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Results of Speciation Determination Measurements of Insulating Sulfur Hexafluoride Gas

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March 2023

SRNL-STI-2024-00142, Revision 0

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Prepared for
U.S. Department of Energy

SRNL-STI-2024-00142
Revision 0

Keywords: *tritium, water, sulfur hexafluoride*

Retention: *Permanent*

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Savannah River National Laboratory is operated by Battelle Savannah River Alliance for the U.S. Department of Energy under Contract No. 89303321CEM000080.

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ACKNOWLEDGEMENTS

The author gratefully acknowledges the funding and support from NNSA NA-231 Mo-99 Program Office. The author would also like to thank Billy Lao of DILO Company Inc. and Pieter Vos of Enervac International ULC for fielding questions about commercial SF_6 purification practices. Thanks to James Folkert in the assistance in traveling to the SHINE Heliopolis facility, setting up the instrumentation, and travelling back. Thanks to Lauren Hanna for computed vibrational modes used in the FTIR spectral interpretation.

EXECUTIVE SUMMARY

Under the direction of the NNSA NA-231 Mo-99 Program, SRNL has provided technical assistance to U.S. companies under cooperative agreements. One such area of technical assistance has been for the recommendation for contamination management methods in the eventuality that tritium contaminates the insulating gas of a high-voltage ion source used in the accelerator-based process developed by SHINE Medical Technologies to produce Mo-99. The SHINE accelerator process uses tritium as the target of a deuterium ion beam, where the ion beam is produced within a high-voltage ion source insulated with sulfur hexafluoride (SF_6), and is separated from the tritium target by an extensive pump train. Normal operating conditions preclude the conditions necessary for contamination of the insulating gas, however certain atypical conditions could result in a tritium contamination event.

Accelerator based processes for medical isotope production represent an anomalous tritium contamination challenge due to the presence of SF_6. SF_6 decomposes through normal use as an insulator gas, and the exact speciation of the decomposition byproducts depends on a variety of factors. This creates a scenario where the anticipation of species present in the insulating gas mixture is difficult. In turn, the fate of tritium within such a chemical system is unclear. This document discusses the results of measurements made *in-situ* of the SF_6 insulating gas in use within a SHINE Medical Technology's high-voltage ion source. This was done in an effort to determine the chemical speciation of the gas, allowing for the recommendation of management strategies in the event that tritium contaminates the insulating gas. The results of the measurements reported in this work suggest that tritium contamination in the insulating gas may result in two chemical forms of tritium in the system, tritiated water and molecular tritium. These two forms of tritium would be captured by two methods. Tritiated water may be captured and removed from the SF_6 by an adsorbent canister already present within the pressure vessel that houses the insulating gas while in use. It may also be separated from the SF_6 during routine purification of the SF_6, which is performed by SHINE personnel routinely. The purification cart will perform all non-embedded systems cleanup necessary to remediate tritiated species in the system. The purification cart passes the insulating gas through several filters, followed by the liquefication of the SF_6. The filters will separate tritiated water from the SF_6 at this stage. It is expected that molecular tritium will accumulate in the gas-phase during the liquification step, which is separated and stored. Tritium may be recovered from this mixture through more conventional hydrogen purification methods such as a palladium diffuser.

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LIST OF ABBREVIATIONS

CF_4	Carbon Tetrafluoride
S_2F_{10}	Disulfur Decafluoride
eV	Electron Volt
FTIR	Fourier-Transform Infrared Spectrometer
^3He	Helium-3
HF	Hydrogen Fluoride or Hydrofluoric Acid (aq)
H_2S	Hydrogen Sulfide
mA	milliampere
Mo-99	Molybdenum-99
m/z	Mass-to-Charge Ratio
NNSA	National Nuclear Security Administration
N_2	Nitrogen
NF_3	Nitrogen Trifluoride
O_2	Oxygen
PCB	Printed Circuit Board
psig	Pound per Square Inch - Gauge
RGA	Residual Gas Analyzer
SO_2	Sulfur Dioxide
SF_6	Sulfur Hexafluoride
SF_4	Sulfur Tetrafluoride
SO_2F_2	Sulfuryl Fluoride
SOF_2	Thionyl Fluoride
SOF_4	Thionyl Tetrafluoride
SRNL	Savannah River National Laboratory
T_2 or ^3H	Tritium
TF	Tritium Fluoride
HTO or T_2O	Tritiated Water
cm^{-1}	Wavenumber

1.0 Introduction

Under the direction of the NNSA NA-231 Mo-99 Program, SRNL has provided technical assistance to U.S. companies under cooperative agreements. One such area of technical assistance has been for the recommendation for contamination management methods in the eventuality that tritium contaminates the insulating gas of a high-voltage ion source used in the accelerator-based process developed by SHINE Medical Technologies to produce Mo-99. The SHINE accelerator process uses tritium as the target of a deuterium ion beam, where the ion beam is produced within a high-voltage ion source insulated with sulfur hexafluoride (SF_6), and is separated from the tritium target by an extensive pump train. Normal operating conditions preclude the conditions necessary for contamination of the insulating gas, however certain atypical conditions could result in a tritium contamination event. Accelerator based processes for medical isotope production represent an anomalous tritium contamination challenge due to the presence of sulfur hexafluoride (SF_6). SF_6 is used as an electrical insulator in accelerator applications due to its high dielectric strength and allows for the construction of smaller accelerator systems compared to other electrical insulation mediums such as air or dry nitrogen.

