#### DEVELOPMENT OF NEW METHODS OF THE DIODE LASER SPECTROSCOPY AND THEIR APPLICATIONS FOR DETECTION MOLECULE IN GAS MIXTURES AND MEASUREMENTS OF THE ISOTOPIC COMPOSITION



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#### Introduction

The department of diode laser spectroscopy (A.M.Prokhorov General Physics Institute of RAS) deals with diode laser (DL) spectroscopy researches and gas analyze over 30 years. There have been created dozens of unique DL gas analyzers and spectrometers in the department for this period. They have been made on the basis of DL in the near and mid IR range for various applied issues. These devices are known as very reliable and easy to use and judging by some parameters they excel other existing analogues. The development of technology of manufacturing diode lasers with fiber output operating in the near IR range makes it possible to create compact, mobile DL gas analyzers with high parameters of sensitivity and speed performance.

The report describes the DL gas analyzers made in the department last years, also considers new methods of measurements the concentration of molecules in different buffer gases including air and estimation of isotopic composition for various molecules in the near and mid IR range.

# Why diode lasers are?

The all necessary components of a DL (active element, pumping, resonator) locate in the single semiconductor chip.



Semiconductor as an active element provides maximum possible performance laser generation in comparison with the other types of lasers. These circumstances define very small size (microns dimensions) of a DL.

Typical performance index for DL is 90 %.

Finally, built-in resonator not only gives absence of technical noises, which are typical for other types of lasers, but also helps to achieve fundamental sensitivity limit due to quantum radiation noises.

# DL and absorption spectra of different molecules in the near IR range.



Practically all molecules have absorption bands in the range 1-2  $\mu$ m (near IR). Blue circles show DL which are used in DLS department GPI RAS.

### **Photodetectors**

Traditionally we use InGa As pin Photodetectors produced by "Hamamatsu".

Spectral response





D\* [1] – detectivity. NEP (Noise Equivalent Power) is a power which is equivalent to noise  $2.10^{-12}$  W/Hz<sup>1/2</sup>

<sup>1</sup>. R.Smith, R.Chasmar, Detection and measurement of infrared radiation, Oxford University, London, (1957)

#### Principles of the molecular detection in DLS

Shapes of acquisition signal with W and without  $W_0$  absorption of a HF.

Absorption spectrum of HF (L=7cm,  $P_0$ =1Bar) Bouguer-Lambert-Beer law.



Noise, interference, disturbance of the receive signal (optoelectronics channel) determine minimal detectable absorption level of molecule.

#### Measurements of impurities CH<sub>4</sub>, C<sub>2</sub>H<sub>4</sub>, H<sub>2</sub>O, NH<sub>3</sub>, H<sub>2</sub>S and CO<sub>2</sub> in process of the highpurity hydrides rectification

- High purity hydrides (NH3, AsH3, PH3, SiH4, GeH4) are important components of modern electronics and optoelectronics technologies
- Requirement: impurities concentration has to be less than <1-10 ppb.</p>
- Real time impurities control is the key element of purification process optimization. DLS system must have next advantages: easy to use, reliability, high sensitivity, selectivity and performance.
- $Ga(CH_3)_3 + AsH_3 \rightarrow GaAs$   $PH_3 \rightarrow GaP$   $NH_3 \rightarrow GaN$   $Al(CH_3)_3 + AsH_3 \rightarrow AlAs$   $In(CH_3)_3 + AsH_3 \rightarrow InAs$



## **Rectification column**

Rectification column (6 m high) view and diagram. Raw material is loaded in rectification column. Due to heater and  $LN_2$ cooling condensation evaporation processes took place inside column. As result impurities are concentrated at bottom (Heavy Fraction - HF) and upper (Light Fraction - LF) parts of rectification column. Central part - pure material.



### TDLS complex block-scheme



# Connection diagram of optical cells in the gas system of rectification columns





Three channels TDLS complex to detect  $H_2O$  and  $CH_4$  in  $NH_3$ 

The cell is embedded in the columns for measuring the concentration of impurities.

LF was directed to cells with L = 2 cm, L = 150 cm and multipath cell with = 15.2 m to measure  $CH_4$  concentration. HF was directed to cell with L = 150 cm, L = 5 cm to measure  $H_2O$  concentration. To remove atmosphere water absorption dry nitrogen flow was used.

