Effect of Nd$_2$O$_3$ doping on spectroscopic characteristics of zinc barium boro-tellurite glass for laser medium application

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Abstract. The ZnO-BaO-B$_2$O$_3$-TeO$_2$ (ZBBoT) glass samples were prepared, and their properties affected by varied concentrations of Nd$_2$O$_3$ doped in were investigated. The traditional melt quenching process was conducted to synthesize the samples which were explored to identify their density, molar volume, refractive index, absorption, photoluminescence (PL) and PL decay spectral profiles. The optical absorption has been used to derive the Judd-Ofelt (J-O) parameters ($\Omega_\lambda$, $\lambda$ = 2, 4 and 6). The trend of J-O parameter is $\Omega_2 > \Omega_6 > \Omega_4$. Moreover, the J-O parameters are used to predict the radiative properties which are significant for the assessment of the suitability of a glass for laser emission. The emission of glasses exhibited 2 peaks at 1060, and 1345 nm. Based on research, it is expected to produce glass material that is applied as an active laser medium and solid-state lighting materials.

Keywords: Neodymium oxide, boro-tellurite glass, Luminescence, J-O parameters

1. Introduction
Since 1961, after Elias Snitzer published his first theoretical explanation of optical modes in glass fibers following by the first operation on Nd: glass lasers [1], several types of neodymium doped glass have been interested by many researchers around the world who investigated Nd$^{3+}$ ion doped fluorides [2–5], chalcogenides [6], aluminosilicates [7], germanates [3], and tellurite glasses [8–10], etc. Neodymium ions provide high intensity of the emitted wavelength 946 nm, 1060 nm and 1319 nm indicating the energy transitions $^{4}F_{3/2} \rightarrow ^{4}I_{9/2}$, $^{4}F_{5/2} \rightarrow ^{4}I_{11/2}$ and $^{4}F_{5/2} \rightarrow ^{4}I_{13/2}$, respectively, where the most powerful one is the emission at 1060 nm, according to a research on high power laser applications [11]. Therefore, the ions are appropriate to be dope into a glass structure for laser enhancing. Oxide glasses have a structure which can be predetermined by properties of prepared compositions. Boron atoms in glass matrix contribute to the properties of glass: low melting point, high transparency, high chemical stability, good glass forming nature, and good RE ion solubility. Whereas tellurium atoms contribute to low melting temperature, large refractive index, and good chemical and thermal stability.
On the other hand, when doping rare earth ions into glass structure, borate one exhibits high phonon energy (~1300–1500 cm⁻¹), indicating high non-radiative relaxations of RE³⁺ ions. While tellurite one which needs fast quenching to form glass structure, exposes low phonon energy (~700 cm⁻¹). To form glass taken moderate quenching time and relatively low phonon energy, the combination as boro-tellurite glass is a good alternative together with other better properties: high chemical durability, high dielectric constant and excellent transparency [12]. Hence, this work was conducted to fabricate samples of boro-tellurite glass doped with Nd³⁺ ion. The physical and the spectroscopic properties of the samples were investigated to identify a proper composition of raw materials and to indicate which neodymium concentration is the most suitable for optical amplifier and laser applications. Additionally, calculations according to Judd-Ofelt theory [13,14] were performed, and consequent parameters were compared with previously published one.

2. Experiment
The samples of boro-tellurite glass doped with Nd₂O₃ with varied concentration (ZBBoT) were produced using melt quenching technique. The glass composition is (35-x)TeO₂-30B₂O₃-10ZnO-25BaO-xNd₂O₃, where x is 0.00, 0.05, 0.50, 1.00 and 1.50 mol%. The mixtures 15 g of compounds was taken in a porcelain crucible and melted at 1150°C for 1.5 h, then the melted sample was poured into preheated graphite block. The glass samples were annealed at 350°C for 3 h for eliminated internal mechanical stress before slowly cooled down to room temperature. The glass samples cut and polished in width 10 mm and length 15 mm, thickness 3 mm dimensions shape for optical measurements. The densities (ρ) were measured by Archimedes principal using distilled water as an immersion liquid at room temperature. After that the molar volumes (Vₘ) were calculated from relation by

\[ V_m = \frac{M_T}{\rho} \]  

where M_T is total molecular weight of glasses. For refractive index measurement were measured by Presidium Refractive Index Meter II. The optical properties were measured with UV-Vis-NIR spectrophotometer (Shimadzu, UV-3600) in the range 200-1200 nm. The Photoluminescence (PL) and PL decay spectral profiles was measured with spectrofluorophotometer Quanta Master (QM-300) from Photon Technology International (PTI)-Horiba. Judd-Ofelt analysis was considered by absorption results while the predicted radiative transition probabilities (Aᵣ), emission cross-section and branching ratio (βᵣ) were determined from J-O parameters and luminescence results.

