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The Heavy Element Volatility Instrument (HEVI)

B. Kadkhodayan, A. Türler, K.E. Gregorich, M.J. Nurmia, D.M. Lee, and D.C. Hoffman

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The Heavy Element Volatility Instrument (HEVI)

B. Kadkhodayan, A. Türler, K. E. Gregorich, M. J. Nurmia,
D. M. Lee, and D. C. Hoffman

Department of Chemistry
University of California
Berkeley, CA 94720

and

Nuclear Science Division
Lawrence Berkeley Laboratory
1 Cyclotron Road
Berkeley, CA 94720

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Abstract

We have constructed the Heavy Element Volatility Instrument (HEVI), an on-line gas chromatography system which is used to continuously separate halides according to their volatility. Both gaseous HBr and HCl have been used as halogenating agents. The detailed design of the apparatus is described and some preliminary results are presented. The experimental results are compared with those from a Monte Carlo code simulation based on a microscopic model for gas thermochromatography in open columns with laminar flow of the carrier gas. There is good agreement between the experimental results and the simulation.

1. INTRODUCTION

Experimental investigation of the chemical properties of the transactinide elements is extremely difficult. The longest lived known isotopes of the transactinide elements have half-lives of a minute or less. These elements have only been produced in heavy ion fusion reactions at rates of a few atoms per minute or less. Furthermore, actinide activities which are produced in high yields in these reactions interfere in the detection of the transactinides, which further complicates chemical studies. Due to the low production rates and short half-lives of these nuclides, very specific and unique chemical procedures have been devised. Some procedures are designed to operate continuously, whereas others allow fast reproducible separations with high repetition rates in order to obtain statistically significant results. On-line isothermal gas chromatography is one of the most unique chemical procedures designed to investigate the properties of these elements. This method takes advantage of the high volatilities of the halides of the group 4, 5 and 6 transition elements to efficiently separate transactinide halides and their lighter homologs from the less volatile trivalent actinides. This chemical separation allows the investigation of nuclear and chemical properties of the transactinide elements.

The first on-line gas chromatography experiments with transactinide elements were performed in 1966 by Zvara et. al. [1-5]. Taking advantage of the fact that bromides and chlorides of the group 4 and 5 elements, and possibly Rf and Ha, form rather volatile species, they performed gas-solid thermochromatography. Based on the deposition temperature in a quartz chromatography column, Rf and Ha halides were separated from the actinide activities and were detected by counting fission tracks in the quartz column itself. However, merely recording the position of fission tracks in a thermochromatographic column is insufficient for identifying the atomic number (Z) or mass number (A) of a fissioning species since all information concerning the Z, A and the half-life of the fissioning nucleus is lost.

In 1986 Brüchle et. al. [6] developed the On-Line Gas chemistry Apparatus (OLGA), which was used to search for volatile superheavy elements. OLGA was capable of separating volatile elements in their oxide or hydroxide forms. This system had the advantage of performing alpha spectroscopy while measuring the half-lives, making it possible to identify the decaying nuclide. Soon after, Gäggeler et. al. developed OLGA II [7], which was especially designed for the separation of volatile halide species of short-lived nuclides. This system was also equipped with an improved detection system and was used in several studies of the halides of hafnium, niobium, tantalum, protactinium, rutherfordium and hahnium [8,9,10].

Motivated by this pioneering research, we have constructed an improved system called the Heavy Element Volatility Instrument (HEVI). HEVI is an on-line gas chromatography system which continuously separates halide species of short-lived nuclides according to their volatility. Major improvements in the system include a longer chromatography column with superior temperature profiles which should result in more distinct separations. Addition of a controlled gas flow system prevents salt buildup inside the column from the He/KCl aerosol gas transport system and allows continuous use of the system without periodic replacement of the column. The new system was designed to prevent or reduce corrosion. Both gaseous HBr and HCl have been used as halogenating agents in our experiments. The experimental results will be published at a later time. The current paper concentrates on the description of the design of HEVI.

2. OPERATION PRINCIPLE

Activity is transported from the target chamber to HEVI with a He/KCl aerosol gas transport system. The KCl aerosols are generated by sublimation of crystalline KCl at approximately 650 ^{o}C inside a quartz tube. Helium gas is passed over the KCl at the rate of 2 liters per minute to sweep the aerosols into a presorter capillary, where the larger aerosol particles are allowed to settle out of the He flow. The presorted aerosols are then transported directly into the target chamber through a 4.8 mm i.d. Teflon capillary. Products of nuclear reactions between an ion beam from the LBL 88-Inch Cyclotron and a fixed target, which recoil out of the target, are stopped in the helium which sweeps out the volume behind the target continuously, and are collected on the aerosols. The activity-laden aerosols are then rapidly transported by the helium through a 1.6 mm i.d. Teflon capillary tube to the chromatography system.

The activity-laden aerosols enter the first section of the quartz column and are stopped on a quartz wool plug placed at the entrance to the chromatography section of the column. This first section of the quartz column is kept at approximately 900 ^{o}C to vaporize the aerosols leaving the activity on the quartz wool. Reactive gases are added at this point to form halide compounds. The volatile species are then carried down a cooler, isothermal section of the column by the helium gas flow. The retention time of a molecule in the isothermal section of the column is related to the number of adsorption/desorption steps between the molecule and the column surface and also the period of time the molecule spends in the adsorbed state. The retention time is then dependent on the adsorption enthalpy, the column temperature, the true volume flow rate of the carrier gas and the column length. If the retention time is relatively long in

comparison to the half-life of a species, it will remain on the surface of the quartz column and decay. However, if the species has a relatively short retention time compared with its half life, it will leave the column and enter the recluster chamber where it is attached to new aerosol particles and transported to a detection system.