## **TDLS** complex



#### Analytical cells



#### TDLS Components (cont.)







L=70 см, 140 см



Herriot cell L= 22 and 17 m,

#### Allan plots



To determine the minimal detectable absorption of impurities, the Allan procedure was used. In Fig.(left ) the Allan variance of the absorption is plotted versus the averaging time. It is seen that the minimal detectable absorption at the path length 2 m approaches 3 10<sup>-7</sup> for the averaging time 3 s. Right- Allan variance instable DI temperature. For these time averaging Allan T approaches 1.5 10<sup>-5</sup> <sup>0</sup>C.

# System operation (modulation regime)



For known parameters of analytical and reference channels concentration of molecule under investigation can be obtained straightforward.

DL is excited by trapezoidal current pulse with modulation (A). In presence of molecular absorption two lines can be observed due to modulation (B). Using these data computer calculates signal looking like line first derivative (C). Two cannels are using in system - analytical and reference ones. Using reference channel both autocorrelation function of reference and correlation function of analytical and reference channels were determined – black and red in Fig.D, respectively

$$C = \frac{\alpha \cdot P_r \cdot L_r}{P_a \cdot L_a} \cdot 10^9, [ppb]$$
$$C_{xy} = \alpha \cdot C_{xx}$$

### System operation (cont.)



Recorded signals in reference (yellow) and analytical (white) channels.

Recorded signals processing in reference (yellow) and analytical (white) channels. Green

- reference model function.

Correlation function of processed recorded signals in reference (yellow) and analytical (white) channels with reference model function.

# Example of real-time concentration measurements using modulation regime



#### Software "Panorama" for recording panorama spectra



a. L

Absorption,

View of the soft "Panorama" for measurements absorption spectrum. DL temperature tuning region (from -5, to +55 °C). Soft uses for define optimal analytical spectral regions for molecular detections.

Time recording -several minute. spectral tuning -30 cm<sup>-1</sup>, resolutions -0,0004 см<sup>-1</sup>

Absorption spectrum of  $AsH_3$ in 1.6  $\mu$ m, L- 150 cm, P- 10 kPa



# Experimental spectra fitting

Fitting software was developed to determine lines parameters using Voigt, Rautian-Sobel'man (hard collisions model), Galatry (soft collision) model functions to fit experimental spectra.



Example of software interface. Left graph – experimental spectrum, spectral range with weak water lines (absorbance < 1 %) was investigated. Operator determines location of lines to be analyzed. In present case 7 lines. Right graph result of fitting (red) and residual (green). Results of fitting for 2 lines are presented on right part of picture.

#### Fitting experimental absorption spectra by model functions



$$\sigma - \sigma_0) = A \cdot \operatorname{Re}\left[\frac{W(x, y+z)}{1 - \sqrt{\pi z} \cdot W(x, y+z)}\right] \quad W(x, y) = \frac{i}{\pi} \int_{-\infty}^{+\infty} \frac{\exp(-t^2)dt}{x - t + iy},$$
$$y = \sqrt{\ln 2} \frac{\gamma_a}{\gamma_d}, \quad x = \sqrt{\ln 2} \frac{\sigma - \sigma_0}{\gamma_d} \quad \gamma_d = 3.5812 \ln 0^{-7} \sqrt{\frac{T}{M} \cdot \sigma_0}$$



(a)-Example of fitting multilinesspectrum using Rutian-Sobel'mancontur.(b)-Residual





#### Measurements of NH<sub>3</sub> near $\lambda$ =1.51 $\mu$ m.



#### Measurements of CH<sub>4</sub> in NH<sub>3</sub> near $\lambda$ =1.65 $\mu$ m.

For H<sub>2</sub>O channel water vapor line 7161.4101 cm<sup>-1</sup> was selected.



Spectral range near 1.65  $\mu$  was determined for  $\text{CH}_{4}$  detection.

In this spectral range both  $CH_4$  and  $NH_3$  have absorption. To determine analytical line to detect  $CH_4$  ammonia and methane spectra were recorded by DL in use.

Methane R4 <u>multiplet</u> (6057.088 cm<sup>-1</sup>) was selected as analytical one.

### Methane concentration



Methane concentration in LF was measured by TDLS in two cells and compared with a gas chromatography.