3. Results and Discussion

3.1 Physical Properties
The physical properties namely density, molar volume, and refractive index of glass samples were demonstrated in Figure 1. The more density, the more Nd₂O₃ concentration, from 0.00 mol% until 1.50 mol%. This can be described as a molecular mass issue. That value of Nd₂O₃ is 336.48 g/mol while that of B₂O₃-TeO₂ is 229.22 g/mol. By added the heavier Nd₂O₃, the molecules scatteringly penetrated into the structure of the lighter B₂O₃-TeO₂. Therefore, the mass per unit volume called density increased with Nd₂O₃ concentration. In contrast, molar volume is conditional on density of glasses, therefore its trend went in opposite direction of that of the density i.e., it decreased with the increment of Nd₂O₃ concentration. This indicated more compactness of glass structure with higher concentration of Nd₂O₃, inserted into atomic space. The values of density (ρ) are in the range of 3.81 to 3.98 g/cm³ while their molar volumes (Vₘ) are in the range of 32.24 to 31.57 cm³/mol. The refractive indices of the samples were increased with the increment of Nd₂O₃ concentration. Additionally, they changed with Nd₂O₃ content in a specific characteristic. This is due to the high polarizabilities and ionic refractivities of component in glass matrix.
Figure 1. Variation of density, molar volume and refractive index with Nd$_2$O$_3$ concentration in ZBBoT glasses

3.2 Optical Properties

The absorption spectra at room temperature were measured covering the wavelength of 200 to 1200 nm. The absorption spectra of Nd$_2$O$_3$ doped ZBBoT glasses in the UV–VIS–NIR are shown in Figure 2. The spectra obtained for all Nd$_2$O$_3$ doped ZBBoT glasses are similar in nature except for the band intensities. It is clearly observed that the absorption intensity of the absorption band increases with the increase of Nd$_2$O$_3$ concentration. The absorption bands represent the transitions from the ground state $^4$I$_{9/2}$ to various excited states. The various spectroscopic transitions observed are as follows: $^4$I$_{9/2}$ to $^2$G$_{7/2}$, $^2$K$_{13/2}$+$^4$G$_{7/2}$, $^4$G$_{5/2}$+$^2$G$_{7/2}$, $^4$F$_{9/2}$, $^4$S$_{3/2}$+$^4$F$_{7/2}$, $^4$F$_{5/2}$+$^2$H$_{9/2}$, and $^4$F$_{3/2}$ [15–17]. The absorption band was corresponded to wavelengths that are 519, 526, 583, 684, 745, 803, and 877 nm respectively. The intensity of absorption spectra increases by the doping Nd$_2$O$_3$ from 0.00 to 1.50 mol% in the glass system. The strongest absorption is come from the wavelength of 583 nm due to $^4$I$_{9/2}$→$^4$G$_{5/2}$+$^2$G$_{7/2}$ transition. The absorption spectra show the present glass was suitable for a device that absorbs wavelength in the blue to near infrared region [18].

Figure 2. The absorption spectra of ZBBoT glasses doped with Nd$_2$O$_3$ concentration
3.3 Photoluminescence Properties

