CONSTRUCTION OF A THERMOLUMINESCENCE READOUT INSTRUMENT

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By

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ABSTRACT

CONSTRUCTION OF A THERMOLUMINESCENCE READOUT INSTRUMENT

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In this study, a double beam spectrophotometer system and an electrical furnace was used as a thermoluminescence reader to detect the visible light emitted from a heated themoluminescence dosemeter (TLD) material. Samples of TLD material (natural CaF_2-17) were placed in the electrical furnace and heated to the desired temperature (about 400 ℃). The light emitted by TLD samples during heating was detected and measured by a spectrophotometer. An X-Y recorder used to plot data as a function of temperature. The same procedure was applied to other samples which were exposed to different intensity of X-ray radiation.

Finally, glow curves, dose response curves and spectrum of the emitted light were obtained from the output of the spectrophotometer and the results are discussed.

Keywords: TLD Instrument, spectrophotometer, dosemeter.

OZET

BIR TERMOLUMINESANS OKUYUCU YAPILMASI

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Bu çalışmada, çift ışıklı spektrofotometre ve bir elektrik fırını kullanılarak bir termoluminesans okuyucu tasarlandı ve geliştirildi. Bu okuyucu ısıtılan dozimetreden yayılan görünür bölgedeki ışınların yoğunluğunu ölçmede kullanıldı. Burada dozimetre malzemesi olarak doğal CaF_2 -17 kullanıldı ve bu madde fırının içine yerleştirilerek ışıma yapması için gerekli olan ısıya kadar ısıtıldı (400 °C civarı). Isınma esnasında dozimetreden yayılan ışınların yoğunluğu spektrofotometre ile ölçüldü. Ölçüm sonuçları, bir X-Y kaydedici kullanılarak ısının fonksiyonu olarak çizildi. Dozimetre farklı şiddetlerde X-ışınlarına maruz bırakılarak değişik ölçümler yapıldı.

Sonuç olarak, kararlılık eğrileri, doza bagımlılık eğrileri ve dozimetreden yayılan ışınların tayfları spektrofotometrenin çıkışından elde edildi ve sonuçlar tartışıldı.

Anahtar sözcükler: Termoluminesans dozimetre aleti, spektrofotometre, dozimetre.

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TABLE OF CONTENTS

PAGE

Abstract iii

Ozet iv

Acknowledgement v

List of Tables viii

List of Figures ix

1. INTRODUCTION 1

2. PRINCIPLES AND METHODS OF THERMOLUMINESCENCE 2

2.1 INTRODUCTION 7

2.2 THE THEORY OF THERMOLUMINESCENCE 8

2.3 GLOW CURVES 11

2.3.1 Introduction 11

2.3.2 The Randall-Wilkins Model 16

2.3.3 Seitz Model 16

2.4 DETERMINATION OF TRAPS DEPTS METHODS 17

2.4.1 Peak Temperature Method 17

2.4.2 Trap Energy Determination method 17

3. THERMOLUMINESCENCE DOSIMETRY (TLD) 21

3.1 INTRODUCTION 21

3.2 REQUIREMENTS OF A SUITABLE THERMOLUMINESCENCE MATERIAL 27

3.3 EMISSION SPECTRA OF THERMOLUMINESCENCE MATERIALS 29

4. THERMOLUMINESCENCE INSTRUMENTATION 31

4.1 INTRODUCTION 31

4.2 READOUT INSTRUMENT 35

4.2.1 Ultraviolet and Visible Spectrophotometer 35

4.2.1.1 System of The Ultraviolet and Visible Spectrophotometer 38

4.2.1.2 Program Structure 39

4.2.2 Heater 42

5. EXPERIMENTS AND RESULTS 43

6. DISCUSSIONS AND CONCLUSION 107

REFERENCES

LIST OF TABLES

Table Page

5.1. For the sample CaF_2 ; 500 nm. 102

5.2. For the sample CaF_2 ; 430 nm. 102

LIST OF FIGURES

Figure Page

2.1. Thermoluminescence, the return of electrons trapped in the metastable state is speeded up by heating the material. 9

2.2. Energy-band scheme for electrons. 10

2.3. (a) Schematic diagram of excitation and thermal stimulation in a phosphor.(b) Typical TL glow curve of CaF_2 (Mn) phosphor. 15

2.4. Typical glow curves for varying amounts of radiation from 1(minimum) to 4(maximum). 15

3.1. Schematic energy level diagram of a phosphor exhibiting thermoluminescence.(a) Exposure to ionising radiation.(b) Electron trap is less stable during heating, and hole trap is the emitting centre.(c) Hole trap is less stable during heating, and electron trap is the emitting centre. 25

4.1. Basic thermoluminescence dosimetry reader. 34

4.2. Block diagram of thermoluminescence readout instrument. 36

4.3. Readout instrument. 37

4.4. Main unit. 37

4.5. Block diagram of the system. 38

5.1. Thermoluminescence light emission spectrum of the main TL peaks in CaF_2 44
5.2 Glow curve of CaF_2 curve B is a second experiment on the same sample after all TL has been removed. The wavelength of the light is 500 nm. 45
5.3. Glow curve of CaF_2 ; 0.479 mg, 500 nm. 46
5.4. Glow curve of CaF_2 ; 0.290 mg, 650 nm. 47
5.5. Glow curve of CaF_2 ; 0.300 mg, 650 nm. 48
5.6. Glow curve of CaF_2 ; 0.320 mg, 650 nm. 49
5.7. Glow curve of CaF_2 ; 0.345 mg, 650 nm. 50
5.8. Glow curve of CaF_2 ; 0.370 mg, 650 nm. 51
5.9. Glow curve of CaF_2 ; 0.375 mg, 650 nm. 52
5.10. Glow curve of CaF_2 ; 0.390 mg, 650 nm. 53
5.11. Glow curve of CaF_2 ; 0.380 mg, 500 nm. 54
5.12. Glow curve of CaF_2 ; 0.434 mg, 500 nm. 55
5.13. Glow curve of CaF_2 ; 0.450 mg, 500 nm. 56
5.14. Glow curve of CaF_2 ; 0.460 mg, 500 nm. 57
5.15. Glow curve of CaF_2 ; 0.450 mg, 500 nm. 58
5.16. Glow curve of CaF_2 ; 0.460 mg, 500 nm. 59
5.17. Glow curve of CaF_2 ; 0.300 mg, 460 nm. 60
5.18. Glow curve of CaF_2 ; 0.270 mg, 460 nm. 61
5.19. Glow curve of CaF_2 ; 0.255 mg, 460 nm. 62
5.20. Glow curve of CaF_2 ; 0.210 mg, 460 nm. 63
5.21. Glow curve of CaF_2 ; 0.195 mg, 460 nm. 64
5.22. Glow curve of CaF_2 ; 0.170 mg, 460 nm. 65
5.23. Glow curve of CaF_2 ; 0.215 mg, 430 nm. 66

5.24. Glow curve of CaF_2 ; 0.294 mg, 430 nm. 67

5.25. Glow curve of CaF_2 ; 0.250 mg, 430 nm. 68

5.26. Glow curve of CaF_2 ; 0.150 mg, 430 nm. 69

5.27. Glow curve of CaF_2 ; 0.192 mg, 430 nm. 70

5.28. Glow curve of CaF_2 ; 0.100 mg, 500 nm. After exposure to X-ray radiation. 71

5.29. Glow curve of CaF_2 ; 0.217 mg, 500 nm. After exposure to X-ray radiation. 72

5.30. Glow curve of CaF_2 ; 0.245 mg, 500 nm. After exposure to X-ray radiation. 73

5.31. Glow curve of CaF_2 ; 0.274 mg, 500 nm. After exposure to X-ray radiation. 74

5.32. Glow curve of CaF_2 ; 0.340 mg, 650 nm. After exposure to X-ray radiation. 75

5.33. Glow curve of CaF_2 ; 0.345 mg, 650 nm. After exposure to X-ray radiation. 76

5.34. Glow curve of CaF_2 ; 0.380 mg, 650 nm. After exposure to X-ray radiation. 77

5.35. Glow curve of CaF_2 ; 0.390 mg, 650 nm. After exposure to X-ray radiation. 78

5.36. Glow curve of CaF_2 ; 0.340 mg, 650 nm. After exposure to X-ray radiation. 79

5.37. Glow curve of CaF_2 ; 0.410 mg, 650 nm. After exposure to X-ray radiation. 80

5.38. Glow curve of CaF_2 ; 0.332 mg, 460 nm. After exposure to X-ray radiation. 81

5.39. Glow curve of CaF_2 ; 0.365 mg, 460 nm. After exposure to X-ray radiation. 82

5.40. Glow curve of CaF_2 ; 0.385 mg, 460 nm. After exposure to X-ray radiation. 83

5.41. Glow curve of CaF_2 ; 0.400 mg, 460 nm. After exposure to X-ray radiation. 84

5.42. Glow curve of CaF_2 ; 0.245 mg, 430 nm. After exposure to X-ray radiation. 85

5.43. Glow curve of CaF_2 ; 0.255 mg, 430 nm. After exposure to X-ray radiation. 86

5.44. Glow curve of CaF_2 ; 0.272 mg, 430 nm. After exposure to X-ray radiation. 87

5.45. Glow curve of CaF_2 ; 0.300 mg, 430 nm. After exposure to X-ray radiation. 88

5.46. Glow curve of teflon dosimeter ; 430 nm. After exposure to X-ray radiation. 89

5.47. Glow curve of teflon dosimeter ; 430 nm. After exposure to X-ray radiation. 90

5.48. Glow curve of teflon dosimeter ; 430 nm. After exposure to X-ray radiation. 91

5.49. Glow curve of teflon dosimeter ; 430 nm. After exposure to X-ray radiation. 92

5.50. Glow curve of teflon dosimeter ; 460 nm. After exposure to X-ray radiation. 93

5.51. Glow curve of teflon dosimeter ; 460 nm. After exposure to X-ray radiation. 94

5.52. Glow curve of teflon dosimeter ; 500 nm. After exposure to X-ray radiation. 95

5.53. Glow curve of teflon dosimeter ; 500 nm. After exposure to X-ray radiation. 96

5.54. Glow curve of teflon dosimeter ; 500 nm. After exposure to X-ray radiation. 97

5.55. Glow curve of teflon dosimeter ; 650 nm. After exposure to X-ray radiation. 98

5.56. Glow curve of teflon dosimeter ; 650 nm. After exposure to X-ray radiation. 99

5.57. Glow curve of teflon dosimeter ; 650 nm. After exposure to X-ray radiation. 100

5.58. Glow curves of CaF_2 , exposed to X-ray radiation. 103

5.59. The dose response curve of CaF_2 . 104

5.60. Glow curves of CaF_2 , exposed to X-ray radiation. 105

5.61. The dose response curve of CaF_2 . 106

CHAPTER 1

INTRODUCTION

Materials and substances that are capable of emitting light, particularly in the visible range, are termed 'luminescent'. There is a wide variety of such substance : minerals, crystals, chemical substance, and bio-materials of plant and animal origin, and they can be natural or manufactured in laboratories. The underlying phenomenon that causes these materials to luminesce is called luminescence.

Luminescence, unlike natural radioactivity, does not occur spontaneously but, generally, requires energy to excite it. The excitation can be caused by ultraviolet light, X-rays, gamma rays, electrons, or alpha particles, all of which can deposit energy into the material that is subsequently re-emitted in the form of visible light. Luminescence phenomenon essentially involve electronic transition of the atom or a molecule in a solid, so that the absorbtion and release of energy is a quantum process. Application of an electric field or even some mechanical stress can also energies electrons or ions.

There are numerous luminescent materials but not all are efficient enough to be put to practical use. The more efficient materials are those which are prepared in laboratories and have a specific chemical composition. These materials exhibit a degree of repeatability and reliability in their performance which qualifies them for practical application.

These materials are exhibited when the material is very hot. Heating agitates the atoms strongly and, by collision with the neighbouring atoms, results in the excitation and subsequent emission of light. To begin with, this radiation is in the invisible far infrared but, at higher temperatures, shifts to the visible region.

A systematic scientific study of the subject of luminescence is, however, of recent origin, dating approximately from the middle of the nineteenth century. The application of luminescence in general and of thermoluminescence in particular,is of still more recent origin. The bulk of investigations leading to various applications have been carried out within the past 25 years or so.

In 1852 the English physicist G.G Stokes identified this phenomenon and formulated his law of luminescence which states that the wavelength of the emitted light is grater than that of the exciting radiation. It was the German physicist E. Weidemann who, in 1888, introduced the term "luminescence" for the observation of excess emission over and above the thermal background. Experimental techniques for the study of emission spectra, efficiency of excitation, and duration of luminescence after glow, were initiated by A.E. Becquerel.

The release, in the form of light, of energy already stored in a phosphor when it is heated is the phenomenon of thermoluminescence. In other words, a thermoluminescence phosphor has been pre-excited by some suitable radiation (ultraviolet, nuclear, cosmic rays) whose energy is retained within the sample; when the phosphor is heated to an appropriate temperature, this energy is released as visible light. The emitted light can easily be detected by a spectrophotometer and associated electronic equipment. Robert Boyle seems to have observed such an emission in diamond as early as 1663. A proper understanding of the thermoluminescence phenomenon developed only after more than two centuries of experimental investigation. In thermoluminescence methods, the phosphorescent solids, after excitation, are heated and the corresponding intensity of the emitted light is recorded as the temperature increases at a linear rate. It is as if the excitation energy remains 'frozen' in a thermoluminescent material until heating stimulates its release in the form of light.

A new instrumental techniques for the determination of thermoluminescent glow curves, especially useful for experimental investigations of the thermoluminescence measurements.

Thermoluminescence measurements provide different kinds of basic information on defects in a large variety of crystals. Until recently, almost all thermoluminescence measurements were made by focusing the light emitted by a sample directly on a photomultiplier tube. But, in this work, the thermoluminescence measurements were made by modifying the spectrophotometer instrument where response changes rapidly with wavelength.

This thesis will describe one of the initial studies utilizing recently developed equipment for measuring emission intensity simultaneously as a function of both light wavelength and sample temperature. One of the principal objectives is the construction of intensity-vs-temperature curves, or glow curves of the thermoluminescence materials by modifying the spectrophotometer instrument.

The thermoluminescence material has studied by using modifyed spectrophotometer instrument determining simultaneously the emission spectrum as a function of sample temperature. The recorded data for each emission spectrum is dependent on the wavelength of the emitted light.

The use of a single photomultiplier tube improves the analytical results, since the differential signals are incident on the same area of the photomultiplier they are subject to the same photomultiplier sensitivity to voltage and temperature changes. It is to be noted that when this differential thermoluminescence technique is employed, it requires no optical filters and there is no need to choose special photomultipliers, such as those that have low response in the red region of the spectrum. In fact, if a rapid scanning spectrophotometer is used with a red - sensitive photomultiplier, this apparatus should give clear information as to the existence and extent of any emissions in this region of the spectrum especially at higher temperature [1].

Thermoluminescence and emission spectra from LIF (TLD-

100) have been studied over the temperature range from 90 to 500 K. Maximum emission spectrum occur at 270 nm and 420 nm [2]. This occurs when the thermoluminescence spectrum changes with temperature, especially when the emission occurs in a region where the photomultiplier tube sensitivity changes rapidly with wavelength. Also, it can occur when the emission contains more than one component and glow peaks represent contributions from undetermined number of emission bands. For these, as well as other reasons, the apparatus mentioned above was developed to determine thermoluminescence intensity simultaneously as a function of both light wavelength and sample temperature [3].

CHAPTER 2

PRINCIPLES AND METHODS OF THERMOLUMINESCENCE

2.1. INTRODUCTION

When a solid material such as limestone or granite is heated in dark place, it is usually observed to emit light during the period of rising temperature. If the material is held at constant temperature, the intensity of luminescence decreases about 90 percent per minute, the rate depending on the temperature. The intensity of the radiation depends upon the sample, the temperature, and the rate of rise of temperature. A rock taken from the ground gives luminescence on first being heated but gives no luminescence if cooled and heated again. If, after having been once heated, the rock is exposed to high-intensity X-rays or gamma rays, the thermoluminescent property is restored. The first heating after X-rays irradiation produces luminescence, and a later heating with no intervening irradiation produces no luminescence.

Some minerals, when from ground, have colour which disappears upon heating. These minerals have both their thermoluminescent property and their colour restored by X-ray or gamma ray irradiation.

2.2. THE THEORY OF THERMOLUMINESCENCE

When a dielectric solid is exposed to ionizing radiation or ultraviolet light, or when it undergoes certain chemical reactions or mechanical stress, a varying percentage of the liberated electrons (or holes) may become trapped at certain imperfections in the lattice which are called electron (or holes) traps. If the temperature is low and/or the traps are deep enough, they may remain trapped for hundreds or thousands of years before they are released by a sufficient "stimulation", which increases their probability of escaping from the traps. Stimulation usually consists of the transfer of optical (light) or thermal energy to the dielectric.

The return of the electrons (or trapped holes) to the stable state, which is called annealing (or fading if it proceeds slowly), is associated with a release of energy, most of which is thermal and results in a minute temperature increase during annealing. A small fraction of the energy, however, may be released as visible or ultraviolet light, which can be observed as a transient "glow" of the pre-irradiated crystal or glass at a characteristic temperature during heating. This effect is called Thermoluminescence (TL), (illustrated in figure 2.1). If several traps of different depths are involved in the process, the recording of the light emission as a function of heating time (or temperature) results in a "glow curve" [10].

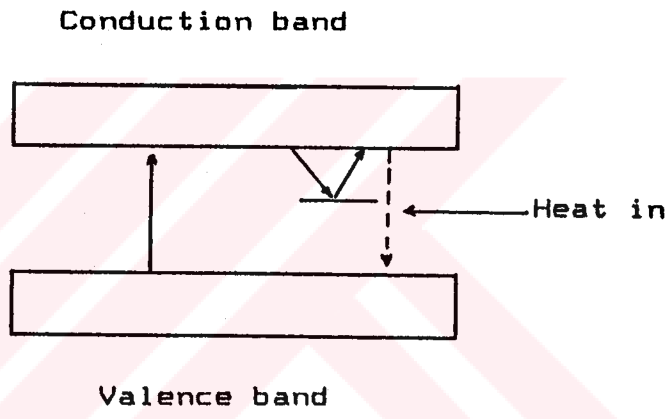


Figure:2.1 Thermoluminescence, the return of electrons trapped in the metastable state is speeded up by heating the material [10].

The production of thermoluminescence in a material by exposure to ionising radiation may be divided into two stages: (i)ionization and electron trapping, and

(ii) electron and hole recombination with photon emission.

Figure 2.2 illustrates the energy band configuration for electrons.

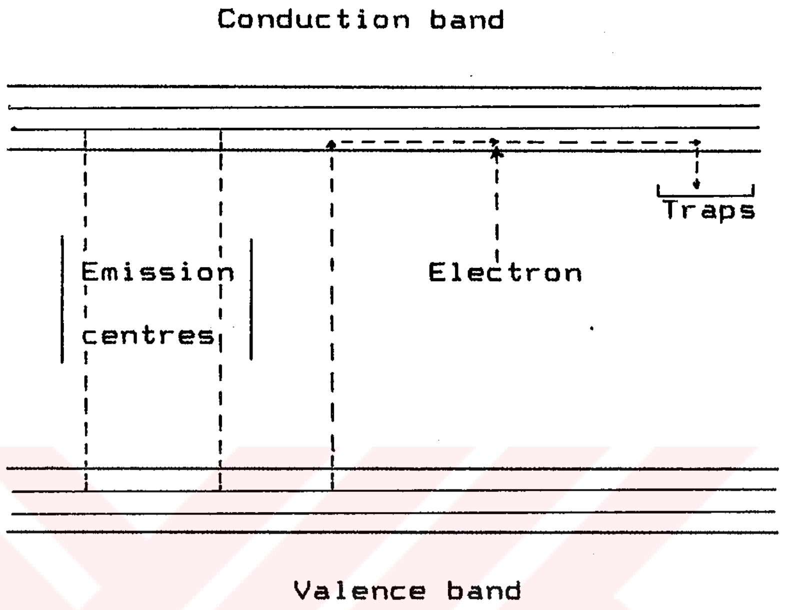


Figure:2.2 Energy-band scheme for electrons [7].

The electrons occupy fixed positions with energies in the normally filled bands. Irradiation with X-rays or gamma rays changes the electrons to a higher energy conduction band. Electrons with energy in the conduction band move through the crystal until they become bound into electron traps, by radiation.

The electrons remain in the traps until they are transferred back to the conduction band through an elevation in temperature of the crystal. Once again in the conduction band, electrons move through the crystal until they reach an emission centre. Then they can lose energy by radiation and become fixed in the crystal at the point of the emission centre, with the gradual increase in temperature the higher-energy electron traps are emptied first, giving rise to the low-temperature peaks in the glow curves. The lower-energy electron traps become emptied the higher temperatures, giving rise to the high-temperature peaks.

The trapped electrons remained in their traps provided that they do not require sufficient energy to escape. This can be determined by two main factors: the depth of the traps, and the temperature of the material.

2.3. GLOW CURVES

2.3.1. INTRODUCTION

Radiation of insulators containing electron as well as hole trapping levels, carriers of both types become mobile in their respective bands, which are then eventually trapped into these levels. These trapped carriers can be released if thermal energy is supplied; this stimulates the carriers to cross the potential barrier of these traps and, in the process, allows them to move to the suitable recombination centres that contain a hole or an electron, resulting in the emission of light. This process of light emission by thermal stimulation is called 'thermally stimulated luminescence' (TSL) [4], although the older but less justified name 'thermoluminescence' (TL) still permits and is more often used. If the probability of transition from a trap to the band is high even at room temperature, the well known phosphorescence process of crystals is observed.

The study of these phenomena is based on models that describe the number of charge carriers present in the traps and centres relative to time and temperature. In order to know the charge carrier population, Boltzmann's statistics are usually applied and probabilities of all possible transitions are deduced. A simple approximate model is that of Randall and Wilkins [5], which assumes that all traps are located at the same level.

At temperature T, the re-trapping of the carriers depends upon the occupied trap density n and the potential barrier (known as the thermal activation energy) E of these traps. The rate of re-trapping, dn/dt, can be expressed as

$$-\frac{dn}{dt} \sim n e^{(-\frac{E}{k_B T})} \qquad 2.1$$

$$-\frac{dn}{dt}=sne^{(-\frac{E}{k_BT})} \qquad 2.2$$

where s is a constant known as the frequency factor and k_B is the Boltzman constant (=0.862 x 10^{-4} eV.K^{1}). Note the dimensionality of s which is time^{-1} ; hence the name frequency factor.

The plot of the thermoluminescence intensity as a function of rising temperature of the thermoluminescence phosphor being heated (at constant rate of increase of temperature, dT/dt=q; constant) exhibits one or more peaks, known as glow peaks.The analysis of these glow curves provides information on:

(i) trap depts (i.e. thermal activation energy corresponding to each glow peak);

(ii) electron/hole trap densities ;

(iii) frequency factor , s ;

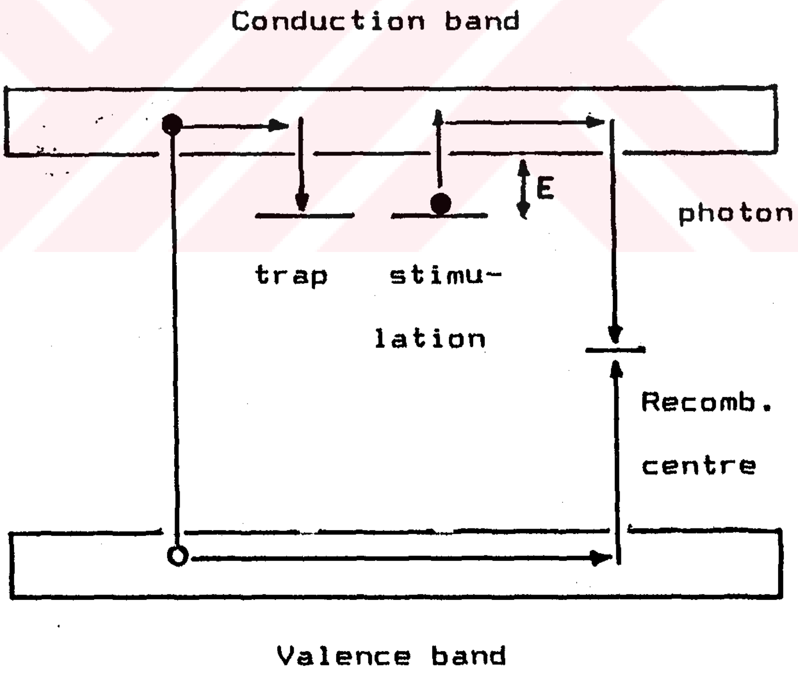
(iv) carrier mobility , and

(v) capture cross section of traps.

In addition to the glow curves if an emission spectrum (thermoluminescence intensity versus wavelength) is also recorded then the nature and characteristics of luminescent centres can also be studied.

The shape of the glow curves depends upon the chemical nature of the crystal, the impurities in it, the number of imperfections in the crystal lattice, and the history of the crystal with respect to X-ray or gamma irradiation [10].

Increasing the quantity of radiation causes a rise, then a fall, in the intensities of some peaks but may result in the stimulation of an additional higher-temperature peak as shown in figure 2.4 [7].



(a)

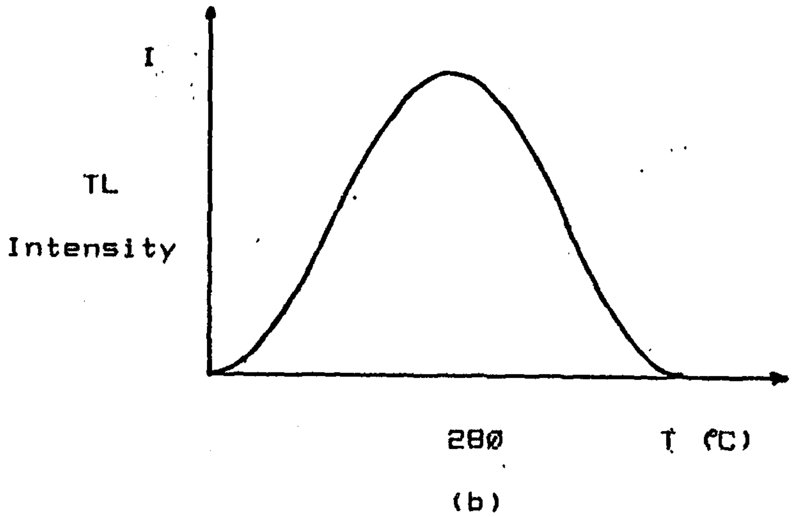


Figure 2.3 (a) Schematic diagram of excitation and thermal stimulation in a phosphor. (b) Typical TL glow curve of $CaF_2(Mn)$ phosphor [10].

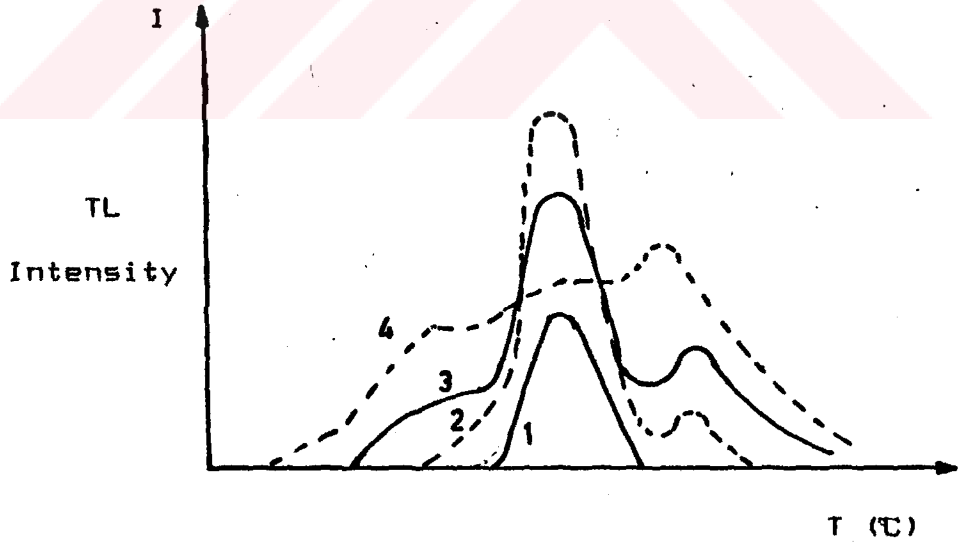


Figure 2.4 Typical glow curves for varying amounts of radiation from 1(minimum) to 4(maximum) [7].

2.3.2.THE RANDALL - WILKINS MODEL

Assume the presence of a single defect (trap) level within the forbidden gap between the valence and conduction bands of the phosphor. When it is assumed that the thermally stimulated electron from the has a negligible chance of re-trapping and go straight to the thermoluminescent centre, it constitutes the basis of the first order kinetics. Figure 2.3 shows the effect following the excitation and subsequent thermal stimulation of a thermoluminescence phosphor [4]. As the temperature increase at a constant rate q, the traps lying close to the main trap at depth E below conduction band become vacated. The peak in the glow curve thus relates to the maximum in the occupied trap density distribution around the main trap of activation energy E, is characteristic of the trapping states.

2.3.3.SEITZ MODEL

According to "SEITZ" [16] model, the electrons which are excited from stable state to the semistable state can be captured by traps or can back to the ground state.

If the electrons release from traps, they make some translations. These transitions are optical transitions that electrons return to the ground level without radiation or translate to the any level by "hv" photon radiation.

2.4. DETERMINATION OF TRAP DEPTS METHODS

2.4.1.PEAK TEMPERATURE METHOD

In this method, the temperature which corresponds to the maximum peak of glow curve is the maximum temperature. For the calculation of the activation energy URBAH [14] made a good approximation

$$E=\frac{T_m(^{\circ}K)}{500} \qquad 2.3$$

2.4.2. TRAP ENERGY DETERMINATION METHOD

The energy distribution of electrons within the trap can be described by the Boltzman distribution, and hence the probability P of a single electron from the trap is given by

$$P=se^{\left(-\frac{E}{k_B T}\right)} \qquad 2.4$$

Assuming that no electrons released from traps are retrapped, but that all undergo thermoluminescence transitions, the intensity of the thermoluminescence glow I depends on the rate of photon emission and therefore on the rate of release of electrons from traps and their rate of arrival at thermoluminescence centres:

$$I=-C\frac{dn}{dt}=Cnse^{(-\frac{E}{k_BT})} \qquad 2.5$$

where C is a constant related to thermoluminescence efficiency.