# Long-term concentration measurements impurities $NH_3$ in phosphine purification



Localization of impurity in heavy and light fractions in the process of cleaning hydrides.



Using the DL gas analyzers was found localization of the impurities which allowed to optimize the cleaning process..



Fig. TDLS ethylene impurity measurements in SiH<sub>4</sub>

#### Table 1 Set of DL gas analyzers for impurities detection in process of the purification hydrides released in the **GPI RAS**

Molecule	Spectral region, µm	Hydrides	Sensitivity,% vol
H <sub>2</sub> O	1,391	NH <sub>3,</sub> PH <sub>3</sub>	<b>1*10</b> <sup>-4</sup>
NH <sub>3</sub>	1,513	AsH <sub>3</sub>	<b>5*10</b> <sup>-5</sup>
$C_2 H_4$	1,635	SiH <sub>4</sub>	<b>1*10</b> <sup>-4</sup>
CH <sub>4</sub>	1,651	SiH <sub>4</sub>	<b>4*10</b> <sup>-5</sup>
CO <sub>2</sub>	1,601	PH <sub>3</sub> , SiH <sub>4</sub> , AsH <sub>3</sub>	<b>1*10</b> <sup>-3</sup>
H <sub>2</sub> S	1,601	AsH <sub>3</sub>	<b>1*10</b> <sup>-3</sup>
$C_2 H_2$	1,531	PH <sub>3</sub> , SiH <sub>4</sub>	<b>1*10<sup>-4</sup></b>

#### **Explosive decay products detection by use DLS**



Using experimental data from GC and MS. (Socorro, NM USA) was found that the **HNCO** – **isocyanic acid** is the decay products of nitrogen's explosives.

For all explosives samples under investigation 3.66 min peak (marked by arrow) was observed (upper picture). Lower picture this peak MS.

Hydrolysis acid in the atmosphere leads to:

 $HNCO+H_2O=NH_3+CO_2$ 

Resume: ammonia is signature of all N containing explosives.



## Explosives detection in laboratory



<u>Field test in</u> <u>Russia.</u>

<u>Non-contact</u> <u>TNT sample</u> <u>detection in</u> <u>laboratory</u> <u>environment,</u> <u>M = 400 g</u>



# Albuquerque, USA

Backpack prototype for remote explosives detection (NH<sub>3</sub>,  $\lambda$ =1.51 µm ) was jointly developed by Aquila (Canberra) and GPI (DLSLAB).





## Moscow, Russia

Backpack prototype for remote explosives detection was developed by GPI (DLSLAB) using electronics manufactured by Aquila (Canberra).



## Minimum detectable concentration



NEC (Noise Equivalent Concentration of  $NH_3$ ) as function of averaging time as it was measured in Moscow.

For both instruments under consideration, NEC was determined by DL quantum noise.

<u>NEC together with explosive decay rate determine minimum detectable</u> <u>explosive mass inside moving vehicle and distance at which sample with</u> <u>given mass can be detected down wind.</u>

## Remote explosives detection

Several remote explosives detection scenarios can be considered.



Field test 1: remote explosives detection inside moving vehicle.



Field test 2: non-contact 2 kg explosive sample finding inside building using gradient of measured explosives signature concentration.

Field test 3: 300 kg explosive sample in wrapping was detected at 120 m down wind.

# TDLS instrument development for medical screening diagnostics

- •Screening approach in medicine is the massive inspection of population or persons with high risk of one or other illness.
- For medical screening diagnosis is important to:
- •Identify normal concentration of a gaseous component in the products of metabolism;
- •Establish the relationship of deviation from the norm with variety of pathological processes in the body.

To solve these problem was creation **TDLS instruments**:

- 1. Screening diagnostics water vapor and ammonia from palm surface.
- 2. Screening diagnostics of  $CH_4$ ,  $CO_2$  and  $H_2S$  in human breath.

# TDL Spectrometer for screening diagnostic $H_2O \rtimes NH_3$ from palm surface



#### Instrument view and block-scheme TDLS





#### Program interface.

## **TDLS Optoelectronics components**







S. M. Chernin's multipath cell with total optical path 39 m.

Electronics

### **Clinic device testing**



Clinical test of the spectrometer were carried out in the city clinical hospital № 12 of Moscow.