3.3.1 Excitation spectra

Excitation spectra investigated from the samples of ZBBoT glass doped with Nd$_2$O$_3$ of which the concentrations ranged from 0.00 to 1.50 mol% were measured in the range of 200 – 1,000 nm as shown in Figure 3. Eight obvious peaks were observed for the given emission wavelength of 1,060 nm, corresponding to the excited wavelength at 429 nm (4$I_{9/2}$$\rightarrow$$^2$P$_{1/2}$), 477 nm (4$I_{9/2}$$\rightarrow$$^2$D$_{3/2}$), 524 nm (4$I_{9/2}$$\rightarrow$$^4$G$_{7/2}$), 575 nm (4$I_{9/2}$$\rightarrow$$^4$G$_{5/2}$), 680 nm (4$I_{9/2}$$\rightarrow$$^2$H$_{11/2}$), 745 nm (4$I_{9/2}$$\rightarrow$$^4$F$_{9/2}$), 803 nm (4$I_{9/2}$$\rightarrow$$^4$F$_{7/2}$$+$$^2$H$_{9/2}$), and 882 nm (4$I_{9/2}$$\rightarrow$$^4$F$_{5/2}$$+$$^2$H$_{9/2}$), where the energy transitions shown in the parentheses. The highest peaks of each the excitation spectrum are at the common wavelength of 575 nm corresponding to the final state of the transition, $^4$G$_{5/2}$ nm [19].

![Excitation spectra of ZBBoT glasses doped with varied Nd$_2$O$_3$ concentration](image)

**Figure 3.** The Excitation spectra of ZBBoT glasses doped with varied Nd$_2$O$_3$ concentration

3.3.2 Emission spectra

Emission spectra investigated from the samples of ZBBoT glass doped with Nd$_2$O$_3$ of which the concentrations ranged from 0.00 to 1.50 mol% were measured in the range of 800 – 1600 nm after excited at 585 nm from xenon flash lamp as shown in Figure 4. Two obvious peaks in the range of NIR were observed. Those were corresponding to the emitted wavelength at 1060 nm ($^4$F$_{9/2}$$\rightarrow$$^4$I$_{13/2}$) and 1345 nm ($^4$F$_{3/2}$$\rightarrow$$^4$I$_{11/2}$), where the energy transitions shown in the parentheses. The intensity of each emission spectrum was at 1060 nm and increased with the concentration of Nd$_2$O$_3$ until 0.50 mol% where was the saturated point of its concentration, and then tended to decrease. This is called concentration quenching effect where Nd atoms were too close, therefore an individual atom tended to absorb the light emitted from the adjacent one leading to dimmer emission. Hence, the most appropriate doping concentration of Nd$_2$O$_3$ for ZBBoT glass samples, is 0.50 mol%.
Since the sample of ZBBoT glass doped with Nd$_2$O$_3$ of which the concentrations 0.50 mol% gave the highest emission intensity, it was investigated emission property following the Judd–Ofelt (J-O) theory. The absorption spectra around 583 nm were analyzed to identify the experimental oscillator strength ($f_{\text{exp}}$) and calculated oscillator strength ($f_{\text{cal}}$). Those parameters: $0.80 \times 10^{-6}$ and $0.90 \times 10^{-6}$, respectively, indicated that the energy transition $^4I_{9/2} \rightarrow ^4G_{5/2}+^2G_{7/2}$ exhibited the strongest oscillator strength among the others as shown in the Table 1. The parameters $\Omega_2$, $\Omega_4$, and $\Omega_6$ of the ZBBoT glass sample was $2.10 \times 10^{-20}$, $0.40 \times 10^{-20}$, and $0.58 \times 10^{-20} \text{ cm}^2$, respectively. The $\Omega_2$ of ZBBoT is more than $0.55 \times 10^{-20} \text{ cm}^2$ of ZBSN4 and $0.05 \times 10^{-20} \text{ cm}^2$ of BBFBNd but less than $4.49 \times 10^{-20} \text{ cm}^2$ of Silicate and $3.86 \times 10^{-20} \text{ cm}^2$ of TZO0.5Nd. Hence, ligands surrounded Nd$^{3+}$ in ZBBoT glass metric were high asymmetry and their bonds were higher covalency than ZBSN4 and BBFBNd, but lower than silicate and TZO0.5Nd. The $\Omega_4$ and $\Omega_6$ of ZBBoT indicated that its viscosity and rigidity tended to lower compared to other types of glass as shown in the Table 2. The energy transition $^4F_{3/2} \rightarrow ^4I_{13/2}$ around 1060 nm showed the highest values of the parameters: radiative transition probability ($A_R$), stimulated cross-section ($\sigma(\lambda p)$), and branching ratios ($\beta R$) for ZBBoT glass sample doped with Nd$_2$O$_3$ as shown in the Table 3. Especially, the ($\sigma(\lambda p)$) value of $6.32 \times 10^{-20} \text{ cm}^2$ ensured that the glass sample can be used to generate high amount of laser emission consuming low energy. The proportion between the experimental branching ratios ($\beta R_{\text{exp}}$) and calculated ones ($\beta R_{\text{cal}}$) is more than 0.50 mol% promising that ZBBoT glass can generate laser light at 1060 nm. Hence, the sample of ZBBoT glass doped with Nd$_2$O$_3$ of which the concentrations 0.50 mol% has an interesting potential to apply as laser medium materials with 1060 nm emission.