If the material is heated at a uniform rate,

$$R=\frac{dT}{dt} \qquad 2.6$$

then

$$\frac{dn}{dt}=\frac{dn}{dT}x\frac{dT}{dt}=Rx\frac{dn}{dT} \qquad 2.7$$

By substitution in equation (2.2),

$$\frac{dn}{dT}=-\frac{1}{R}(ns)\,.\,e^{(\frac{E}{kT})} \qquad 2.8$$

or

$$\frac{dn}{n}=-(\frac{s}{R})\,.\,e^{(-\frac{E}{k_BT})}dT \qquad 2.9$$

Integrating gives

$$\ln\left(\frac{n}{n_0}\right)=-\int_{T_0}^{T}\left(\frac{1}{R}\right)s.e^{\left(-\frac{E}{k_B T}\right)}dT \qquad 2.10$$

where n_0 is the number of electrons present in the trap at time t_0 and temperature T_0.

By substituting for n in equation (2.5),

$$I=n_0 C.e^{-\left[\int_{T_0}^{T}\left(\frac{1}{R}\right)s.e^{\left(-\frac{E}{kT}\right)}\right]}.s.e^{\left(-\frac{E}{kT}\right)} \qquad 2.11$$

or

$$I_0=Cn_0 s.e^{\left(-\frac{E}{kT}\right)} \qquad 2.12$$

Therefore, finally substituting for I_0 in equation (2.11),

$$I=I_0.e^{-\left[\int_{T_0}^{T}\left(\frac{1}{R}\right)s.e^{\left(-\frac{E}{kT}\right)}\right]} \qquad 2.13$$

$$\frac{I}{I_0}=e^{-\left[\int_{T_0}^{T}\left(\frac{1}{R}\right)s.e^{\left(-\frac{E}{kT}\right)}\right]}dT \qquad 2.14$$

In real phosphor many different trapping levels will be present, each one due to a particular lattice defect or complex of defects. Each trapping level give rise to an associated glow peak maximum which may or may not be resolved during readout. The area and peak height of each glow peak depends on the number of associated electron traps present. This in turn depends on the number of lattice defects and, for real phosphors on the type and amount of impurity atoms present, as well as on the thermal history and treatment of the material [9].

CHAPTER 3

THERMOLUMINESCENCE DOSIMETRY (TLD)

3.1. INTRODUCTION

There are many materials which exhibit thermoluminescence: The problem is, therefore, not to find such a material but rather to select the most appropriate thermoluminescence phosphor for a purpose from the many available. The application of thermoluminescence phosphors in various fields are expanding and progressing rapidly and there is a constant search for suitable thermoluminescence materials.

When a thermoluminescent phosphor is exposed to ionising radiation at a low (room) temperature, many of the free electrons become trapped in lattice imperfections in the crystalline solid. These electrons may remain trapped for long periods when the crystals are stored at room temperature. If the temperature is raised, the probability of release is increased and electrons may be released from the traps and return to stable energy states with the emission of light.

In thermoluminescence dosimetry (TLD) this property is used to measure the radiation dose to which the phosphor has been exposed. This is done by means of a thermoluminescence dosimetry reader, consisting of a controlled heating element and a photomultiplier system which determines the light fluency emitted during the heating of the dosemeter material. In most thermoluminescence dosimetry readers the integrated light intensity is measured as s function of the heating temperature cycle. Thus, thermoluminescence dosimetry consists of two steeps: (i) radiation exposure, which leaves some excited electrons in metastable traps, and (ii) readout, which consists of controlled heating of the exposed thermoluminescence dosimetry and measurement of the emitted light intensity.

The integrated amount of light emitted from the phosphor is proportional to the number of electrons in the trapped state. Therefore, the quantity of radiation or the exposure can be directly related to the amount of light emitted by the phosphor when heated. This is the principle by which the thermoluminescent material can be used for dosimetry.

The basic phenomenon can be explained by using a hypothetical energy diagram of an insulating crystal exhibiting thermoluminescence due to ionising radiation. This is illustrated in figure 3.1 to depict the fundamental process that occur in the phosphor.

When a crystal is exposed to ionising radiation, electrons are released from the valence band to the conduction band. This leaves a hole (a positively charged site) in the valence band. The electron and the hole move through the crystal, until they recombine or until they are trapped in metastable states. These metastable states are throughout to be associated with defects in the crystal such as the impurity sites. At this point, there are two possible ways in which a thermoluminescence photon may be emitted. As the crystal is heated, sufficient energy may be given to the electron so as to raise it from its trapping site to the conduction band. The electron may move around in the crystal until it recombines with a trapped hole and a thermoluminescence photon is emitted. Alternatively, the hole trap may be less stable than the electron trap and, when the crystal is heated, the hole receives sufficient energy to wander until it recombines with a trapped electron and a thermoluminescence photon is emitted.

The energy gap between the trap and conduction band determines the temperature required to release the electron and produce the thermoluminescence, and is characteristic of the particle material employed. In the practical situation, many trapped electrons and holes are produced. As the temperature of the crystal is increased, the probability of releasing any electron from a trap is increased; thus the emitted light will be weak at low temperature, pass through one or more maxima at a higher temperature, and then decrease again to zero as no more filled traps remain.

The deliberate introduction of impurities into crystals increases the number of traps and can increase the thermoluminescent efficiency of the material. Most thermoluminescence dosimetry materials contain these 'activators'. The centres to which they give can have their own characteristic energy levels and transitions between them give to emissions of photons of corresponding characteristic energies.

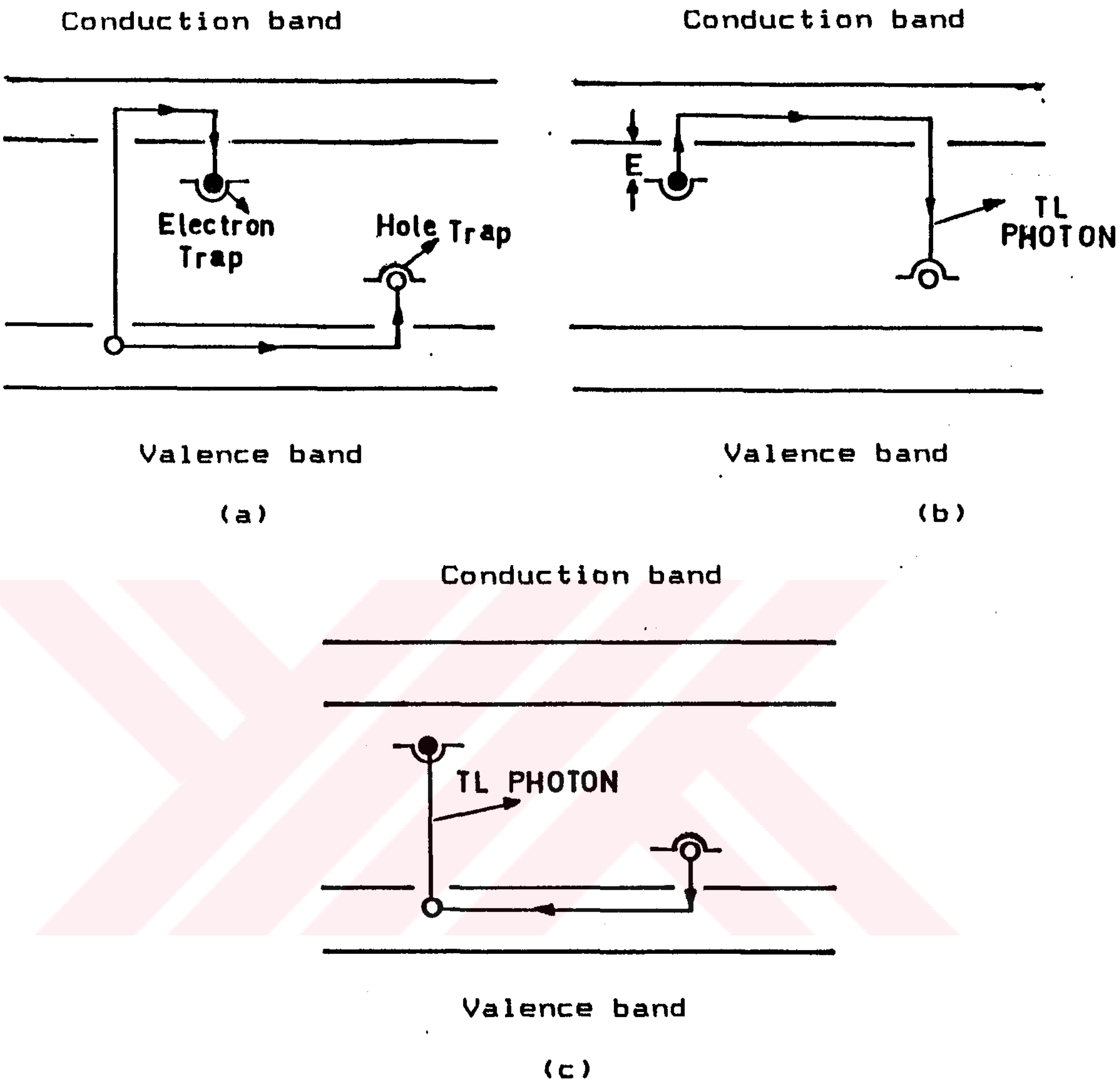


Figure 3.1 Schematic energy level diagram of a phosphor exhibiting thermoluminescence. (a) Exposure to ionising radiation. (b) Electron trap is less stable during heating, and hole trap is the emitting centre. (c) Hole trap is less stable during heating, and electron trap is the emitting centre [10].

As the temperature of a thermoluminescent material that has been exposed to radiation is increased, more and more trapped electrons are raised to the conduction band and then emit radiation on falling to lower energy levels. If the thermoluminescence is plotted against temperature a glow curve is obtained as illustrated in figure 2.4. The higher temperature part of the glow curve corresponds to electrons in deeper traps.

The materials used for thermoluminescence dosimetry give a measure of the energy absorbed by the materials themselves and not of the absorbed dose in some other material such as water or tissue. Let us suppose that it is absorbed dose in water that is required. If both the water and thermoluminescence dosimetry material are irradiated by photons under conditions of electronic equilibrium, then the ratio of the absorbed doses in the two media will be the same as the ratio of their mass energy absorbtion coefficients. The more the atomic number of the thermoluminescence dosimetry material differs from that of water the greater this ratio will be. It is for this reason that a material such as $CaSO_4$ although having high sensitivity for thermoluminescence dosimetry, has not been used as much for medical dosimetry as materials such as LiF and $Li_2B_4O_7$ which are closer to water or soft tissue in atomic number.

The response of thermoluminescence dosimetry materials can be affected by, amongst other things, their previous thermal history, their previous radiation history, the time and nature of storage between exposure and readout, and the gas surrounding them during readout. Commercial readout equipment endeavour to control some of these variables. Despite some of these unpromising features thermoluminescence dosimetry has found valuable applications. The great merit of thermoluminescence dosimetry is that the dosimeters can be very small and flexible in shape and can therefore be introduced into materials in which the absorbed dose is required without appreciably perturbing field. They need no leads or connections and are very rugged [8].

3.2. REQUIREMENTS OF A SUITABLE THERMOLUMINESCENCE MATERIAL

A thermoluminescence phosphor for dosimetric use should combine several properties which, as a result, limit the choice to only a few inorganic compounds. Some of the more desirable properties of a thermoluminescence dosimetry phosphor are [11] :

i) A high concentration of electron (or hole) traps and a high efficiency of light emission associated with the recombination process.

ii) A sufficient storage stability of the electron (or hole) traps to cause no undesirable fading even during extended storage at ambient or slightly increased temperatures (such as tropical or desert climates) or, in the case of biomedical implantation studies, the body temperature. This should also be true for the opposite extreme of climate within the Arctic Circle where temperature is low.

iii) A spectrum of the emitted thermoluminescence light to which the detector system (photomultiplier and filter combination) responds well, and which interferes as little as possible with the incandescence (infrared) emission of the heated phosphor and its immediate surroundings.

iv) The main peak temperature (T_m) lies between 180 and 250 °C. At higher temperatures the infrared emission from the hot sample and sample holder increasingly interferes with the measurement of low doses. The spectrum wavelengths between about 300 and 500 nm are most desirable unless systems specially sensitive to Ultraviolet are used.

v) A trap distribution that does not complicate the evaluation procedure due to the presence of additional rapidly fading low temperature peaks or high temperature peaks and due to the retrapping process. The phosphor should preferably be completely during the readout procedure itself with no changes in its sensitivity and/or background reading or linearity [10].

3.3. EMISSION SPECTRA OF THERMOLUMINESCENCE MATERIALS

The emission spectrum can be measured using a wide spectrum photomultiplier, with an photocathode, and sets of low-pass filters with progressive cut-off wavelengths. The following emission spectra refer mainly to the major families of phosphors such as LiF, $Li_2B_4O_7$, $CaSO_4$, and CaF_2.

i) LiF (Mg), (Ti)

The spectral emission peaks of both Li :Mg, Ti and thermoluminescence phosphor are at 400 nm in the blue region with full width at half maximum (FWHM) of approximately 115 nm. This emission corresponds with the peak maximum efficiency of bi-alkali photomultiplier (PM) tubes [12].

ii) $Li_2B_4O_7$ (Mn)

The spectral emission from the 200 C glow peak of $Li_2B_4O_7$ (Mn) phosphor is yellow-orange in colour, and the maximum appears at 600 nm, while that for the $Li_2B_4O_7$ (Cu) phosphor it is at about 360 nm [13].

iii) $CaSO_4$ (Tm) and $CaSO_4$ (Dy)

The spectral emission peak of $CaSO_4$ (Tm) phosphor is at 452 nm, and at 480 nm and 570 nm for the Dy dopant. It has been shown that Dy gives essentiall the same emission spectrum regardless of the host lattice.

iv) $CaF_2(Dy)$

The spectral emission peaks are at 460 and 480 nm in the blue and 570 nm in the yellow region.

v) Others

(a) BeO. The spectral emission of BeO phosphor extends from the blue region of the visible spectrum into the ultraviolet, down to a 200 nm wavelength. A higher detection sensitivity can be achieved using a photomultiplier tube fitted with a quartz window and ultraviolet transmitting optics.

(b) Al_2O_3. The spectral emission varies considerably with the type of Al_2O_3. The spectral emission for ruby [Al_2O_3 (Cr)] is at 700 nm in the red region, while an emission peak at 425 nm in the blue is associated with those phosphors containing titanium [10].

CHAPTER 4

THERMOLUMINESCENCE INSTRUMENTATION

4.1. INTRODUCTION

Back in the years 1930 - 1950, the technique of measuring thermoluminescence by glow curves was developed. Since then this technique has been simplified so that many thermoluminescence materials can be studied easily. Thermoluminescence can be observed visually for example, by pouring an irradiated phosphor onto a heated electric iron in the dark. Instrumentation is therefore not very complicated; it consists of a heat source and a quantitative light detector. During the early days, most researchers in the field built their own readout equipment, usually based on electrical resistance heaters, photomultipliers (PM), and filter systems for discrimination against spurious signals and the infrared emission from heated components.

Instruments for the study and quantification of the thermoluminescence from a phosphor are described in many reports; some are available commercially, and the commercial readers are of most interest to the average thermoluminescence dosimetry user. They fall into two types: those specific to a particular design of dosemeter, and those which can accept a variety of dosemeter forms or phosphor types. They may be manual, semi-automated, or fully automated systems. The essential features common to every thermoluminescence dosimetry reader are: (1) a phosphor heating system, (2) a light collection and detection system, (3) a signal-measuring system, and (4) a display and recording system. A schematic illustrating features common to all thermoluminescence dosimetry readers is shown in figure 4.1.

A simple thermoluminescence dosimetry reader has been described by Cameron et. all. (1967) [6].

A few simple, general rules for the design of a thermoluminescence dosimetry reader can be given here. For the heating of the phosphor, electric resistance heating and hot gas heating have been widely used. The reader should be designed in such a way that the photomultiplier tube sees as much of the phosphor, and as little of the other heated areas as possible. The spectral response of the photomultiplier tube should cover spectrum of the thermoluminescence to be measured. Most thermoluminescence readers nowadays employ an integrator because the integrated thermoluminescence light is more closely related to the close and easier to measure reproducible than the thermoluminescence peak height. Additional provisions for an X-Y glow curve recorder is available. The diagram of all thermoluminescence dosimetry is shown in figure 4.1 [10].

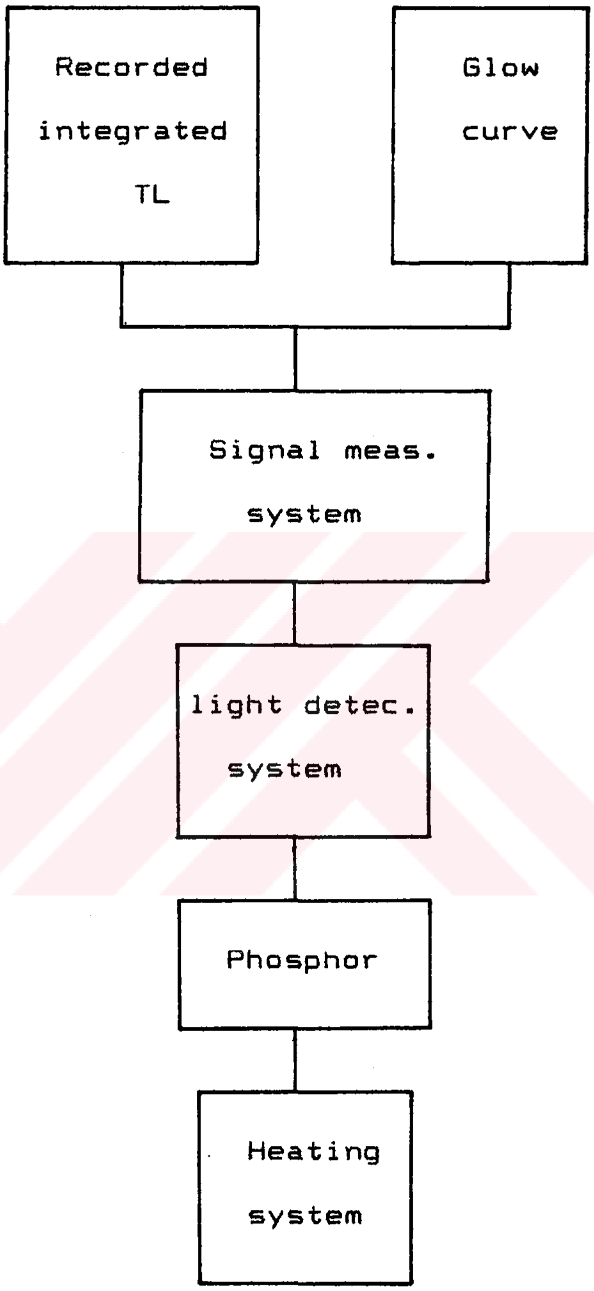


Figure 4.1 Basic thermoluminescence dosimetry reader.

4.2. READOUT INSTRUMENT

In our readout instrument has utilized the thermoluminescence measurement of the light signal emitted by the phosphor. Then is plotted the thermoluminescence intensity simultaneously as a function of both light wavelength and sample temperature. The resulting curve is required to as the glow curve. Figure 4.2 shows a block diagram of the components of the readout instrument and figure 4.3 shows the readout instrument. There are three components of the instrument [10].

i) Ultraviolet and visible spectrophotometer
ii) Heater
iii) Recorder

4.2.1.ULTRAVIOLET AND VISIBLE SPECTROPHOTOMETER

This instrument measures the transmittance and the absorbtion of a sample in the ultraviolet/visible/near infrared region of 200 to 1100 nm wavelengths to make quantitative analysis. It can also measure the absorbtion and the transition spectrum, as a matter of course. It uses a deuterium lamp in the ultraviolet region and a tungsten iodine lamp in the visible - to - near infrared as the light source. The light from the source, after passing through a high order light cut-off filter, goes to the monochromator that uses a concave diffraction grating. The light is monochromated by the monochromator and is split into two beams by the beam splitter. One of the beams through the sample and the other the reference sample such as a solvent or other and is incident on the silicon photocell detector. Figure 4.4 shows the mane unit of this instrument [19].

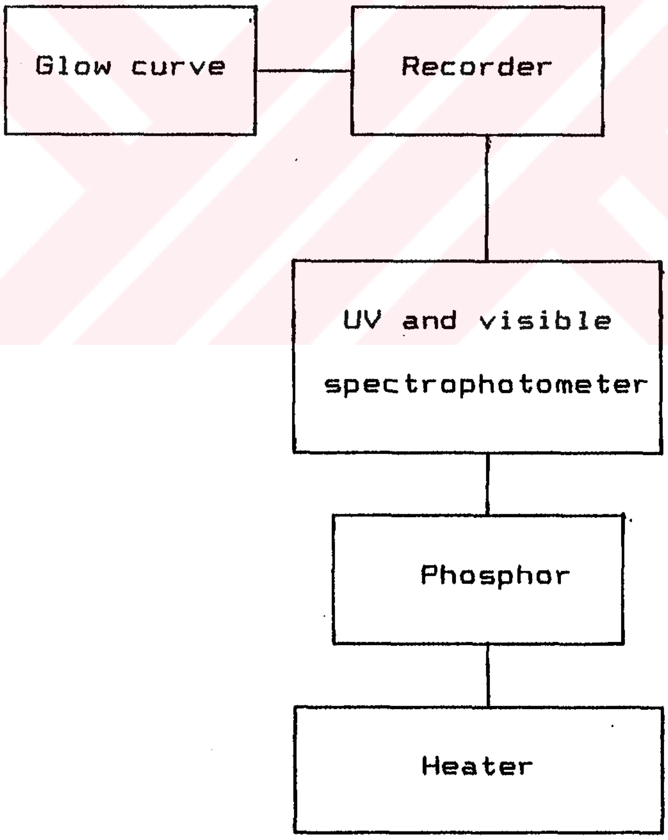


Figure 4.2 Block diagram of thermoluminescence readout instrument

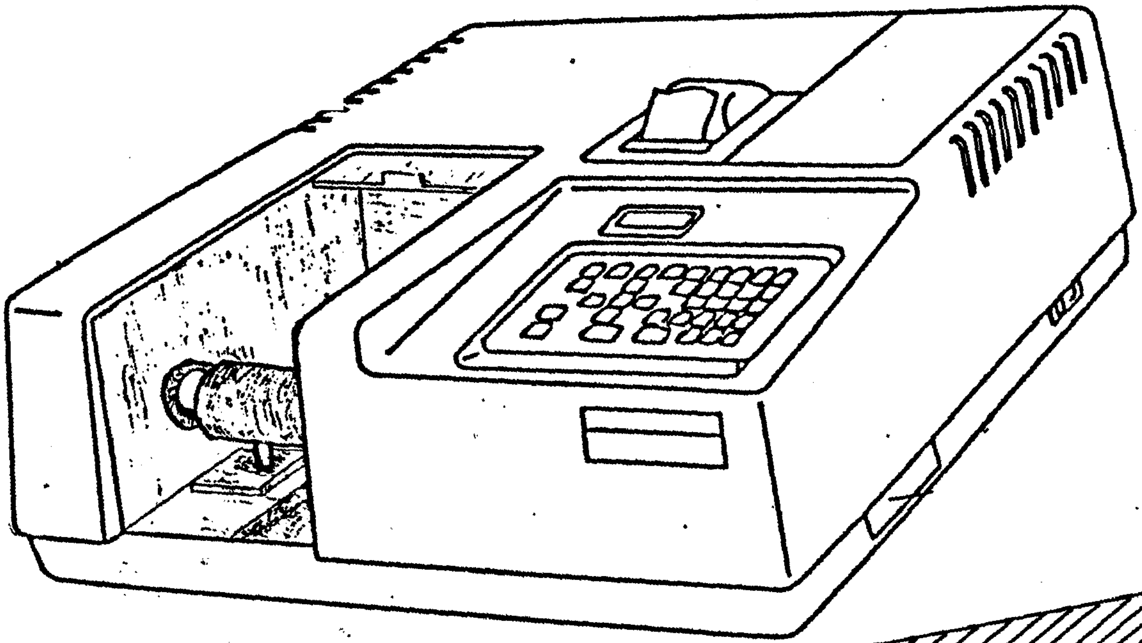
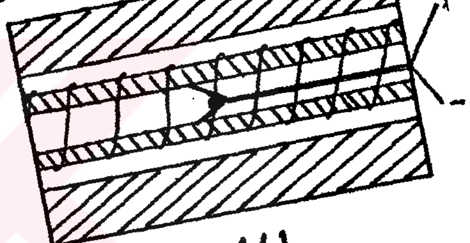


Figure 4.3 Readout Instrument



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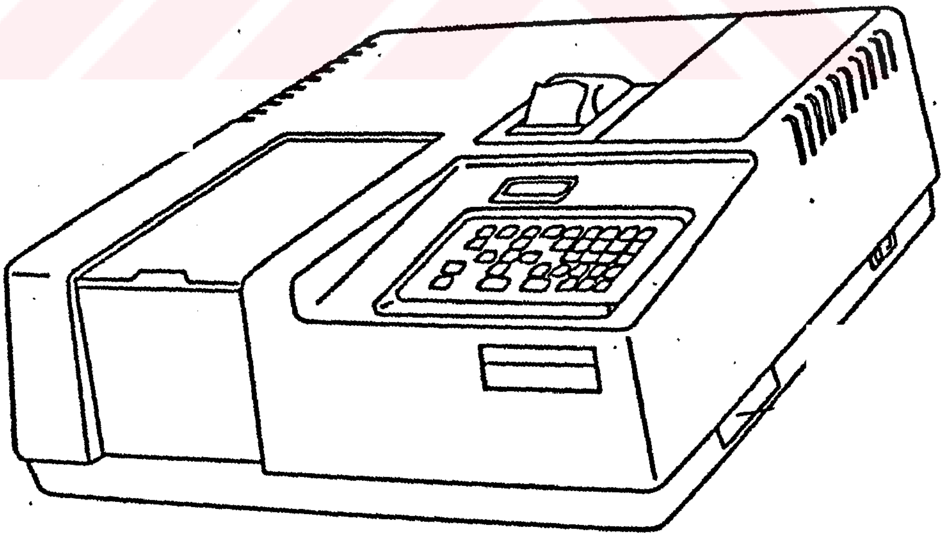


Figure 4.4 Main Unit

4.2.1.1.SYSTEM OF THE UV AND VISIBLE SPECTROPHOTOMETER

Figure 4.5 shows the block diagram of this system. After the light signal is converted into an electrical signal by the detector and amplified, it is subjected to A-D conversion. The data thus converted is subjected to necessary processing by a microcomputer and is output.

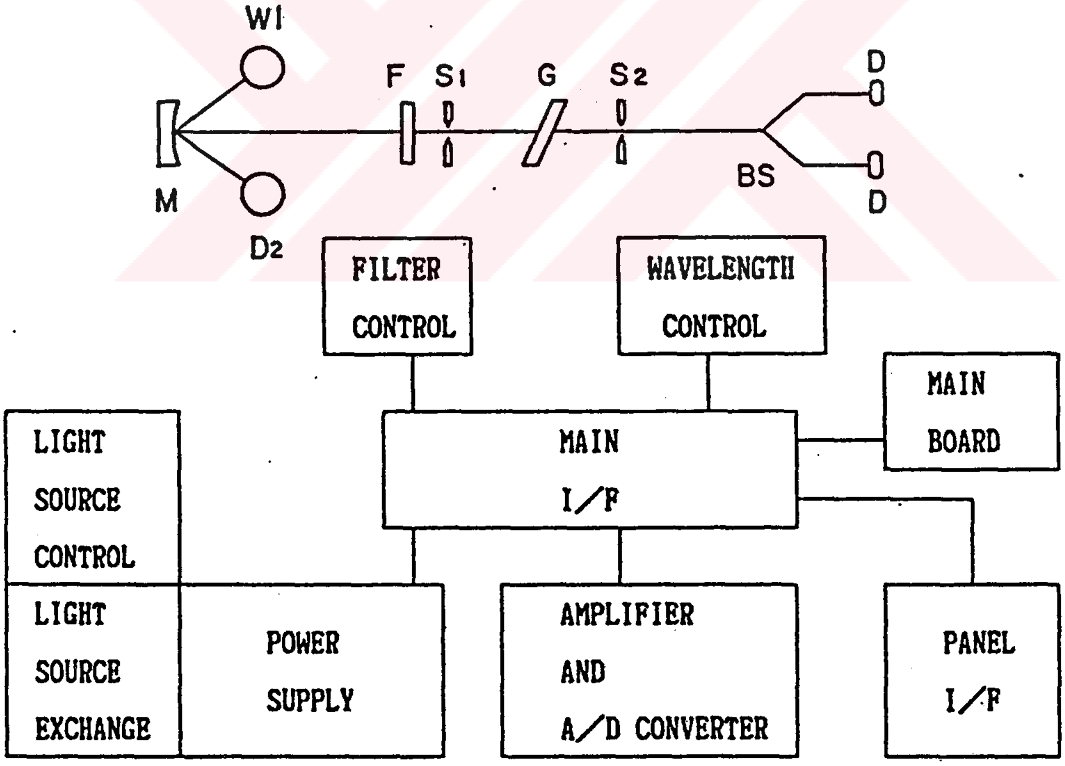


Figure 4.5 Block diagram of the system

4.2.1.2.PROGRAM STRUCTURE

The programs include the following four.

(1) Standard measurement program

This program is supplied with the instrument as standard and includes Method 1 through 5.

Method 1: Measurement at fixed wavelength (%T, Abs)

Method 2: Measurement of change with time at fixed wavelength (stored in memory)

Method 3: Quantitative analysis

Method 4: Measurement of spectrum data

Method 5: Measurement of change with time at fixed wavelength (not stored in memory)

(2) Data processing program

This program comes with CRT. The following methods are included.

Method 11: Spectrum data processing (Addition, subtraction, multiplication, division; derivative processing; smoothing processing; peak detection; mode conversion; expansion and reduction of scale; etc.

Method 15: Comment function

Method 16: Command link function (simple program function)

If CRT is not supplied, the memory program will be supplied instead. This program permits move of the memory contents, addition, subtraction, multiplication, division and data printout.

(3) Program for setting the spectrophotometer and control of input/output unit.

i) Program for setting the spectrophotometer

Method 21: Setting of spectrophotometer

Method 22: Self-diagnosis

Method 23: Baseline correction and wavelength correction

ii) Program related to input/output unit

Method 31: Control of external device (option)

Method 32: Positional adjustment of thermal plotter head

(4) Application program (option)

Application programs are all included in

Method 50: One program is stored on a piece of ROM cartridge or floppy disk.