Characteristic measurement time

for single patient is around 30 sec.

## **Experimental results**

In screening experiment was tested control group patients (more than 50 people).





Measurement was carried out before and after dinner . Each peak on graph correspond to measurement of the single patient . The last peak –wipe. Fragment of the test. Measurement value is the ratio down and medium graphs.

### TDL Spectrometer for screening diagnostics of CH<sub>4</sub>, CO<sub>2</sub> and H<sub>2</sub>S in human breath







# TDLS for screening diagnostics of CH<sub>4</sub>, CO<sub>2</sub> and H<sub>2</sub>S in human breath (new version)



View of the experimental prototype TDLS for screening diagnostic  $CH_4$ ,  $CO_2$  and  $H_2S$  (new version)

Program interface.

In the 12 Moscow clinical hospital now tests 3-components TDLS for screening diagnostics exhalant components  $CH_4$ ,  $CO_2$  and  $H_2S$ 

### **Program interface**



Soft for simultaneous measurements  $CH_4$ ,  $CO_2$  and  $H_2S$  concentrations. Modulation regime gen. and correlation processing of spectrum

#### Analytical line of an absorptions



## **Clinical tests results**



•Clinical test of the spectrometers were carried out in the clinical hospital № 12 in Moscow.

•All measured gases have different concentrations before and after meals. •Abnormally high concentration of  $CH_4$ , exceeding the norm (0,3 ppm) on the order, was discovered on patients with chronic renal failure (поч. недостат.)

- In some cases (type II diabetes or bronchitis) abnormal concentration of  $H_2S$  were also measured.
- Big deviation of CO<sub>2</sub> was not discovered.
- •

• To increase number of detectable gases (NH<sub>3</sub>,  $C_2H_4$  H<sub>2</sub>O, HDO) it is planned to upgrade TDL spectrometer using NI RIO real time controller.

An aircraft system based on near-IR diode lasers for continuous measurements of the concentration of methane, carbon dioxide, water, and its isotopomers





Model YAK-42D [1]		
Cruising speed:	810 km/h	
Distance:	4100 кm	
Height:	<b>10</b> кт	

DL complex

The diode laser spectrometer developed for the aircraft laboratory YAK-42D. The spectrometer provides real-time measurements of  $CO_2$ ,  $CH_4$ , and  $H_2O$  content in the troposphere at a background concentration level ( $H_2^{16}O$ ,  $H_2^{18}O$ , HDO,  $H_2^{17}O$ ),  $CO_2$ ,  $CH_4$  [2].

[1] http://www.aviaport.ru/directory/aviation/jak42d/

[2] Ia. Ponurovskiy, et all, TDLS complex development for airplane-laboratory "Atmosphere", Abst. of Papers 7th International 2 Conference on Tunable Diode Laser Spectroscopy, p43, 2009.

#### Altitude profile of concentrations $H_2O$ (isotopomers), $CO_2$ , $CH_4$ , CO



Molecule	Variation concentrations from Earth surface up to 25 km altitude , ppm
Isotopomers H <sub>2</sub> O	3-15000
CO <sub>2</sub>	363-365
CH <sub>4</sub>	1.6-1.8
СО	0.05-0.12

•Interesting information : The spatial and temporal variability of the content of water vapor isotopes - HDO  $\mu$  H<sub>2</sub><sup>16</sup>O – in the atmosphere is of great interest for meteorological studies of the Earth's hydrological cycle.

#### **Experimental Setup**



Block-diagram of the aircraft DL spectrometer. The present system includes three modules: DL1 is designed to measure concentrations of water vapor and its isotopomers. DL2 measures carbon dioxide concentration. DL3 measures methane concentration



General view of vacuum Chernin multipath optical cell . The basic length of the cell is 25 cm, the volume of the cell is 2 liters, the number of paths is 156 with 39 m optical path length.



The total weight of the instrument is 25 kg, the height is 45 cm, and the width and depth correspond to the standard dimensions of a 19" telecommunication rack.

#### Experimental Setup (cont.)



Electronics with DL and reference channel





Air flow through the analytical cell is formed with a sample preparation module (Pressure stabilisator ) flow rate = 0.2 L/sec,  $T_{flow} = 23 \, {}^{0}C$ Total pressure inside cell Pcell -40 mBar



Design of the "Turbulence channel" Channel to measure the parameters of atmospheric turbulence. Turbulence was determined through measurements of fluctuations in the distribution of water vapor concentrations.