**Figure 4.** The Emission spectra of ZBBoT glasses doped with varied Nd$_2$O$_3$ concentration

### 3.4 Potential for Laser medium material according to the Judd–Ofelt theory

Since the sample of ZBBoT glass doped with Nd$_2$O$_3$ of which the concentrations 0.50 mol% gave the highest emission intensity, it was investigated emission property following the Judd–Ofelt (J-O) theory. The absorption spectra around 583 nm were analyzed to identify the experimental oscillator strength ($f_{\text{exp}}$) and calculated oscillator strength ($f_{\text{cal}}$). Those parameters: $0.80 \times 10^{-6}$ and $0.90 \times 10^{-6}$, respectively, indicated that the energy transition $^4I_{9/2} \rightarrow ^4G_{5/2}+^2G_{7/2}$ exhibited the strongest oscillator strength among the others as shown in the Table 1. The parameters $\Omega_2$, $\Omega_4$, and $\Omega_6$ of the ZBBoT glass sample was $2.10 \times 10^{-20}$, $0.40 \times 10^{-20}$, and $0.58 \times 10^{-20} \text{ cm}^2$, respectively. The $\Omega_2$ of ZBBoT is more than $0.55 \times 10^{-20} \text{ cm}^2$ of ZBSN4 and $0.05 \times 10^{-20} \text{ cm}^2$ of BBFBNd but less than $4.49 \times 10^{-20} \text{ cm}^2$ of Silicate and $3.86 \times 10^{-20} \text{ cm}^2$ of TZO0.5Nd. Hence, ligands surrounded Nd$^{3+}$ in ZBBoT glass metric were high asymmetry and their bonds were higher covalency than ZBSN4 and BBFBNd, but lower than silicate and TZO0.5Nd. The $\Omega_4$ and $\Omega_6$ of ZBBoT indicated that its viscosity and rigidity tended to lower compared to other types of glass as shown in the Table 2. The energy transition $^4F_{3/2} \rightarrow ^4I_{13/2}$ around 1060 nm showed the highest values of the parameters: radiative transition probability ($A_R$), stimulated cross-section ($\sigma(\lambda p)$), and branching ratios ($\beta R$) for ZBBoT glass sample doped with Nd$_2$O$_3$ as shown in the Table 3. Especially, the ($\sigma(\lambda p)$) value of $6.32 \times 10^{-20} \text{ cm}^2$ ensured that the glass sample can be used to generate high amount of laser emission consuming low energy. The proportion between the experimental branching ratios ($\beta R_{\text{exp}}$) and calculated ones ($\beta R_{\text{cal}}$) is more than 0.50 mol% promising that ZBBoT glass can generate laser light at 1060 nm. Hence, the sample of ZBBoT glass doped with Nd$_2$O$_3$ of which the concentrations 0.50 mol% has an interesting potential to apply as laser medium materials with 1060 nm emission.
Table 1. Experimental oscillator strength ($f_{\text{exp}}$) and calculated oscillator strength ($f_{\text{cal}}$) of each energy transition for ZBBoT glass sample doped with Nd$_2$O$_3$ of which the concentrations 0.50 mol%.