The following programs are available,

i) Wavelength program (spread sheet system)

ii) Printout of measurement data at fixed wavelength intervals.

iii) Kinetics (for enzyme routine)

iv) Kinetics (for enzyme study)

v) Colour difference calculation program

vi) Multi-component analysis program

vii) Derivative quantitative analysis program

viii) Saving data and measurement parameters onto floppy disk

ix) Dissolution test program

Monochromators is very important part for recording emission spectra which is used to supply a beam of light having some desired narrow range of wavelength. It is part of a spectrophotometer. In that case, it has the following components parts: (1) an entrance slit producing a well defined beam of heterochromatic radiation, (2) a prism or diffraction grating dispersing the incident radiation into a continuous spectrum, (3) some device to rotate the prism or grating so that the desired wavelengths of exit radiation are obtained and (4) an exit slit allowing a narrow band of wavelengths. A monochromator becomes a spectrophotometer if it is preceded by a source of continuous radiation and followed by a detector, an amplifier and a device for measuring the amplified output signal.

The common form resembles a prism spectroscope. While light from a heated thermoluminescence dosimetry, entering the fixed collimator as usual through a narrow slit, is dispersed by a special four-sided prism after one internal reflection. The image of the resulting spectrum falls on a metal plate, and a second, fixed narrow slit in this plate allows light of approximately only a single wavelength to emerge. The purity of this beam depends on the two slits, also upon diffraction at the slits and scattering by the prism. The prism is mounted so that it can be rotated by a tangent screw, thus bringing different parts of the spectrum to the second slit as desired. The tangent screw may be provided with a graduated head reading directly in wavelengths; but to much reliance must not be placed on the indicator, since the adjustment is affected by mechanical factors [19].

4.2.2.HEATER

Heater provides to heat of the thermoluminescence phosphor. It can be called the oven. The temperature of the oven can be controlled by the thermocouple. The purpose of heating cycle is to heat phosphor until the electrons liberated in traps. Heater and the phosphor are have to locate in the dark to be free because it is all aimed to measure the intensity of light. The emitted light is applied to the ultraviolet spectrophotometer.

CHAPTER 5

EXPERIMENTS AND RESULTS

The experimental apparatus used in recording thermoluminescence phenomena allows of obtaining thermoluminescence emission spectra and dose response curve. The sample was mounted by a cylindrical glass which was coincide with a cylindrical non-metal which is screwed to a face of a aluminium block. Thermoluminescence phenomena was detected by a spectrophotometer which ic capable to detect the wavelength of the light emitted by the phosphor. And the transmittance was then measured and plotted on the Y-axis of an X-Y recorder. The intensity of the light was proportional to the temperature of the sample which is plotted on the X-axis of the recorder.

The thermoluminescence of CaF_2 dosimeter crystal has been studied using the spectrophotometer for determining simultaneously the emission spectrum as a function of the transmittance of the sample is shown in figure 5.1. The X-ray source used in these experiments was operated at 100 kV and had an effective energy about of 90 keV.

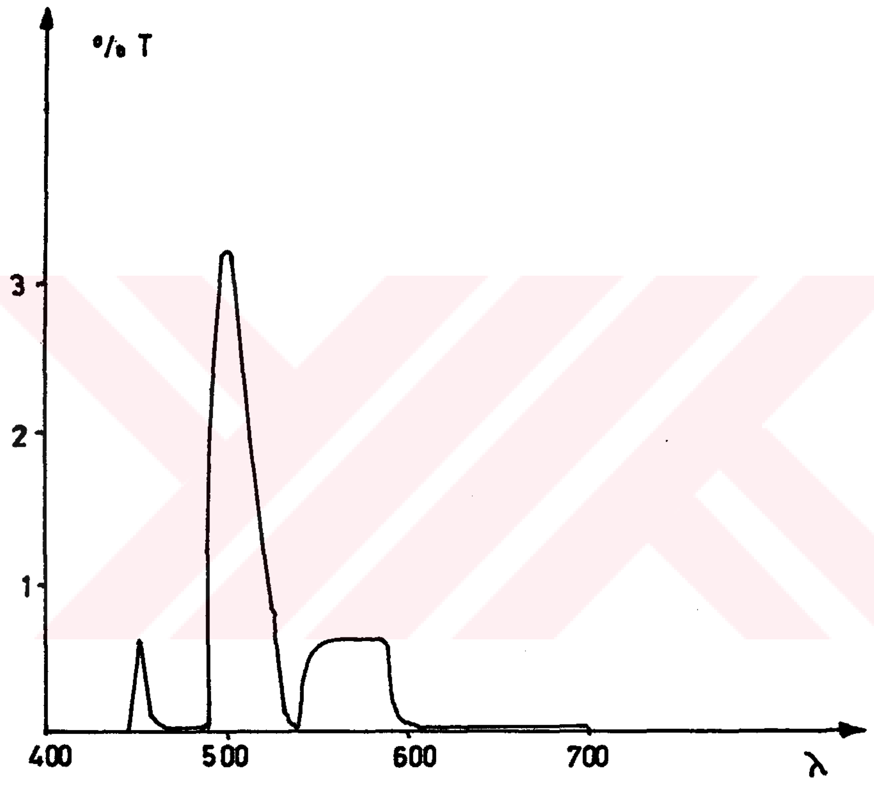


Figure 5.1 Thermoluminescence light emission spectrum of the main thermoluminescence peaks in CaF_2 .

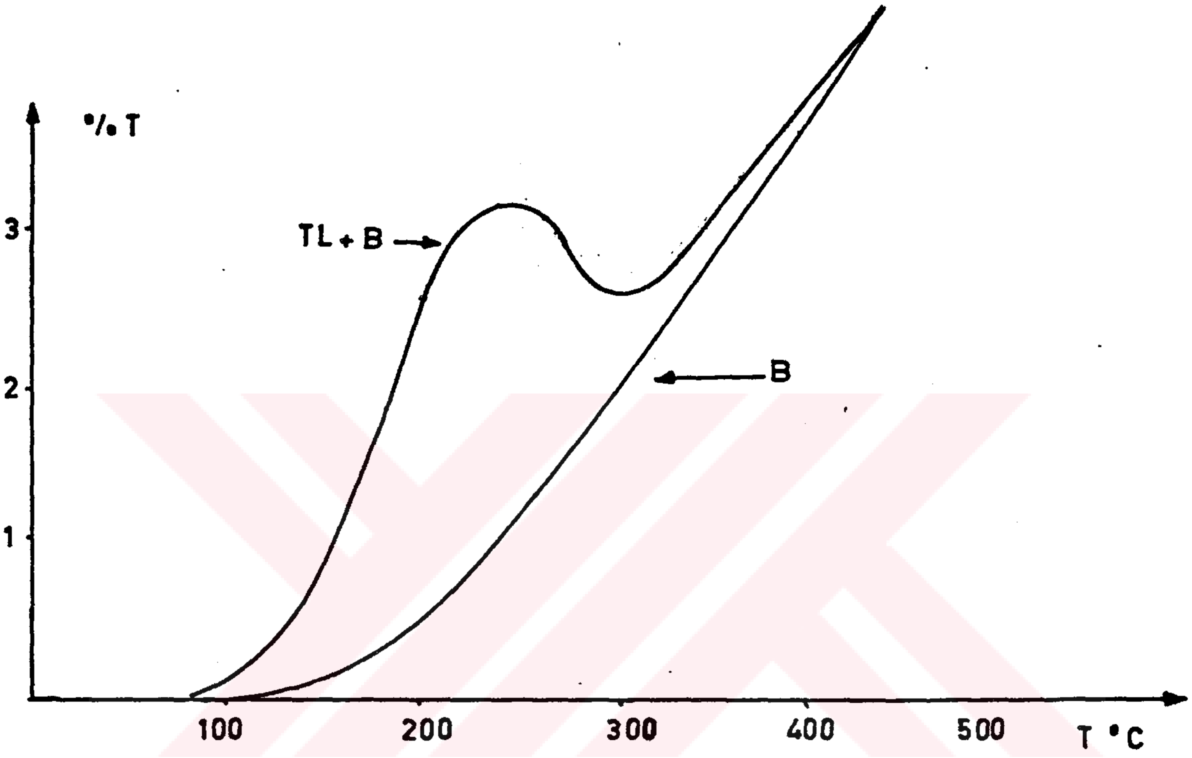
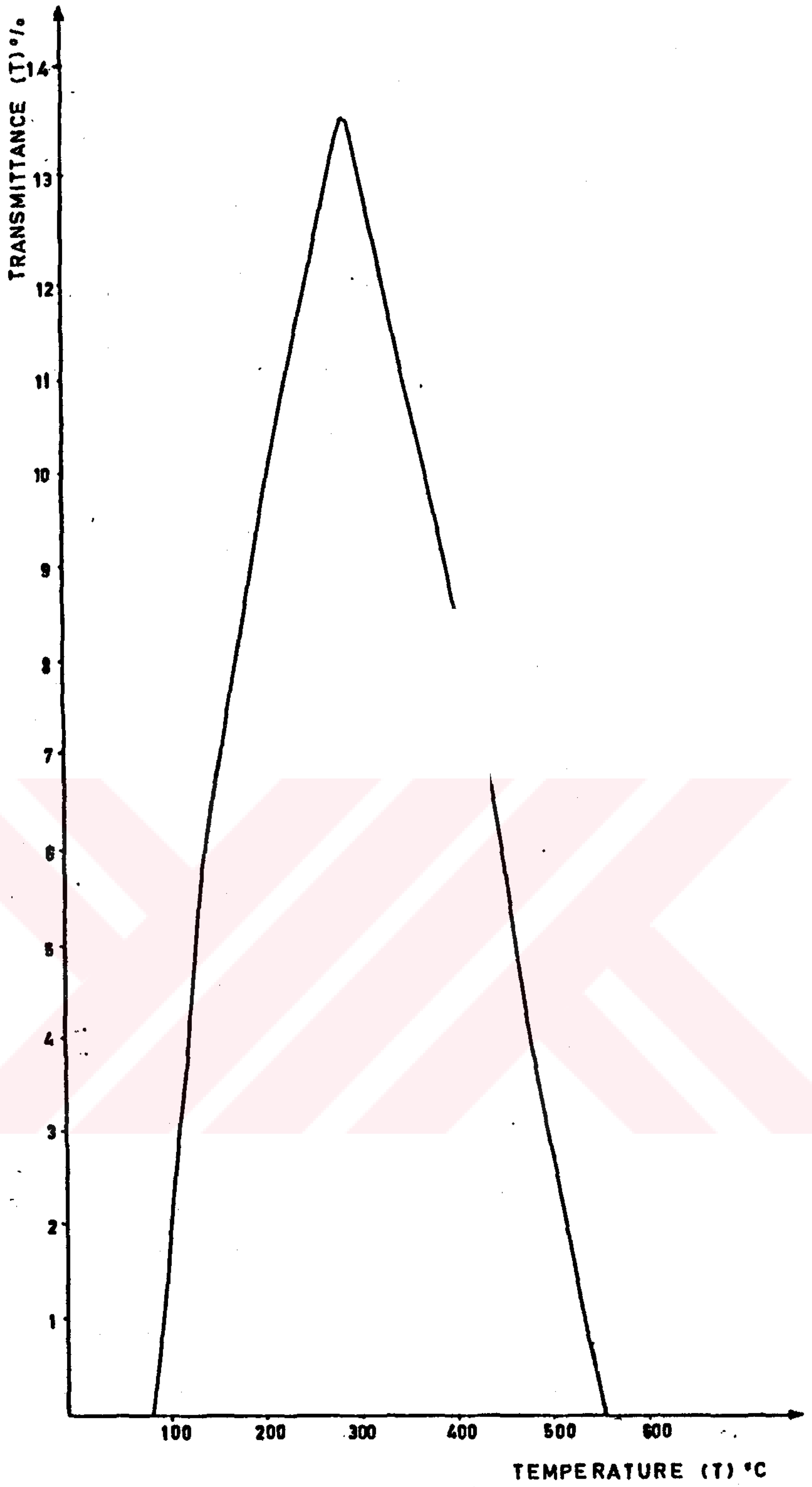


Figure 5.2. Glow curve of CaF_2 curve B is a second experiment on the same sample after all thermoluminescence has been removed. The wavelength of the light is 500 nm.

Figure 5.2 shows a simple thermoluminescence glow curve for a signal having a Gaussian-shape curve. Subtraction of the blackbody contribution, B, would give the desired output which is shown in figure 5.3.



-igure 5.3 Glow curve of CaF_2; 0.479 mg, 500 nm.

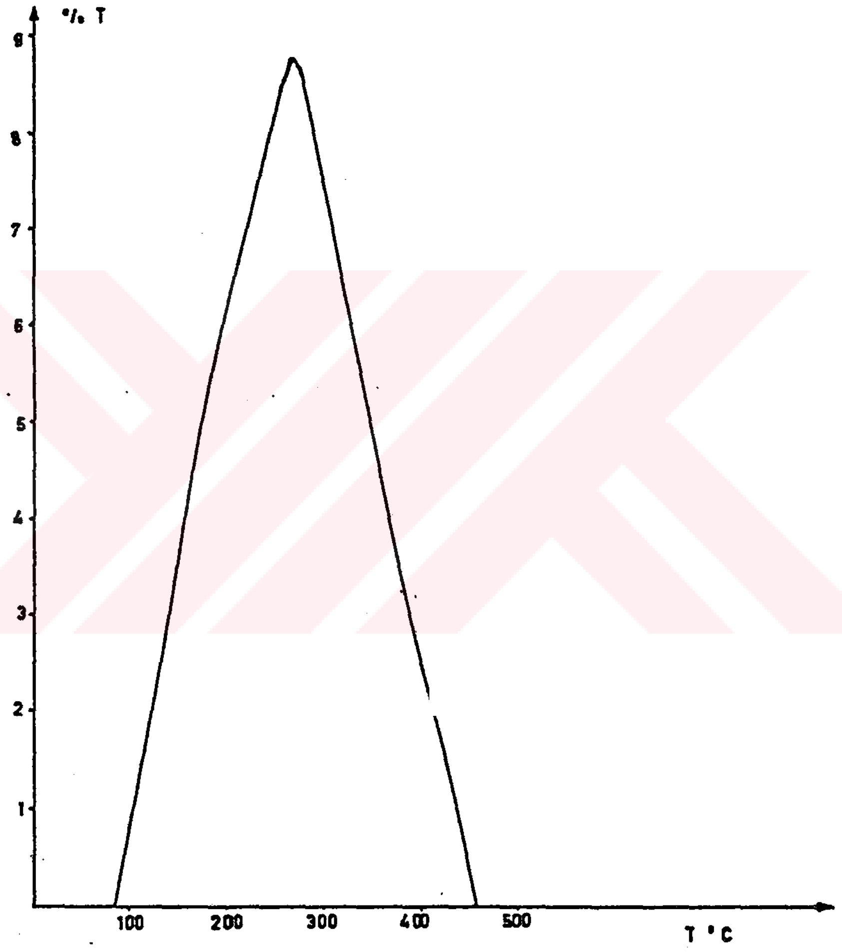


Figure 5.4 Glow curve of CaF_2; 0.290 mg, 650 nm.

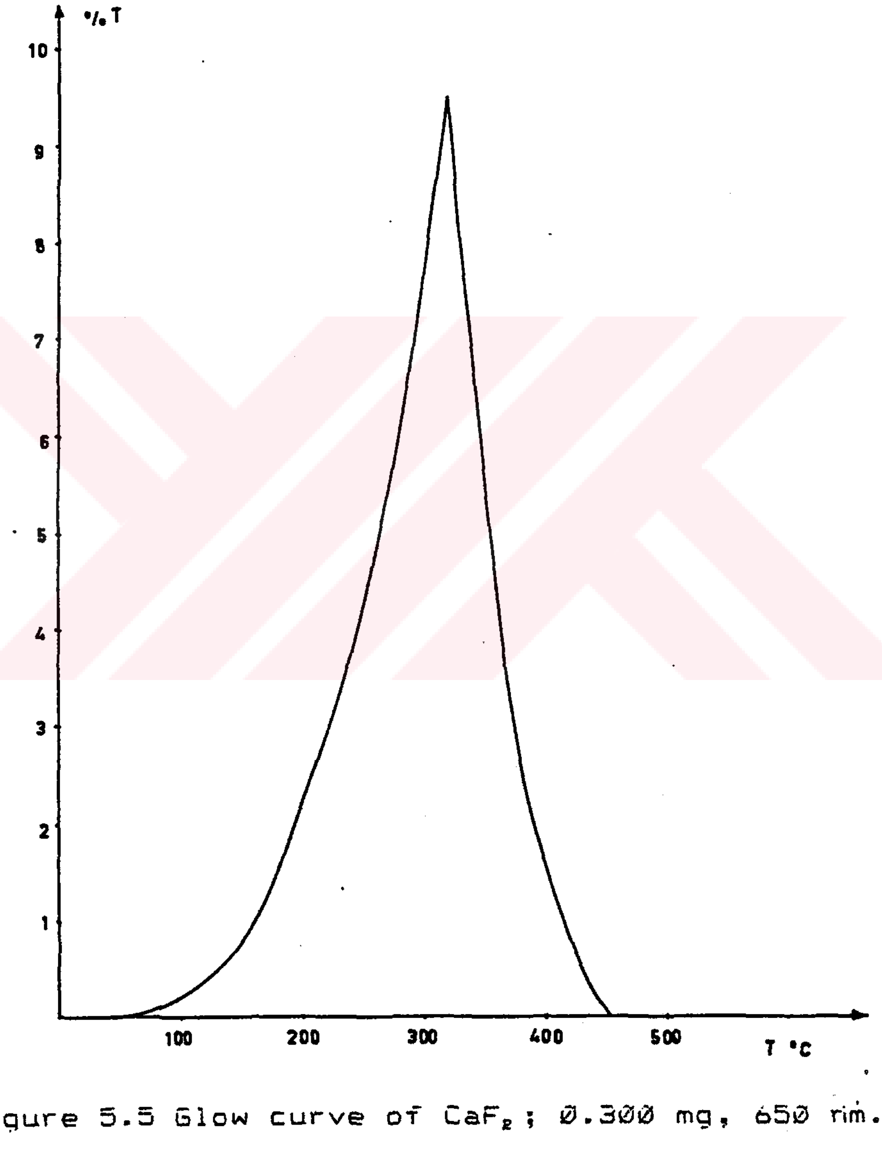


Figure 5.5 Glow curve of CaF_2; 0.300 mg, 650 nm.

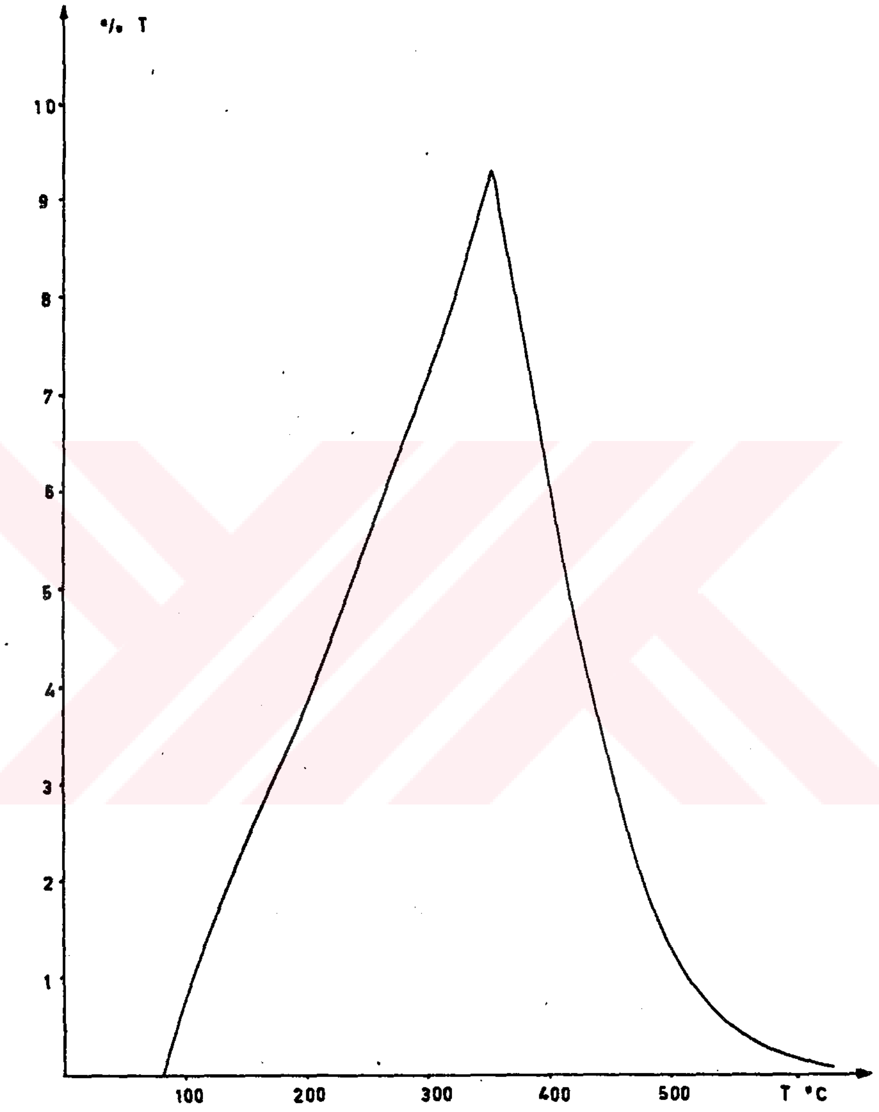


Figure 5.6 Glow curve of CaF_2 ; 0.320 mg, 650 nm.

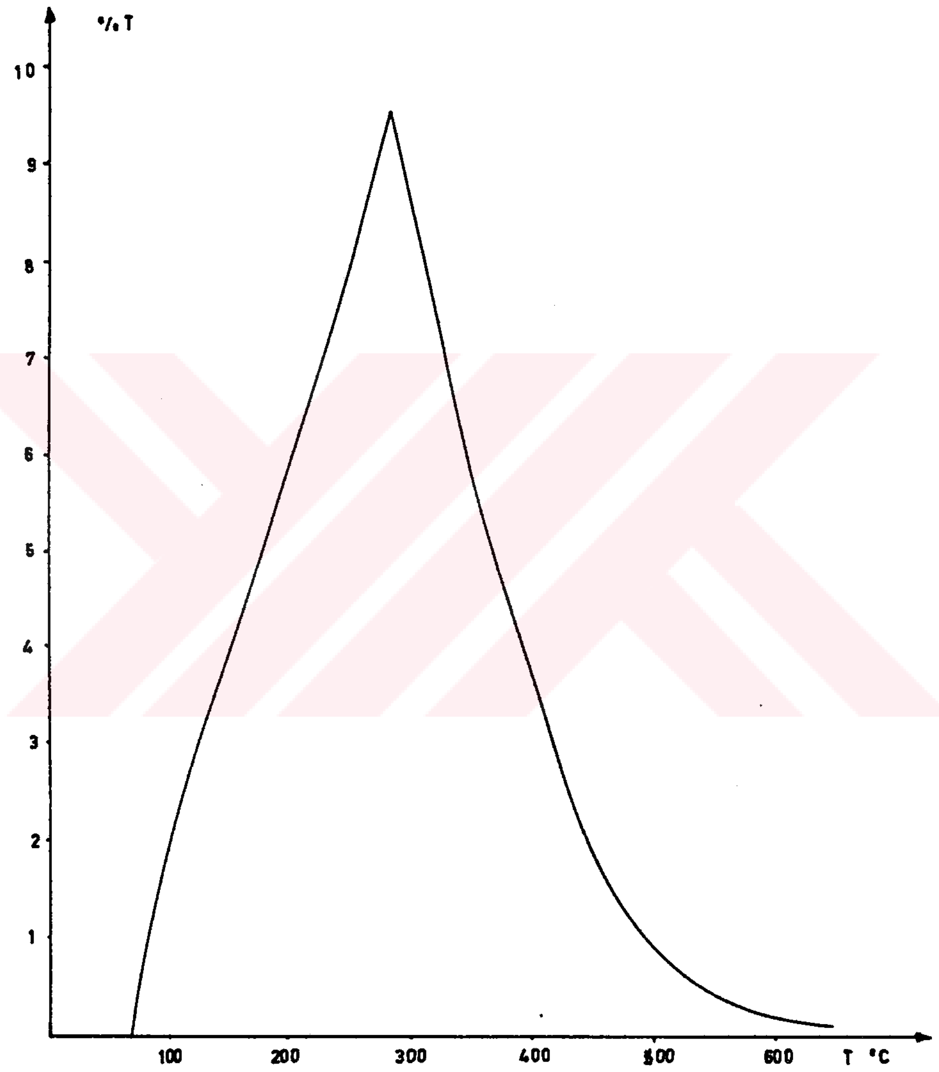


Figure 5.7 Glow curve of CaF_2, 0.345 mg, 650 nm.

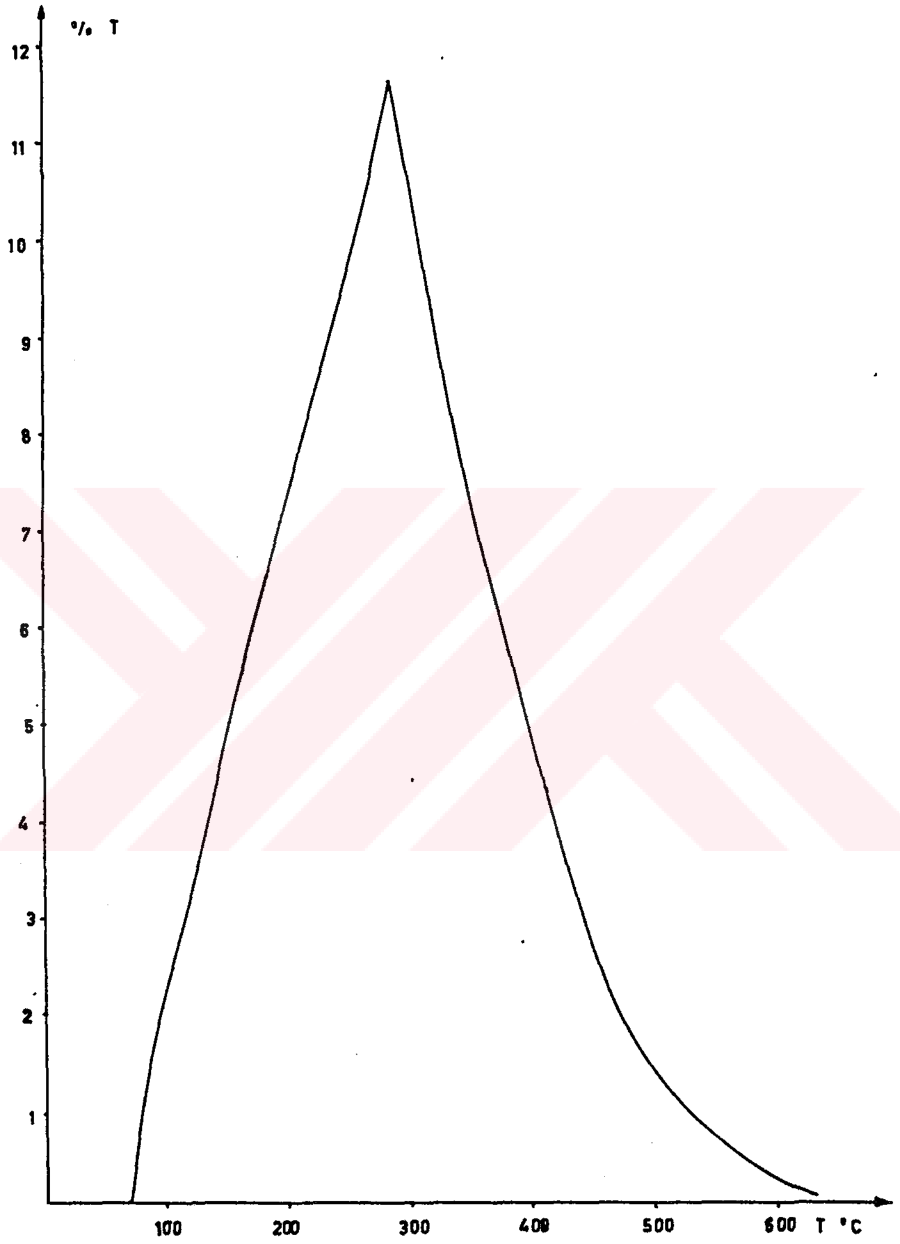


Figure 5.8 Glow curve of CaF_2; 0.370 mg, 650 nm.

