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MODERNIZATION OF A THERMAL DESORPTION SPECTROSCOPY SETUP FOR HIGH-SENSITIVITY ANALYSIS OF HYDROGEN AND HELIUM ISOTOPES

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Abstract. This paper presents the results of an engineering and technical development aimed at modernizing a thermal desorption spectroscopy (TDS) system to enhance the sensitivity of gas-phase mass spectrometry analysis. The measurement system was integrated with a high-sensitivity DLS-20 mass spectrometer operating in conjunction with an RGA-100 quadrupole residual gas analyzer. A vacuum circuit separation scheme utilizing an adjustable leak valve was implemented, providing precise gas flow control and ensuring the stable operation of the DLS-20 analyzer in the low-pressure regime. The relevance of this work stems from the investigation of processes and the determination of gas release mechanisms from tritium-breeding materials, where hydrogen isotopes (primarily as HT molecules) and helium are generated simultaneously. During mass spectrometry analysis, their signals overlap at the mass-to-charge ratio of $m/z \approx 4$, which complicates the interpretation of TDS experimental data. It is demonstrated that adjusting the opening degree of the mechanical leak valve allows for a more than 30-fold reduction in the gas flow entering the analyzer. The system was calibrated using helium-deuterium mixtures, revealing that the detection sensitivity for deuterium is approximately 2.7 times higher than that for helium. Reliable resolution of the helium, HT, and D₂ peaks was achieved. The obtained results demonstrate the feasibility of utilizing the modernized setup for high-sensitivity investigations of tritium, hydrogen isotope, and helium release processes, as well as for improving the reliability of TDS spectra interpretation.

Keywords: thermal desorption spectroscopy (TDS), mass spectrometry, tritium-breeding materials, gas evolution mechanisms.

1. Introduction

The method of thermal desorption spectroscopy (TDS) is widely applied to investigate the processes of gas retention and release from solid materials [1,2]. This method provides comprehensive information on the mechanisms of gas diffusion, trapping, and detrapping, as well as the energetic characteristics of gas atom interactions with crystal lattice defects [3,4]. TDS studies acquire particular relevance when examining materials intended for use in nuclear and thermonuclear power plants. In such materials, neutron irradiation simultaneously generates hydrogen isotopes, including tritium, alongside helium produced via nuclear reactions [5,6]. Investigating the retention and release processes of these gases represents a pivotal task in evaluating the performance and operational lifetime of materials under service conditions [7-9]. One of the primary challenges in the mass spectrometry analysis of gas evolution is the overlap of signals from different gases with close mass numbers. Specifically, in studies of tritium-breeding materials, tritium release frequently occurs in the form of HT molecules, which are detected at the mass-to-charge ratio of $m/z \approx 4$. Since the helium

signal is recorded in the exact same region, interpreting the results using standard quadrupole mass spectrometers becomes significantly hindered. An additional complexity arises from the presence of background hydrogen in vacuum systems, which can substantially exceed the flux of hydrogen isotopes evolving from the investigated sample. This constrains the sensitivity of the method and reduces the reliability of analyzing gas release processes. Consequently, there is a clear need to enhance the sensitivity of TDS measurement systems and develop approaches for a more accurate analysis of the gas phase composition, particularly in cases involving overlapping signals of helium and hydrogen isotopes. From an engineering perspective, this problem can be addressed by modernizing the TDS setup through the integration of high-sensitivity mass spectrometers and the implementation of managed gas flow separation schemes between different components of the vacuum system. The objective of the present work is to upgrade the existing thermal desorption spectroscopy system by integrating a DLS-20 high-resolution mass spectrometer, and to investigate the capabilities of the proposed measurement scheme for registering low-intensity gas flows and analyzing hydrogen isotopes and helium, including tasks associated with tritium release.