| State        | Wavelength (nm) | Energy (cm$^{-1}$) | $f_{\text{exp}}$ (10$^{-6}$) | $f_{\text{cal}}$ (10$^{-6}$) |
|--------------|-----------------|--------------------|-------------------------------|------------------------------|
| $^4I_{9/2} \rightarrow ^4G_{7/2}$ | 519             | 19267.8            | 0.40                          | 0.30                         |
| $^4I_{11/2} \rightarrow ^4K_{13/2}$ | 526             | 19011.4            | 0.19                          | 0.18                         |
| $^4I_{11/2} \rightarrow ^4G_{5/2}$ | 583             | 17152.7            | 0.80                          | 0.90                         |
| $^4I_{13/2} \rightarrow ^4F_{9/2}$ | 684             | 14619.9            | 0.03                          | 0.04                         |
| $^4I_{13/2} \rightarrow ^4F_{7/2}$ | 745             | 13422.8            | 0.57                          | 0.10                         |
| $^4I_{13/2} \rightarrow ^4F_{5/2}$ | 803             | 12453.3            | 0.70                          | 0.55                         |
| $^4I_{13/2} \rightarrow ^4F_{3/2}$ | 877             | 11402.5            | 0.24                          | 0.36                         |

Table 2. J-O parameters ($\Omega_2$, $\Omega_4$ and $\Omega_6$) for the sample of ZBBoT glass doped with Nd$_2$O$_3$ of which the concentrations 0.50 mol% compared with those for other systems of glass doped with Nd$_2$O$_3$.

| Glass system | $\Omega_2$ (×10$^{-20}$) | $\Omega_4$ (×10$^{-20}$) | $\Omega_6$ (×10$^{-20}$) | Tendency | Reference |
|--------------|---------------------------|---------------------------|---------------------------|----------|-----------|
| ZBBoT        | 2.10                      | 0.40                      | 0.58                      | $\Omega_2 > \Omega_6 > \Omega_4$ | The Sample |
| ZBSN4        | 0.55                      | 2.55                      | 2.01                      | $\Omega_4 > \Omega_6 > \Omega_2$ | [20]       |
| silicate     | 4.49                      | 3.13                      | 3.53                      | $\Omega_2 > \Omega_6 > \Omega_4$ | [21]       |
| BBFBNd0.05   | 2.02                      | 0.41                      | 0.60                      | $\Omega_2 > \Omega_6 > \Omega_4$ | [22]       |
| TZO0.5Nd     | 3.86                      | 4.02                      | 3.79                      | $\Omega_6 > \Omega_4 > \Omega_2$ | [23]       |

Table 3. Radiative transition probability ($A_R$), stimulated cross-section ($\sigma(\lambda_p)$), and calculated branching ratios ($\beta_R$) for the sample of ZBBoT glass doped with Nd$_2$O$_3$ of which the concentrations 0.50 mol%.

| Energy state | $\lambda_p$ (nm) | $A_R$ (s) | ($\sigma(\lambda_p)$) × 10$^{-22}$ (cm$^2$) | $\beta_R$ | $\beta_{R\text{exp}}$ | $\beta_{R\text{exp}}$ |
|--------------|------------------|----------|---------------------------------|----------|----------------------|----------------------|
| $^4I_{11/2}$ | 1060             | 305.10   | 6.32                           | 0.63     | 0.58                 |                      |
| $^4I_{13/2}$ | 1345             | 60.12    | 2.95                           | 0.01     | 0.06                 |                      |

