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# ANALYSIS OF ISOTHERMAL DECOMPOSITION OF QUARTZ THERMOLUMINESCENCE INTENSITY

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***Abstract:*** This study examines the intricacies of isothermal decay analysis applied to thermoluminescence (TL) peaks, with an emphasis on determining kinetic parameters. The study asks whether the trap responsible for ITL signals follows first-, second-, or general kinetics, as evidenced by the inconsistent decay pattern and the detection of two overlapping first-order TL peaks.

This work contributes to the understanding of TL peaks and advances progress in luminescence dosimetry research by establishing a robust methodology for characterizing luminescence mechanisms in materials. These observations lead to the conclusion that the TL data originate from more than one trap, and based on the existing literature, it is concluded that there are two overlapping first-order TL peaks.

The study includes consideration of isothermal degradation data at different temperatures (T = 250, 260, 270, 280 and 290 °C) and explores the difficulties associated with obtaining an accurate linear approximation for different values of kinetic order (b). The nature of degradation is interpreted on the basis of monomolecular theory, which assumes a first-order process. The ITL curves were converted into two exponential decay curves.

The slopes of the regression lines give the activation energies (E) for curve 1 and curve 2, respectively: E1 = 0.99 ± 0.16 eV and E2 = 1.32 ± 0.18 eV. The frequency factor (s) is determined by the intersection of the regression line: $s1 = 1.32 \times 10^{8}$ $s^{-1}$ and $s2 = 1.77 \times 10^{12}$ $s^{-1}$.

***Keywords:*** isothermal decomposition, quartz, activation energy.

## 1. Introduction

Natural minerals play an increasingly important role in modern science and technology, and many of them have thermoluminescence (TL) properties that contribute to our understanding of damage processes and safety during radiation events. Among these minerals, quartz is the most important, cost-effective and abundant material with numerous advantages for radiation, environmental and clinical radiological studies [1–9]. Irradiated quartz grains exhibit different luminescence curves when heated to temperatures above room temperature. These curves depend on the chemical composition, types and concentrations of impurities, defects, geological origin, irradiation, sensitivity and other experimental conditions [2, 3].

Numerous TL luminescence peaks in the temperature range 333–753 K were recorded by different authors for various quartz samples [4–6]. According to these studies, high-temperature TL peaks show greater stability after irradiation than low-temperature peaks, which degrade faster due to shorter lifetimes. Despite the varied abilities of natural quartz, a comprehensive understanding of the detailed irradiation response, defect formation and distribution, and thermoluminescence mechanism remains unclear [10–15].

The problem arises from the difficulty of comparing TL measurements between studies because quartz samples of different origins, conditions, and impurities can show inconsistencies.

Therefore, in this study, TL properties and defect formation in natural quartz exposed to high doses of gamma radiation (8 kGy) are investigated [16].

Luminescence-based measurements in retrospective dosimetry require estimation of accumulated charge in localized defect states using external stimuli such as heat or light. In thermoluminescence (TL), the specific excitation mechanism is thermal energy. Isothermal signals, called phosphorescence or isothermal TL (ITL), are used to estimate the equivalent dose (De) and determine trap parameters such as thermal and optical trap depth (in eV), frequency factor (in $s^{-1}$), as well as thermal assistance and thermal extinguishing energy. Therefore, a comprehensive understanding of the properties and origin of isothermal signals (ITL) is critical. Signals resulting from a constant flux of excitation energy are expected to exhibit a consistent, often exponential decay pattern.

Contemporary interest in the thermoluminescence of quartz [17–25] in building materials such as mortar and concrete requires accurate determination of the thermoluminescence parameters associated with the intermediate luminescence peaks of this mineral, especially for dose reconstruction purposes [7, 8].

The isothermal TL signal from deep traps opens up retrograde dosimetry opportunities [7]. Recently, there have been indications that the high temperature signal TL (325 °C) exhibits a significantly higher saturation dose compared to OSL. Consequently, efforts have been made to use isothermal TL at 310 and 320 °C to develop methods for measuring dose in a single fraction.