Tritium contaminated SF_6 creates a material without any obvious processes for managing the contamination, reuse, or disposal of the contaminated SF_6. Potential strategies documented in the FY21 work scope[1] included liquefaction, hydrates, dissolution, and caustic scrubbing-based approaches which all have potential to remove tritium from SF_6 and its decomposition byproducts by sequestering the tritium in water, which may then be treated by water detritiation methods. Bench- and pilot-scale testing to determine the degree of SF_6 byproduct generation and the efficiency of detritiation treatment methods is needed to make a recommendation on the best suited treatment method for accelerator-based Molybdium-99 production. To this end, SRNL developed an experimental plan during FY22[3] and in July-August 2022 travelled to SHINE's Heliopolis facility to make measurements of the SF_6 byproducts created during normal operation of the neutron generator used in the Mo-99 isotope production process. Issues during the initial start-up of the neutron generator resulted in no measurements being made during the trip. The experimental plan and discussion of the outcome of the plan during the July-August 2022 trip are outlined in two correspondence documents: SRNL-L1110-2022-00010[2] and SRNL-L1110-2022-00012[4].

Briefly, the plan comprised of a set-up stage and a measurement stage at the SHINE facilities. The goal of the set-up stage was for analytical instrumentation, supporting equipment, and a manifold to be transported to and prepared for use within the SHINE Heliopolis facility in Fitchburg, WI. The second stage of the plan was for SHINE personnel to begin operation of a neutron generator in the facility, during which measurements were made on the SF_6 in the ion source of the neutron generator using the equipment set up in the first stage of the plan.

The set-up stage was executed without issue, resulting in analytical instrumentation, supporting equipment, and a manifold ready for use within the SHINE Heliopolis facility in Fitchburg, WI. The second stage of the plan required SHINE personnel to begin operation of a neutron generator in the facility, during which measurements would be made on the SF_6 in the neutron generator using the equipment set up in the first stage of the plan. The second stage was not completed, as operation of the SHINE neutron generator was never established during this timeframe.

In July-August 2023, SRNL personnel again traveled to SHINE's Heliopolis facility to make measurements of the SF_6 byproducts created during normal operation of the neutron generator used in the Mo-99 isotope production process, beginning with SF_6 which had been cleaned using an SF_6 purification cart and running the ion source in order to generate SF_6 decomposition byproducts. Results of the July-August 2023 trip is outlined in the correspondence document: SRNL-L1110-2023-00005[4]. The original plan outlined in SRNL-L1110-2022-00010 was followed and measurements were taking during the operation of the neutron generator within the SHINE facility. The number of measurements totaled 14, each approximately 1.5 to 2 hours in length. 110 spectra were collected across 21 hours and 43 minutes of beam time.

Due to tritium permeation/diffusion through accelerator process and confinement materials, there is the possibility that tritium can contaminate the electrical insulation medium of the accelerator. Tritium contaminated SF_6 creates a material without any obvious processes for managing the contamination, reuse, or disposal of the used SF_6. This document will discuss the results of the measurements made on the SHINE neutron generator's insulating SF_6, and the resulting recommendations of management strategies for tritium contaminated SF_6 for accelerator-based processes for Molybdenum-99 (Mo-99) production.

2.0 Sulfur Hexafluoride Properties and Impurities

SF_6, used as an insulating gas for high voltage applications, is a fairly inert, stable, non-flammable, non-toxic gas. Insulating grade SF_6 may contain common impurities such as oxygen, nitrogen, and water on the ppm scale. Carbon tetrafluoride (CF_4) is also a common impurity of SF_6, as it is difficult to separate and does not greatly impact the insulating properties of the SF_6. Electric discharge within the SF_6 gas can decompose the gas into toxic sulfur-oxy-fluoro species[1]. These impurities exist independent of exposure to tritium, and when tritium is present, represent many mechanisms by which tritium can "contaminate" a gas that is typically treated as non-reactive.

There are numerous studies in the literature on the decomposition byproducts of SF_6, however the exact speciation of the byproducts depends greatly on the availability of oxygen, nitrogen, and water to the decomposition reaction. Common byproducts of SF_6 decomposition include hydrogen fluoride (HF), thionyl fluoride (SOF_2), thionyl tetrafluoride (SOF_4), sulfur tetrafluoride (SF_4), disulfur decafluoride (S_2F_{10}), sulfuryl fluoride (SO_2F_2), sulfur dioxide (SO_2), hydrogen sulfide (H_2S), nitrogen trifluoride (NF_3), and carbon tetrafluoride (CF_4).

The potential chemical reservoirs of tritium in such a chemical system are primarily in tritiated water, isotopologues of hydrogen sulfide, and tritium fluoride (TF). Potential strategies for isolating and treating contaminated SF_6 are outlined in SRNL-STI-2021-00313[1]. It was determined that there was not enough information about the decomposition byproduct speciation within the SHINE system to recommend a treatment approach. A research plan was developed, and measurements were made in late FY23 to determine the speciation of the decomposition byproducts in an effort to inform such an approach.

3.0 In-Situ Measurements of SF_6

SF_6 decomposition byproducts are generated during the normal operation of the high voltage system within the ion generator. As the ion source continues to operate, the concentration of decomposition byproducts increases[5]. Of the potential and expected decomposition byproduct species, an evaluation of analytical methods to determine the speciation of these byproducts determined that a combination of mass spectrometry and infrared spectroscopy would be capable of detecting all of the most likely species present, except S_2F_{10}, SF_4, and HF. S_2F_{10} fractionates into the same mass fragments as SF_6, and the absorbance spectrum of the molecule within an SF_6 matrix would not be distinguishable. The same issue is a concern for SF_4, as the fractionation pattern and absorbance spectra are similar. While HF is readily detectable by infrared spectroscopy and the mass 20 ion measured by mass spectrometry is indicative of HF, it is expected that HF will either react with the manifold and pressure vessel, and/or decompose to H^+ and F^+ ions in the mass spectrometer, which are not informative when detected in an SF_6 matrix. The other decomposition byproducts will be able to be uniquely identified by the two methods, provided that they reach sufficient concentrations to be detected.

