)} \left(\exp(-E/KT) \exp(-E/KT) dE ----- (3.22) \right)$$

Emm

If we consider N to be constant over an energy range from Zero to intensity (I) Then as per distribution following three cases possible (a) Uniform trap distribution (b) Quasi uniform trap distribution (c) Experimental trap distribution.

a) Uniform trap distribution :

The distribution of traps is said to be uniform if a phosphor contains an equal number of traps of all the depths in the range of energy zero to infinity. Under this condition the integration of equation (3.22) gives.

$$I = \frac{NKT}{t} \left[1 - \exp(-st)\right]$$

If $St \gg 1$, the intensity after first microsecond above equation becomes.

$$t = \frac{NKT}{t}$$
 ----- (3.24)
= NK t⁻¹ (3.25)
= Const. t⁻¹

Thus phosporescence intensity (I) is inversely proportional to decay time (t) and a plot of I Versus t^{-1} should be a straight line. More over, the plot of log I against log t should be a straight line with a slope equal to -1.

b) Quasi uniform trap distribution :

The phosphors having number of traps N, which donot vary too rapidly with values of E in a complete distribution are said to be having quasi uniform trap distribution. Here the intensity of phosphorescence decay at any time t is proportional to the number of electrons in traps of mean life time given by

 $T = S^{-1} \exp(E/KT)$ ----- (3.24)

From the equation (3.24), the product of $(I_{\rm b})$ is proportional to E. Thus plot of (I-t) against logt for given phosphorescence decay will represent the variation of N with t and gives an approximate idea about the trap distribution.

c) Exponential trap distribution :

If we have N \swarrow exp. (- βE), as found in some

experimental causes, then for $s_{t} > 1$ we have

 $I = const. t^{-} (BKT+1)$ (3.26)

i.e. Power of t varies linearly with the temperature of phosphor. The constant β (which is +ve always) can be found out experimentally and also found from the shape of the thermoluminescence curve.

When BKT=1 the equation (3.26) is correlated with the bimoleculer law and when BKT=0, the case becomes of uniform trap distribution i.e. reciprocal law hold-good. Many Phosphors at room temperature show the decay of the type given by equation (3.26) with any trap distribution over a wide range, have decay is affected by slight variation of temperature. This is because, with rising temperature deeper traps supply electrons at the same rate at which shallow traps supply electrons at room temperature. However at room temperature, at time t , most of the light emission by a phosphor with continuous trap distribution is due to traps in which the electrons will have the mean life time $T = \frac{1}{2}$.

3.4 RESULTS AND DISCUSSION

3.4.1 Decay curves :

Decay curves are plotted after exciting the sample for 200 seconds under ultraviolet source. The typical decay curves for varing percentage of activator and co-activator are (Sm : Nd, Dy : Tb) are shown in figure(3.1 to 3.5). It is seen that for all curves PL intensity falls down rapidly at initial stage and then followed by slow rate of decay. These are same general nature is reported for alkaline earth sulphide phospors by other workers (7,12).

The first step in the analysis of decay curve is to decides the nature of decay is exponential, hyperbolic or power Law, This can be decided by graph (I_{rt}), (log I_{rt}) and (Log I Vs log t).

respectively. These graphy also give information about the order of kinetics.

The posibility of an exponential decay with a single trap depth can be examined with the help of equation (3.5) graph of Log I versus t are plotted. The plots are found to be non linear for all samples, which indicates non exponential decay with single trap. However graph of Log I Vs log t are almost linear for all samples typical graphs are shown in Fig. (3.6 to 3.11), which suggest that the decay may be hyper-bolic or power law. The observed decay can be represented by the equation of the form (3)

$$I = I_0 t^{-D}$$

Where I is intensity at any time t I_0 is intensity at time t=0 and b is the decay constant.

3.4.2 Correlation coefficient :

The degree of linearity between Log I and Log t is confirmed by evaluating correlation coefficient (r) using the equation.

$$r = \frac{N \sum x y - \sum x \cdot \sum y}{\left(N \sum x^{2} - (\sum x)^{2}\right)^{\frac{1}{2}} \left[N \sum y^{2} - (\sum y)^{2}\right]^{\frac{1}{2}}} ---(4.27)$$

Where $x = \log t$ and $y = \log t$ for N set of observations. The magnitude of r signifies the closeness of the relationship while its sign indicates whether y increases or decreases with x. When the relationship is linear, the values or r equal to unity.