If you want the TL to be safe, you can change the TL's isothermal setting if it is installed in sunlight. When selecting parameters, you must turn off the TL function to activate the energy (E), additional factors (s) and kinetic changes TL (b).

## 2. Materials and methods

Quartz samples used in this experiment were obtained from beach sand using traditional chemical separation methods. The sand was sieved to isolate fractions ranging in size from 80 to 120 microns. This granular fraction was then subjected to hydrochloric acid (HCl) treatment, heavy liquid separation, and etching in 40% hydrofluoric acid (HF) solution. The precipitated fluorides were dissolved with HCl. Before subsequent irradiation, the samples were heated to 600 °C for an hour to eliminate residual thermoluminescence (TL) centers. Irradiation occurred at dose levels reaching 8 KGy at ambient temperature using a $^{60}$Co source.

In a standard isothermal decomposition experiment, the established procedure involves rapidly heating the irradiated sample to a certain temperature and maintaining it for a certain period of time. For measurement purposes, quartz grains were attached to a 0.1 mm thick aluminum disk using silicone spray. Isothermal TL (ITL) measurements were performed using a Harshaw 3500 handheld reader, holding the sections at a constant temperature for 50 seconds. Preheating was carried out at a rate of 2 $°C^{-1}$ in a nitrogen atmosphere (N2), ITL curves were recorded immediately upon reaching the measurement temperature. Under these conditions, we observed monotonically decreasing ITL signals that showed no noticeable degradation due to thermal lag. Any significant thermal delay will manifest itself as an initial rise to a maximum before subsequent degradation [9]. The emitted light, called phosphorescence decay, is observed over a period of time, allowing the decay rate of the captured electrons to be estimated. Graphs showing the correlation between thermoluminescence (TL) intensity and time at a constant temperature are known as isothermal decay curves.

Garlick and Gibson demonstrated a methodology for analyzing isothermal decomposition using first-order kinetics [10]. When isothermal decay curves are examined at a specific

temperature (Ti) for TL peaks corresponding to first order kinetics, the resulting plots show an exponential relationship with time, as shown in the equation:

$$\mathrm{It} = I_0 exp(-sexp^{\left(-\frac{E}{kT_i}\right)}t\,) \tag{1}$$

Where
$I_0$ = initial TL intensity, It = the TL intensity at time t, s = effective frequency factor, E = activation energy, T = temperature of isothermal decay.

This equation means that a plot of ln(I) versus time will show a linear relationship for peaks determined by first-order kinetics. Additionally, the slope of this line graph will be determined by:

$$slope = m_i = -sexp^{\left(-\frac{E}{kT_i}\right)} \tag{2}$$

Taking the natural logarithm of the equation yields:

$$(|slope|) \; = lns - \frac{E}{kT_i} \tag{3}$$

The graph depicting ln(slope) versus 1/kT is expected to be a straight line with a slope equal to -E and a Y-intercept corresponding to ln(s).

Equations describing thermoluminescence processes were provided by Randall-Wilkins for first order, Garlick-Gibson for second order, and May-Partridge for general order kinetics [11]:

$$I(t) = -\frac{dn}{dt} = nsexp^{\frac{-E}{kT}} \tag{4a}$$

$$I(t) = -\frac{dn}{dt} = \frac{n^2}{N}sexp^{\frac{-E}{kT}} \tag{4b}$$

$$I(t) = -\frac{dn}{dt} = n^b s' exp^{\frac{-E}{kT}} \tag{4c}$$

Where n is the trapped charged population.