2. Methods

Experimental Setup

The key concept behind modernizing the thermal desorption spectroscopy (TDS) setup was to expand the existing measurement scheme by integrating an additional high-sensitivity DLS-20 mass spectrometer. The upgraded system comprises two mass spectrometers: an RGA-100 quadrupole residual gas analyzer [10] (Stanford Research Systems) and a DLS-20 mass spectrometer (Hiden Analytical Limited) [11], each connected to its own vacuum system equipped with independent turbomolecular pumps. This configuration ensures optimal operating conditions for each analyzer and enhances measurement stability across a wide pressure range. The connection between the TDS chamber and the DLS-20 mass spectrometer chamber is established via an adjustable leak valve (L1, serving as a throttling element), which allows for precise control of the gas flow entering the high-sensitivity analyzer. The implementation of independent vacuum circuits and the adjustable leak valve represents a key element of the modernization, enabling the DLS-20 mass spectrometer to operate in the low-pressure regime regardless of the pressure level inside the TDS chamber. This feature is of paramount importance for registering low-intensity gas flows and performing high-sensitivity measurements. The RGA-100 mass spectrometer is capable of recording gas spectra in the mass range from 1 to 100 amu. The principle of gas detection is based on the ionization of a small fraction of gas molecules; the resulting positive ions are subsequently separated by mass and measured using a Faraday cup as the detector.

The DLS-20 is a quadrupole mass spectrometer featuring ultra-high mass resolution (up to $\Delta m \approx 0.005$ amu) and an increased quadrupole rod diameter of 20 mm (which provides enhanced sensitivity). It offers two operational modes:

1. RGA (Zone 1): a wide mass range mode (up to ~ 200 amu);
2. zhRGA (Zone H): an ultra-high-resolution mode (up to ~ 22.5 amu).

The instrument utilizes an ion-counting secondary electron multiplier (SEM) as a detector, which ensures high sensitivity combined with a low background noise level. Since the recommended maximum operating pressure for the SEM detector is 5×10^{-6} Torr, the pressure within the DLS-20 mass spectrometer chamber was maintained below this threshold throughout the experiments. To achieve this operating regime, a gas flow regulation scheme between the setup chambers was implemented.

The vacuum system comprised the following components:

- RGA-100 quadrupole mass spectrometer;
- DLS-20 quadrupole mass spectrometer;
- Turbomolecular pumps (assigned to each vacuum circuit);
- Scroll (roughing) pump;
- Ionization vacuum gauges;
- Diaphragm vacuum gauge;
- Gas inlet system.

A schematic diagram of the modernized setup is depicted in Figure 1. The newly integrated elements associated with the DLS-20 mass spectrometer and its corresponding vacuum system are highlighted in red.