In present investigations, the values of r for all the samples are calculated and are very close to -1 (table 3.1). The negative sign or r implies that intensity diminishes with time

3.4.3 Decay constant

The decay rate is generally indicated by the value of decay constant (b). Faster the decay higher is value of decay constant

and vice-versa It also provides the information about the relative population of traps at various depths.

The value of decay constant is calculated by the method of least square (13) using the relation.

$$b = \frac{N\Sigma(x \cdot y) - \Sigma x \cdot \Sigma y}{N\Sigma(x) 2 - (\Sigma x)^2}$$

Where $x= \log t$ and $y=\log I$ and N being number of observations. Table (3.1) shows the values of decay constants calculated using above formula. The same table includes the value of b obtained from the slopes of staight lines on log I - log t plots. It is seen that both these values are in good agreement with each other.

3.4.4 <u>Variation of decay constant with the percentage of activator</u> and co-activator :

The variation of decay constant (b) with percentage of Nd in host material [by keeping suitable percentage of Sm activator] is plotted, fig.(3.12) which shows bhatthere is no relation between decay constant and percentage of activator. Similarly decay constant and percentage of activator Dy by keeping suitable percentage of co-activator Tb constant plot in fig. (3.12) shows there is no relation between decay constant and activator. 3.4.5 ' Peeling off ' of decay curves :

The decay in present investigation could be represented by the equation $I = I_0 t^{-b}$. The values of b from calculated as well from the graph is not equal to 2 or exactly equal to 1 for $t \gg 1$, as required by equation 4.10. This excludes the possiblity of hyperbolic decay with single trap depth. However, the observed power Law decay can be explained on the basis of mono molecular super position theory suggested by Randall and Wilkin (3) and followed by other workers(14,15,16) According to this such a decay results due to the superposition of various exponentials corresponding to different traps and is expressed by the equation such as

$$I = I_0 t^{-D}$$

= $I_{01} \exp(-P_{1}t) + I_{02} \exp(P_{2}t) + I_{01} \exp(-P_{1}t)$

Where $I_{o_1} I_{o_2}$ are starting contributions to luminescence intensity first, second ..., th exponential components and $P_1 = S \exp(-E_1/KT)$, $P_2 = \exp(-E_2/KT)$ $P_n = S \exp(-E_n/KT)$ are the transition probabilities of an electron escaping from the traps of depth $E_1 E_2$ En respectively.

It is possible to split each decay curve into a set of exponentials by the method of successive substraction as suggested by Bube (17) and others(18,19,20). In present investigation the same procedure is adopted and the decay curves were split into three exponentials fig. (3.13 to 3.18). The activation energies (trap depths) corresponding to these exponentials are calculated by using the slopes P_1, P_2, P_3 of straight lines on semilog graph between I and t. The values of E thus evaluated for slowest, middle and fastest exponentials are denoted by E_1, E_2 and E_3 respectively and are shown in table (3.2). The value of S used in the above calculations were obtained from the thermoluminescence studies of the sample. (estimated using Randall and Wilkin's formula) described in chapter W (Ref. table 4.7 and 4.8).

3.4.6 Life times and starting contribution :

A) Life times : The slopes P_1 , P_2 , P_3 of three straight lines on semilog graph between I and t represents the transition probabilities. The reciprocal of these transition probabilities gives the values of

life times T_1 . T_2 and T_3 respectively of trapped electrons contributing to slowest, Middle and fastest exponential components respectively. Which are shown in table (3.2).

B) <u>Starting contributions</u>: Three straight lines into which the semilog plot is resolved are extrapolated to cut the intensity axis. The values of I corresponding to these points. I I and I of 0_2 of 0_3 represents the starting contribution to intensity at t = o second by slowest, middle and fastest exponentials.

3.4.7 Activition Energy :

The values of trap depth calculated for all samples (table 3.2) for slowest vary from .57 to .63 @vfor Middle. .48 to .53 ev. and .43 to .44 ev for fastegt exponential.