3.4 Lifetime

The lifetime profile of present glass sample is presented in Figure 5. This is measured under $\lambda_{\text{em}} = 1060$ nm and $\lambda_{\text{ex}} = 585$ nm and observed in the $^4F_{3/2} \rightarrow ^4I_{11/2}$ transition. The curve is fitted by single exponential. The experimental lifetime decrease by increasing Nd$_2$O$_3$ concentration in the glass system. These are 676.21, 529.13 146.74, 93.48, 60.11 µs for 0.05Nd, 0.1Nd, 0.5Nd, 1.0Nd and 1.5Nd respectively. The increasing Nd$_2$O$_3$ concentration affects the clustering in the glass system. It leads to increase the intensity of non-radiative transition in the glass system. The experimental lifetime for quenching concentration (0.50Nd) is 146.74 µs which is higher than 122, 95.8, and 72 µs for TeNd05 [4], Bi-60 [15], and SLBNd0.5 glass [16] respectively.
The ZnO-BaO-B$_2$O$_3$-TeO$_2$ (ZBBoT) glass samples were prepared, and their properties affected by varied concentrations of Nd$_2$O$_3$ doped in were investigated. The traditional melt quenching process was conducted to synthesize the samples. The glass composition is (35-x)TeO$_2$-30Ba$_2$O$_3$-10ZnO-25BaO-xNd$_2$O$_3$, where x is 0.00, 0.05,0.50, 1.00 and 1.50 mol%. The density of the samples increased with the increment of Nd$^{3+}$ ion concentration from 0.00 to 1.50 mol%, while the molar volume decreased. The absorption spectra demonstrated seven obvious peaks centered at 519 nm ($^4G_{5/2}$), 526 nm ($^2K_{13/2}$), 583 nm ($^2G_{5/2}$+G$^{3/2}$), 684 nm ($^4F_{9/2}$), 745 nm ($^4S_{3/2}$+F$^{7/2}$), 803 nm ($^4F_{5/2}$+$^2H_{9/2}$) and 877 nm ($^4F_{7/2}$), where the energy states specified in the parentheses. Most of the absorptions occurred in visible and infrared ranges from 450 nm to 1000 nm. The intensity of the spectra increased with the increment of Nd$_2$O$_3$ concentration. The excitation spectra showed eight obvious peaks for the given emission wavelength of 1.060 nm, corresponding to the excited wavelength at 429 nm ($^2I_{9/2}$→$^2I_{13/2}$), 477 nm ($^2I_{9/2}$→$^2D_{3/2}$), 524 nm ($^2I_{9/2}$→$^2G_{7/2}$), 575 nm ($^2I_{9/2}$→$^4S_{5/2}$), 680 nm ($^2I_{9/2}$→$^2H_{11/2}$), 745 nm ($^2I_{9/2}$→$^4F_{9/2}$), 803 nm ($^2I_{9/2}$→$^4F_{7/2}$+$^2S_{1/2}$), and 882 nm ($^2I_{9/2}$→$^4F_{5/2}$+$^2H_{9/2}$), where the energy transitions specified in the parentheses. The highest peaks of each the emission spectrum are at the common wavelength of 575 nm corresponding to the final state of the transition, $^4G_{5/2}$ nm. The emission spectra showed two obvious peaks in the range of NIR. Those were corresponding to the emitted wavelength at 1060 nm ($^4F_{3/2}$→$^4I_{11/2}$) and 1345 nm ($^4F_{5/2}$→$^4I_{15/2}$), where the energy transitions shown in the parentheses. The intensity of each emission spectrum was at 1060 nm and increased with the concentration of Nd$_2$O$_3$ until 0.50 mol%. In the J-O analysis, the sample of ZBBoT glass doped with Nd$_2$O$_3$ of which the concentrations 0.50 mol% was selected to investigate its emission. The absorption spectra around 583 nm were analyzed to identify the experimental oscillator strength ($f_{\text{exp}}$) and calculated oscillator strength ($f_{\text{calc}}$). Those parameters: 0.80 x10$^{-6}$ and 0.90 x10$^{-6}$, respectively, indicated that the energy transition $^2I_{9/2}$→$^4G_{5/2}$+$^2G_{7/2}$ exhibited the strongest oscillator strength among the others. By the comparison of the parameter $\Omega_2$ to the previous studies, it was concluded that ligands surrounded Nd$^{3+}$ in ZBBoT glass matrix were high asymmetry and their bonds were higher covalency than ZBSN4 and BBFBNd, but lower than silicate and TZO0.5Nd. The parameters $\Omega_2$ and $\Omega_6$ of ZBBoT indicated that its viscosity and rigidity tended to lower compared to other types of glass. The energy transition $^2F_{3/2}$→$^2I_{13/2}$ around 1060 nm showed the highest values of the parameters: radiative transition probability ($A_{\text{R}}$), stimulated cross-section ($\sigma(\lambda_p)$), and branching ratios ($\beta R$) for ZBBoT glass sample doped with Nd$_2$O$_3$. Especially, the ($\sigma (\lambda_p)$) value of

![Figure 5](image-url)
6.32 \times 10^{-20} \text{ cm}^2 ensured that the glass sample can be used to generate high amount of laser emission consuming low energy. The proportion between the experimental branching ratios ($\beta_{\text{Exp}}$) and calculated ones ($\beta_{\text{Cal}}$) is more than 0.50 mol% promising that ZBBoT glass can generate laser light at 1060 nm. Hence, the sample of ZBBoT glass doped with Nd$_2$O$_3$ of which the concentrations 0.50 mol% has an interesting potential to apply as laser medium materials with 1060 nm emission.

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