These equations provide a method for calculating E. The use of isothermal analysis in this context allows the determination of the kinetic order denoted by b. Keeping the temperature constant and integrating the general order equation (4c) with respect to time (t), we obtain the following expression:

$$I_t = \; I_0 \; \left[1 + s' n_0^{b-1} \, (b-1) t exp^{\left(-\frac{E}{kT}\right)}\right]^{\frac{b}{1-b}} \tag{5}$$

Where $I_0$ and $n_0$ represent the initial TL intensity and the initial concentration of trapped charges, respectively, $I_t$ is the TL intensity at time t. By rearranging Equation (5), we obtain:

$$I_0 = \; s' n_0^b exp^{\left(-\frac{E}{kT}\right)} \tag{6}$$

where
$s'$ = s/N = effective frequency factor, N is the number of traps.
$n_0$ = initial trapped charged population.

This equation assumes that a graph of quantity versus time should have a linear trend when a suitable value of b is determined. Different temperatures of isothermal decomposition form a series of straight lines with different slopes.

## 3. Results and discussions

In Fig. 1 shows the reduced data corresponding to five different temperatures: T = 250, 260, 270, 280 and 290 °C. As described in detail previously, isothermal decay curves of thermoluminescence (TL) peaks corresponding to first-order kinetics follow exponential functions of time.

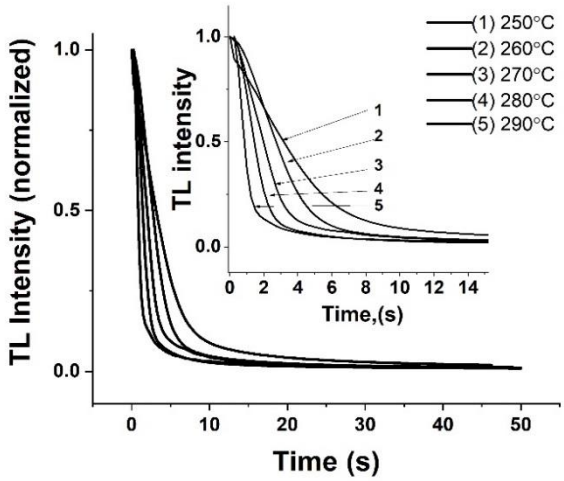


*Fig. 1. Isothermal TL decay curves of quartz at different temperatures. Quartz irradiated at 8 kGy. Insert is a magnified part of the spectrum from 0 to 14 seconds.*

According to [12], a plot of ln(I) versus time (t) will show a linear correlation with the slope of the line given by equation (3) for first-order kinetic peaks. A plot of ln(|slope|) versus 1/kT is expected to show a linear model with a slope equal to -E and a y-intercept equal to ln s if the TL isothermal data provided are consistent with first-order kinetics. We initially calculate ln(TL) for each isothermal curve and plot ln(TL) versus time.

Subsequently, regression lines are calculated for the graph with T = 250 $^{o}$C in Figure 2.

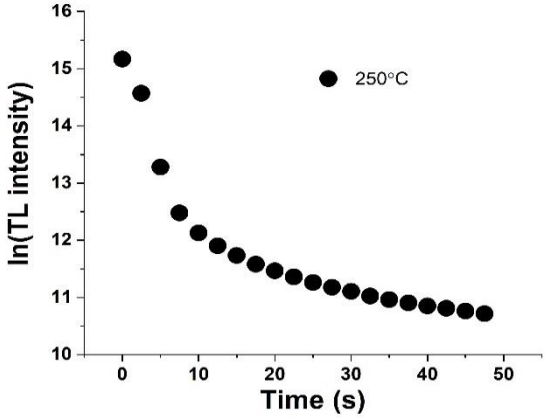


*Fig. 2. The isothermal decay curves on the semilog scale for the isothermal decay curve of quartz at 250 $^{o}$C.*

Figure 2 depicts the plot of ln(TL) against time (t), with T = 250 $^{o}$C representing the temperature while recording isothermal decay curves. First-order kinetics can be ruled out by

examining the plot of ln(TL) versus time, as shown in Figure 2. The resulting plots exhibit nonlinearity, indicating that the data are not consistent with first-order kinetics.