3.4.8 Variation of activation energy with percentage of RE₄ and RE₂ in host material :

From table (3.2), it is seen that there is no significant change in trap depth with activator concentration. This suggests that change of percentage of RE_p, RE₂ (Cas:Sm:Nd, Cas; Dy:Tb) does not introduce any new trapping levels. The observed minute changes in the value of E could be due distribution of trap densities, (10,18,19)

3.499 Kinetics of Luminescence :

The kinetics involved in the decay process may be either monomolecular (fist order), bimodecular (second order) or intermediate, depending upon relative location of traps and luminescence centres. The monomolecular decay is exponential represented by equation (3.5). The bimolecular process involves the hyperbolic decay given by the equation (3.9) while the power Law decay ($I = I_0 / (1 + \frac{t}{T})^{-1}$) results when process is neither first nor second order. Thus a straight line on semilog plot between I and t represents a first order Kinetics where as a straight line between reciprocal of square root of intensity and time indicates second order kinetics. Further for second order and intermediate kinetics a plot between log I and log t assumes a slope of -2 and -1 respectively at large value of t An examination of decay obtained in present investigation reveals that they are neither exponential (fig.3.13 to 3.18) nor hyperpolic fig.(3.19) and slope of log I versus log t \neq -2. However, the decay can be represented by a powerlaw equation of the type I = $I_0 \stackrel{-b}{t_0}$. But the value of b is not exactly equal to unity as required by intermediate process resulting from traps of single depth. Which excludes the possibility of inter-mediate kinetics. On the other hand, the observed power law decay could be well explained on the basis of monomolecular superposition of theory suggesting thereby the kinetics involved is likely to be monomolecular.

3.4.10 Distribution of traping levels :

The trapping levels in a phosphor are distributed in their depths. The distribution may be uniform, Quasi-uniform or exponential. (3) for the present system the distribution is ascertained as follows:

For uniform trap distribution, over a wide range, the relation between phosporescence intensity I and t is given by the equation (3.23); Viz.

$$I = \frac{NKT}{t} \left[1 - \exp(-st) \right]$$

Phosphorescence, in present case lasts for few minutes and frequency factor S, is of the order of 10^9 sec^{-1} . Hence St>> 1 and above equation reduces to

 $I = \frac{NKT}{t}$ $= NKT t^{-1}$ $= I_0 t^{-1}$

The relation is similar to $I = I_0 t^{-b}$, and for uniform trap distribution b should be unity. However observed values of b are not exactly equal to unity but are close to unity (Table 3.1) This indicates that the trap distribution is not uniform but probably quasi uniform. Also referring to equation. (3.26) I= constant $t^{-(BKT+1)}$ for an exponential trap distribution b = (BKT+1) which means b> 1 for the proposed samples b fluctuates (.81 to 1.19). The plot I verses t^{-1} fig.(3.20) for Cas:Sm:Nd and plot(3.21) Cas:Dy:Tb are not exactly straight line. Which rules gut the possibility of exponential trap distribution.

Above conclusion is supported by a plot between I.t and log t obtained for few samples The plots are neither parallel to log t axis nor exponential as required by uniform and exponential trap distribution respectively (fig.3.22) i.e for CaS:Sm:Nd(I.t)increases attainsmaximum value and decreaseand becomes parellel to logt -axis, while in fig (3.23) Cas:Dy:Tb : I.t decreases and becomes parallel to log t axis, suggesting here by the trap distribution to be non uniform. The curve also gives an idea about the density of traps at various depths. Since(I.t) is a measure of trap density and log t is of trap depth.

3.5 SUMMARY :

The important features of this chapter are to be summarised as follows :

1) Phosporescence decay observed for integrated intensity is of the form $I=I_0 t^{-b}$.

2) Nature of decay is of power law type and can be explained in terms of superposition of various exponentials of mono-molecular type.

3) The value of decay constant is not exactly equal to unity which excludes the possibility of the uniform trap distribution. The distribution may be quasi-uniform.

4) The trap depth of effective level are relatively insensitive to the percentage of activators. (Sm:Nd, Dy :Tb).
 But there is no relation between them.