3.1 Mass Spectrometric Analysis

The anticipated mass-to-charge ratio (*m/z*) for the expected and potential species in the insulating gas are detailed in Table 3-1. The nominal mass of S_2F_{10} is not expected to be able to be observed, as the electron impact ion source of the mass spectrometer would fragment the molecule, leaving only fragments at the same mass as the fragments of SF_6.

Table 3-1. Anticipated *m/z* ratios for expected and potential decomposition byproducts.

Component Name	Nominal Mass	Fractionation Peak *m/z* (primary mass peaks in bold)					z=2 Peaks	
H_2O	**18**	17	16					
CO_2	**44**	28						
SF_6	**146**	127	108	89	70	51		
SOF_2	86	**67**	48	32	19	16	24	
SOF_4	124	**105**	86	67				
SF_4	108	**89**	70					
S_2F_{10}	254	**127**	108	89	70	51	**127**	54
SO_2	**64**	48	32	16				
NF_3	71	**52**	33	19	14			
CF_4	88	**69**	50					
SiF_4	104	**85**						
COF_2	66	**47**	28	31				
OF_2	54	**35**	19	16				
HF	20	19						
H_2S	**34**	33	32					

3.2 Infrared Spectroscopic Analysis

The anticipated infrared absorption peaks for the expected and potential species in the insulating gas are detailed in Table 3-2. Because the insulating gas is primarily SF_6, the absorption peaks for SF_6 are expected to saturate the wavenumber regions at and around the 620 and 960 cm^{-1} bands, as well as combination and overtone bands at wavenumbers near 1240, 1580, and 1920 cm^{-1}. Because the absorption bands of the SF_6 will be so saturated, it is expected that infrared absorption will be useful for detecting H_2O, CO_2, HF, H_2S.

Table 3-2. Anticipated infrared absorption peaks for expected and potential species present in the insulating gas[6, 7].

Component Name	IR Primary Band	Other IR Bands	
H_2O	3280	1660	
CO_2	2345	3600	3700
SF_6	960	620	
SOF_2	808	747	1330
SOF_4	821	752	1383
SF_4	891.5	728	867
S_2F_{10}	827	940	1256
SO_2	1375	1125	1175
NF_3	905	1033	1929
CF_4	1212		
SiF_4	980		
COF_2	1894	1180	829
OF_2	928	1731	831
HF	3875		
H_2S	2612	2626	1178

3.3 Experimental

The manifold assembled at the SHINE Heliopolis facility is shown in Figure 3-1. Briefly, the manifold supplies purge gas and SF_6 from the pressure vessel surrounding the SHINE ion source. This vessel is maintained at 30 psig, and contains the ion source, water cooling lines, electronics including PCBs containing elements such as carbon and silicon, and polyethylene ductwork used to circulate the SF_6. It also contains a cartridge of adsorbent used to remove water from the circulating SF_6. The SHINE technicians allowed the ion beam to run throughout the days that testing occurred, so that the ion source would be in use within the SF_6. This created the necessary conditions under which the SF_6 insulating gas could decompose.

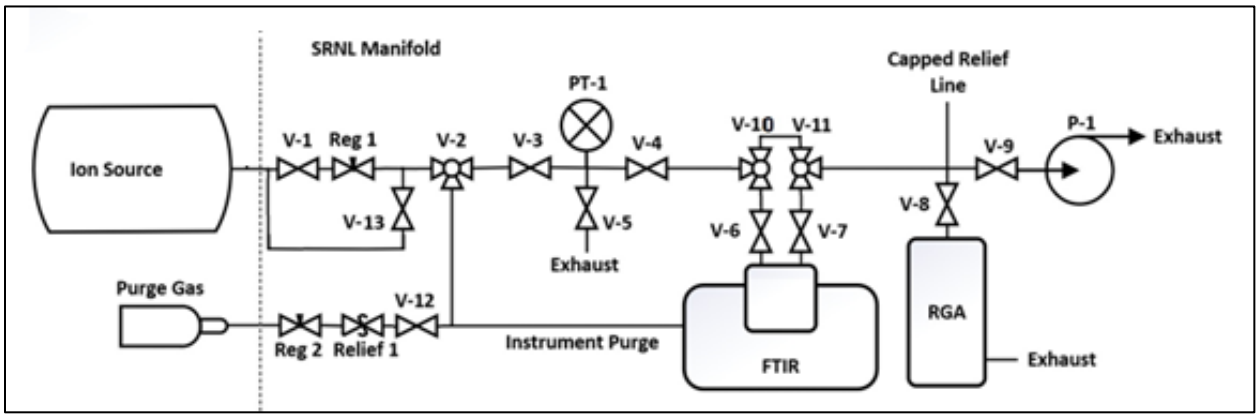

Figure 3-1. Manifold diagram for FTIR and RGA measurements on the insulating gas used in the SHINE ion generator pressure vessel.

The SF_6 flows from the pressure vessel containing the ion source to a regulator, where the pressure is stepped down to atmospheric pressure. The gas is then routed for measurements within a long-pass gas cell in the sample compartment of a Fourier-Transform Infrared (FTIR) spectrometer. The gas cell is filled to 760 Torr as measured by a pressure transducer (Paroscientific Series 2000) and then an FTIR measurement is taken. The gas cell is then pumped down to 400 Torr, where FTIR measurements are made a second time. The SF_6 is sampled by the residual gas analyzer (RGA) by flowing the gas through the gas cell at 760 Torr. A measurement round consisted of an argon purge monitored by the RGA to determine when the manifold was clear of the insulating gas from the previous round, the measurement of the insulating gas at 760 Torr by FTIR and RGA, the measurement of the insulating gas at 400 Torr by the FTIR, and lastly an evacuation and backfill of the manifold using the pump and argon gas. Measurements were made roughly every 1.5 to 2 hours across three days for a total of 14 rounds of measurements. The 14^{th} round of measurements were made after 21 hours and 43 minutes of ion beam time.