$$(\frac{I_t}{I_0})^{\frac{1-b}{b}} = 1 + s'n_0^{b-1}(b-1)texp^{(-\frac{E}{kT})} \quad (7)$$

This equation assumes that the graph of $(I_t / I_0)^{(1-b)/b}$ versus time t should form a straight line when the corresponding value of b is determined. Having determined the value of b, we plot $(I_t / I_0)^{(1-b)/b}$ versus time t for five different decomposition temperatures, resulting in a series of straight lines with a slope (m) given by:

$$m = s'n_0^{b-1}(b-1)exp^{(-\frac{E}{kT})} \quad (8)$$

The activation energy E and the effective frequency coefficient s" = $s'n_0^{b-1}$ will be determined from the slope and intercept of the graph as a function of ln(m) versus 1/kT.

In Fig. Figure 3 shows $(I_t/I_0)^{(1-b)b}$ for isothermal decomposition data at T = 260°C, taking into account four different values of the kinetic order parameter (b = 1.9, 2.0, 2.1 and 2.2) as a function of time t. It is clear that none of the four graphs provides a satisfactory linear fit. This highlights a potential problem when working with isothermal decay data: achieving an accurate estimate of the optimal linear fit can be challenging due to subtle changes in the plot for different values of b. The calculated R values show that plots corresponding to different b values do not provide a valid linear fit and confirm the consistency of the TL data provided with second-order kinetics. Parallel analysis was applied to all four other isothermal degradation data sets and gave the same results.

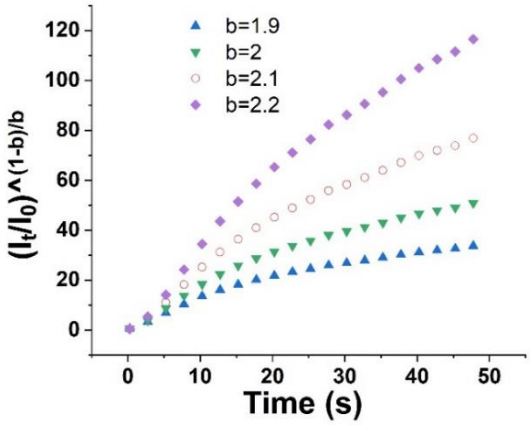


*Fig. 3. Isothermal decay data at a temperature of 260 °C calculated for several values of kinetic order b as a function of time.*

The degradation observed in the present study is explained by the monomolecular superposition theory (first order). This type of distortion results from the superposition of exponential numbers associated with various traps and is represented mathematically by the equation [13]:

$$I_t = I_{01}\exp(-P_1 t) + I_{02}\exp(-P_2 t) + \cdots$$

where $I_{on}$ is the intensity of electron phosphorescence in the Ion energy traps.

$E_n$, $P_n$ = s exp (- $E_n$/kT) is the probability of the electron escaping from the trap, k is the Boltzmann constant, s is the escape frequency coefficient. Therefore, each decay curve can be

decomposed into a series of exponential numbers using a "solve" procedure to calculate the E values corresponding to each exponential.

It can be seen that each decay curve can be decomposed into two exponential values, as shown in the isothermal decay curve at 20 °C shown in Figure 4. For the first curve $I_1$ = 4.84634 $E_6$ and $P_1$ = 0.96471, for the second curve $I_2$ = 114707 and $P_2$ = 0.06435.

Thus, the nature of the degradation can be interpreted on the basis of monomolecular theory, assuming that the luminescence kinetics corresponds to a first-order process.