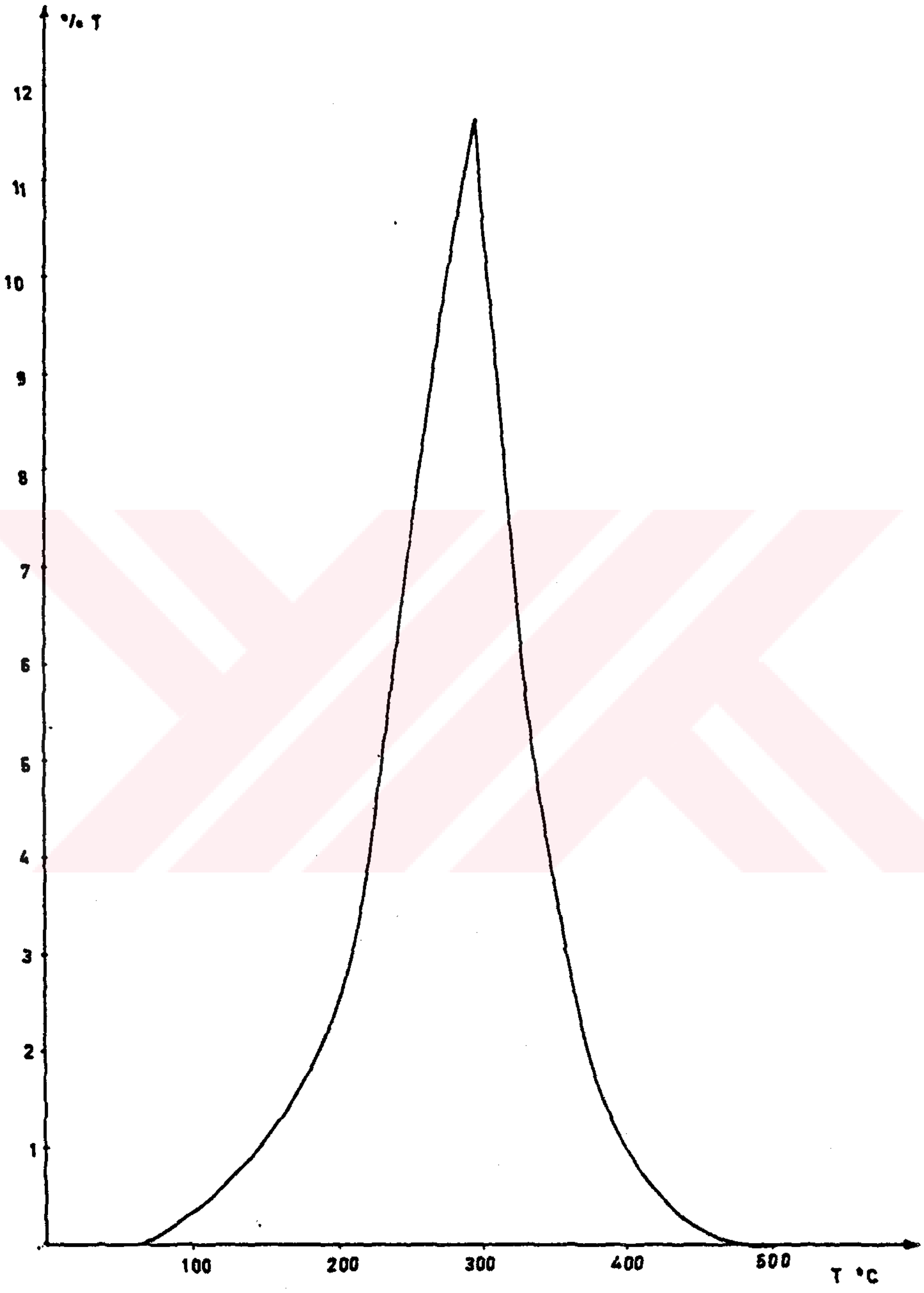


Figure 5.9 Glow curve of CaF_2: 0.375 mg. 650 nm.

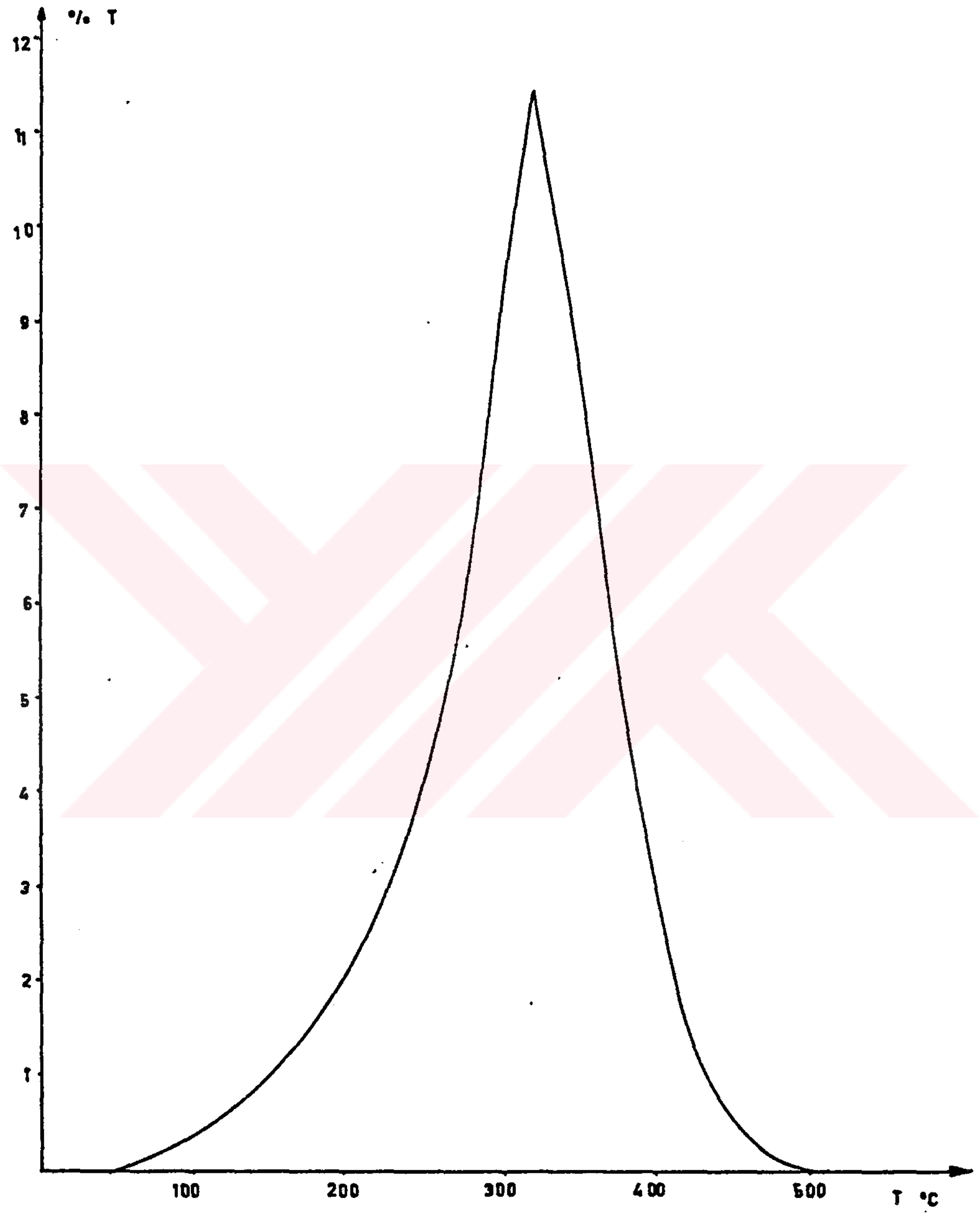


Figure 5.10 Glow curve of CaF_2 ; 0.390 mg, 650 nm.

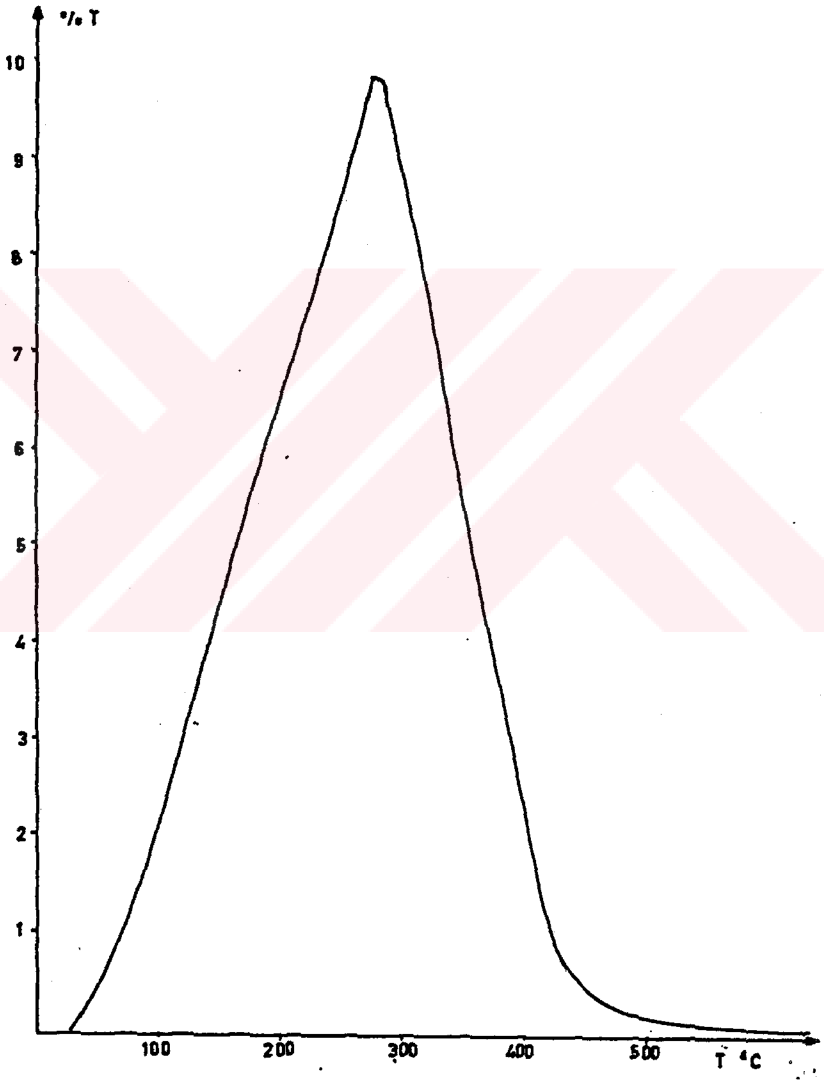


Figure 5.11 Glow curve of CaF_2 ; 0.380 mg, 500 nm.

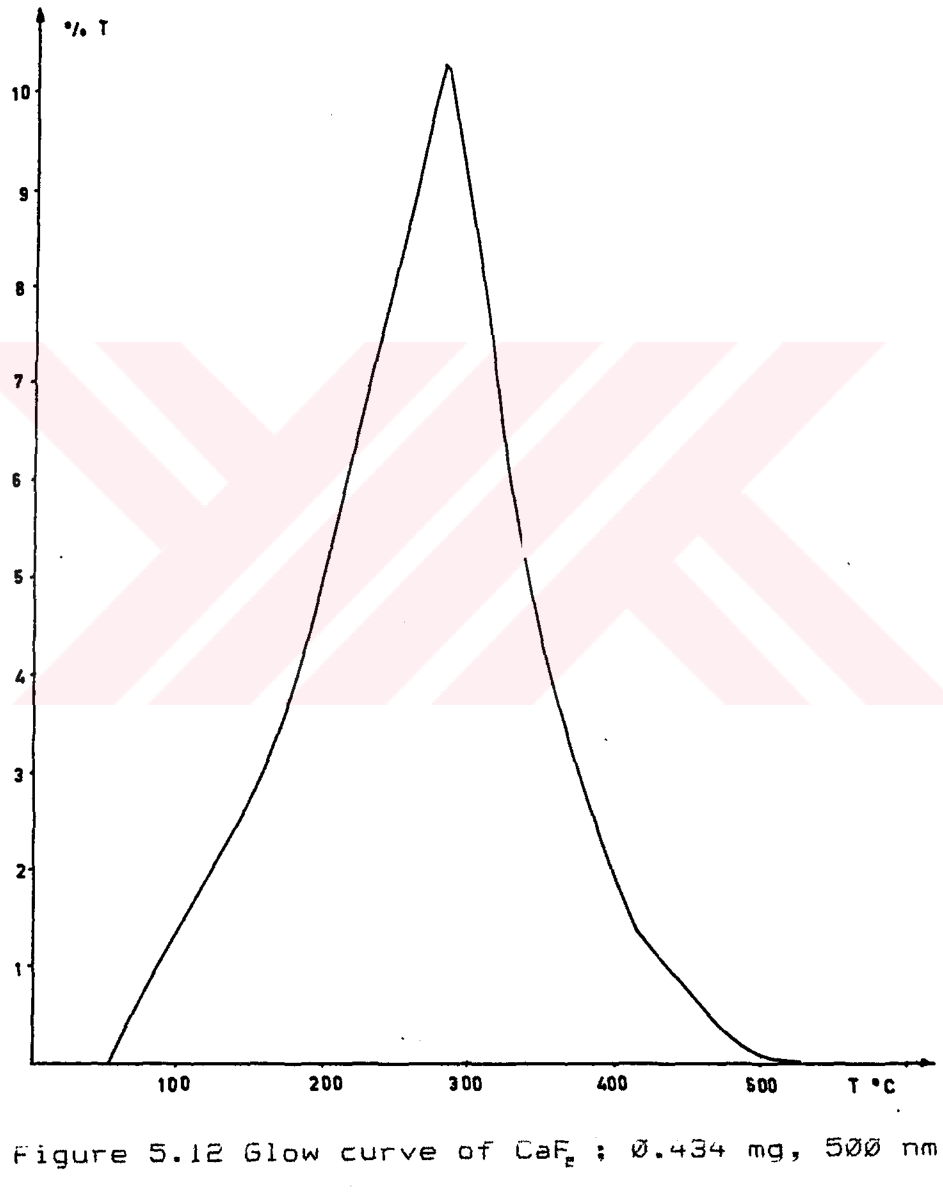


Figure 5.12 Glow curve of CaF_2 ; 0.434 mg, 500 nm.

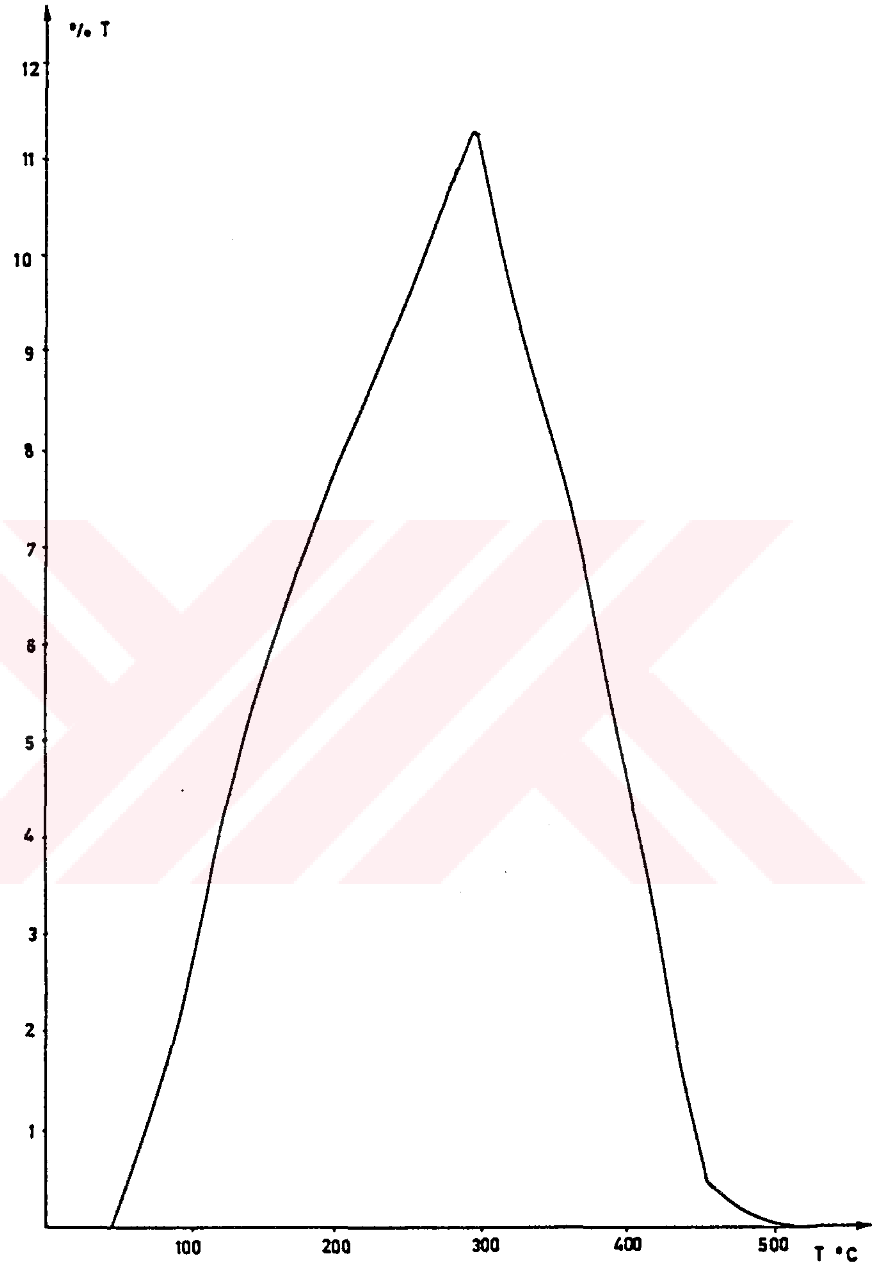


Figure 5.13 Glow curve of CaF_2 ; 0.450 mg, 500 nm.

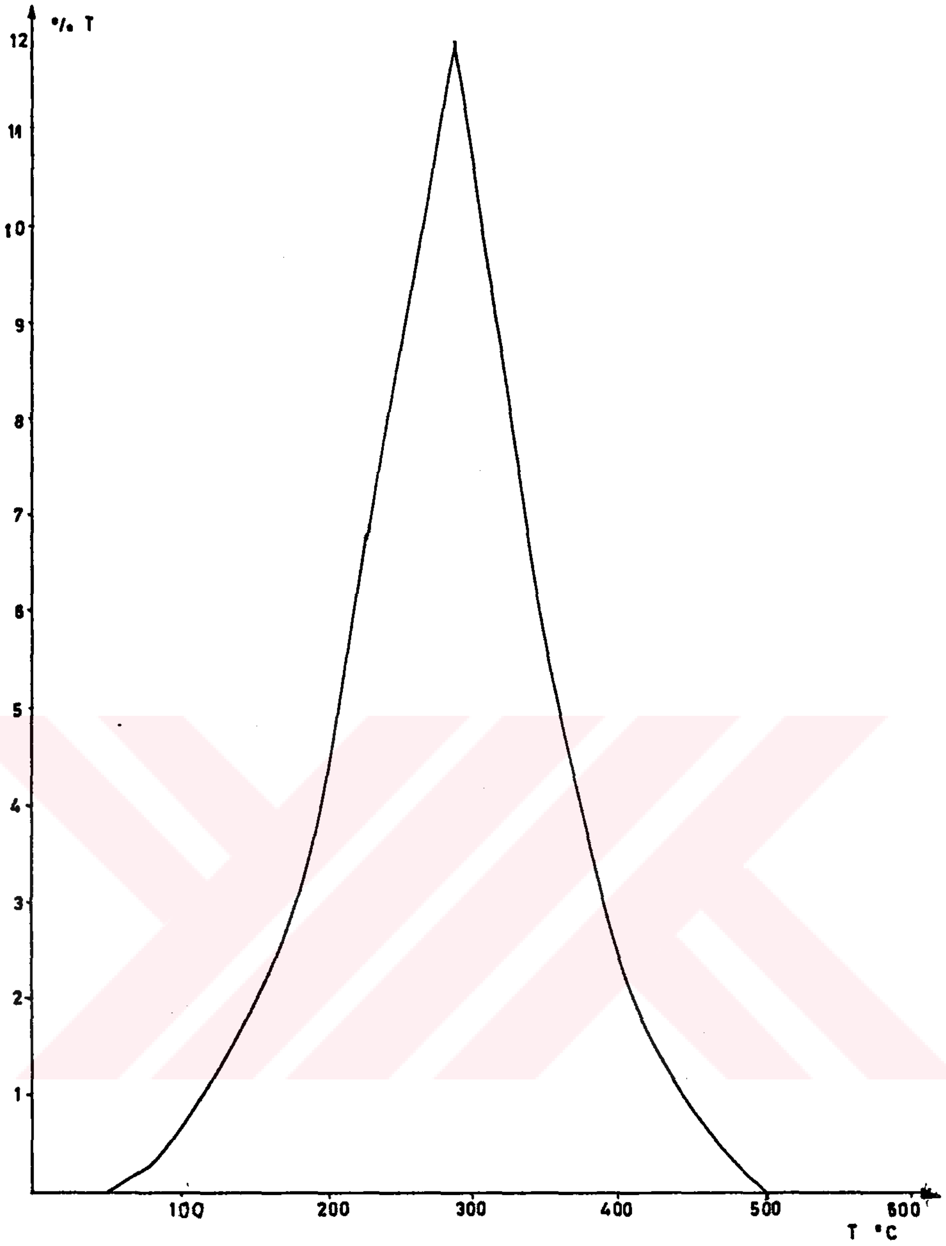


Figure 5.14 Glow curve of CaF_2 ; 0.460 mg, 500 nm.

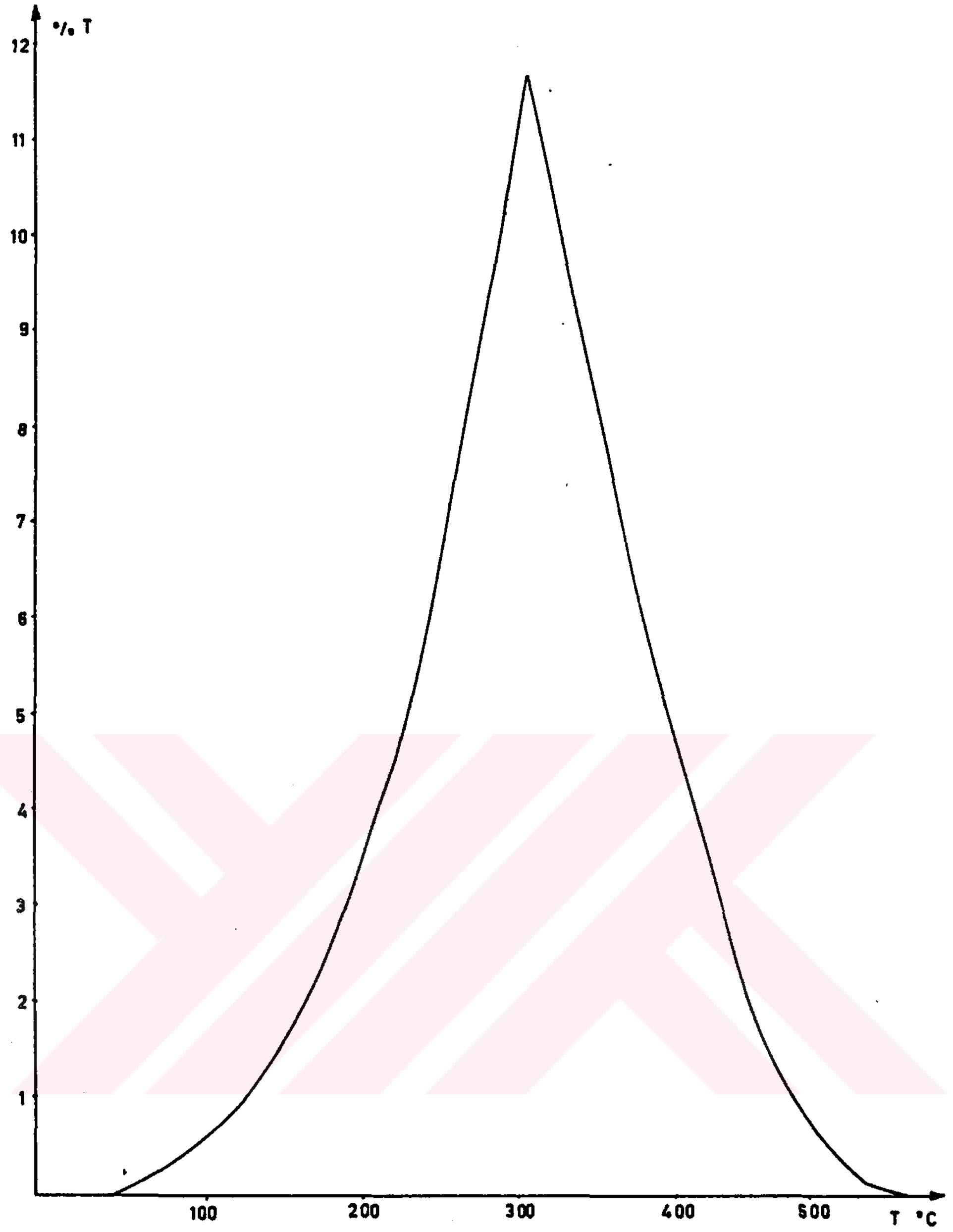


Figure 5.15 Glow curve of CaF_2 ; 0.450 mg, 500 nm.

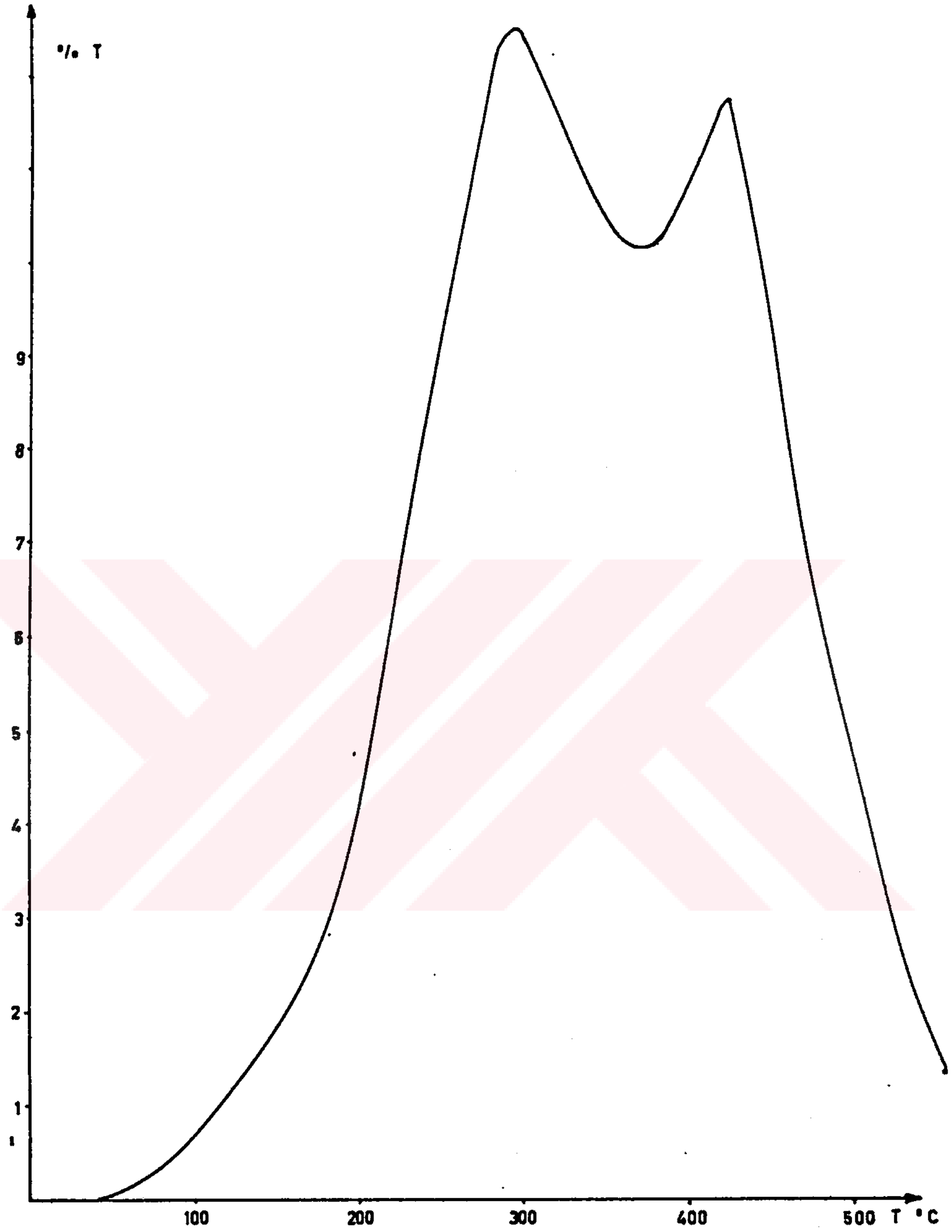


Figure 5.16 Glow curve of CaF_2 ; 0.460 mg, 500 nm.

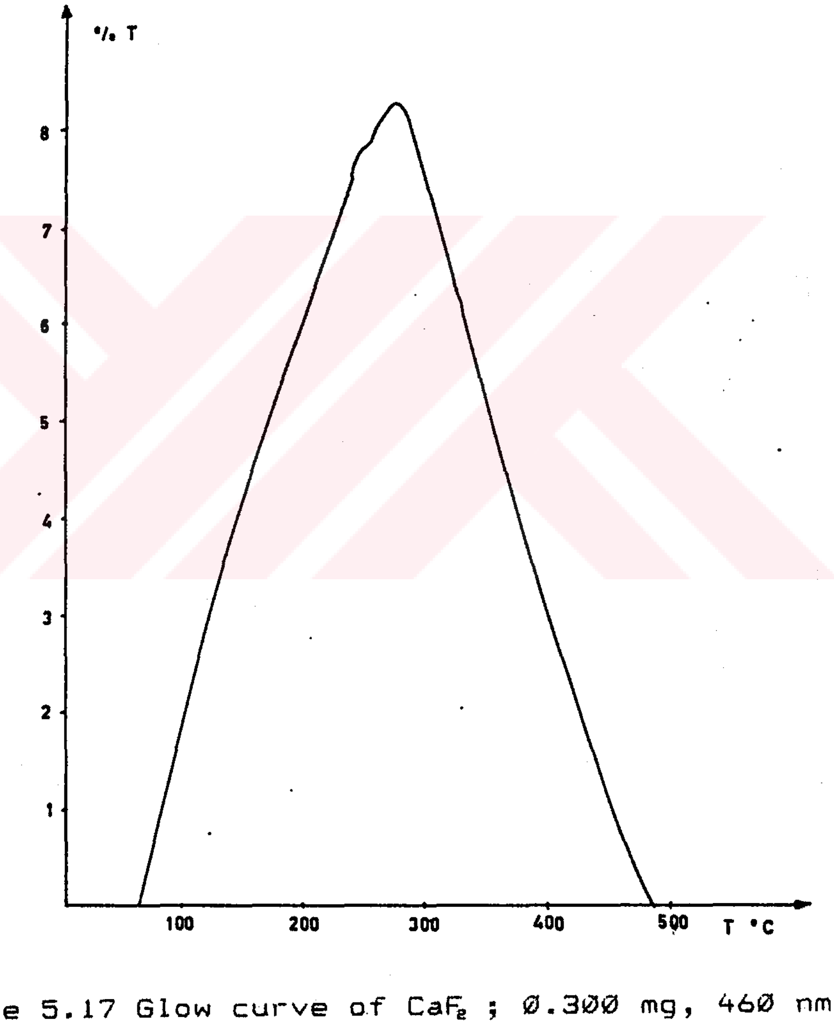


Figure 5.17 Glow curve of CaF_2 ; 0.300 mg, 460 nm.