The FTIR is a JASCO FT/IR 6800 infrared spectrometer equipped with a long-path gas cell (Pike Technologies, 2.4 meter pathlength). Wavenumbers 500 to 4000 cm^{-1} were recorded at 4 cm^{-1} resolution. FTIR results were collected in single beam mode and transformed to absorbance spectra by referencing an argon background measurement made between each measurement run. The RGA is an MKS Cirrus 3, scanning from *m/z* 1 to 150 units with 1.0 mA emission current and 40 eV electron energy. The gas sampling from the manifold to the RGA is performed via silica capillary transfer line.

4.0 Results and Discussion

4.1 Mass Spectrometry Results

The mass spectrum of the insulating gas at 760 Torr after 21.6 hours of beam time is shown in Figure 4-1.

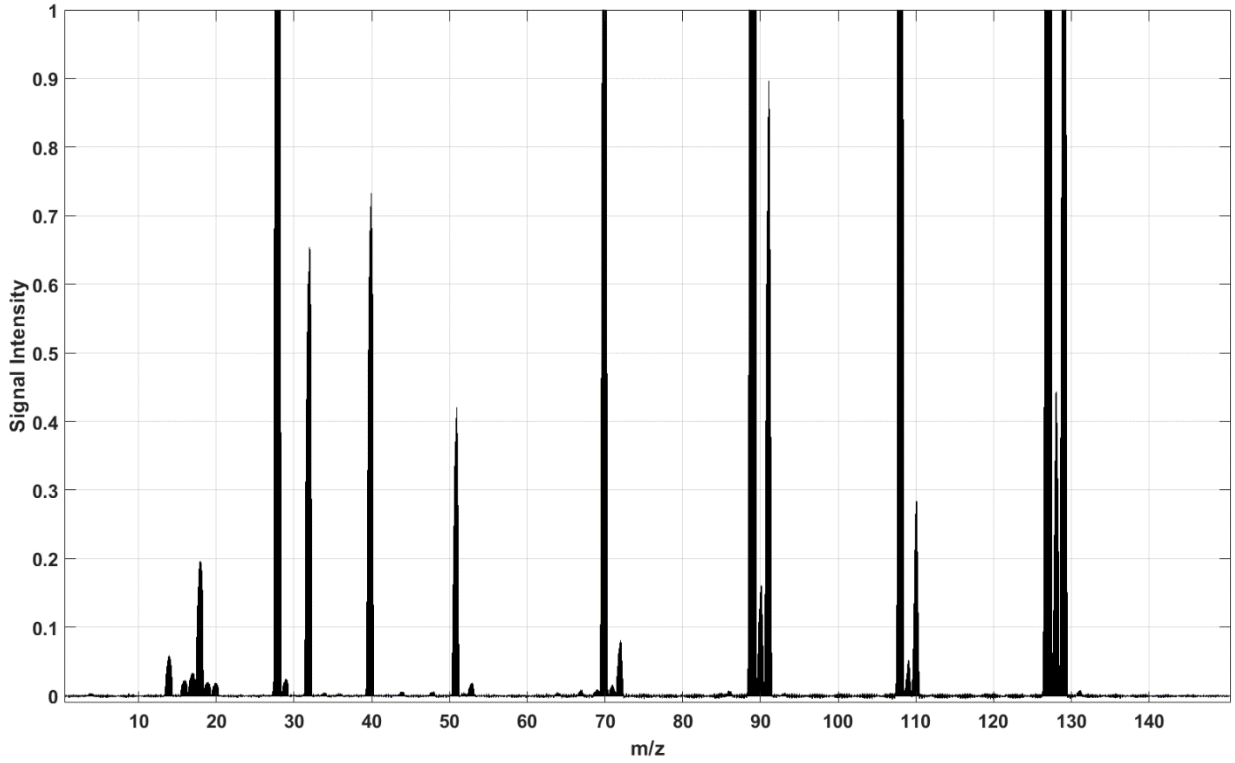

Figure 4-1. The mass spectrum collected at 21.6 hours of beam time. The fragmentation pattern of the SF_6 is present for all fragments, but not the parent ion at *m/z* 146. Peak assignments are detailed in Table 4-1.

The parent ion peak of SF_6 at *m/z* 146 is not present, which is expected. The most predominant peak is at *m/z* 127 for the SF_5^+ ion. Each SF_6 ion fragment shows a signature triplet response due to the natural isotopic abundance of ^{33}S and ^{34}S. Table 4-1 lists each *m/z* where a peak was detected, along with the relative intensity and potential species that the *m/z* indicates. The mass spectrometric measurements are not quantitative, however give an indication of the relative concentrations of the masses detected. The peak intensities of m/z 44, 48, 64, 67, 69, and 86, relative to the peak intensities of m/z 28 and those associated with the SF6 fragmentation (32, 51, 70, 89, 108, and 127) indicate that the major species present in the insulating gas are N_2 and SF_6, with minor concentrations of SO_2, SOF_2, CO_2, and CF_4.

Table 4-1. Assignments of fragments detected in the mass spectrometry data.