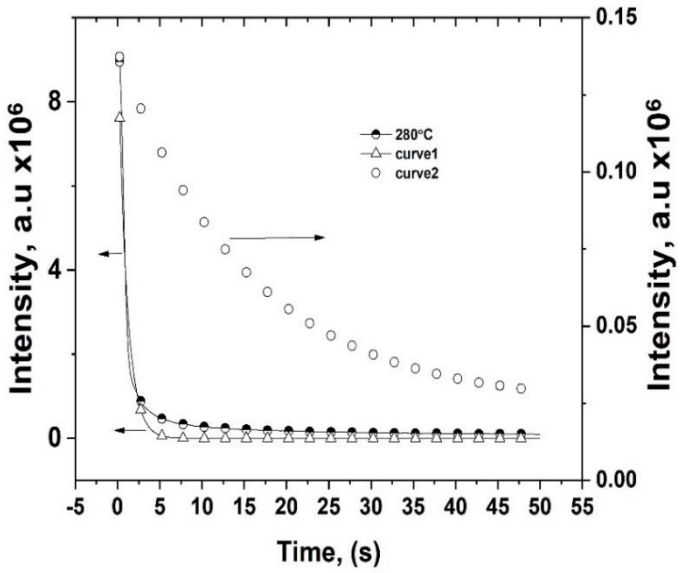


*Fig. 4. Isothermal decay data for the 280 °C deconvoluted into two exponential decay curves*.

*Consideration of first-order kinetics*

It is verified by checking the linearity of the ln (TL1 curve) and ln (TL2 curve) plots with respect to time, as shown in Figure 5. The resulting plots for Curve 1 and Curve 2 show linearity, indicating that the data are consistent with first-order kinetics.

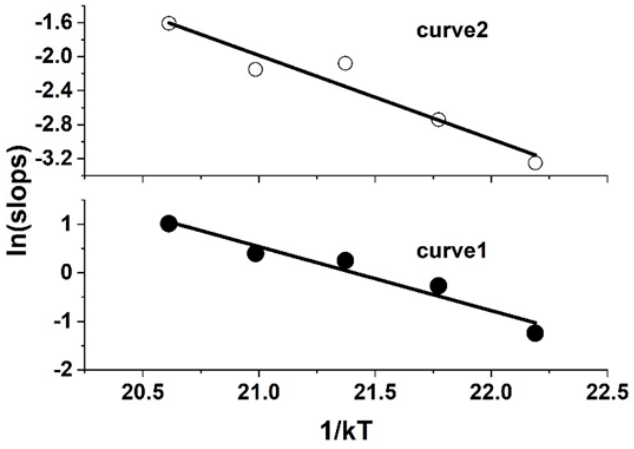


*Fig. 5. The ln(slope) versus 1/kT graph to determine E for TL data of convoluted curves 1 and 2.*

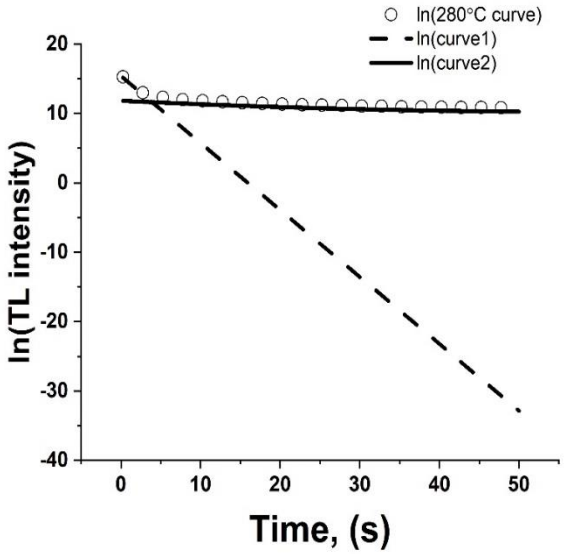


*Fig. 6. The isothermal decay curves on the semilog scale for the isothermal decay curve of quartz at 280 °C and the two deconvoluted cures 1 and 2.*