1, 2, 4 belongs to TDLS complex air receiving module. Part of water channel DL radiation is directed to turbulence channel. Turbulence channel contains part located in box 3. Fiber output (DL) is located close to illuminator (4). DL radiation is reflected by mirror (5) and detected by PD located close to illuminator. Optical length of atmosphere under investigation (out of airplane) is L = 40 cm.



# TDLS complex for airplane-laboratory in the telecommunication rack



#### Electronic module



#### Cell module



# Analytical spectral ranges



carbon dioxide - R 30e line of the 30013 << 0001 band.

Spectral range 7182.5 – 7185 cm<sup>-1</sup> for isotopomers  $H_2O$ 

## CH<sub>4</sub> and CO<sub>2</sub> channels interface



For known parameters of analytical and reference channels concentration of molecule under investigation can be obtained straightforward. DL is excited by special excitation current pulse with modulation to measure both strong and weak lines.

A – recorded signal.

B - signal after passing Odd-Even program module looks like absorbance first derivative. C – B after passing optimal filter looks like absorbance second derivative. Optimal filter is Gaussian monoimpuls (G).  $C_{XY}$  - convolution signal B with G.

 $C = \frac{\alpha \cdot P_r \cdot L_r}{P_a \cdot L_a} \cdot 10^9, [ppb]$  $C_{xy} = \alpha \cdot C_{yy}$ 

# H<sub>2</sub>O channel interface



•Linearization recorded water signal using Fabry-Perrot interferometer  $(D^* = 0.04933 \text{ cm}^{-1}))$ •Execution operations A, B and C (like CH<sub>4</sub> and CO<sub>2</sub>) with recorded water signal. Calculation function C<sub>XY</sub> using G-filter.

•Synthesis 4 basis functions of a isotopomers:  $(H_2^{16}O, H_2^{17}O, H_2^{18}O, H_2^{18}O)$  ) using Hitran database . Model Parameters: Length 39 m, (like multipath cell), Totall pressure 40 mBar, (air flow through cell), 1% concentration  $H_2^{16}O$ . String of synthesis: Absorption spectrum >> Derivative >>  $C_{XXi}$  functions by use Gfilter.

•Linear regression  $C_{XY}$  with respect to  $C_{XXi}$  determination concentration isotopomers of a water vapor .

## **Program Interface**



 This system can be employed for real-time measurements of the concentration of water vapor isotopes, carbon dioxide, and methane.

# Preliminary results of concentrations measurements



Measurements CO<sub>2</sub> and CH<sub>4</sub>



Windows of the water vapor concentrations isotopomers measurements

The minimal detectable concentrations (preliminary tests): 20 ppb –CH<sub>4</sub>, 2-3 ppm -  $CO_2$ . Water isotopomers: 10 ppm (H<sub>2</sub><sup>16</sup>O), 30 ppm (H<sub>2</sub><sup>18</sup>O), 300 ppm (H<sub>2</sub><sup>17</sup>O), and 1100 ppm (HDO).

Single time measurements of all concentrations - 50 ms. At aircraft speed 800 km/h the spatial concentration resolution achieve 12 m. At present undergoing pre-flight testing the all component gas analyzer

### "Turbulence channel" field tests

by GPS.



10

L, m

0.1

0.01 0.1

Allan dev, mBar

Field test . Operator used Leninsky prospect and Kiev highway. A –start point (GPI location), B – final point. Distance between A and B – 40 km. By blue line trajectory is shown as was measured



Water partial pressure as measured during this field test. These information used to determine Allan deviation plot.

•Allan plot of fragment presented above. To obtain this graph GPS data were used to transform time dependence to spatial one.

100

• For big averaging lengths, a drift due to the large-scale non-uniformity of water vapor distribution over the automobile route can be observed.

1000

•A broad maximum centered at 3 m, which is possibly due to the distance between the automobiles in the flow of traffic.