Peak Detected (*m/z*)	Assignments
14	N^+
16	O^+
18	Along with *m/z* 17 and 16, indicates water
19	F^+
20	HF or Ar^{2+}
28	N_2 and/or a CO^+ fragment from CO_2
29	Possibly an isotope peak, approximately 1% of carbon is ^{13}C
32	Sulfur ion fragment, along with *m/z* 34 and 36, which are isotope peaks
36	Either argon or sulfur isotope
40	Argon
44	Carbon dioxide
48	SO^+
51	SF^+, *m/z* 53 is the isotope peak for this ion
64	SO_2
67	SOF_2
69	CF_4
70	SF_2^+, *m/z* 71 and 72 are isotope peaks
86	SOF_4 and/or SOF_2
89	SF_3^+, *m/z* 90 and 91 are isotope peaks
108	SF_4^+, *m/z* 109 and 110 are isotope peaks
127	SF_5^+, *m/z* 128, 129, and 131 are isotope peaks

4.2 Infrared Spectroscopy Results

The infrared absorbance spectrum of the insulating gas at 400 Torr after 21.6 hours of beam time is shown in Figure 4-2. The spectra are saturated with SF_6 modes, as expected. Numerous smaller modes are observed, however. In particular, the SO_2 peak at 1386 cm^{-1}, the water vapor band in the 3500-4000 cm^{-1} range. The majority of the bands in the FTIR data appear to be combination bands of the SF_6 peaks at 620 and 960 cm^{-1}.

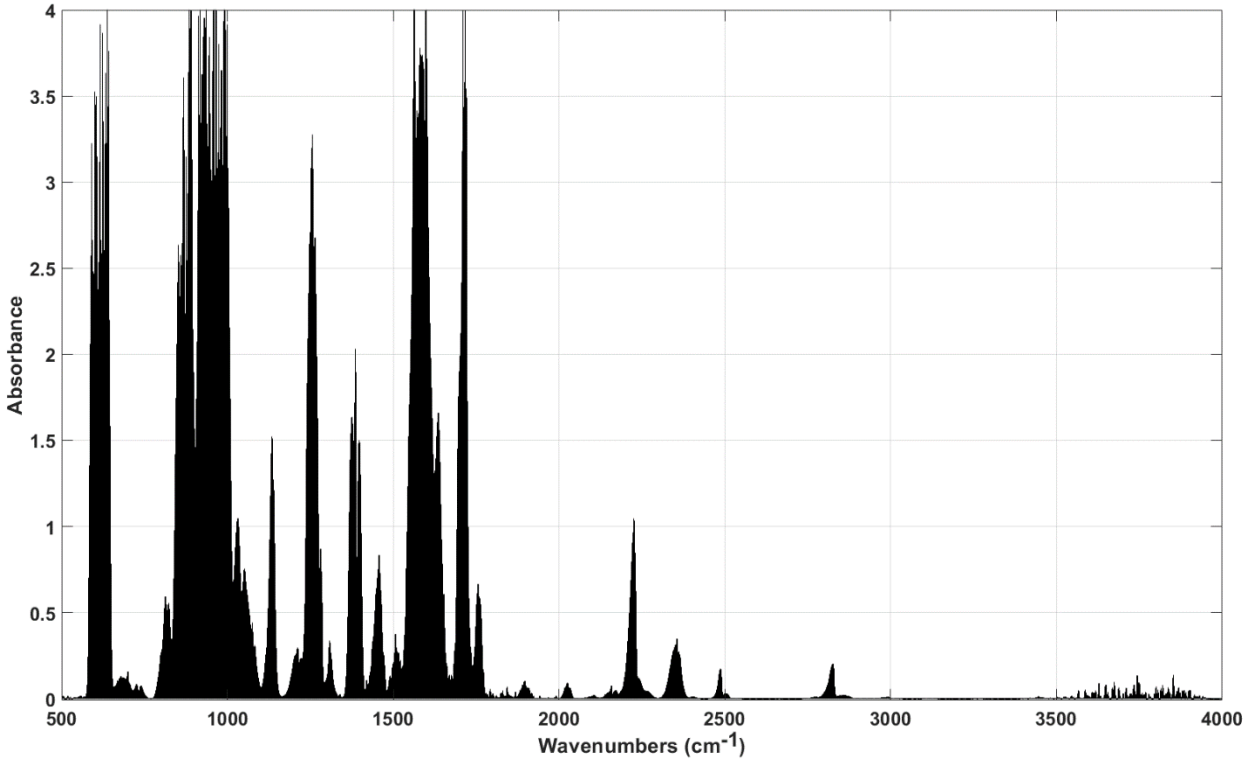

Figure 4-2. FTIR absorbance spectrum of the insulating gas after 21.9 hours of beam time. The SF_6 modes are saturated, as expected.

Figure 4-3 shows the difference in the absorption measurements made at the beginning of the measurement campaign and the end of the measurement campaign. The infrared spectra highlight the increase of water vapor in the insulating gas over time. Notably, the infrared absorption measurements do not show a change in the spectral features over time of the decomposition byproducts. This indicates that these species are either too low in concentration to detect by this method, or that the species were present in the insulating gas prior to the beginning of the measurement campaign.