**Table 1**

The slopes of linear isothermal graphs and their natural logarithms ln(slope)

| Temperature °C | 1/kT ($eV^{-1}$) | Curve1 | | Curve2 | |
|---|---|---|---|---|---|
| | | slope ($s^{-1}$) | ln(slope) | slope ($s^{-1}$) | ln(slope) |
| 250 | 22.19 | 0.2888 | -1.24202 | 0.0388 | -3.24934 |
| 260 | 21.77 | 0.76471 | -0.26826 | 0.06455 | -2.74032 |
| 270 | 21.37 | 1.28707 | 0.25237 | 0.12493 | -2.08 |
| 280 | 20.99 | 1.48454 | 0.3951 | 0.11639 | -2.15081 |
| 290 | 20.61 | 2.75118 | 1.01203 | 0.20012 | -1.60884 |

In the next step, we plot the slopes of these line graphs in Table 1 and calculate the natural logarithm of the slopes, denoted ln(slope), for all five ITL curves corresponding to temperatures T = 250, 260, 270, 280 and 290 °C. The graph in Figure 6 shows the dependence of ln (slope) of curve 1 and curve 2 on 1/kT; where T represents the temperature (in Kelvin) when recording isothermal decomposition curves.

The slope of the regression line provides the activation energy E and the intercepts for curve 1 and curve 2, respectively, are:

E1 = 0.99 ± 0.16 eV; intercept1 = 18.7 ± 3.4 and

E1 = 1.32 ± 0.18 eV; intercept1 = 28.2 ± 4.2

The frequency factor s can be found from the intercept of the regression line:

$intercept1 = ln(s1)$ = 18.7; s1 = exp(18.7) = $1.32 \times 10^8$ $s^{-1}$and

$intercept2 = ln(s2)$ = 28.2; s2 = exp(28.2) = $1.77 \times 10^{12}$ $s^{-1}$

As reported in the literature, most natural sedimentary quartz grains exhibit two thermoluminescence (TL) peaks at 325 and 375 °C when the grains are heated from 300 to 400 °C

at a rate of 20 °C/s [14], and their luminescence is reflected in the photomultiplier tube. It is observed through blue and near-ultraviolet glass filters in front of it. Previous studies have shown that at a heating rate of 5 °C/s, peaks appear at 305 and 350 °C, respectively [6]. Our observations also lead to the conclusion that the ITL data described in Figure 1 are due to more than one trap and, based on the existing literature, there are two overlapping first-order TL peaks.

## 4. Conclusions

A study of the analysis of isothermal decay of thermoluminescence (TL) peaks has provided valuable information on the kinetic parameters governing this luminescence phenomenon. The difficulties associated with obtaining accurate linear fits for different values of kinetic order (b) highlight the complexity of the analysis. Therefore, it can be concluded that the trap responsible for the ITL signals at 250, 260, 270, 280, and 290 °C does not obey first-, second- , or general-order kinetics. This result is verified as follows: (a) Attenuation behavior of ITL signal recorded at 250, 260, 270, 280, and 290 °C, Eq. (5) for b values from 1 to 2 (see Figures 2 and 3). (B) These observations lead to the conclusion that the TL data shown in Figure 4 arises from more than one trap and, based on the available literature, that there are two overlapping first-order TL peaks [26–29].

This comprehensive analysis contributes to the understanding of TL peaks and establishes a reliable methodology for characterizing luminescence mechanisms in materials. Consistent application of these analytical methods to isothermal decomposition data at different temperatures increases the reliability and applicability of our results. In summary, our study contributes to the understanding of the kinetic parameters of TL peaks and provides a basis for future studies in the field of luminescence dosimetry.

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## АНАЛИЗ ИЗОТЕРМИЧЕСКОГО РАЗЛОЖЕНИЯ ИНТЕНСИВНОСТИ ТЕРМОЛЮМИНЕСЦЕНЦИИ КВАРЦА

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***Резюме:*** Это исследование углубляется в тонкости анализа изотермического распада, применяемого к пикам термолюминесценции (TL), с упором на определение кинетических параметров. Исследование ставит под сомнение соответствие ловушки, ответственной за сигналы ITL, кинетике первого, второго или общего порядка, что подтверждается несоответствующей картиной затухания и выводом о двух перекрывающихся пиках TL первого порядка.