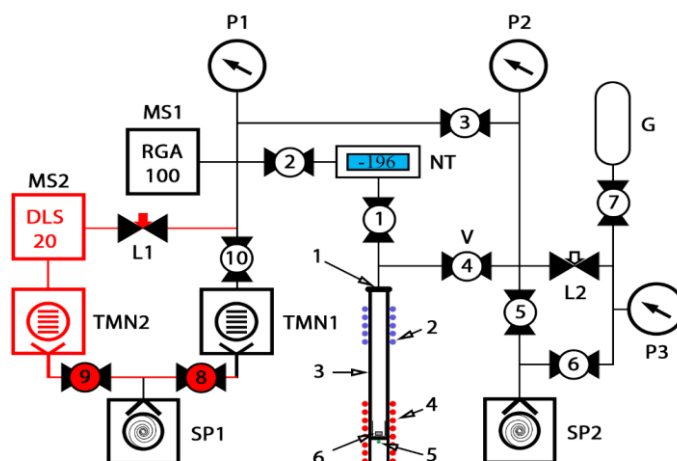


Fig 1. Schematic diagram of the modernized TDS setup integrated with the high-sensitivity DLS-20 mass spectrometer: MS1 – RGA-100 mass spectrometer; MS2 – DLS-20 mass spectrometer; TMN1 – turbomolecular pump of the RGA-100 vacuum circuit; TMN2 – turbomolecular pump of the DLS-20 vacuum circuit; SP1 – scroll (roughing) pump of the RGA-100 vacuum circuit; SP2 – scroll (roughing) pump of the DLS-20 vacuum circuit; NT – nitrogen trap; P1 – ionization vacuum gauge in the mass spectrometer chamber; L1 – leak valve between the TDS and DLS-20 chambers; L2 – leak valve for the controlled inlet of gas (atmospheric air or the helium-deuterium mixture) into the TDS chamber; P2 – ionization vacuum gauge in the experimental device chamber; P3 – diaphragm vacuum gauge; V – vacuum valves; G – replaceable gas cylinder; 1 – loading flange; 2 – cooling system; 3 – experimental device; 4 – heating element; 5 – thermocouple; 6 – crucible with the sample.”

3. Results

Investigation of the Leak Valve Opening Influence on Gas Flow

At the first stage of the work, experiments were performed to evaluate the feasibility of regulating gas flows from the TDS + RGA-100 chamber into the chamber of the high-sensitivity DLS-20 mass spectrometer operating in the wide-range (RGA) mode. Mass spectrometry measurements were conducted at various opening degrees of the adjustable leak valve (L1) connecting the chambers of the setup.

The following operating modes were considered:

1. Leak valve fully open;
2. Leak valve fully closed;
3. Leak valve open by one turn;
4. Leak valve open by a half turn;
5. Leak valve open by a quarter turn.

At an initial system pressure of approximately 1.3×10^{-6} Torr, the mass spectra of the main gas-phase components were recorded. The dependence of the gas partial pressures within the setup chambers on the various opening degrees of the leak valve is shown in Figure 2.

The quasi-equilibrium readings of the mass spectrometers are summarized in Table 1. Data for signals with low intensity and high noise levels ($m/z = 2$ and $m/z = 44$) were omitted, as they were not utilized in the subsequent analysis. During the experiments, a controlled inlet of atmospheric gases into the TDS chamber was established via a mechanical leak valve (L2). This approach ensured a quasi-stationary composition of the gas phase, which was then used to analyze the changes in gas flows between the setup chambers. Consequently, further evaluations of the reduction coefficients for gas flows from the TDS chamber to the DLS-20 mass spectrometer chamber were carried out for the atmospheric components.

Nitrogen N_2 was selected as the primary tracer gas because it provided the lowest error in determining the flow reduction coefficients. Water vapor exhibited a prolonged stabilization time to reach a quasi-equilibrium state, which led to an increase in measurement error. In the case of oxygen at a small leak valve opening degree (1/4 turn), the recorded flow was insufficiently high, causing a significant increase in the relative error (Table 2). Thus, it is demonstrated that adjusting the opening degree of the leak valve allows for a more than 30-fold reduction in the gas flow entering the DLS-20 mass spectrometer chamber. This operating mode can be utilized when the pressure inside the TDS chamber increases, thereby ensuring stable operating conditions for the high-sensitivity analyzer.