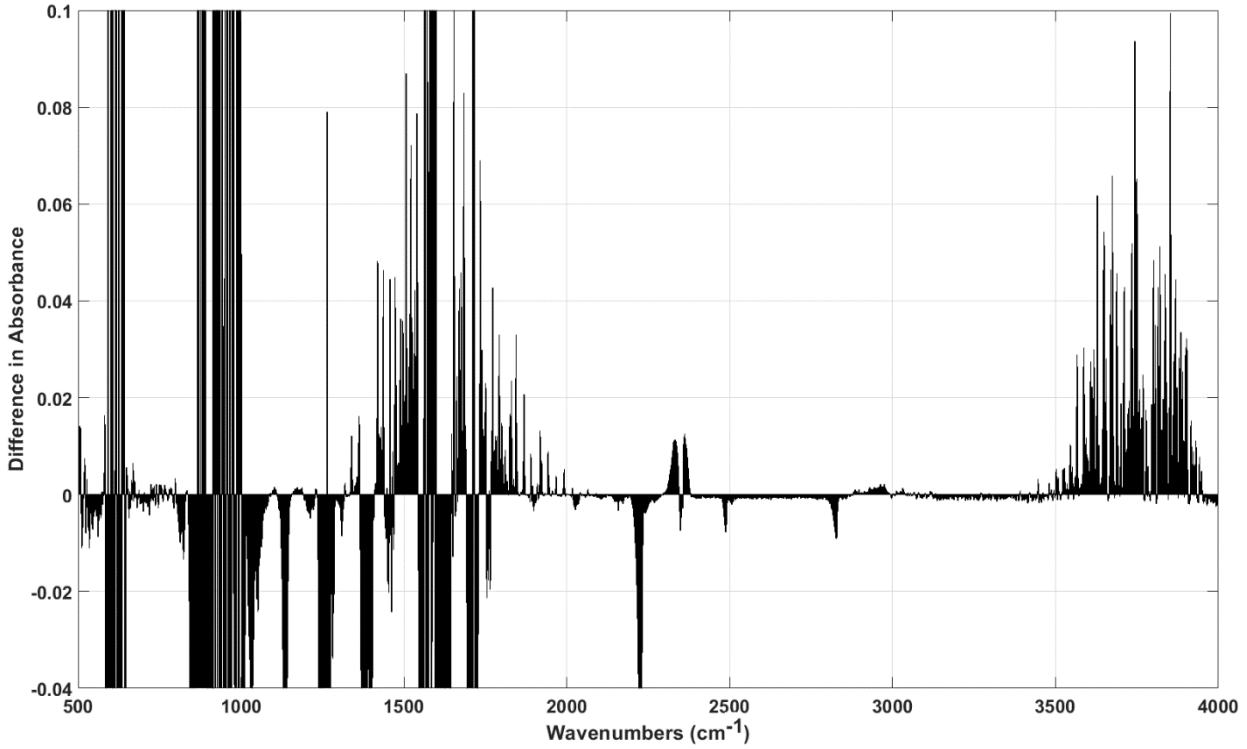

Figure 4-3. The difference in absorbance of the first FTIR measurement of the insulating gas at 400 Torr (prior to any beam time), and the last FTIR measurement of the insulating gas at 400 Torr. Positive peaks indicate an increase in the concentration of absorbing species throughout the measurement campaign. Areas of peak saturation (where the SF_6 absorbance bands are strongest) result in extreme positive and negative values and are artifacts of the noise present in the measurement.

4.3 Insulating Gas Speciation

The species indicated by the FTIR and RGA data are SF_6, SO_2, and atmospheric contaminants such as water, N_2, and CO_2. There are indications of small amounts of SOF_2 and CF_4. Because argon is the purge gas used for the manifold, it is not included as one of the contaminants, although it was detected. Baseline measurements of the insulating gas prior to operation of the beam, and of the manifold in between measurements do not show atmospheric gases present. This indicates that the gases leaked into the insulating gas over the course of the measurement campaign. This is expected. The adsorbent canister inside the pressure vessel is one method to remove these impurities, as well as a purification cart designed to liquefy the SF_6 under pressure, pass it through an activated carbon filter, an activated alumina filter, and a particle filter, respectively[8]. This typically removes the majority of contaminants, however it does not efficiently remove CF_4.

While the peak at *m/z* 19 and 20 together can indicate HF, it is likely that HF is not present. The presence of HF in an environment with access to silicon will generate SiF_4, which was not observed. Additionally, HF was not observed in the FTIR data. It is possible that HF is generated in the SF_6 decomposition process but is short lived. As described in SRNL-STI-2021-00313, isotopic exchange with HF and water are the most likely reservoirs for tritium that contaminates the insulating gas[1]. Since it is not expected that HF is present in an appreciable quantity, it may be expected that upon contamination of the insulating gas, tritium exists as T_2, HT, HTO, and T_2O. The adsorbent canister within the pressure vessel will accumulate the tritiated water. In the event that tritium in the form of HT or T_2 contaminates the insulating gas, it will be separated from the SF_6 during the liquefaction clean-up process of an SF_6 purification cart. The carbon and/or alumina filters of the purification cart will remove tritiated water from the insulating gas. The

elemental tritium may then be separated from the other impurities using conventional hydrogen purification methods, such as a palladium diffuser, commercially used in hydrogen clean-up.

5.0 Conclusions

The results of the measurements reported in this work suggest that the species present in the insulating gas include SF_6, N_2, H_2O, CO_2, SO_2, SOF_2 and CF_4. Tritium contamination in the insulating gas may result in two chemical forms of tritium in the system, tritiated water and T_2. These two forms of tritium would be captured by two methods. Tritiated water may be captured and removed from the SF_6 by an adsorbent canister already present within the pressure vessel that houses the insulating gas while in use. It may also be separated from the SF_6 during routine purification of the SF_6, which is performed by SHINE personnel to routinely. The purification cart will perform all non-embedded systems cleanup necessary to remediate tritiated species in the system. The purification cart passes the insulating gas through several filters, followed by the liquefication of the SF_6. The filters will separate tritiated water from the SF_6 at this stage. It is expected that molecular tritium will accumulate in the gas-phase during the liquification step, which is separated and stored. Tritium may be recovered from this mixture through more conventional hydrogen purification methods such as a palladium diffuser.