Эта работа расширяет понимание пиков TL и устанавливает надежную методологию для характеристики механизмов люминесценции в материалах, способствуя достижениям в исследованиях люминесцентной дозиметрии. Эти наблюдения приводят к выводу, что данные TL происходят из более чем одной ловушки, и на основании существующей литературы делается вывод о наличии двух перекрывающихся пиков TL первого порядка.

Исследование включает в себя рассмотрение данных изотермического распада при различных температурах (T = 250, 260, 270, 280 и 290 °C) и исследует проблемы, связанные с достижением точной линейной подгонки для различных значений кинетического порядка (b). Природа распада интерпретируется на основе мономолекулярной теории, предполагающей приверженность процессу первого порядка. Кривые ITL были преобразованы в две кривые экспоненциального затухания.

Наклоны линий регрессии дают значения энергии активации (E) для кривой 1 и кривой 2 соответственно: E1 = 0,99 ± 0,16 эВ и E2 = 1,32 ± 0,18 эВ. Частотный коэффициент (s) определяется по пересечению линии регрессии: $s1 = 1{,}32 \times 10^{8}$ $с^{-1}$ и $s2 = 1{,}77 \times 10^{12}$ $с^{-1}$.

***Ключевые слова:*** изотермический распад, кварц, энергия активации, частотный фактор.

# KVARSIN TERMOLÜMINESSENSIYA PİK İNTENSİVLİYİNİN İZOTERMİK PARÇALANMASININ TƏHLİLİ

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***Xülasə:*** Bu tədqiqat kinetik parametrlərin müəyyən edilməsinə yönələrək termolüminessensiya (TL) zirvələrinə tətbiq edilən izotermik parçalanma analizinin incəliklərini araşdırır. Tədqiqat ITL siqnallarına cavabdeh olan tələnin birinci, ikinci və ya ümumi kinetikaya uyğun olub-olmadığını sorğulayır. Bu uyğun olmayan parçalanma nümunəsi və üst-üstə düşən iki birinci dərəcəli TL zirvəsinin tapılması ilə öz təsdiqini tapır.

Bu iş TL zirvələrinin başa düşülməsini təkmilləşdirir və materiallarda lüminesans mexanizmlərini xarakterizə etmək üçün möhkəm metodologiya yaradır, lüminesans dozimetriya tədqiqatında irəliləyişlərə töhfə verir. Bu müşahidələr TL məlumatlarının birdən çox tələdən qaynaqlandığı qənaətinə gətirib çıxarır və mövcud ədəbiyyat əsasında iki üst-üstə düşən birinci dərəcəli TL zirvəsi olduğu qənaətinə gəlinir.

Tədqiqat müxtəlif temperaturlarda (T = 250, 260, 270, 280 və 290 °C) izotermik parçalanma məlumatlarının nəzərdən keçirilməsini əhatə edir və kinetik nizamın müxtəlif dəyərləri üçün dəqiq xətti uyğunluğun əldə edilməsi ilə bağlı problemləri araşdırır (b). Parçalanmanın təbiəti birinci dərəcəli prosesi nəzərdə tutan monomolekulyar nəzəriyyə əsasında şərh olunur. ITL əyriləri iki eksponensial parçalanma əyrisindən əmələ gəlir.

Reqressiya xətlərinin yamacları müvafiq olaraq əyri 1 və əyri 2 üçün aktivləşmə enerjilərini (E) verir: E1 = 0,99 ± 0,16 eV və E2 = 1,32 ± 0,18 eV. Tezlik əmsalı (s) reqressiya xəttinin kəsişməsi ilə müəyyən edilir: s1 = 1,32 × 108 $s^{-1}$ və s2 = 1,77 × 1012 $s^{-1}$.

***Açar sözlər:*** izotermik parçalanma, kvars, aktivləşmə enerjisi, tezlik faktoru.