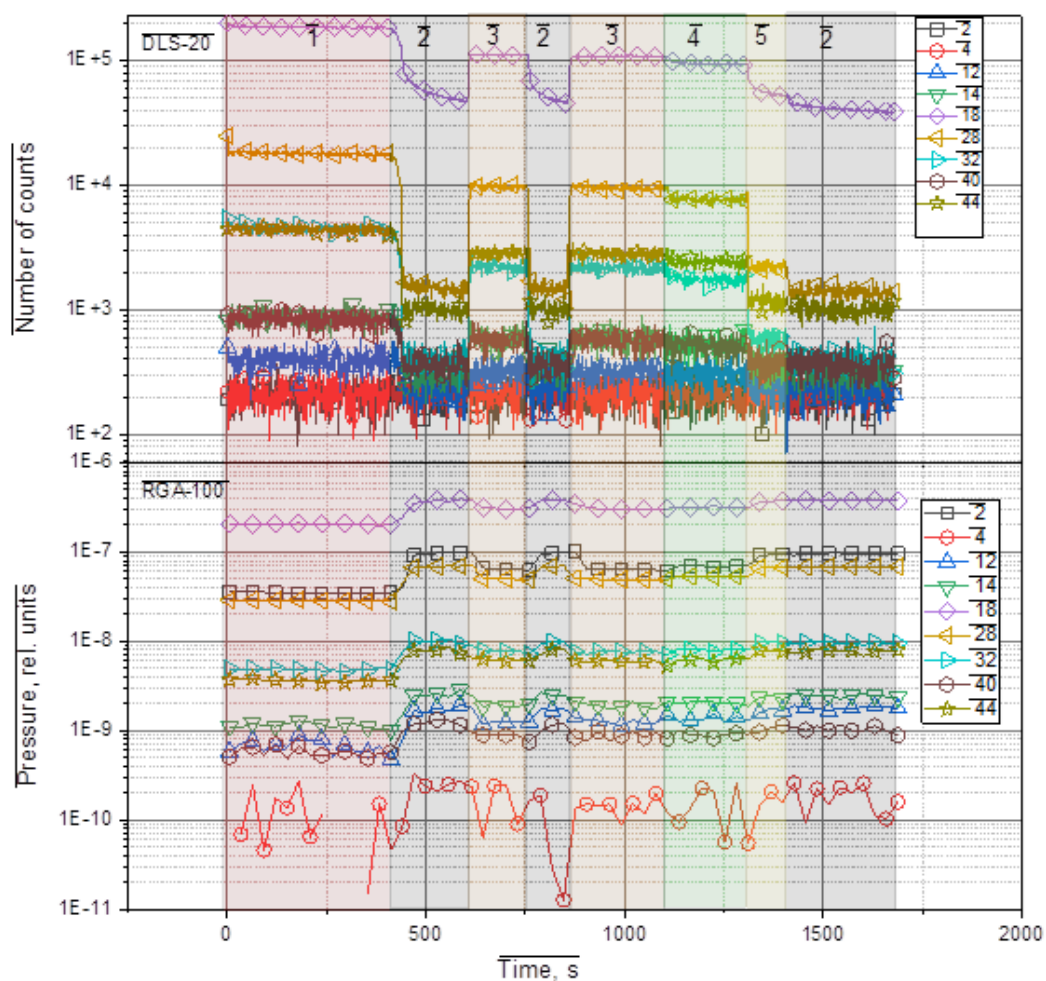


Fig 2. Diagram of changes in the partial pressures of various gases within the setup chambers at different positions of the valve between them.

Table 1. Quasi-equilibrium readings of the mass spectrometers at different stages of the experiment.

M	2	18	28	32	44
Gas	H ₂	H ₂ O	N ₂	O ₂	CO ₂
RGA-100 (1)	3,55E-08	1,97E-07	2,79E-08	4,93E-09	3,70E-09
RGA-100 (2)	1,02E-07	3,81E-07	6,90E-08	9,88E-09	7,90E-09
RGA-100 (3)	6,40E-08	3,03E-07	4,90E-08	7,60E-09	5,00E-09
RGA-100 (4)	6,90E-08	3,15E-07	5,30E-08	8,11E-09	6,30E-09
RGA-100 (5)	9,30E-08	3,72E-07	6,72E-08	9,76E-09	7,80E-09
DLS-20 (1)	-	1,87E+05	1,71E+04	4,45E+03	-
DLS-20 (2)	-	3,90E+04	1,40E+03	4,00E+02	-
DLS-20 (3)	-	1,10E+05	1,00E+04	2,50E+03	-
DLS-20 (4)	-	9,30E+04	7,75E+03	1,50E+03	-
DLS-20 (5)	-	5,30E+04	1,92E+03	6,00E+02	-

Table 2. Flow reduction factors (k) from the TDS chamber to the DLS-20 chamber at different positions of the connecting valve.