6.0 References

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Appendix A. FTIR and RGA Spectra Over Time

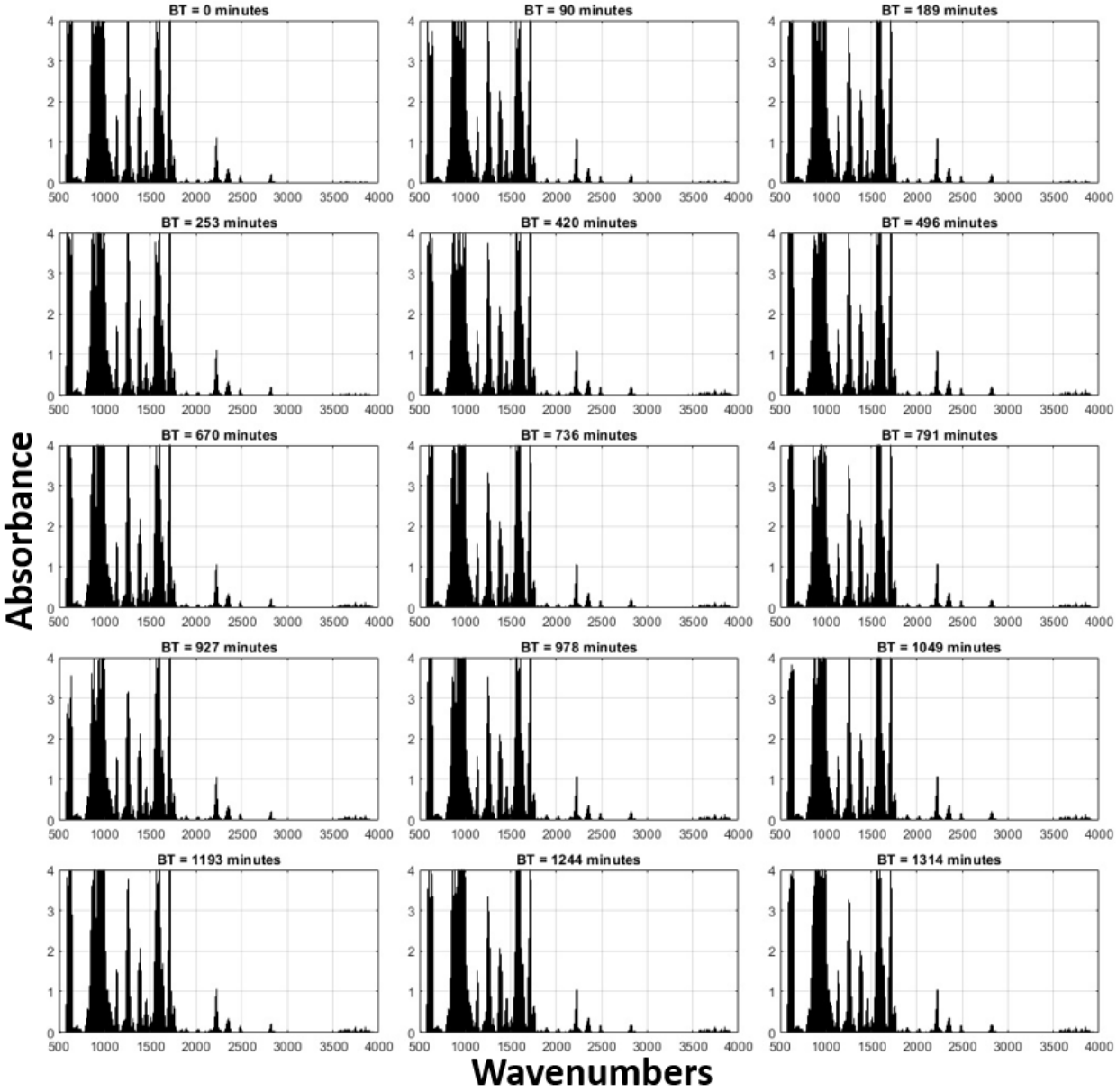

Figure 6-1. FTIR absorbance spectra of the insulating gas at 400 Torr. Each spectrum is labeled according to the beam time (BT) elapsed. The last panel is the spectrum discussed in Figure 4-3.