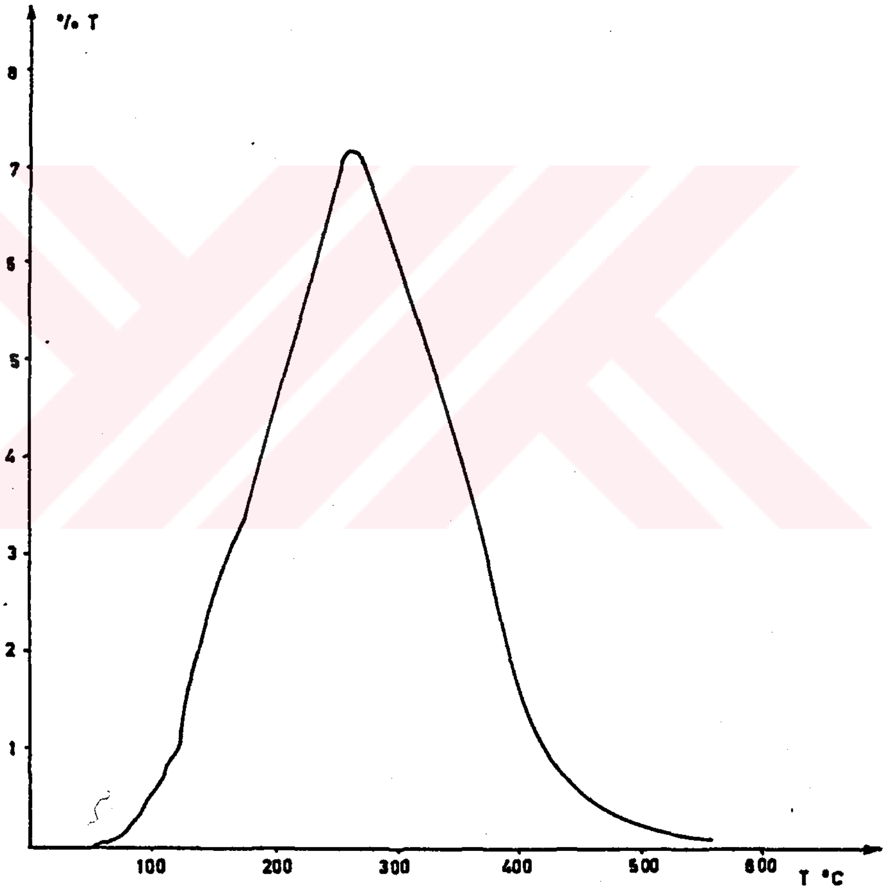


Figure 5.18 Glow curve of CaF_2 ; 0.270 mg, 460 nm.

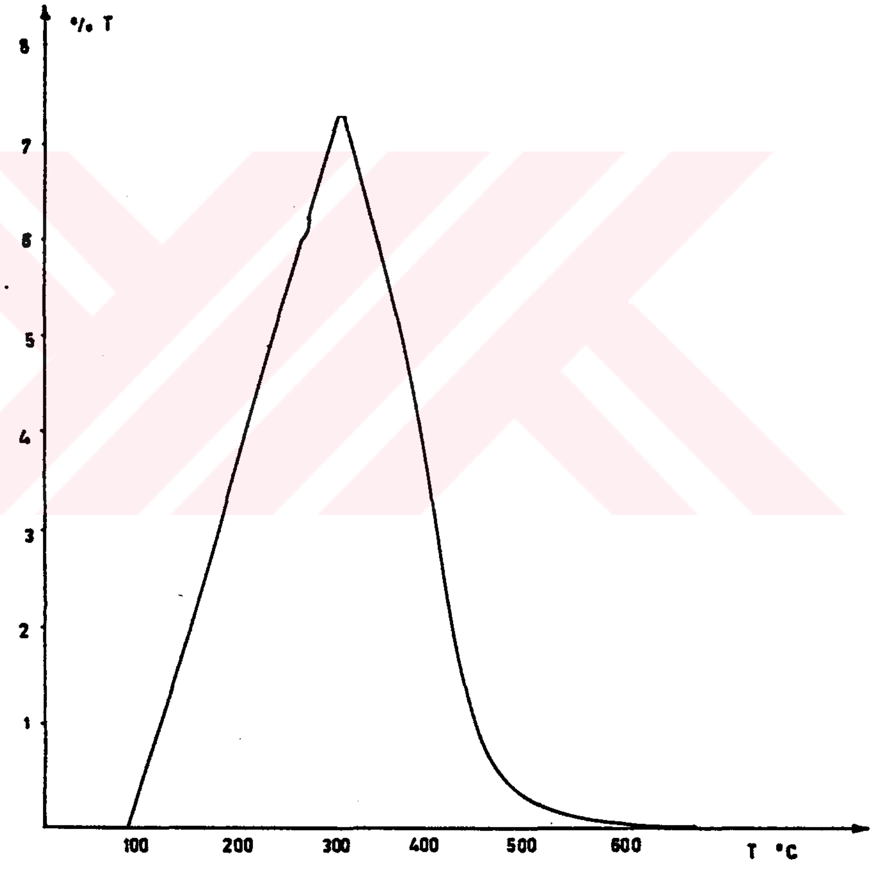


Figure 5.19 Glow curve of CaF_2 ; 0.255 mg, 460 nm.

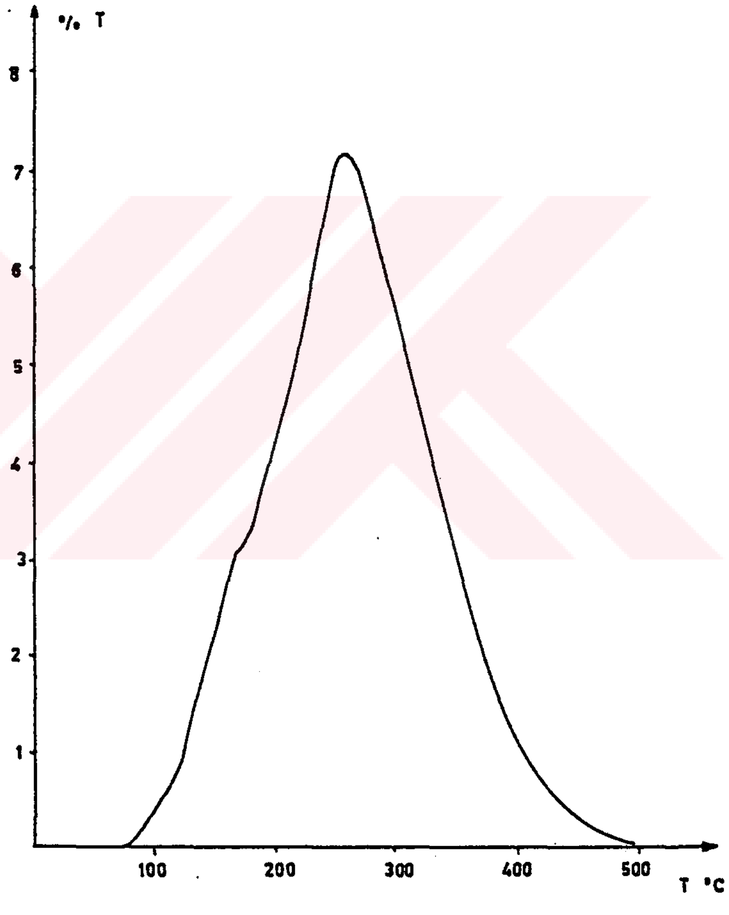


Figure 5.20 Glow curve of CaF_2 ; 0.210 mg, 460 nm.

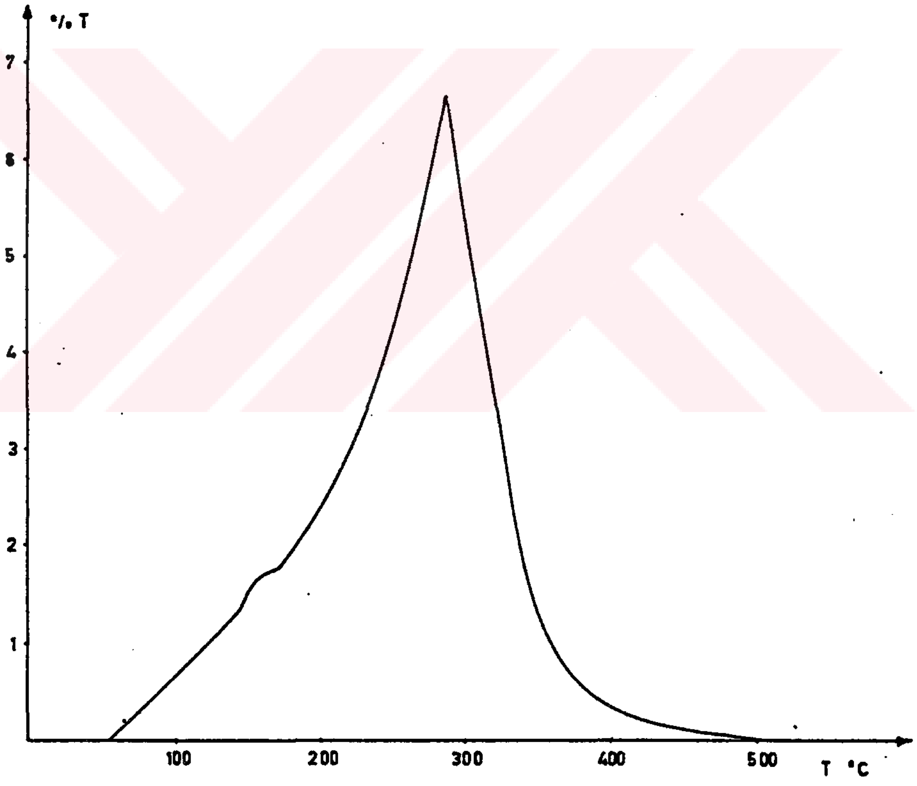


Figure 5.21 Glow curve of CaF_2 ; 0.195 mg, 460 nm.

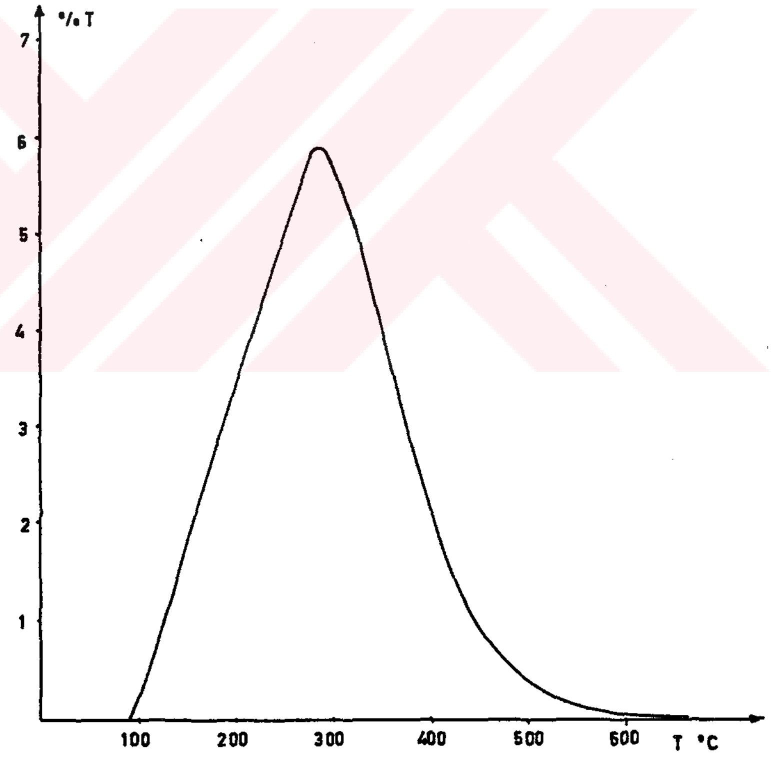


Figure 5.22 Glow curve of CaF$_2$; 0.170 mg, 460 nm.

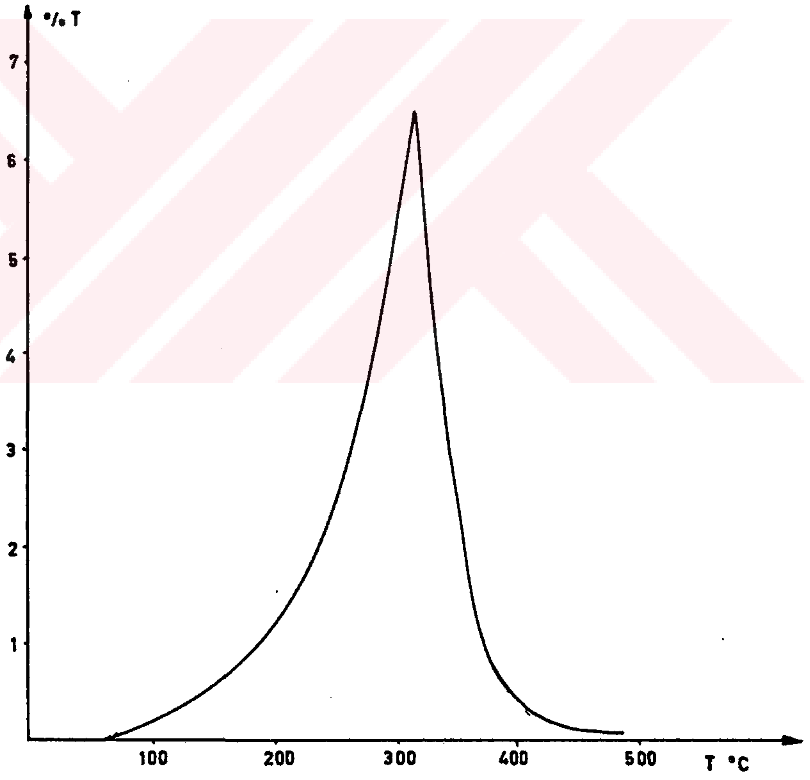


Figure 5.23 Glow curve of CaF_2 ; 0.215 mg, 430 nm.

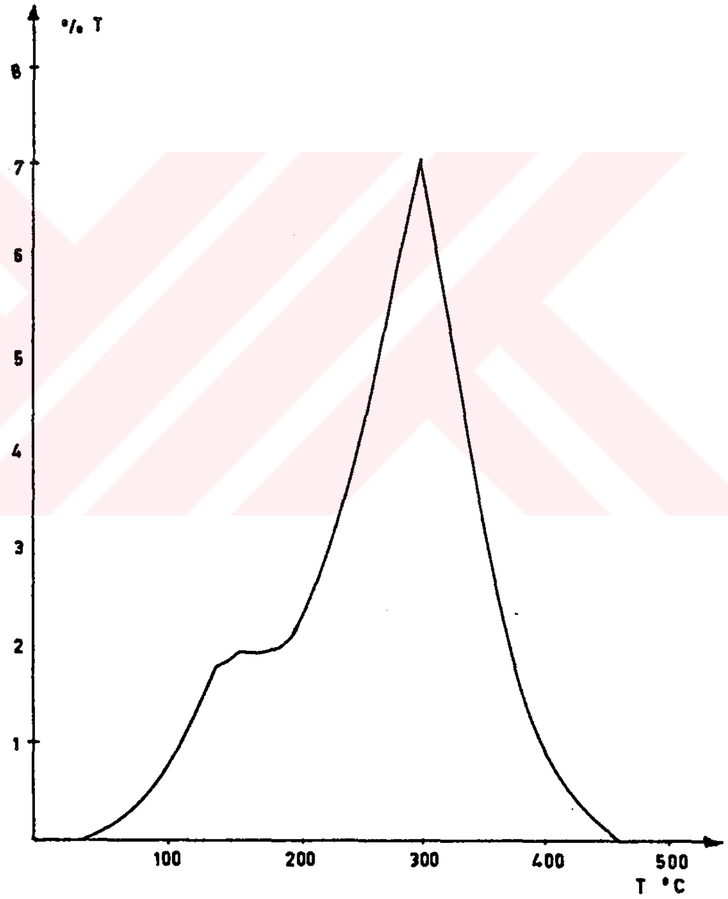


Figure 5.24 Glow curve of CaF_2 ; 0.294 mg, 430 nm.

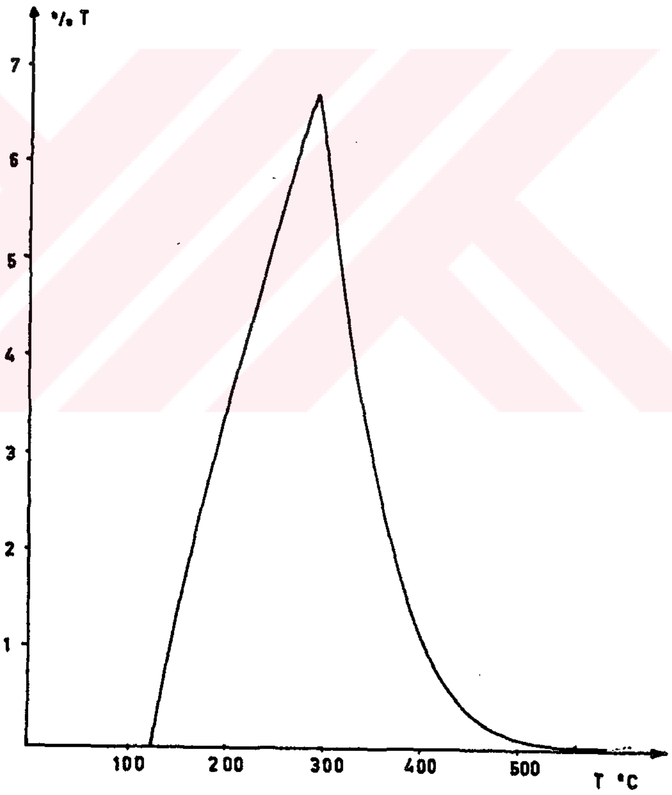


Figure 5.25 Glow curve of CaF_2 ; 0.250 mg, 430 nm.

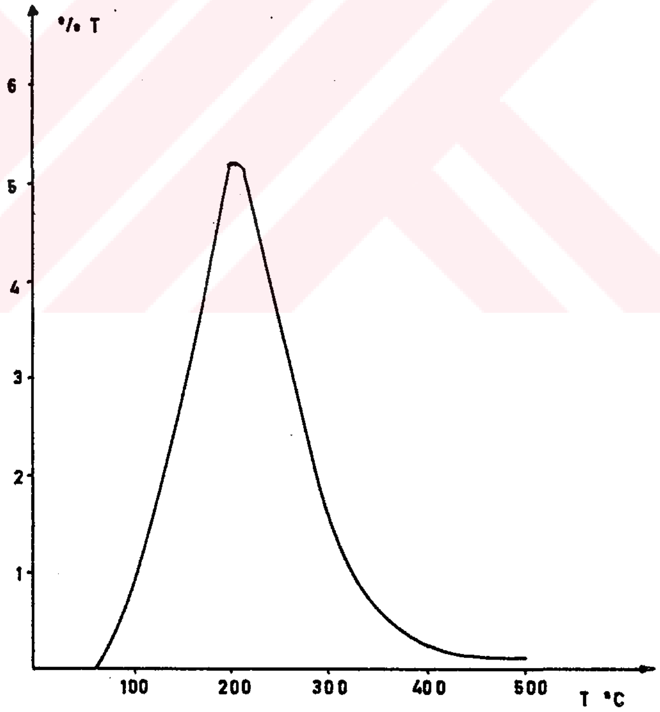


Figure 5.26 Glow curveof CaF_2; 0.150 mg, 430 nm.

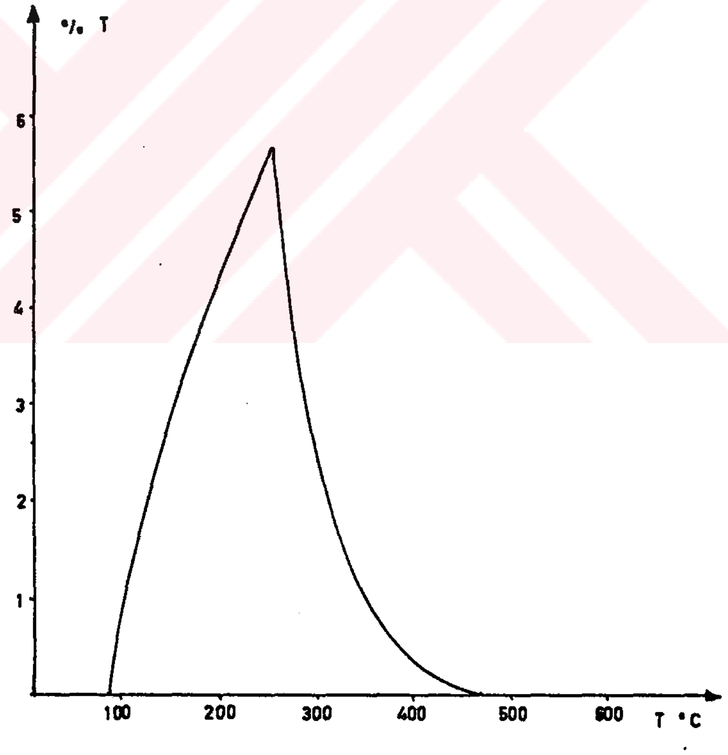


Figure 5.27 Glow curve of CaF_2 ; 0.192 mg, 430 nm.

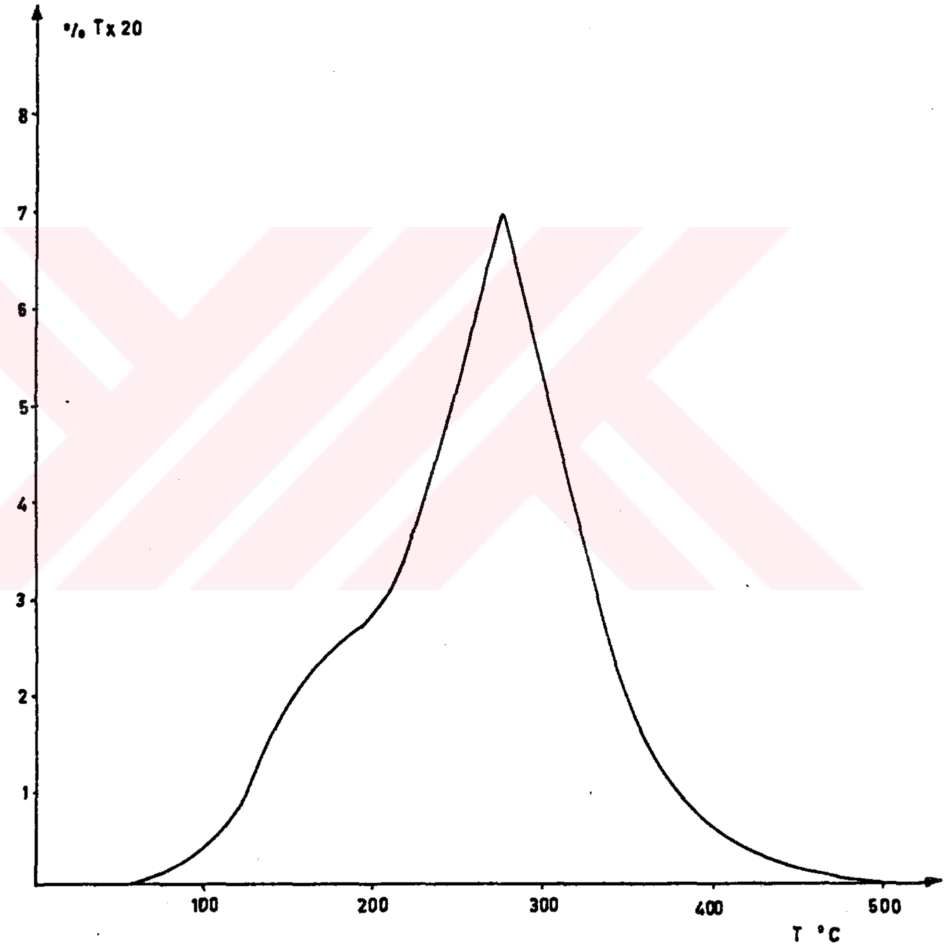


Figure 5.28 Glow curve of CaF_2 ; 0.100 mg, 500 nm. After exposure to X-ray radiation.

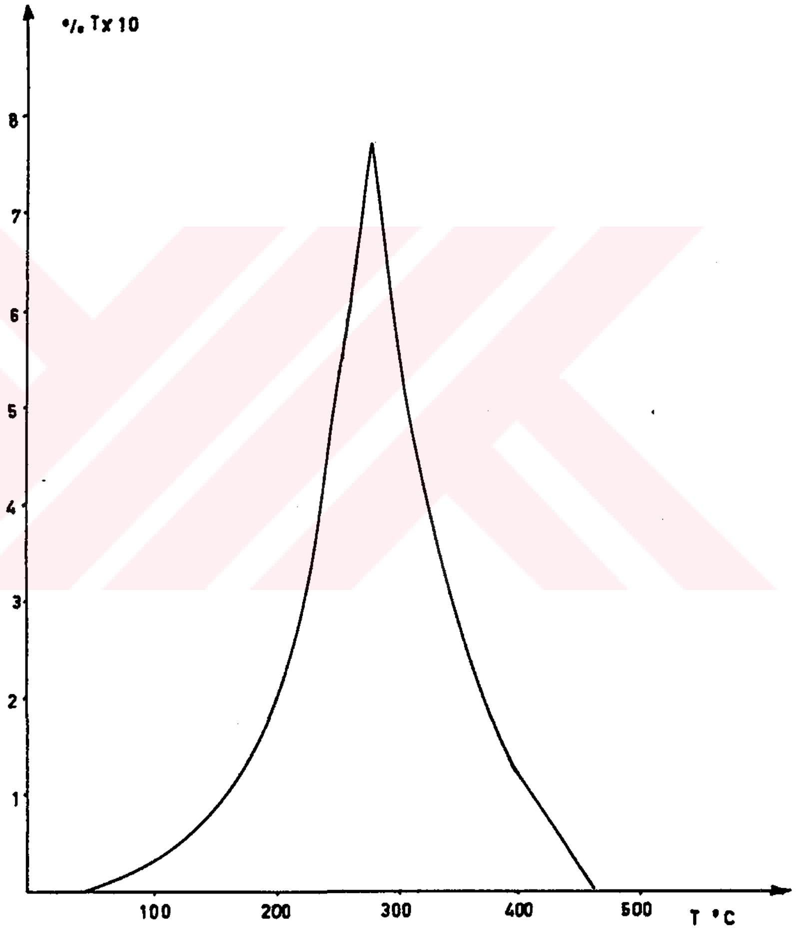


Figure 5.29 Glow curve of CaF_2 ; 0.217 mg, 500 nm. After exposure to X-ray radiation.

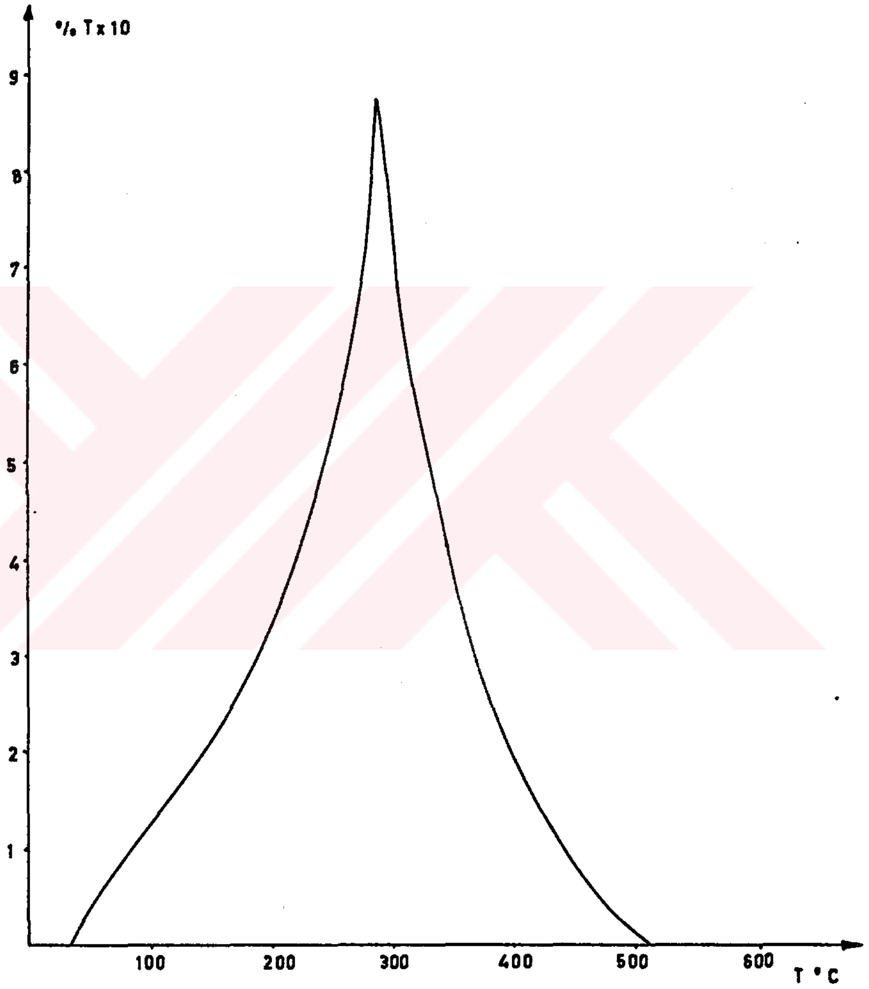


Figure 5.30 Glow curve of CaF_2 ; 0.245 mg, 500 nm. After exposure to X-ray radiation.