Gas type	28	32	28		32	
	N2	O2	N2		O2	
	Flow into the system, rel. units (during registration)		k	Error %	k	Error %
1-the system is open	1,57E+04	4,15E+03	1,000	1,3	1,000	4,8
3 system is open by 1 turn of the valve	8,60E+03	2,20E+03	0,548	2,3	0,530	9,1
4 the system is open by half a turn of the valve	6,35E+03	1,45E+03	0,405	3,1	0,349	13,8
5 the system is open by ¼ turn of the valve	5,20E+02	2,00E+02	0,033	38,5	0,048	100,0

In the considered mode, the measurement noise level was approximately 200 arbitrary units (arb. u.), which corresponds to a pressure of the order of 2×10^{-10} Torr according to the RGA-100 readings. Meanwhile, the minimum detectable signal that provides an acceptable measurement accuracy is around 1000 arb. u., corresponding to a pressure of about 1×10^{-9} Torr. It should be noted that as the opening degree of the leak valve decreases, the pressure detection sensitivity in the TDS chamber drops proportionally to the flow attenuation factor k. For instance, when the leak valve is open by 1/4 turn, the flow reduction factor is $k \approx 30$, which corresponds to the effective signal recording by the DLS-20 mass spectrometer at pressures above 3×10^{-8} Torr in the RGA-100 chamber. The operational parameters of the DLS-20 mass spectrometer in this mode are shown in Table 3.

Table 3. Operational parameters of the DLS-20 mass spectrometer in the wide-range (RGA) mode during the experiment.

Group	Parameter	Value	Units
Detector	discriminator	-10	%
	multiplier	1700	V
Filter	focus	-90	V
Quad	delta-m	0	%
	pre-post-bias	0.0	V
	resolution	0	%
Source	cage	3.0	V
	electron-energy	70.0	V
	emission	1.0	µA
Other	CRV_range	7	–
	CRV_resolution	6	bit
	DeadBandTime	0	ms
	mass	30.0	amu
	mode-change-delay	1000	ms

Experiments with Helium-Deuterium Mixtures

RGA-100 partial pressures in Torr are obtained by multiplying the raw registered (relative-unit) signal by the gas-specific ionization coefficient, and giving an approximate, order-of-magnitude comparison between the DLS-20 raw count rate and the corresponding RGA-100 partial pressure under the reported operating conditions. At the next stage of the work, calibration experiments for the DLS-20 mass spectrometer operating in the ultra-high resolution zhRGA mode were performed using pre-prepared helium-deuterium mixtures. Gas mixtures with a specified component ratio were prepared in volumes V1 and V1+V2 (with a volume ratio of $V2/V1 \approx 1.75$) and subsequently transferred into the replaceable gas cylinder (G). Further experiments were conducted using this cylinder as the source of the working gas. The gas was supplied to the vacuum chamber of the setup via a mechanical leak valve (L2) in a controlled continuous inlet mode. During the experiments, the signals from both the RGA-100 and DLS-20 mass spectrometers were recorded simultaneously, allowing for a comparison of the readings from the two analyzers and an evaluation of the gas-phase composition in the system. Typical mass spectra of helium and deuterium recorded in the setup chamber are shown in Figure 3.