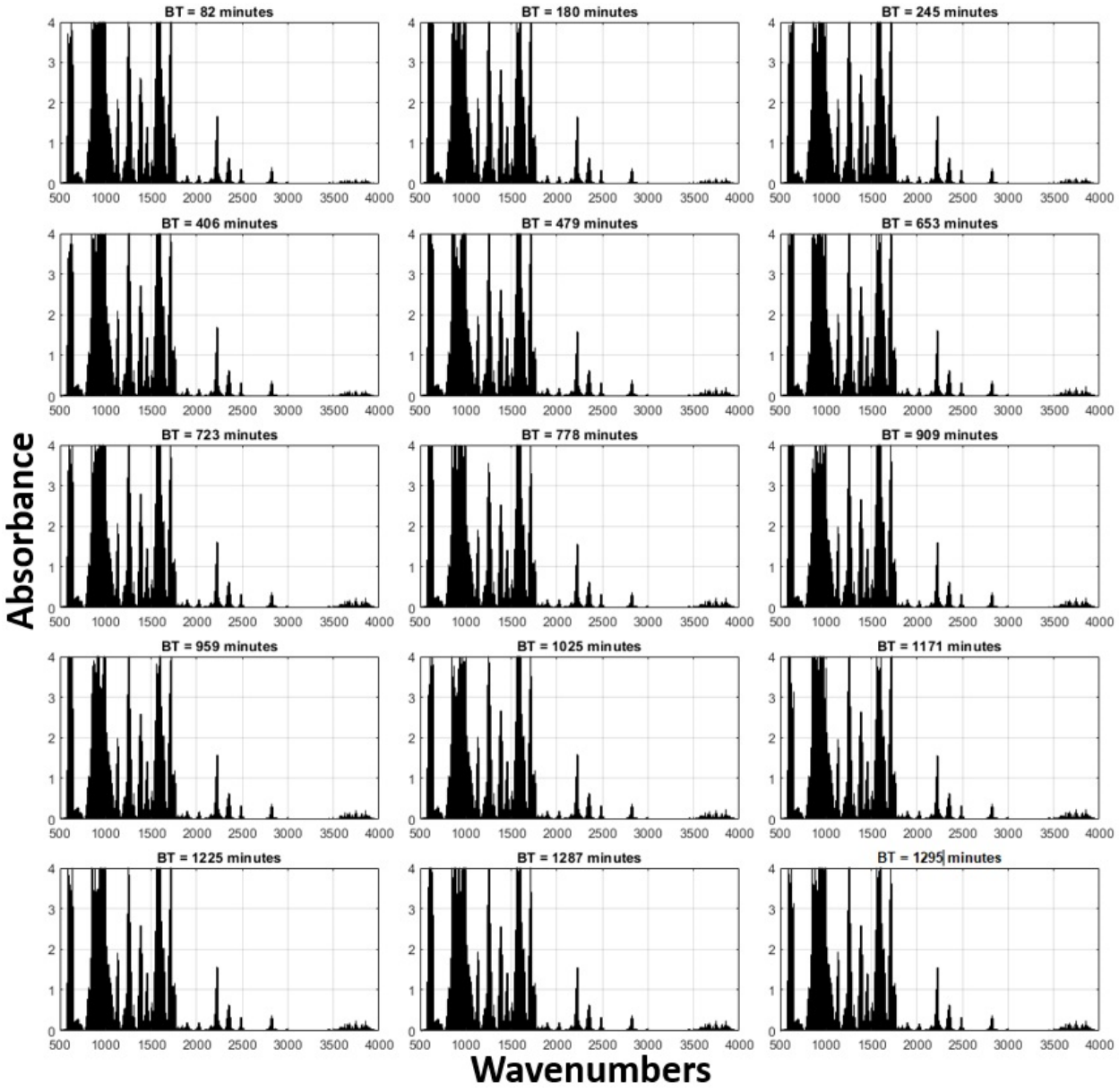

Figure 6-2. FTIR absorbance spectra of the insulating gas at 760 Torr. Each spectrum is labeled according to the beam time (BT) elapsed.

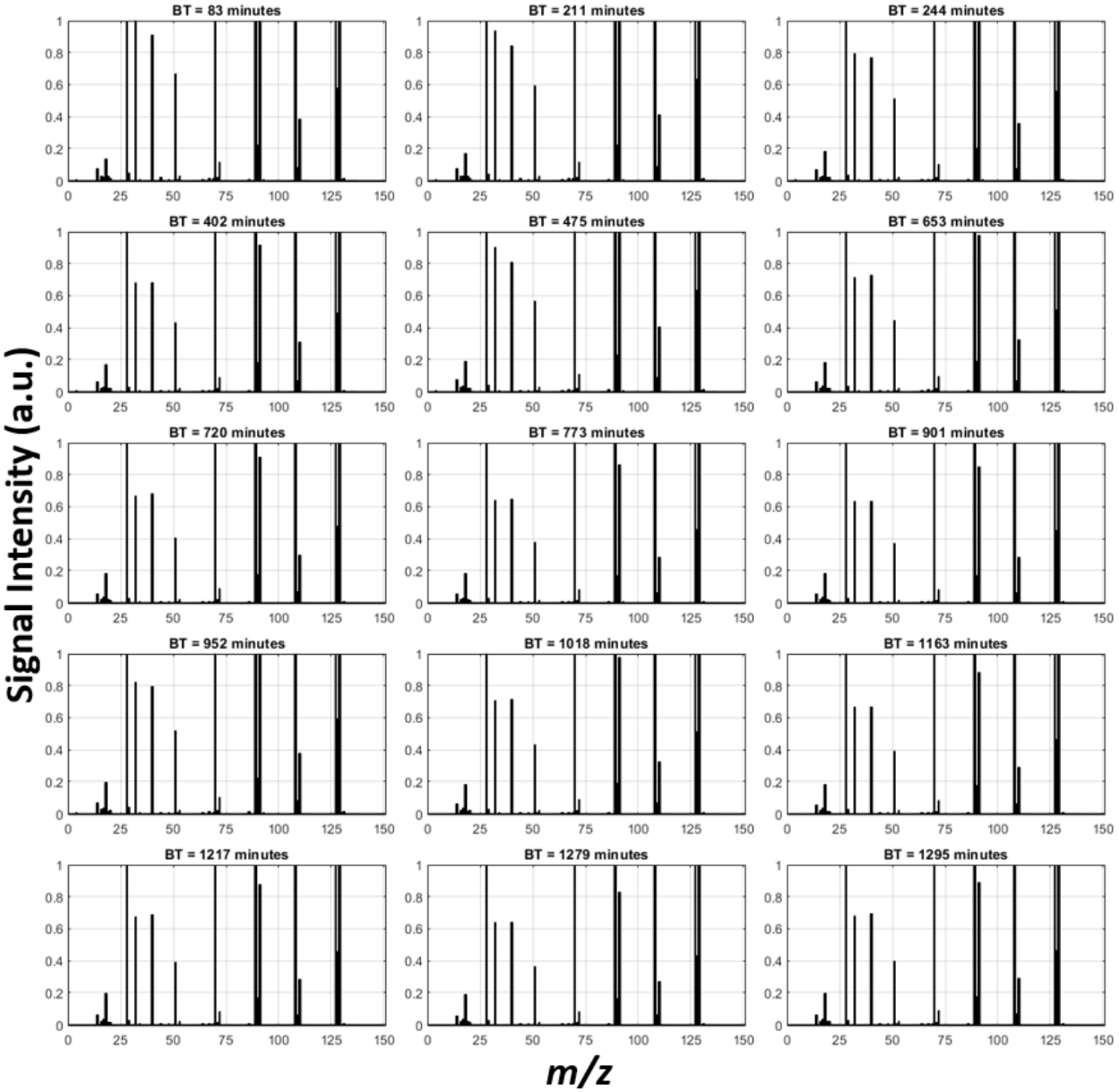

Figure 6-3. Mass spectra of the insulating gas. Each spectrum is labeled according to the beam time (BT) elapsed.

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