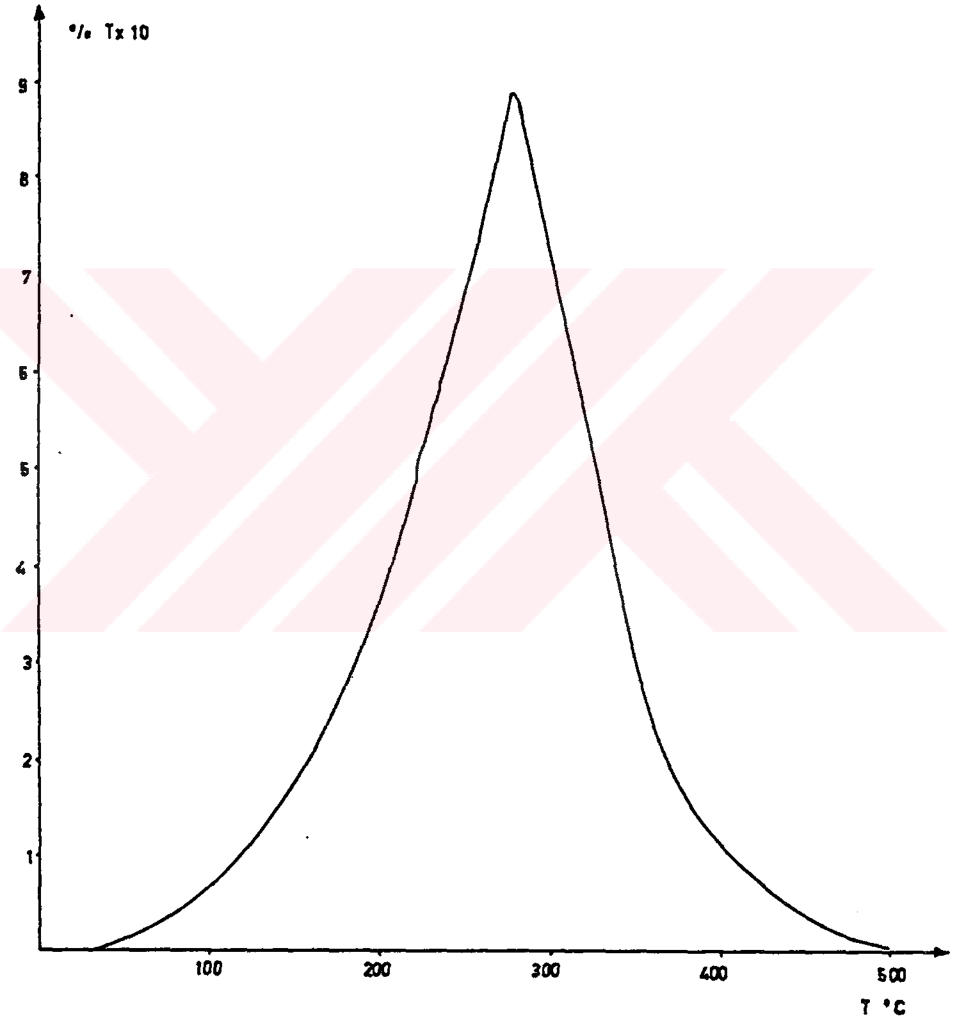


Figure 5.31 Glow curve of caF$_2$; 0.274 mg. 500 nm.
After exposure to X-ray radiation.

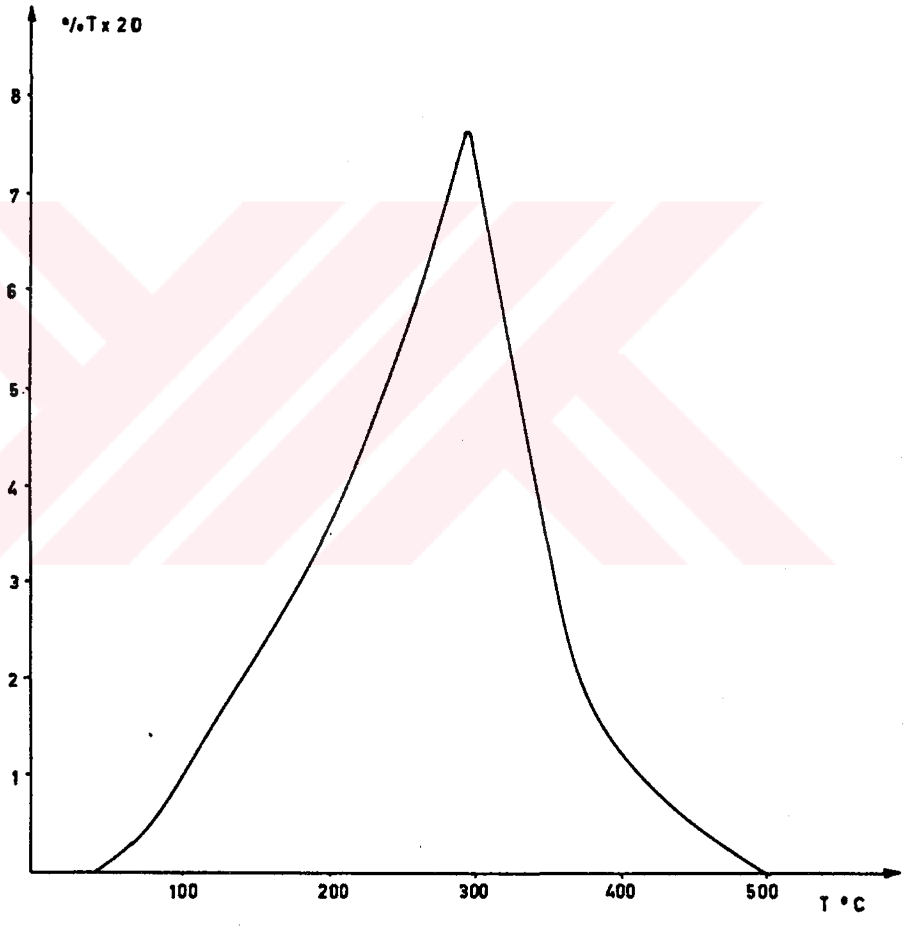


Figure 5.32 Glow curve of CaF_2 ; 0.340 mg, 650 nm. After exposure to X-ray radiation.

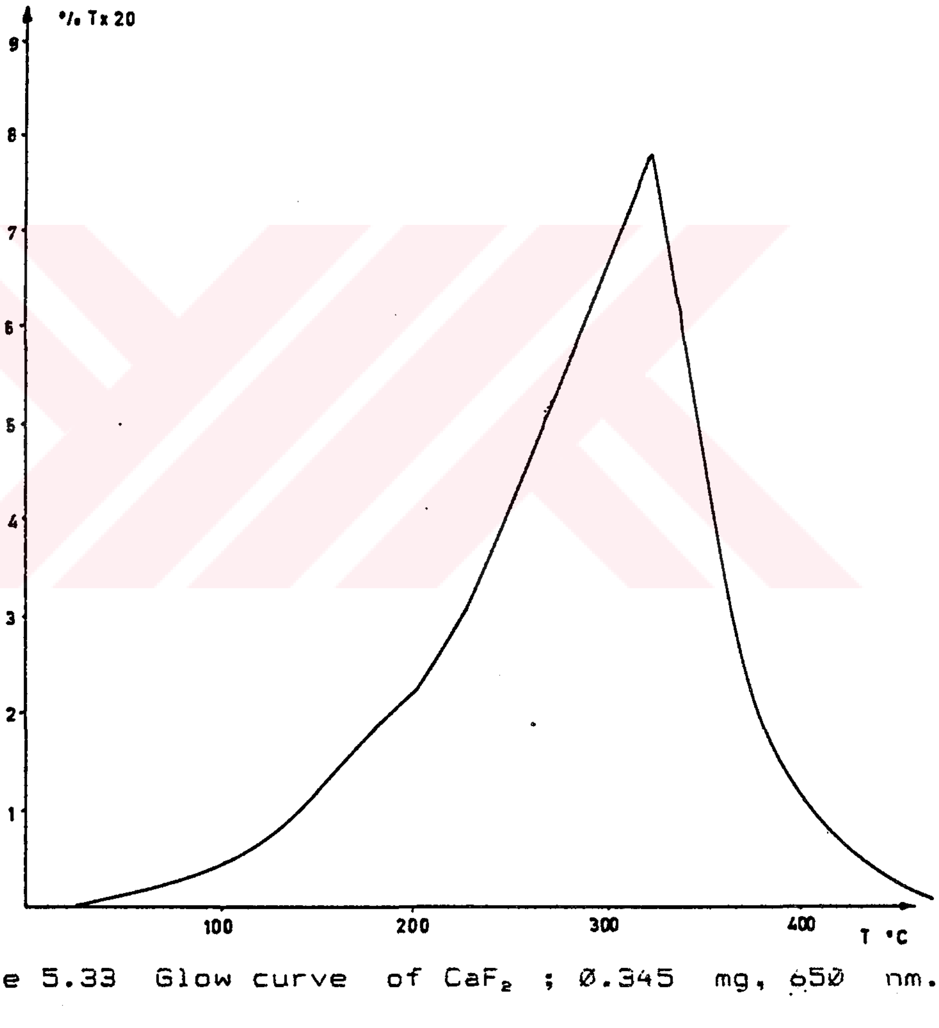


Figure 5.33 Glow curve of CaF_2 ; 0.345 mg, 650 nm. After exposure to X-ray radiation.

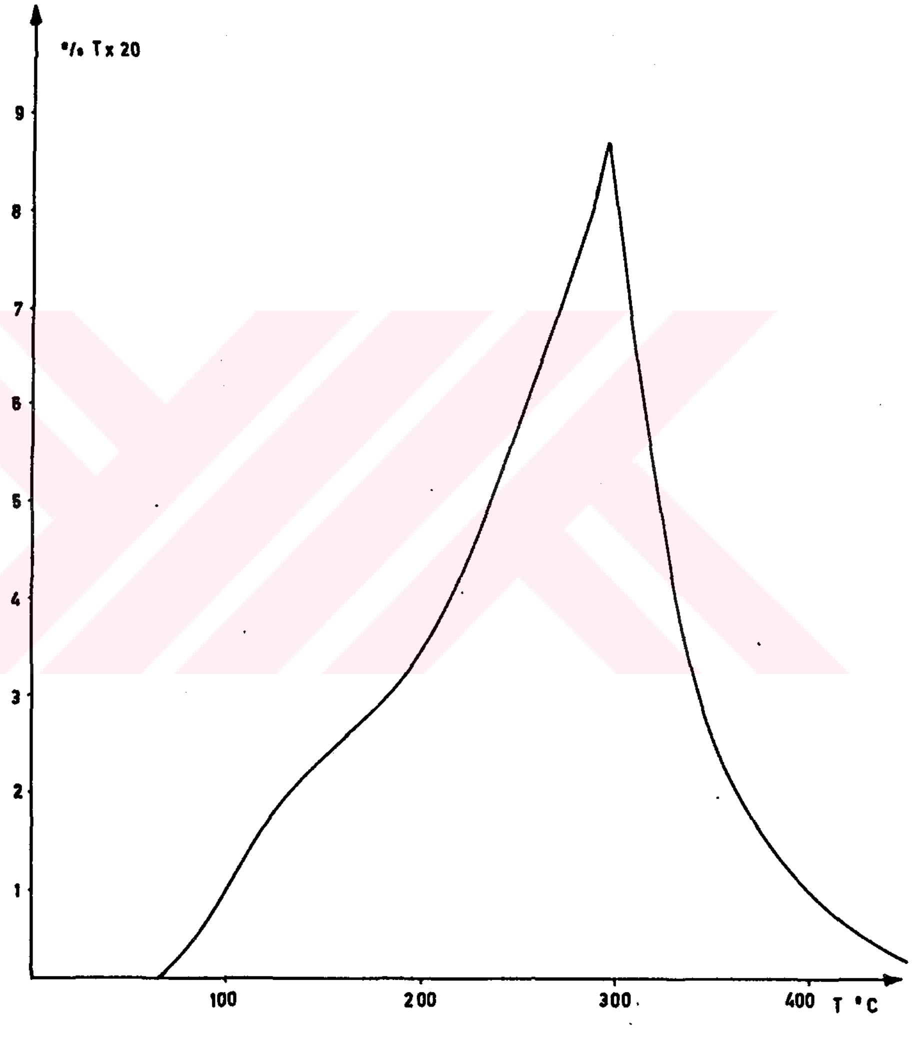


Figure 5.34 Glow curve of CaF_2 ; 0.380 mg, 650 nm. After exposure to X-ray radiation.

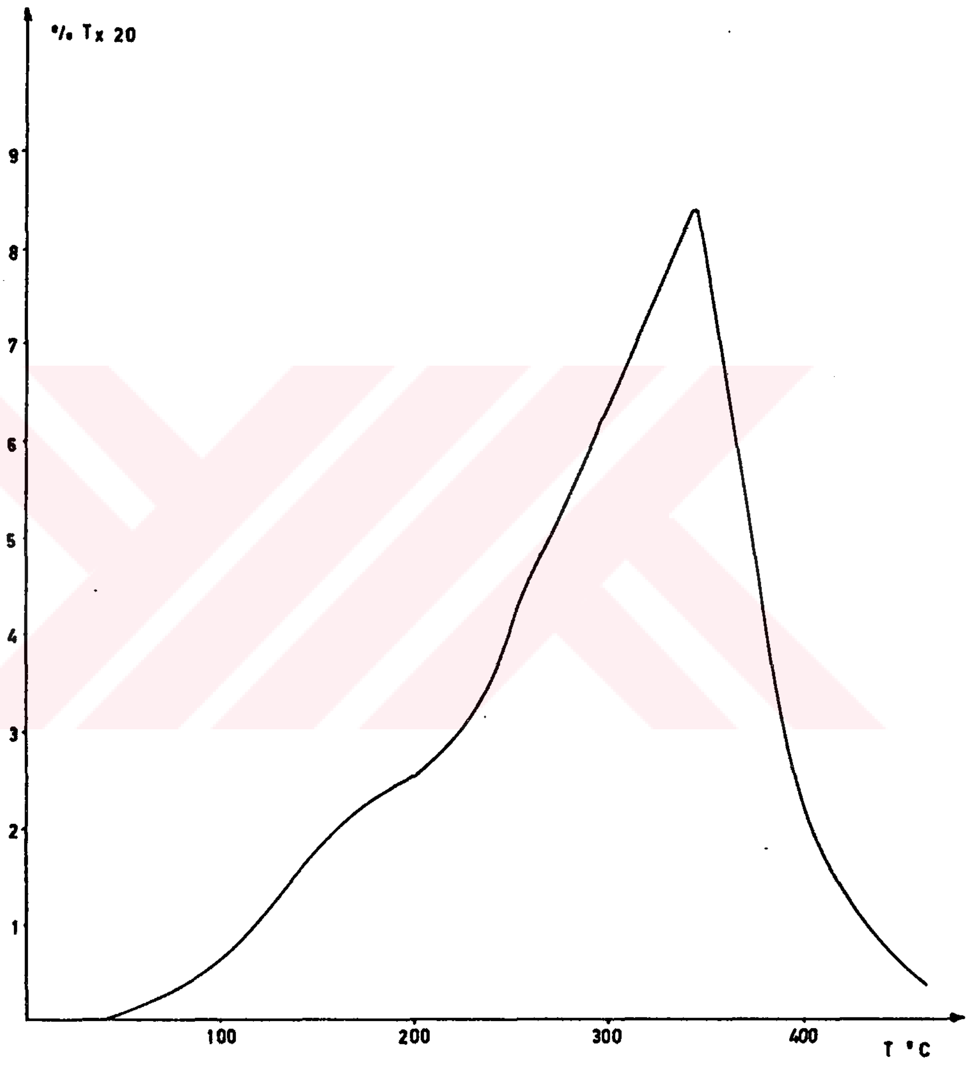


Figure 5.35 Glow curve of CaF_2 ; 0.390 mg, 650 nm. After exposure to X-ray radiation.

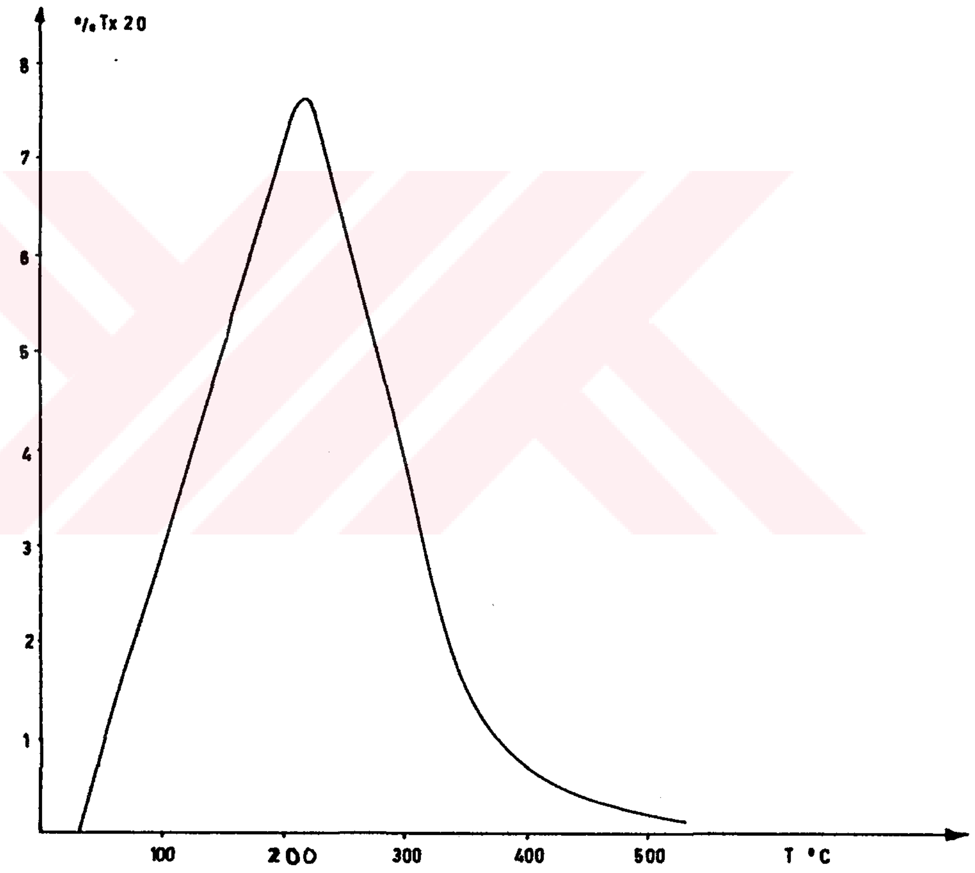


Figure 5.36 Glow curve of CaF_2 ; 0.340 mg, 650 nm. After exposure to X-ray radiation.

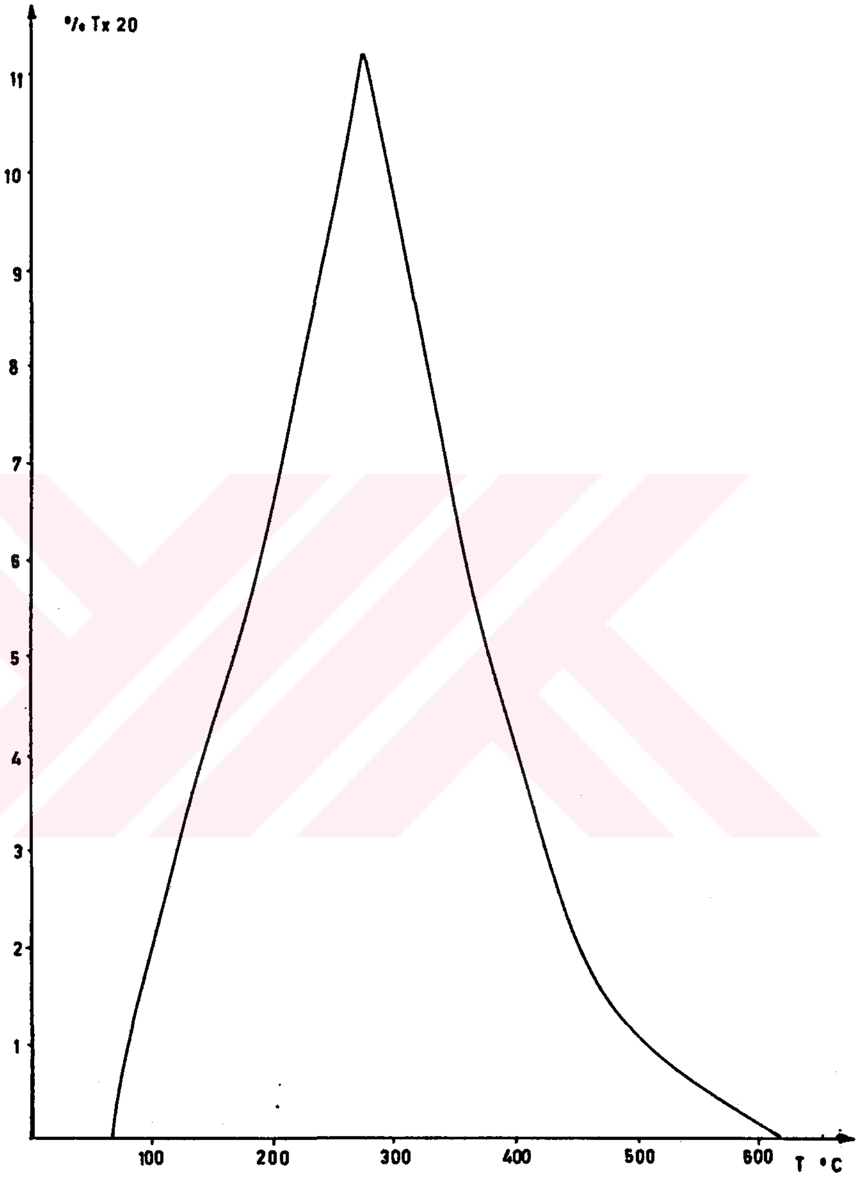


Figure 5.37 Glow curve of CaF_2 ; 0.410 mg, 650 nm. After exposure to X-ray radiation.

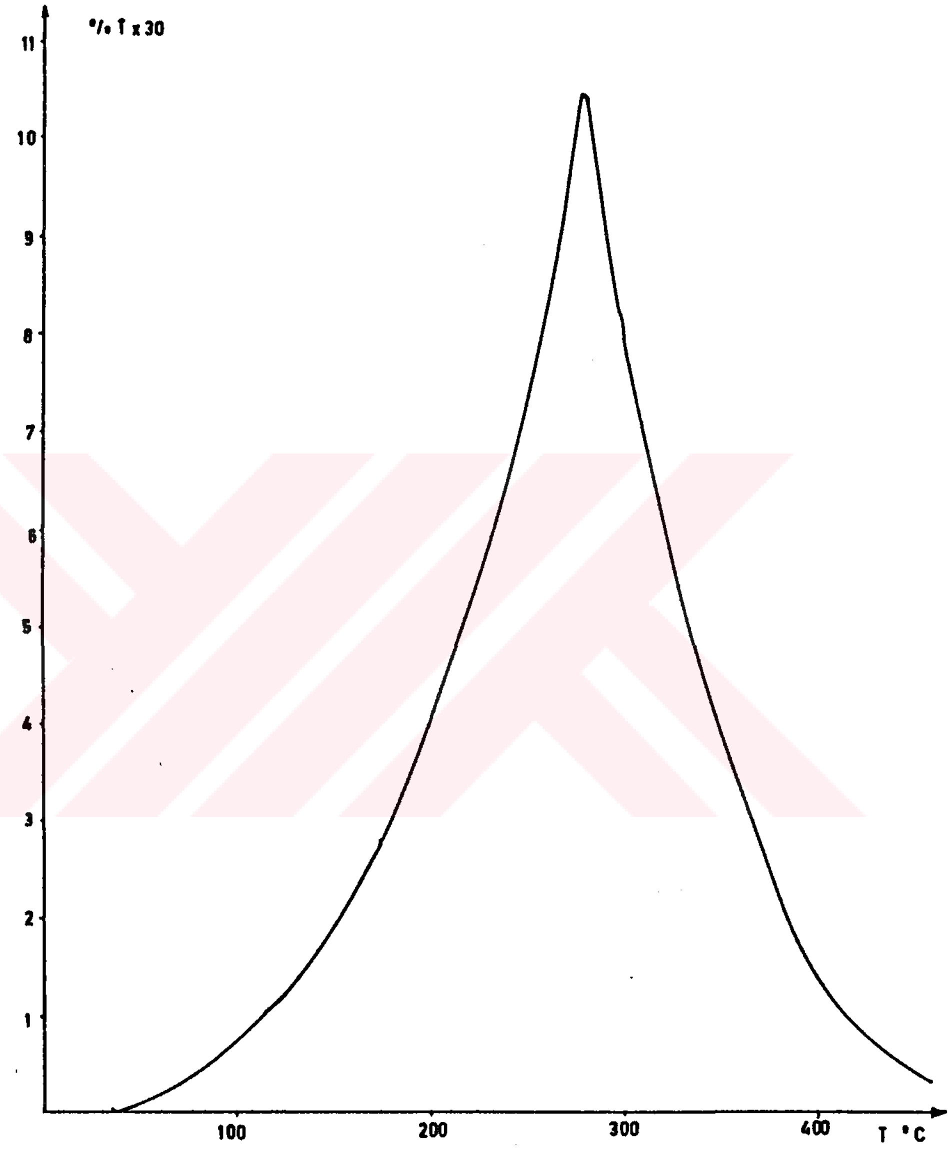


Figure 5.38 Glow curve of CaF_2 ; 0.332 mg, 460 nm. After exposure to X-ray radiation.

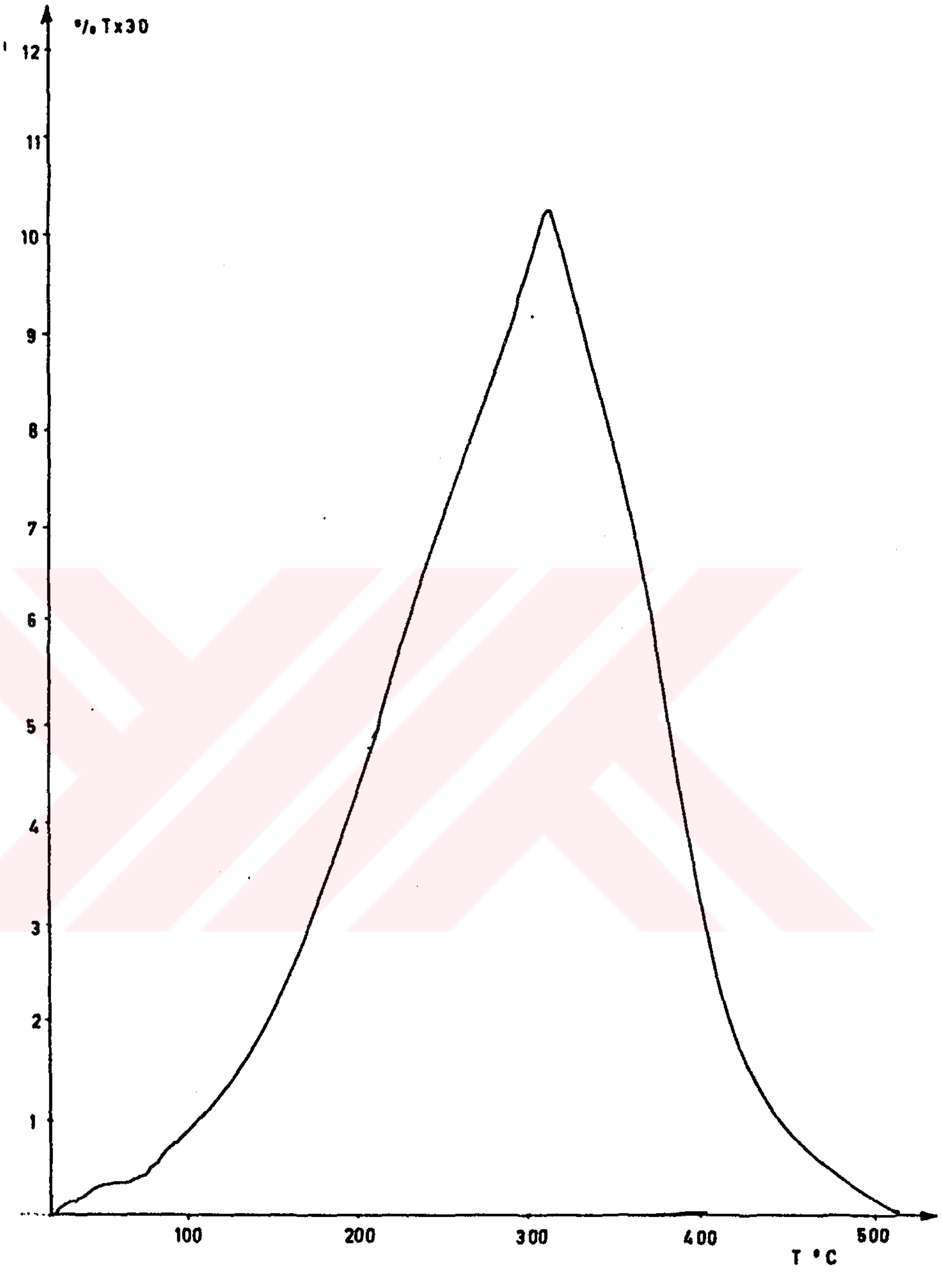


Figure 5.39 Glow curve of CaF_2 ; 0.365 mg, 460 nm. After exposure to X-ray radiation.

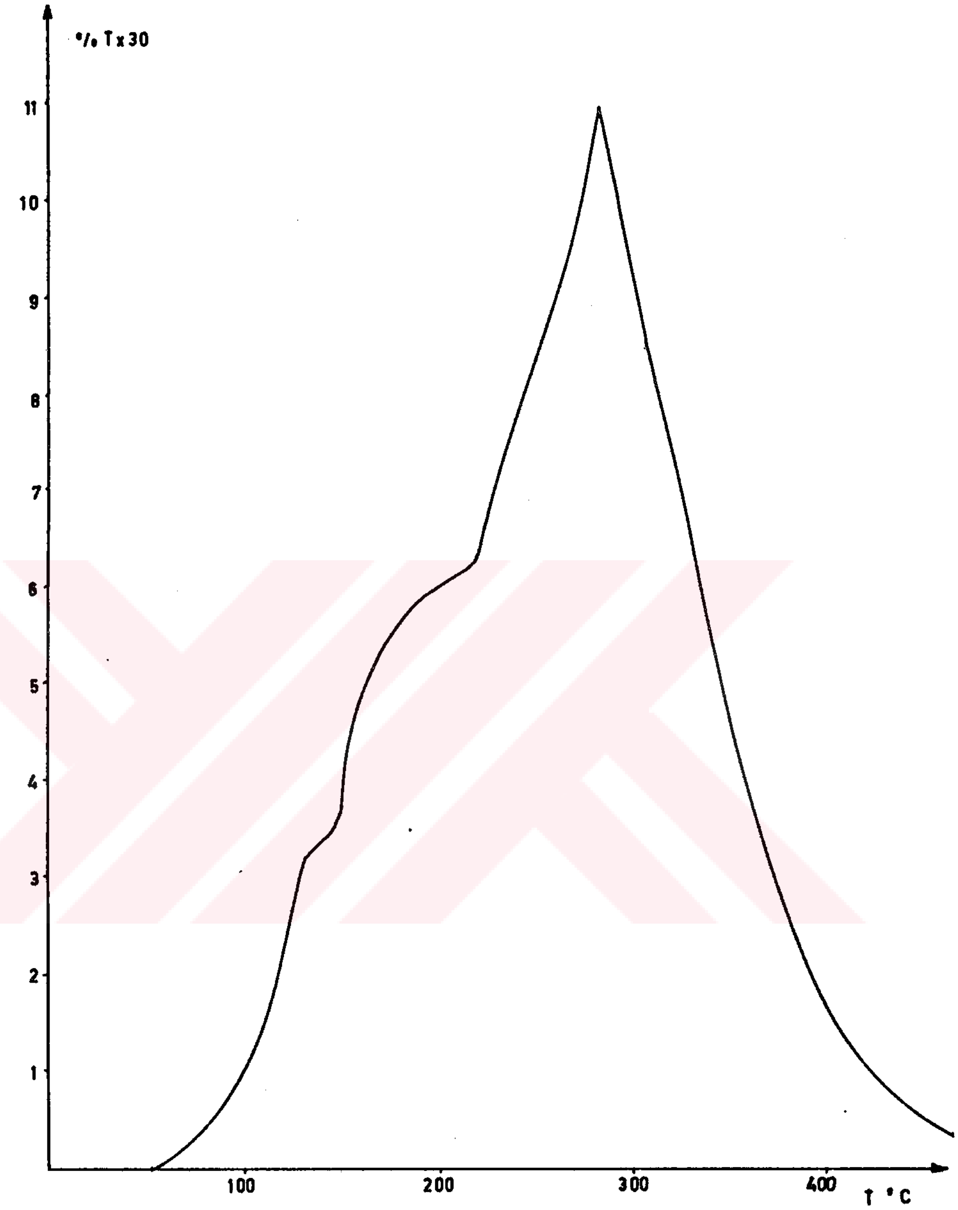


Figure 5.40 Glow curve of CaF_2 ;0.385 mg, 460 nm. After exposure to X-ray radiation.