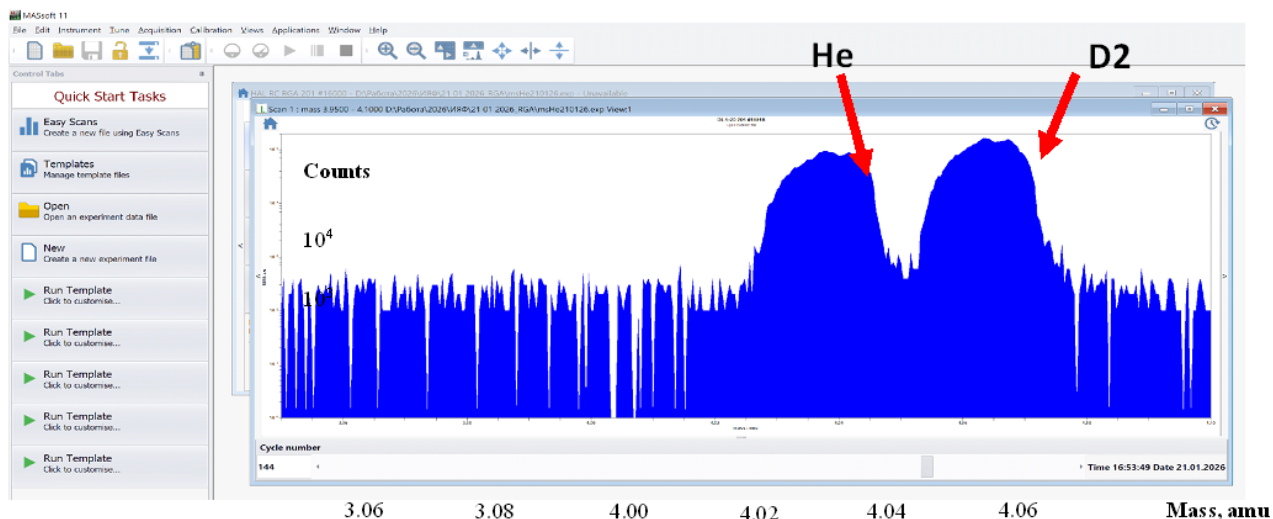


Fig 3. Typical mass spectra of helium and deuterium within the setup chamber.

According to the recorded mass numbers, when the helium-deuterium mixture is supplied to the setup chamber, the following values are obtained via the DLS-20: m/z (He) ≈ 4.038 and m/z (D2) ≈ 4.068 .

During the calibration of the DLS-20, the ultra-high resolution zhRGA mode differs from the standard wide-range RGA mode (see Figure 3) only by the following parameters:

- delta-m (0 $\rightarrow$ 20 %): Determines the effective mass peak width (Δm) and its narrowing degree (decreasing this value enhances the resolution and improves the separation of close masses);
- pre-post-bias (0 $\rightarrow$ 15 V): Sets the potentials before and after the quadrupole to adjust ion energy, which influences transmission efficiency and detection sensitivity;
- resolution (0 $\rightarrow$ 50 %): A tuning parameter for the quadrupole resolution; increasing this value improves mass separation but may reduce signal intensity;
- mass (46 $\rightarrow$ 5.5 amu):

The mass scanning range in the zhRGA mode, optimized for the analysis of light gases and isotopes; emission (1 $\rightarrow$ 100 μ A): The emission current of the electron gun in the ion source, which determines ionization intensity; increasing the current elevates the signal but accelerates cathode degradation. The experimental diagram along with a qualitative description of the stages are presented in Fig.4 and Table 4.

Table 4. Measured helium and deuterium flow into the system for various inlet modes.

	Inlet pressure He, Torr	Pressure D2 at the inlet	He-D2 ratio	Chamber pressure P	M4 RGA Pressure	M6 RGA Pressure	He (DLS20)	D ₂ (DLS20)	He-D2 ratio (according to DLS-20)	Note Working with:
1	707-672	0	1-0	1,7E-5	1.2E-5	Back-ground	37970	200 (Background)	1-0	pure He.
	707~672	0	1-0	3,6E-5	$\sim 3E-5$	Back-ground	44050-38540	200 (Background)	1-0	
	247	0	1-0	9,1E-6	$\sim 8,6E-6$	Back-ground	12360	200 (Background)	1-0	
2	90	440	0.17-0.83	6,2E-5	$\sim 5.6E-5$	$\sim 5.25E-7$	4690	57110	0.076-0.924	a mixture of He-D2 gases.
3	0	690	0-1	-	9,5E-5	1,61E-6		102990	0-1	pure D ₂ .
		251	0-1	-	2,7E-5	1,52E-7		28890	0-1	
4	440	89	0,83-0,17		2,81E-5	3,65E-8	20300	12690	0.61-0.39	a mixture of He-D2 gases.
	158	31	0,83-0,17		6,26E-6	4,37E-9	6160	4280	0.59-0.41	