5) The probable kinetics of decay process is likely to be monomolecular.

6) Percentage of activators (Sm : Nd , Dy : Tb)influences the distribution of trap densities.

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TABLE 3.1 : DECAY CHARACTERISTICS OF SAMPLES STUDIED 3000k

Sample No.	'Co-relation	Decay Con	stant b (-ve)
	co-efficient (r-Ve)	Calculated	. Slope of graph
KD 13	1.01	1.09	1.06
KD 14	.99	1.13	1.12
KD 15	.99	1.05	1.00
KD 16	.99	1.05	1.00
KD 17	.99	1.06	1.02
KD 18	.99	.91	.91
KD 19	.98	.60	.70
KD 20	.96	1.05	1.05
KD 21	.99	1.11	1.06
KD 22	1.00	1.10	1.06
KD 23	.94	1.05	1.04
KD 24	.99	1.01	1.00
KD 25	.99	1.19	1.14
KD 26	.99	1.02	1.00
KD 27	.99	.91	.95
KD28	.98	.94	.95
KD 29	.99	1.10	1.05
KD 30	.99	1.10	1.03
KD 31	.99	1.10	1.10
KD 32	.99	1.01	1.05
KD 33	.99	1.02	1.02
KD 34	.99	1.01	1.00
KD 35	.99	1.01	1.02
KD 36	.99	.88	.83
KD 37	.99	.77	.86
KD 38	.99	1.03	1.05
KD 39	.99	1.02	1.00
KD 40	.98	1.03	1.04
KD 41	.99	1.03	1.06
KD 42	.99	1.07	.99
KD 43	.99	1.10	1.10
KD 44	.99	1.10	1.10

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Sample No.	Slowest exponential		Middle Exponental		Fastest exponental	
	E ₁ (ev)	T ₁ sec	E ₁ (ev)	T ₂ sec	E ₂ (ev)	T2Sec.
KD13	.61	16.9	.52	.41	.43	.015
KD14	.63	27.7	.52	.42	.43	.015
KD15	.62	22.7	.52	.46	.43	.015
KD16	.63	26.3	. 52	.42	.43	.015
KD17	.61	13.3	.52	.37	.43	.015
KD18	.66	90.9	.51	.23	.49	.015
KD19	.62	45	.49	.24	.43	.015
KD 2 0	.64	22.7	.51	.31	.43	.015
KD21	.62	16.6	.51	.111	.43	.015
KD22	.60	11.3	.51	.3	.43	.015
KD23	.62	21.2	.50	.24	.43	.015
KD24	.60	10	.51	.27	.43	.015
KD25	.62	19	.51	.27	.43	.015
KD26	.62	13.8	.53	.5	.44	.015
KD27	.61	12.6	.51	.29	.43	.015
KD 28	.62	17.2	.51	. 25	.44	.015
KD29	.62	21.2	.53	.56	.44	.015
KD 3 0	.62	13.3	.52	.36	.44	.015
KD 31	.62	19.2	.53	.06	.44	.015
KD 32	.60	8.4	.52	.44	.44	.015
KD 33	.63	25	.52	.71	.44	.015
KD 34	.62	15.7	.52	.71	.44	.015
KD 35	.62	15	.52	.31	.43	.015
KD 36	.60	7.9	.51	.23	.44	.015
RD 37	.60	7.0	.51	.24	.43	.015
KD 38	.63	25.7	.52	.48	.43	.015
KD 3 9	.63	22.7	.52	.47	.44	.015
KD 4 0	.59	6.9	.48	•1	.43	.015
KD 41	.57	2.6	.50	.21	.44	.015
KD 42	.59	4.3	.51	.3	.44	.015
KD 43	.61	5.01	•53	.12	.43	.015
KD44	.57	2.5	.50	.2	.43	.015

TABLE 3.2 : DECAY CHARACTERISTICS OF SAMPLES STUDIED AT 3000K

Trap depth and life time from peeling off decay curves

















3 2 log I 1 KD₂₇ KD₂₈ 2 (0,0) 1 log t OF log I Vs log t FIG. NO: 3.9. PLOT