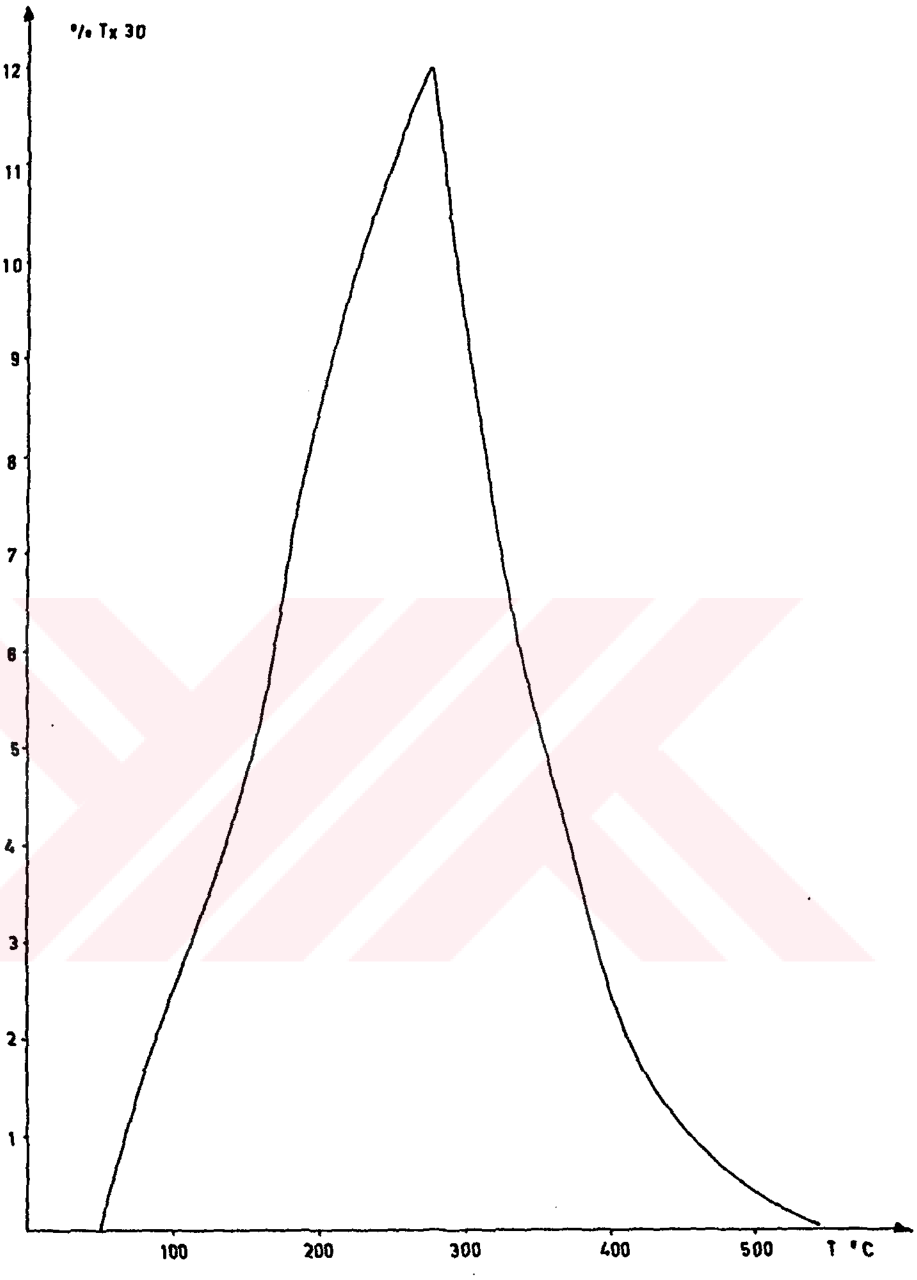


Figure 5.41 Glow curve of CaF_2 ; 0.400 mg, 460 nm. After exposure to X-ray radiation.

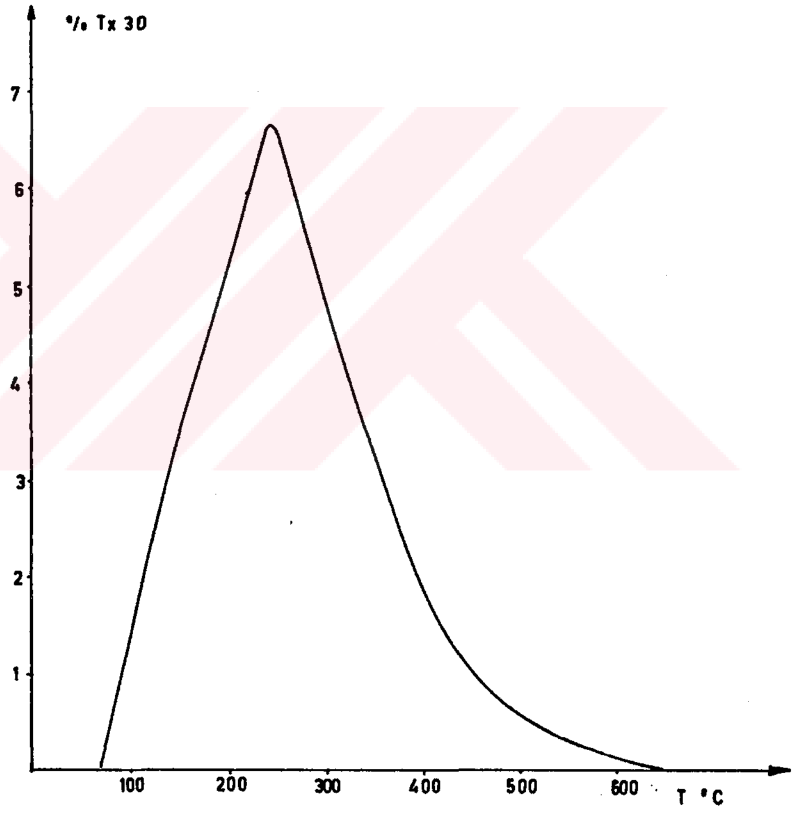


Figure 5.42 Glow curve of CaF_2 ; 0.245 mg, 430 nm. After exposure to X-ray radiation.

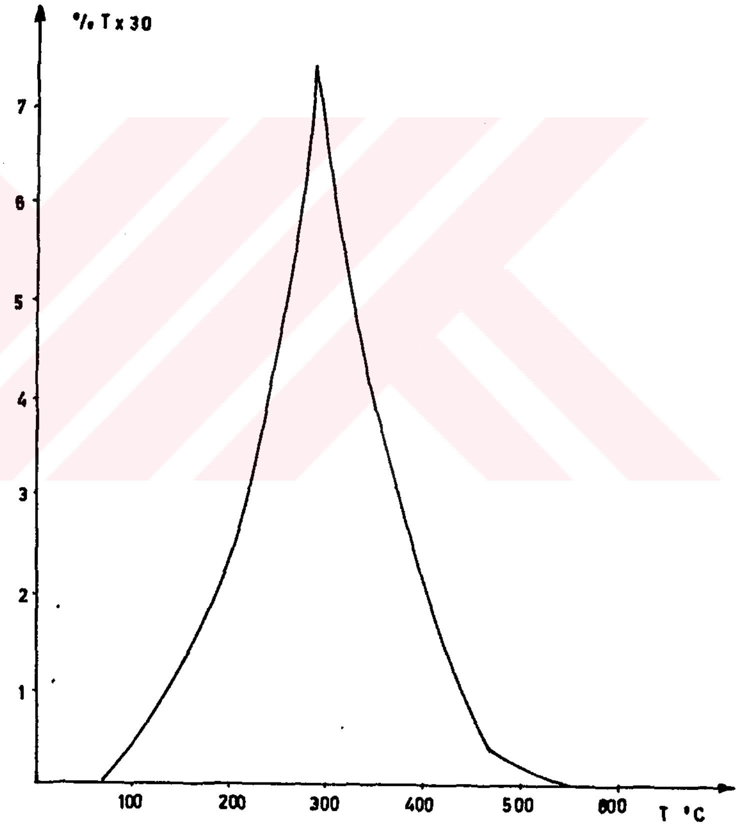


Figure 5.43 Glow curve of CaF_2 ; 0.255 mg, 430 nm. After exposure to X-ray radiation.

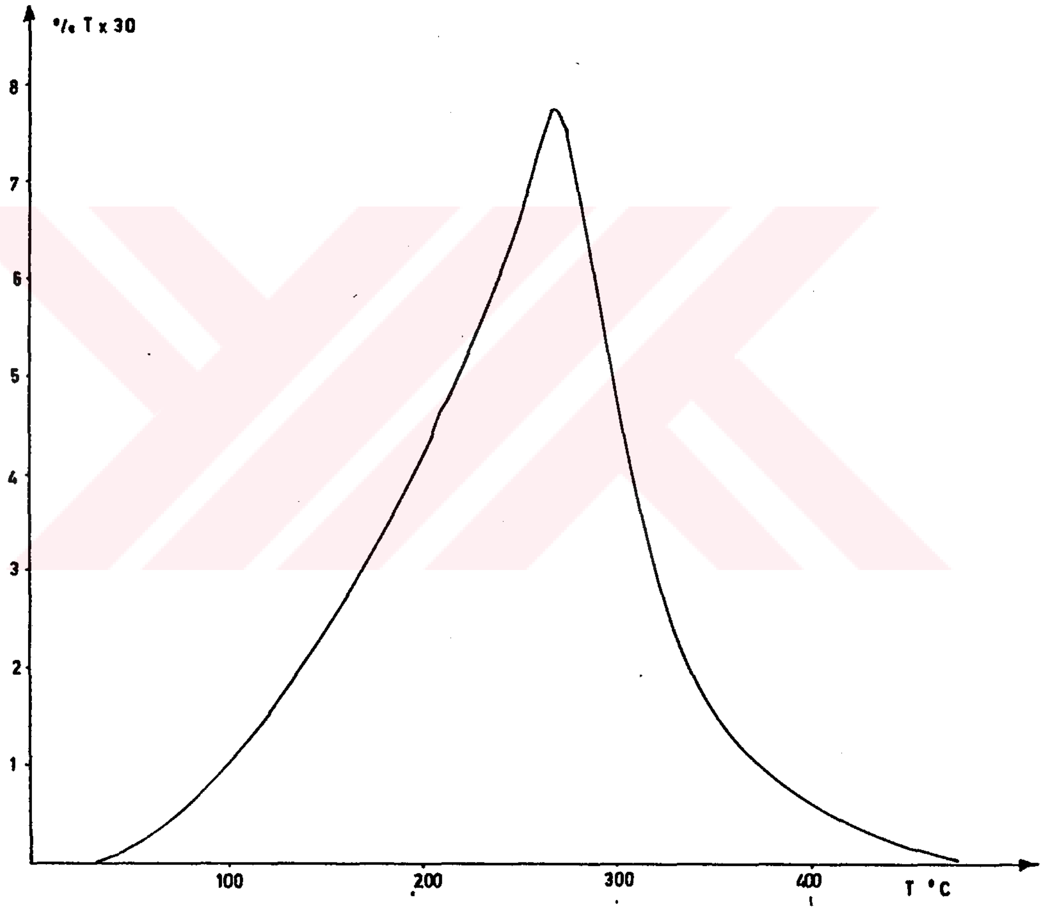


Figure 5.44 Glow curve of CaF_2 ; 0.272 mg, 430 nm. After exposure to X-ray radiation.

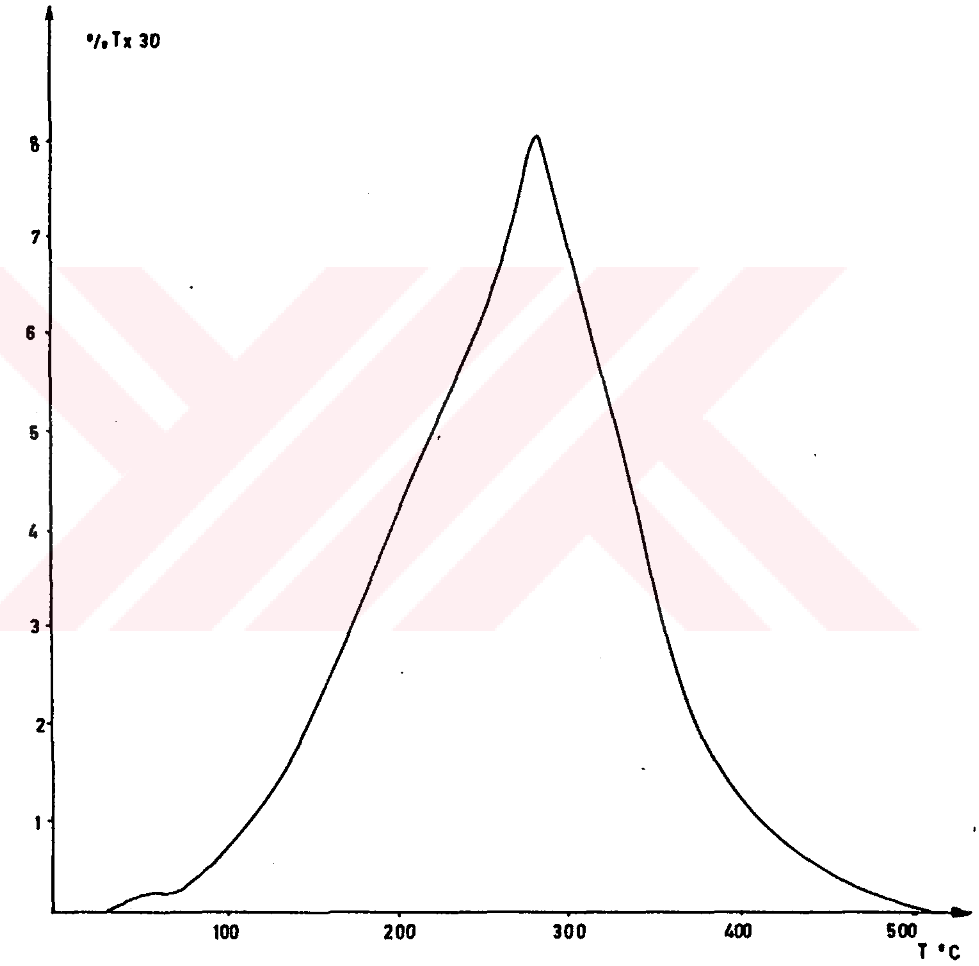


Figure 5.45 Glow curve of CaF_2 ; 0.300 mg, 430 nm. After exposure to X-ray radiation.

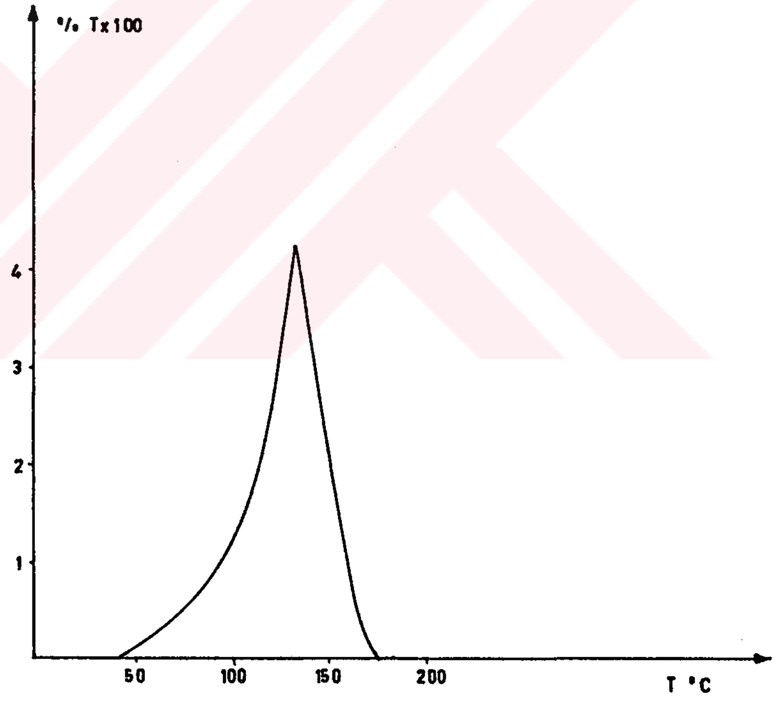


Figure 5.46 Glow curve of teflon dosimeter; 430 nm. After exposure to X-ray radiation.

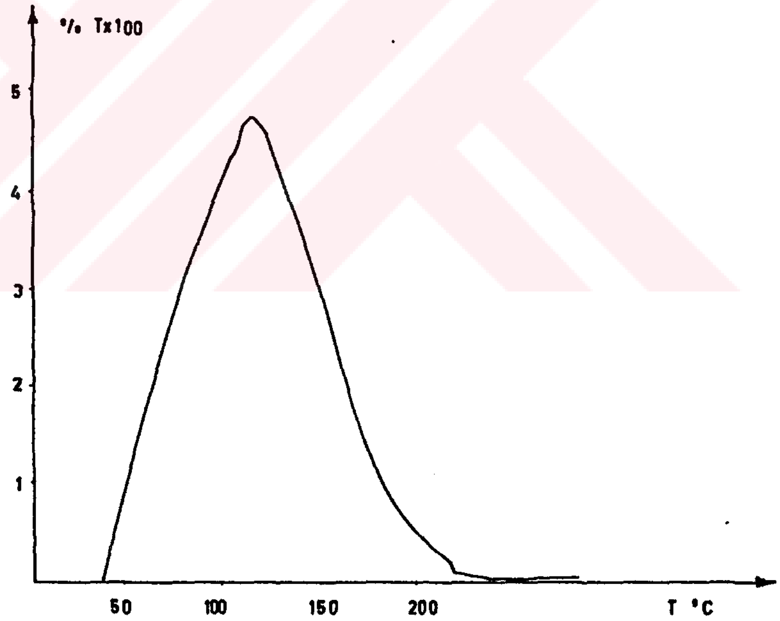


Figure 5.47 Glow curve of teflon dosimeter; 430 nm. After exposure to X-ray radiation.

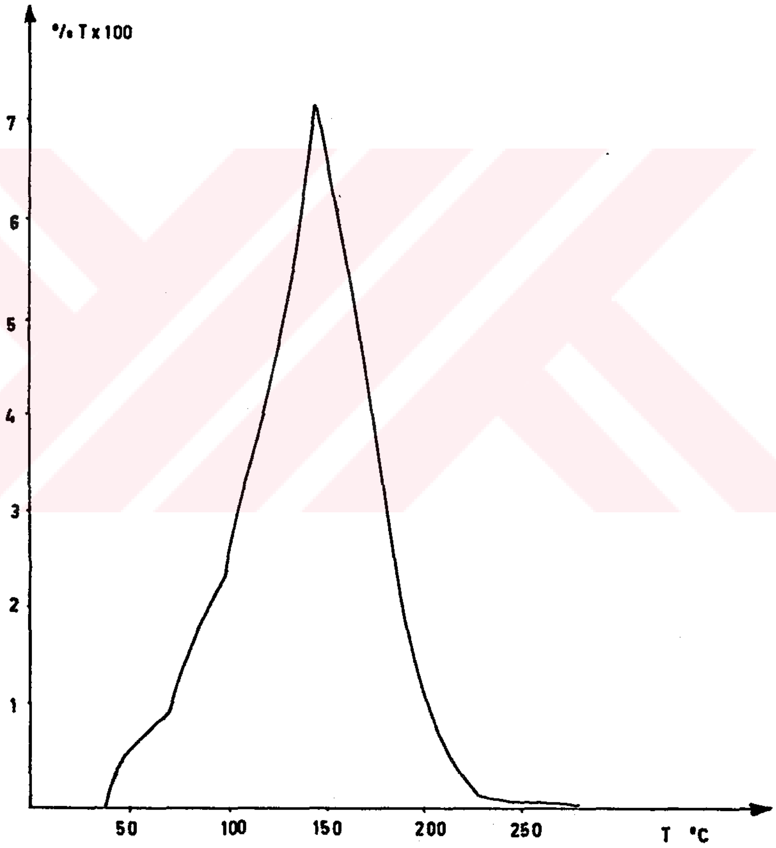


Figure 5.48 Glow curve of teflon dosimeter; 430 nm. After exposure to X-ray radiation.

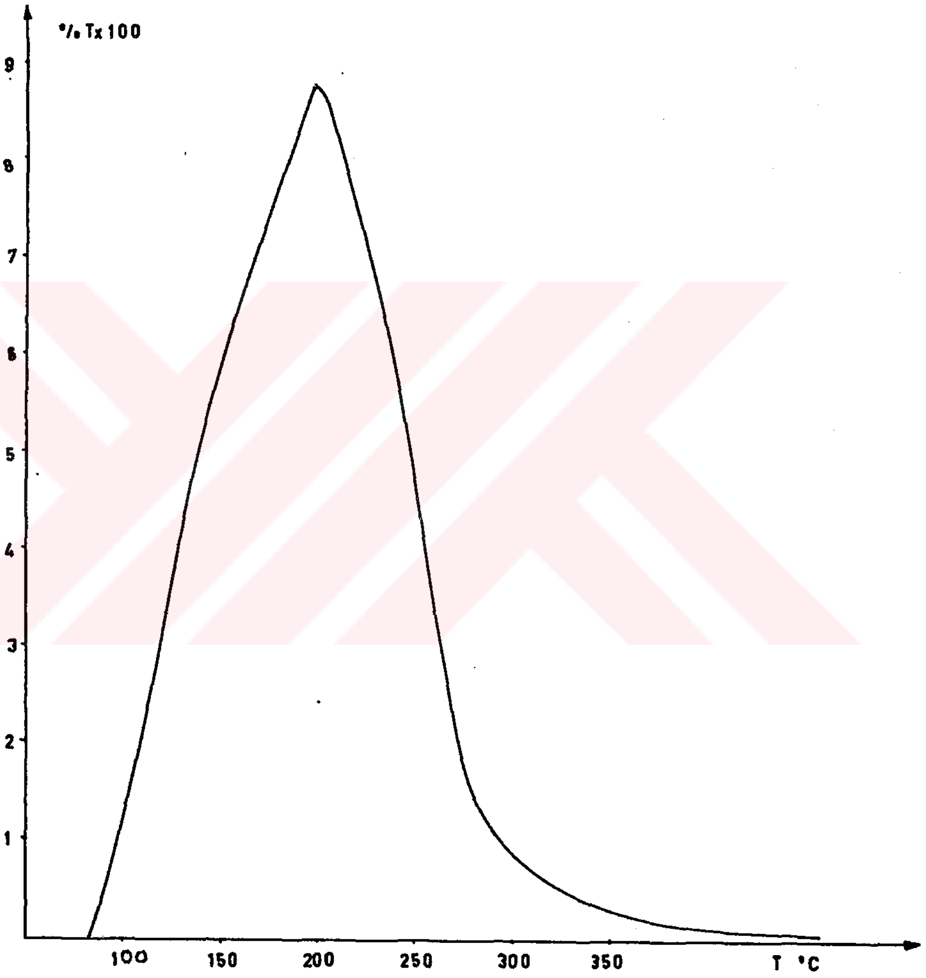


Figure 5.49 Glow curve of teflon dosimeter; 460nm. After exposure to X-ray radiation.

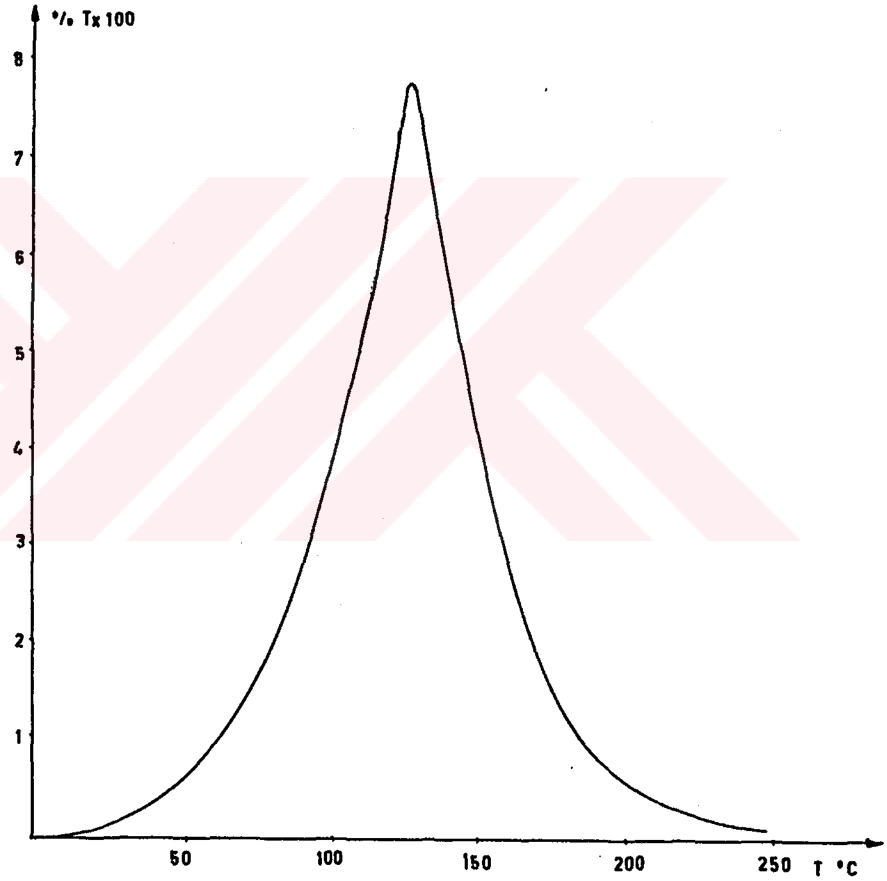


Figure 5.50 Glow curve of teflon dosimeter; 460 nm. After exposure to X-ray radiation.

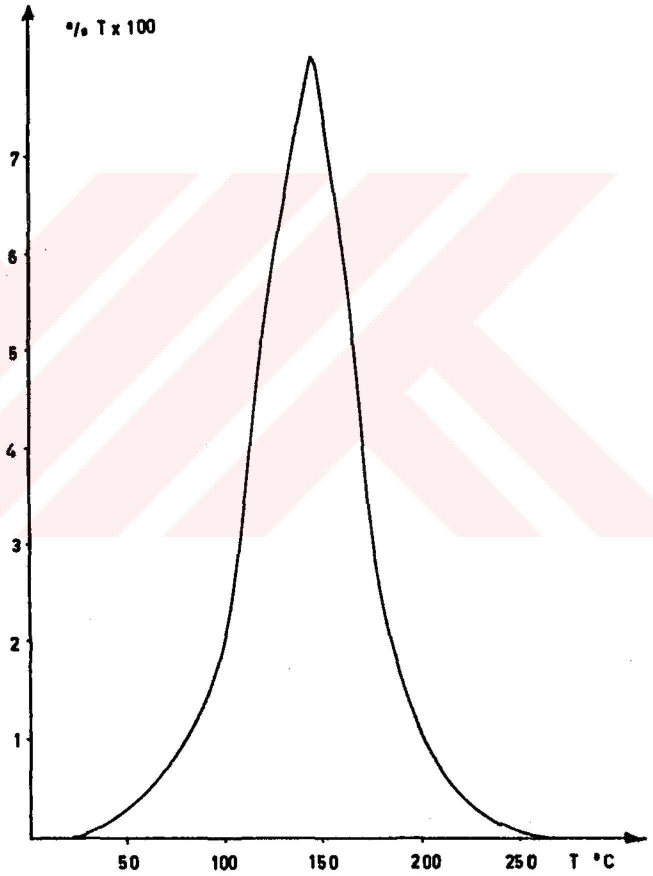


Figure 5.51 Glow curve of teflon dosimeter; 460 nm. After exposure to λ-ray radiation.