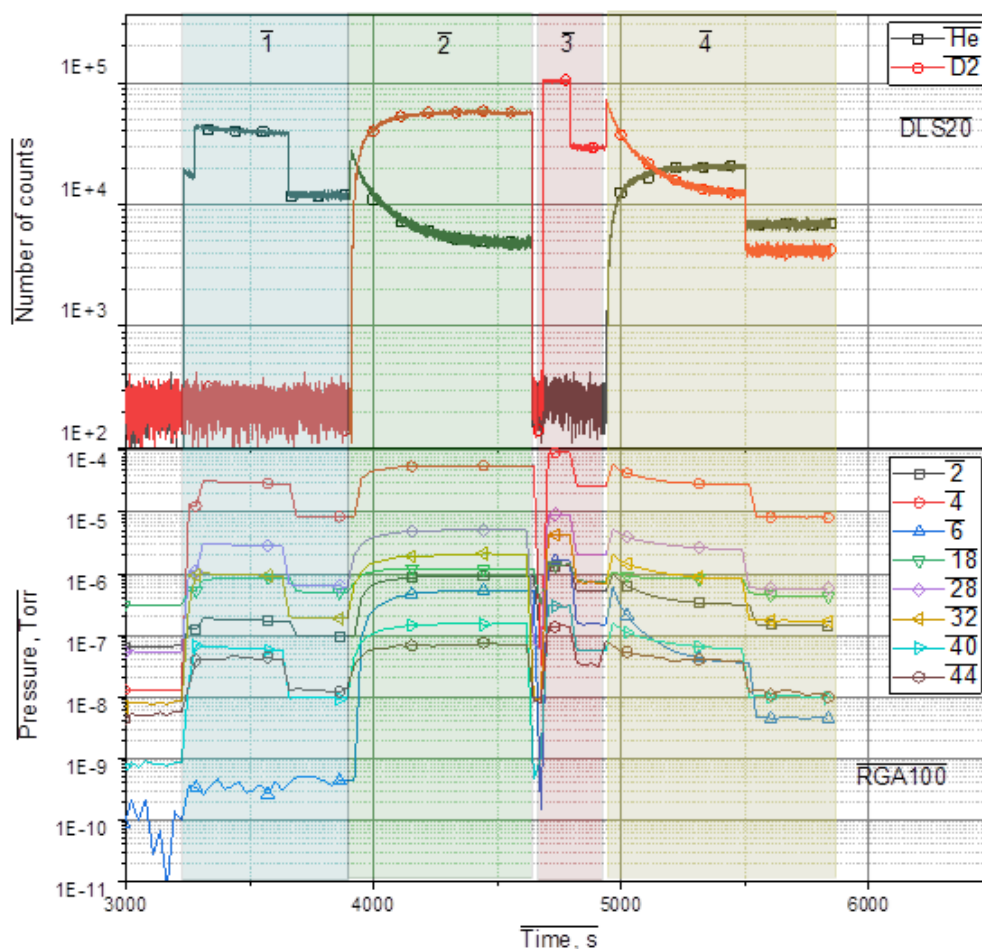


Fig 4. Diagram of experiments with the inlet of helium-deuterium mixtures into the setup chamber.

Based on the obtained data (Table 4), the intensities of the helium and deuterium mass spectrometry signals recorded by the DLS-20 mass spectrometer were plotted as a function of the inlet pressure of the corresponding gases (Figure 5). Analysis of the results revealed that both the sensitivity and the calibration factor for deuterium exceed those for helium by approximately 2.67 times.