# Investigation of <sup>235</sup>UF<sub>6</sub> and <sup>238</sup>UF<sub>6</sub> spectra in the middle IR region with a quantum-cascade diode laser

A precision study of UF<sub>6</sub> spectra in the middle IR–band is required for activities for the benefit of IAEA aimed at prompt determination of the degree of uranium hexafluoride enrichment with the TDLS method [1]. Formerly, we investigated UF<sub>6</sub> IR spectra using a Fabry-Perot diode laser (FP DL) [2]. The laser generated near the maximum of absorption band  $v_1+v_3$  ( $F_{1u}$ ) of a UF<sub>6</sub> molecule at the wavelength of  $\lambda$ =7.746 µm and ensured tuning of ≈2.5 cm<sup>-1</sup> in the range of 1289.76 – 1292.26 cm<sup>-1</sup>. The mean laser power was 0.3 mW and the lasing line width was 5·10<sup>-4</sup> cm<sup>-1</sup>. It was shown [3] that the frequency range employed was not sufficiently effective for the determination of UF<sub>6</sub> enrichment degree. A conclusion was driven at of the need in a DL operating in the optimal spectral range with the centre at near 1293 cm<sup>-1</sup> and the continuous tuning range of ~4-5 cm<sup>-1</sup>.

This paper discusses the results of a study of UF<sub>6</sub> IR spectra with the use of a home designed spectrometer based on a quantum-cascade laser (QCL) of the "Hamamatsu" company make. The QCL generated at the wavelength of  $\lambda$ =7.734 µm with the mean laser power equal to 10.0 mW, the lasing line width of 1.0·10<sup>-4</sup> cm<sup>-1</sup> and ensured current tuning of ≈3.5 cm<sup>-1</sup> over the range of 1290.0 – 1293.50 cm<sup>-1</sup>. **References** 

1. G. Grigoriev, S. Malyugin, Sh. Nabiev, A. Nadezhdinskii, Ya. Ponurovskii, D.Stavrovskii, Yu. Ponomarev, *Atmospheric and Oceanic Optics*, **18**, 797-806 (2005).

2. A. Berezin, G. Grigoriev, S. Malyugin, Sh. Nabiev, A. Nadezhdinskii, Ya. Ponurovskii, D.Stavrovskii, Yu. Shapovalov, *Spectrochim. Acta*, **66A**, 796-802 (2007).

3. G. Grigoriev, S. Malyugin, Sh. Nabiev, A. Nadezhdinskii, Ya. Ponurovskii, Atomic Energy, 104, 302-306 (2008).

### Experimental setup view (new ver.)



Block-scheme

- 1. DL in cryostat; 2. Reference PD;
- 3. Reference gas cell; 4. Analytical PD;
- 5. Analytical gas cell; 6. FP etalon;

Analytical channel with gas cell (5) containing gas under investigation. Reference channel with cells (3) containing methane and acetylene, and with Fabry-Perot etalon (6) were used to determine laser frequency tuning.



### View of UF<sub>6</sub> gas distribution system (National Research

Center "Kurchatov Institute")



Containers with UF<sub>6</sub> samples of different degree of enrichment.

8. Connection to TDLS system

#### Temperature and current frequency tuning QCL



FP res. Free spectra region  $D^* = 0.05 \text{ cm}^{-1}$ 

#### Experimental spectrum $^{238}UF_6$ , $^{235}UF_6$



#### Experimental spectrum of the $UF_6$ (cont.)



Pressure dependence spectrum of UF<sub>6</sub> White -10 Topp Read-20 Topp Green-30 Topp  $L_{cell}$  -20 cm

> Fine structure different White <sup>238</sup> UF<sub>6</sub> Red <sup>235</sup> UF<sub>6</sub>

> > Plot 0

4500

5000

Plot1 🔼



#### Fine structure "next level" (diff. sign) different isotopomers UF<sub>6</sub>

White  $^{238}$  UF<sub>6</sub> Red  $^{235}$  UF<sub>6</sub>



Corr. Function White  $^{238}$  UF<sub>6</sub> Red  $^{235}$  UF<sub>6</sub>







# Estimation of the <sup>235</sup>UF<sub>6</sub> enrichment degree from fine structure of the absorption spectrum



 $^{235}$ UF<sub>6</sub> enrichment degree defined by linear fitting Cross. Corr. Functions of the unknown sample UF<sub>6</sub> and natural isotopomer of the  $^{238}$ UF<sub>6</sub>

#### Our collaborations

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