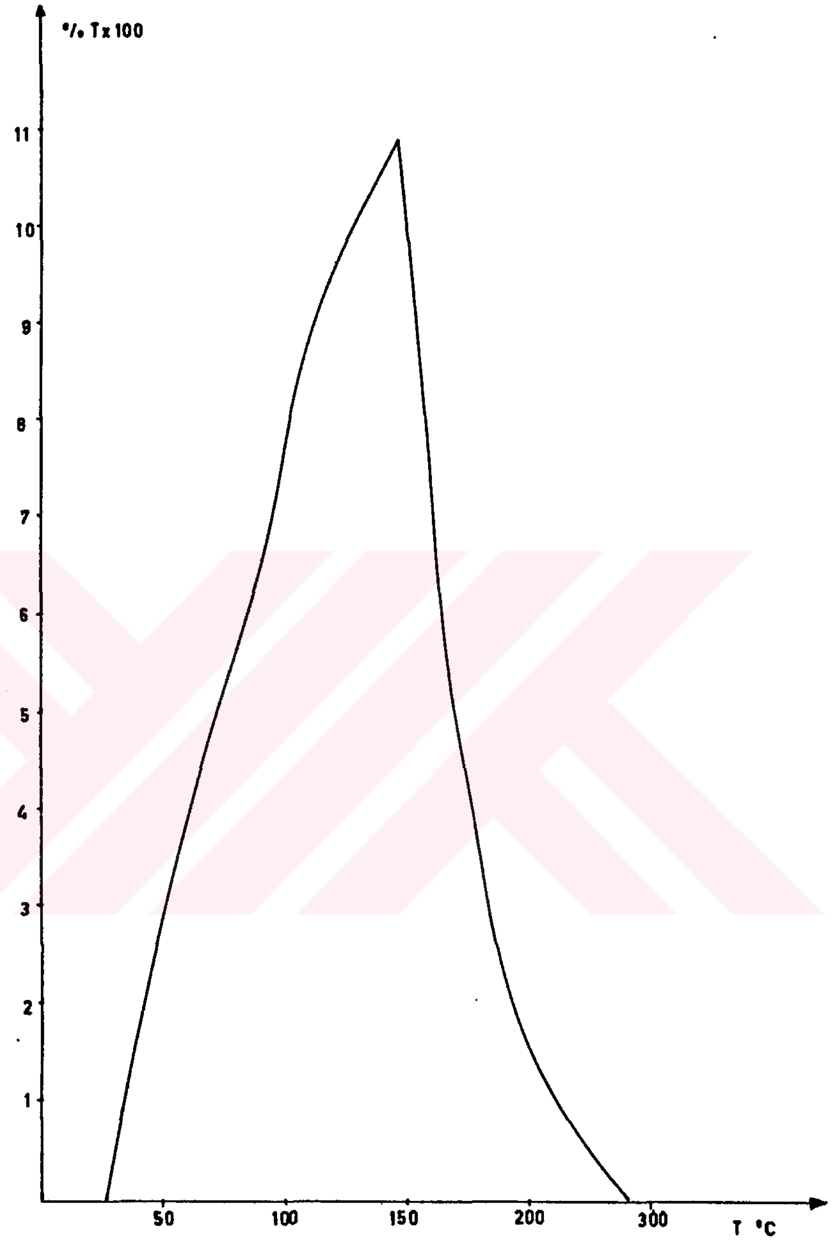


Figure 5.52 Glow curve of teflon dosimeter; 500 nm. After exposure to X-ray radiation.

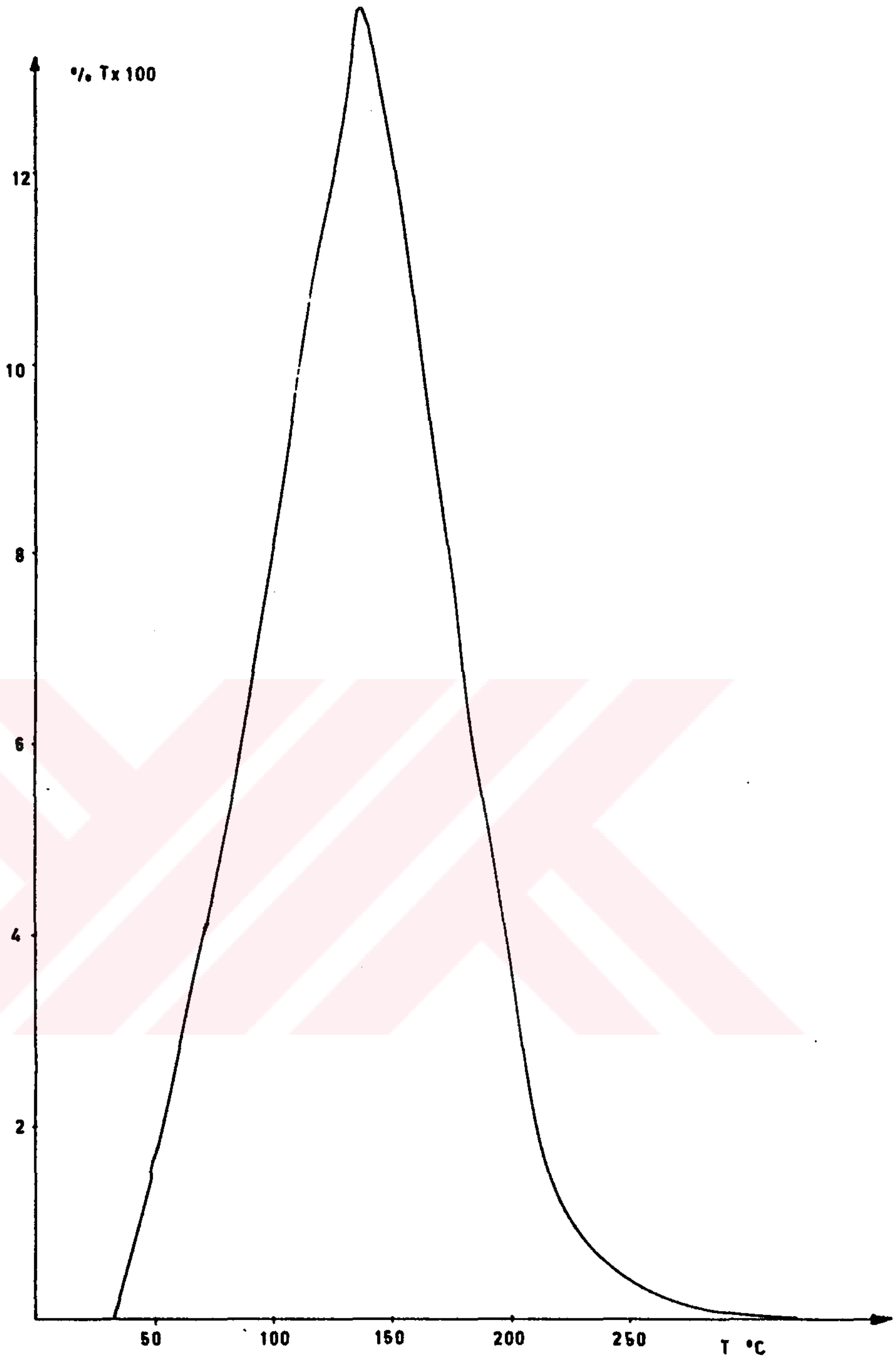


Figure 5.53 Glow curve of teflon dosimeter; 500 nm. After exposure to X-ray radiation.

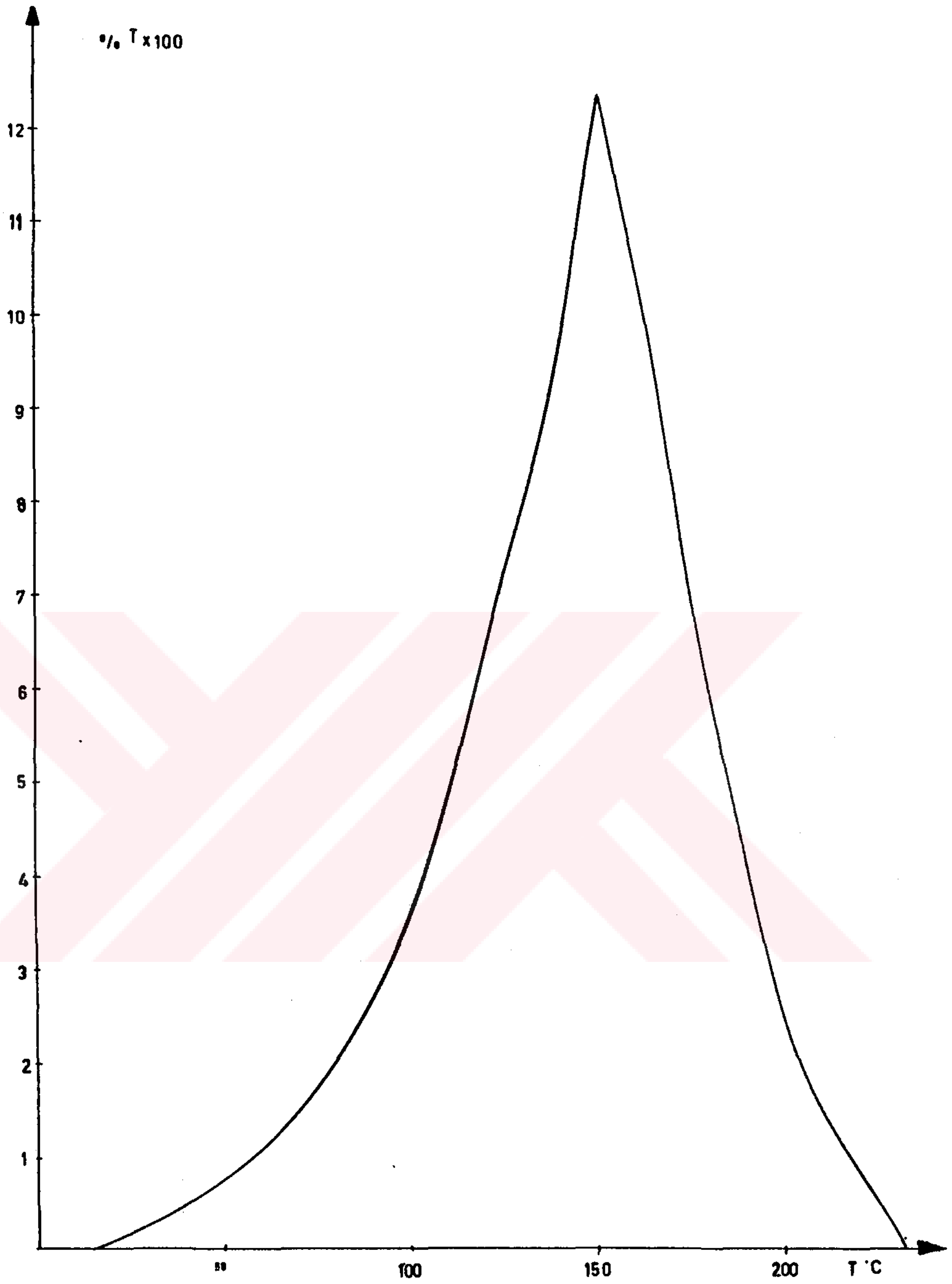


Figure 5.54 Glow curve of teflon dosimeter; 500 nm. After exposure to X-ray radiation.

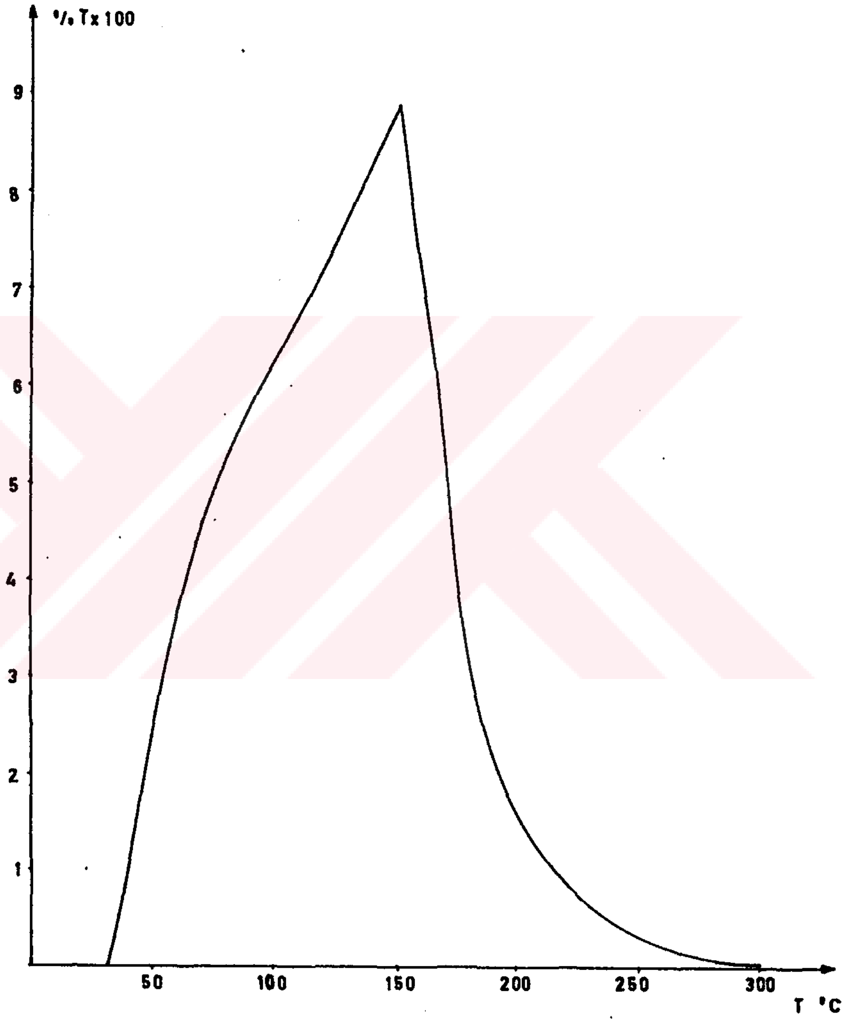


Figure 5.55 Glow curve of teflon dosimeter; 650 nm. After exposure to X-ray radiation.

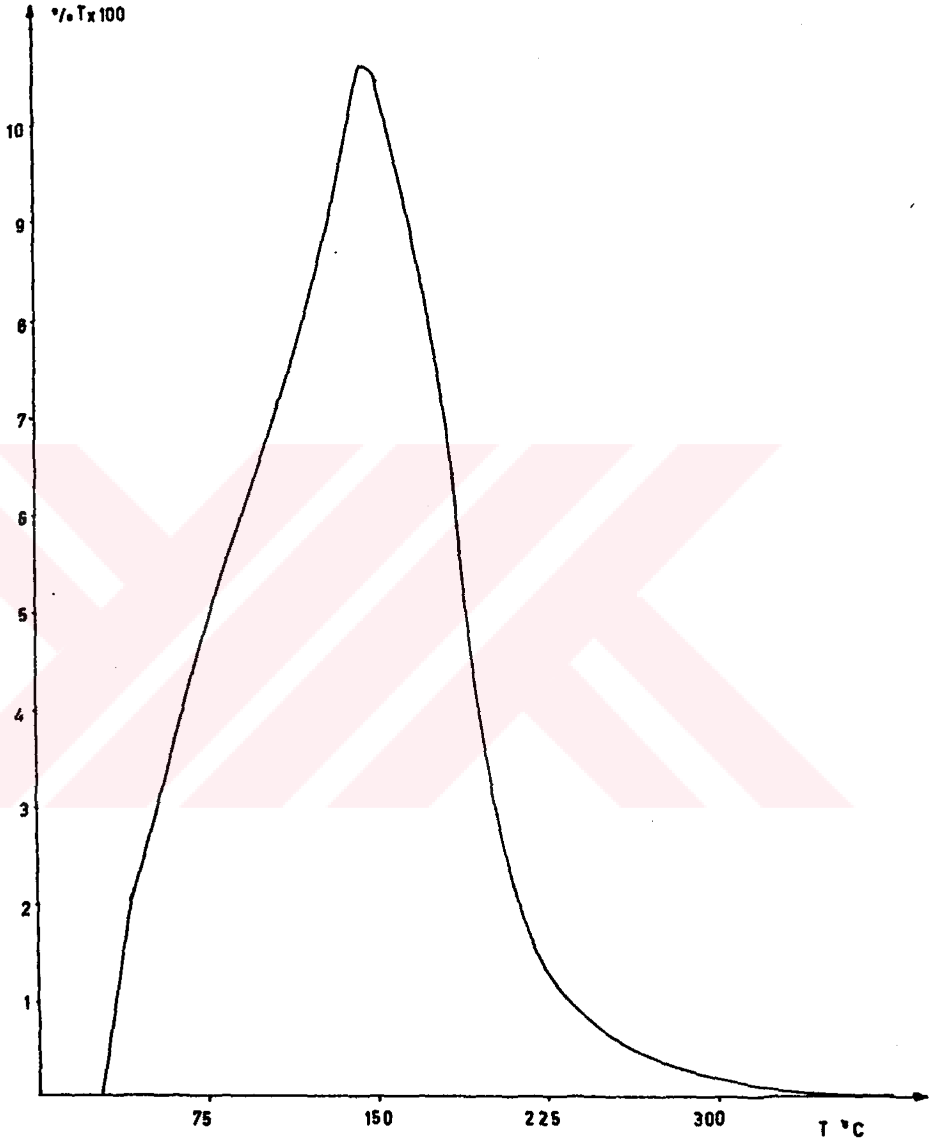


Figure 5.56 Glow curve of teflon dosimeter; 650 nm. After exposure to X-ray radiation.

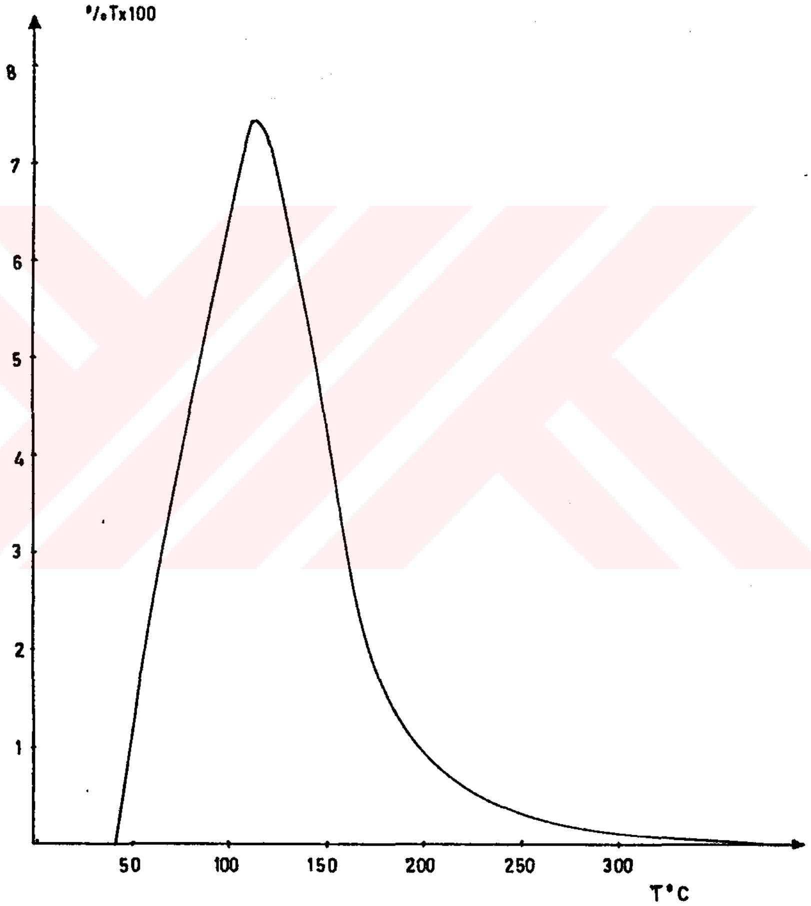


Figure 5.57 Glow curve of teflon dosimeter; 650 nm. After exposure to X-ray radiation.

The sample CaF_2 is exposed to X-ray radiation, 70 kV, 85 kV, 90 kV, 95 kV and 100 kV respectively. And then the transmittance of this sample measured by the spectrophotometer according to the constant wavelength 500 nm. Results of these experiment are shown Table 5.1. The glow curves are shown in figure 5.58. The dose response curve is shown in figure 5.59, which is drawn dose (kV) versus the weight of this sample (mg). Then the sample CaF_2 is again exposed to X-ray radiation, 70 kV, 85 kV, 90 kV, 95 kV and 100 kV respectively. And the transmittance of this sample measured by the spectrophotometer according to the constant wavelength 450 nm. Results of these experiment are shown Table 5.2. The glow curves are shown in figure 5.60. The dose response curve is shown in figure 5.61, which is drawn dose (kV) versus the weight of this sample (mg).

Table 5.1 For the sample CaF_2 ; 500 nm.

Dose (kV)	Weight (mg)	Transmittance (%T)
70	0.443	2
85	0.715	2.5
90	0.829	3.0
95	0.951	3.5
100	1.025	4.0

Table 5.2 For the sample CaF_2 ; 430 nm.

Dose (kV)	Weight (mg)	Transmittance (%T)
70	0.339	1.5
85	0.435	1.8
90	0.469	2.5
95	0.514	3.0
100	0.595	3.5

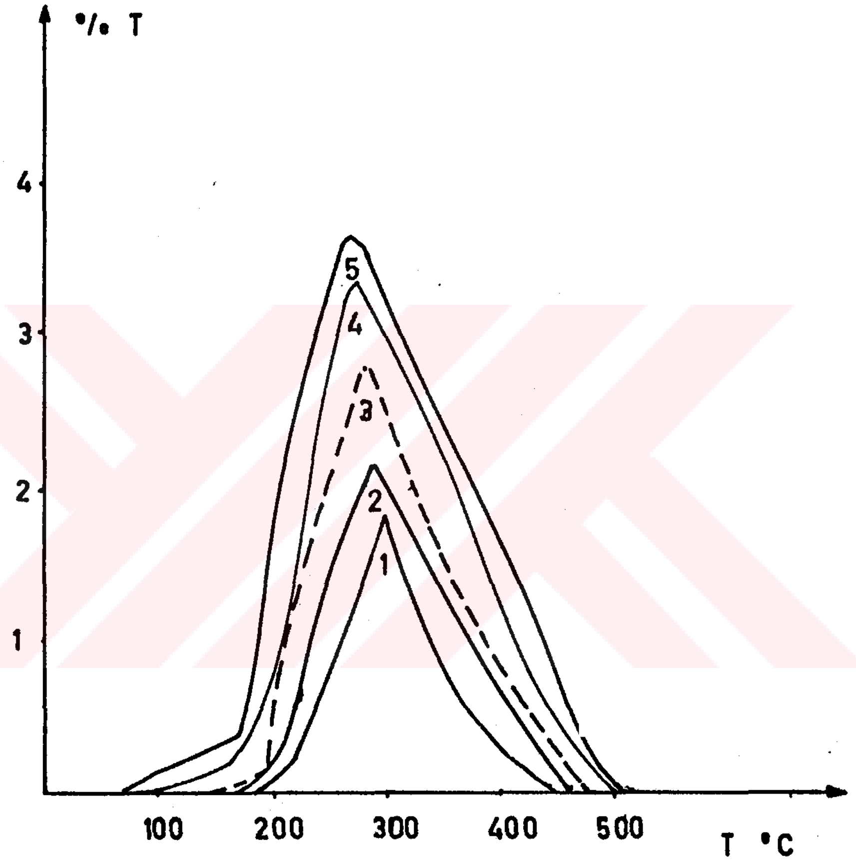


Figure 5.58 Glow curves of CaF_2, exposed to X-ray radiation;

1) 70 kV

2) 85 kV

3) 90 kV

4) 95 kV

5) 100 kV

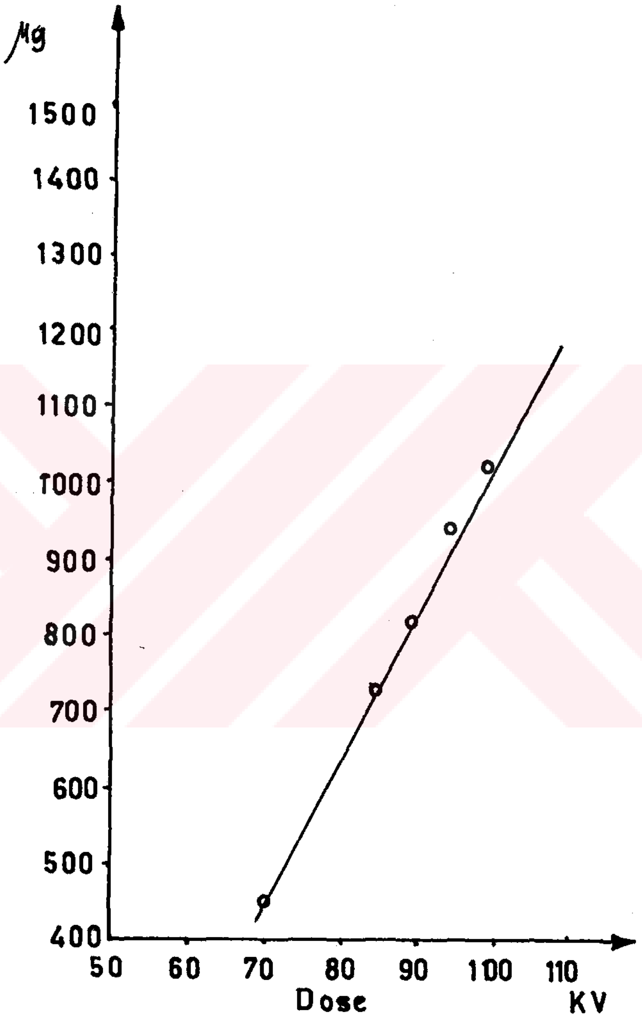


Figure 5.59 The dose response curve of CaF_2 .

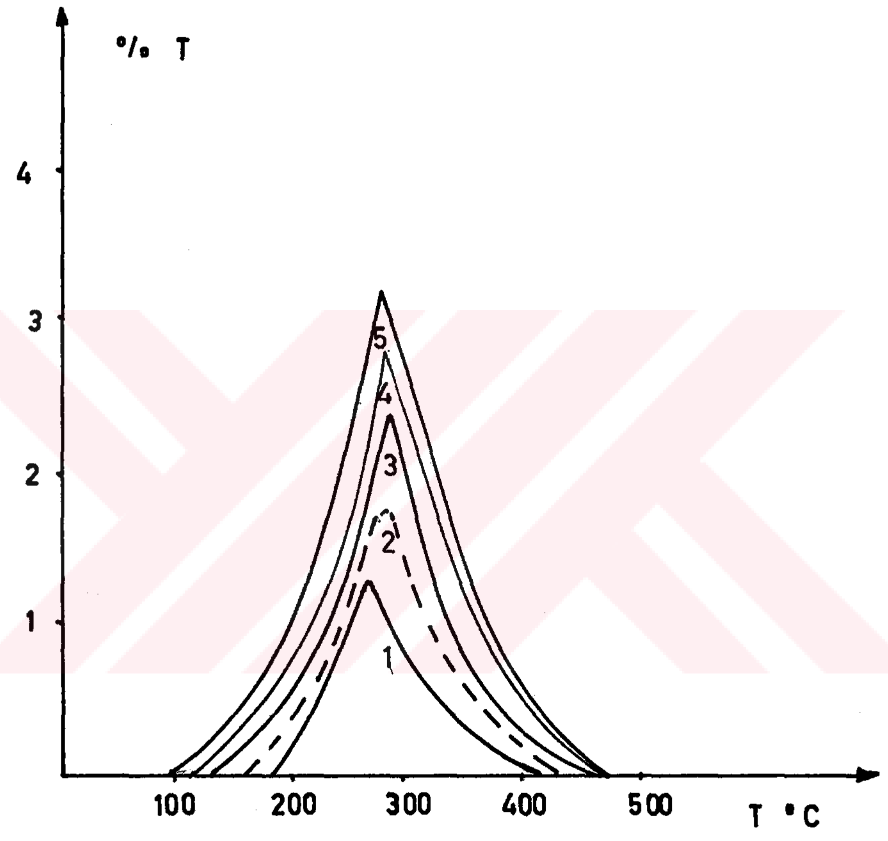


Figure 5.60 Glow curves of CaF_2, exposed to X-ray radiation;

1) 70 kV

2) 85 kV

3) 90 kV

4) 95 kV

5) 100 kV

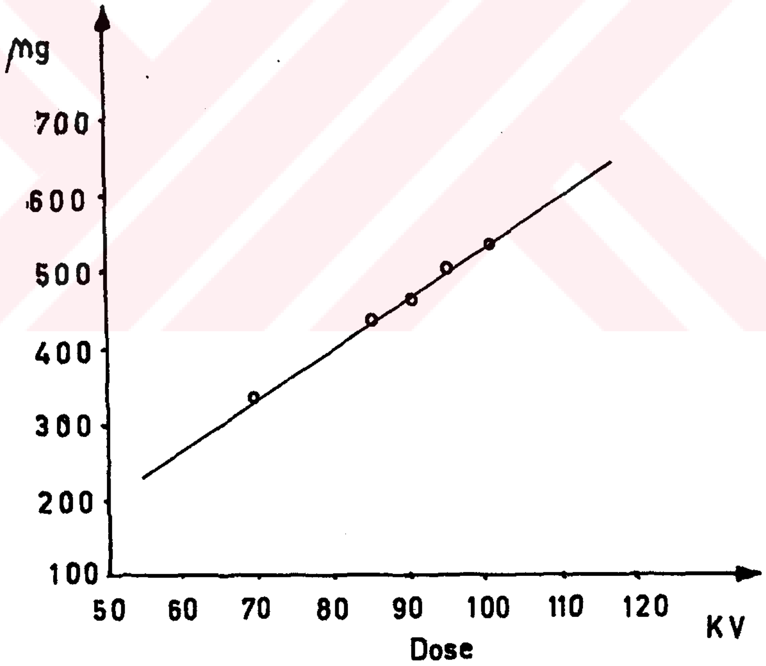


Figure 5.61 The dose response curve of CaF_2 .

CHAPTER 6

DISCUSSIONS AND CONCLUSION

The light emitted by the phosphor always being the source of interest when the thermoluminescence phenomena is concerned. Modification of the double beam spectrophotometer as thermoluminescence reader has provided the detection of the wavelengths of the light emitted by the phosphor by comparing reference wavelength which can be changed manually or sweeps automatically in approximately 15 seconds from 300 nm to 600 nm and 45 seconds from 200 nm to 1100 nm. This specification provides to separate the phosphors from each other since the wavelengths of the lights emitted are characteristics of the thermoluminescence phosphor. In addition to that it is possible to control the quality of the dosemeter material that produced in the laboratory detecting the light emitted by the phosphor.

This instrument can be used in investigation of the trap energy levels described in thermoluminescence phenomena not only in the visible region but ultraviolet and near infrared regions too. During the production of the artificial thermoluminescence phosphors the traps can be produced by changing the impurities and also crystal growth processes controlling by the lights emitted.

Since the instrument can be able to work as a differential thermoluminescence, the background light intensity of the furnace material in infrared region cancelled by the instrument when readings are performed and stored in memory one with and other without the phosphor. This results expands the facilities of the instrument which can be used in age determination of the antiques since the high temperatures should be used.

The experiments performed in that work also showed that the dosimetric characteristics as glow curves dose-response curves are obtained and the results are the same as obtained in the early work $CaF_2$17 [18].

UV-Visible spectrophotometer can be turned into the thermoluminescence instrument in a few minutes by including very simple and cheap tool to the system. So instead only one instrument a wide range research instrument is obtained without any change of the main spectrophotometer.

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