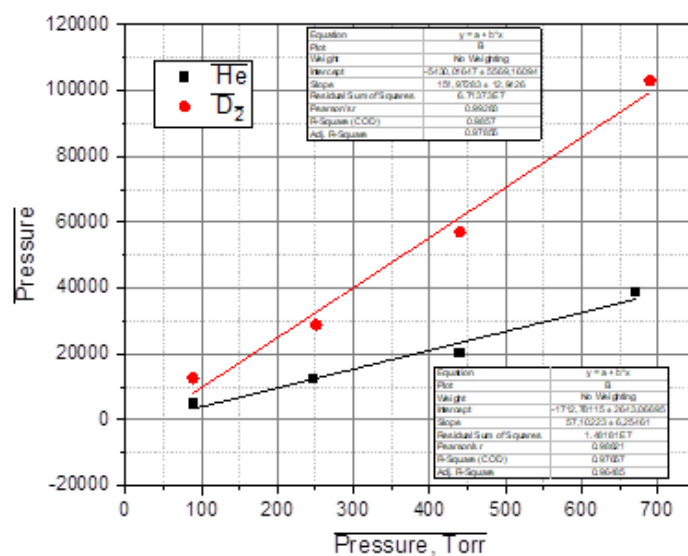


Fig 5. Peak intensities for helium and deuterium measured by the DLS-20 as a function of the inlet pressure of the corresponding gases.

This discrepancy must be taken into account during the quantitative interpretation of mass spectrometry data. The experiments were conducted with the leak valve opening degree corresponding to 1/4 turn.

4. Discussion

The conducted experiments demonstrated that the use of an adjustable leak valve allows for effective control over the gas flow entering the chamber of the high-sensitivity DLS-20 mass spectrometer from the investigated sample. It was established that decreasing the opening degree of the leak valve can reduce the gas flow by a factor of more than 30. This ensures stable operation of the analyzer within its optimal pressure range and prevents its overload during pressure increases inside the TDS chamber.

Calibration experiments with helium-deuterium mixtures indicated that the intensities of the helium and deuterium mass spectrometry signals recorded by the DLS-20 depend linearly on the inlet pressure of the corresponding gases. The obtained relationships confirm the validity of the proposed measurement scheme and the feasibility of performing a quantitative analysis of the gas-phase composition. A comparison of the detection sensitivities for different gases revealed that the calibration factor for deuterium is approximately 2.67 times higher than that for helium. This result must be taken into account during the quantitative interpretation of mass spectrometry data, particularly when analyzing mixtures containing hydrogen isotopes.

The proposed measurement scheme can be employed in studies of materials exhibiting simultaneous release of helium and hydrogen isotopes, including tritium. Under conditions where signals overlap in the mass-to-charge ratio region of $m/z \approx 4$, the modernized system enhances the reliability of gas-phase analysis and reduces uncertainty in the interpretation of TDS experimental results.

5. Conclusion

The thermal desorption spectroscopy setup was modernized by integrating a high-sensitivity DLS-20 mass spectrometer and implementing a vacuum circuit separation scheme using an adjustable leak valve. The proposed solution expands the range of detectable gas flows and enhances measurement stability. The conducted studies confirmed the performance of the proposed scheme and the feasibility of its application for the quantitative mass spectrometry analysis of the gas phase. It is demonstrated that the upgraded setup enables the detection of low-intensity gas flows and allows for sensitivity calibration across various components. The practical significance of this work lies in the applicability of the developed measurement scheme to investigate gas evolution processes from materials characterized by the simultaneous release of hydrogen isotopes and helium (e.g., beryllium-containing materials). Under conditions where signals overlap in the mass-to-charge ratio region of $m/z \approx 4$, the proposed approach improves the reliability of TDS experimental data interpretation.

Conflict of interest statement

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

CRediT author statement

Kulsartov T.V.: Conceptualization, Methodology, Writing Original Draft; **Akhanov A.M.:** Data Curation, Investigation; Software; **Askerbekov S.K.:** Methodology, Resources, Formal analysis; **Shaimerdenov A.A.:** Funding acquisition, Project administration, Supervision; **Chikhray Ye.V.:** Methodology, Validation, Data curation; **Bugybay Zh.T.:** Visualization, Writing – review & editing. The final manuscript was read and approved by all authors.

Statement on the use of Artificial Intelligence.

During the preparation of this manuscript, artificial intelligence tools were used solely for language editing and grammatical improvement. No AI tools were used to generate scientific content, analysis, results, or conclusions.

Data Availability Statement

The data that support the findings of this article are openly available.

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