Two detection systems are used to detect either alpha/spontaneous fission (SF) activities or gamma ray activities. The horizontal rotating wheel system [11], the MG, can be used to detect alpha/sf activities and a Ge detector used in conjunction with a special collection site is used to detect gamma or X-ray activities. Both systems are described in detail in subsequent sections of this paper.

3. DESIGN DESCRIPTION

HEVI consists of several constituents (Fig.1): (i) the split shell furnaces (Fig.2), and controllers, which heat the various sections of the quartz column; (ii) the chromatography column assembly consisting of the Inconel jacket (Fig. 3), the quartz chromatography column and the graphite/ceramic pieces; (iii) the heat sink, which provides cooling between the high temperature section of the system and the cooler isothermal section; (iv) the recluster components (Fig.4), which allow the transport of the separated volatile species to our detection system; (v) the gas flow system consisting of the mass flow controllers and gas switches (Fig.5), which control the continuous flow of gases to the system; and (vi) the detection systems.

3.1. THE SPLIT SHELL FURNACES AND CONTROLLERS

The system is heated by four split shell furnaces (Teco F-6-1000-H-1.5-1V-SSL) mounted end to end on a 15 x 94 cm rotating table, (Fig. 1). The furnaces are 16.51 cm in length with a 12.8 cm o.d., (Fig. 2). The heated length in each furnace is 15.2 cm. The furnaces are designed for operation up to a maximum temperature of 1000 ^{o}C. They are provided with a 2.54 cm i.d. vestibule to allow clearance for the Inconel jacket. The control device for each furnace (Teco/Sigma MDC4E) is capable of accurately controlling the rate of ascent or descent to appropriate temperature settings.

3.2. THE CHROMATOGRAPHY COLUMN ASSEMBLY

Inconel alloy 600 is a good material for use in severely corrosive environments at elevated temperatures. It is resistant to oxidation at temperatures up to 1180 ^{o}C. The alloy has excellent mechanical properties at cryogenic as well as elevated temperatures. Because of its resistance to chloride-ion stress-corrosion cracking and corrosion by high purity water, it is also used in nuclear reactors. The Inconel jacket is a tube approximately 78 cm long with a 2.54 cm o.d. and a 2.21 cm i.d. (Fig.3). Stainless steel flanges are welded on the ends of the Inconel jacket and copper water cooling coils have been soldered to both ends of the tube. Cooling of the flanges is essential for vacuum tight connections with adjacent system constituents.

A tube adapter has been connected to the upstream end of the Inconel jacket to increase its diameter from 2.5 cm to 7.5 cm, in order to allow clearance to insert and seal the 8 mm o.d. quartz chromatography column. The length of the 6 mm i.d. quartz column is 82.2 cm. At the downstream end, there is a 5.1 cm tip of 1 mm i.d.. A 3 mm i.d. indentation is placed 28.2 cm from the beginning of the column (Fig. 3) to hold a small amount of quartz wool. The quartz wool acts as a filter for the activity laden KCl aerosols which are transported from the target chamber via the He gas jet as explained above. This section of the quartz column is kept at approximately 900 ^{o}C by the first furnace, and the remaining 59.1 cm length of the column is kept isothermal at lower temperatures, ranging from 50 ^{o}C to 650 ^{o}C by the other three furnaces. The activity laden aerosols are transported directly to the quartz wool by a 27 cm long x 2.0 mm i.d. aluminium oxide tube, on the quartz column. The aerosols are collected on the quartz wool, and are vaporized at 900 ^{o}C, leaving the activity inside the quartz wool. Brominating or chlorinating agents, such as HBr or HCl gases, are added at this point at a rate of 100 ml/min. Volatile halide species which are formed will then flow down the isothermal length of the column at the appropriate isothermal temperature and enter the recluster chamber. Nonvolatile species decay inside the quartz column.

To keep uniform isothermal temperature profiles in our quartz chromatography column, a series of graphite and ceramic tubes, with a 2.10 cm o.d. and a 9.0 mm i.d., are carefully arranged inside the Inconel jacket (Fig. 3). High density graphite is used for its excellent heat conductivity properties and ceramic is used for its good heat insulation properties. A 7.1 cm ceramic piece insulates the 900 ^{o}C section from the upstream end of the Inconel jacket. This piece is followed by a 7.6 cm graphite piece which assures a uniform temperature at the quartz wool position. Next is a 5.1 cm ceramic piece which insulates the isothermal section from the 900 ^{o}C section. Finally a 52.7 cm graphite tube

surrounds the isothermal section of the quartz tube insuring a uniform temperature. The end of this tube is reduced to a 1.55 cm o.d. where a 2.54 cm length and a 3.2 mm ceramic washer is placed between the graphite and the Inconel wall, to avoid contact between the graphite and the water cooled Inconel tube. This reduces cooling of the graphite (and the column) and ensures uniform temperature profiles at the tip of the column. A 100 ml/min flow of nitrogen gas is maintained inside the Inconel jacket and around the graphite/ceramic arrangement. This assures a slight positive pressure inside the Inconel jacket, preventing the back flow of corrosive gases inside the jacket.

3.3. THE HEAT SINK

To further minimize the transfer of heat from the 900 ^{o}C section to the adjacent isothermal section a removable heat sink was constructed. The heat sink is a water cooled 6.5 x 6.0 x 1.0 cm copper block. It is used around the Inconel jacket between the 900 ^{o}C furnace and the isothermal section for all temperature settings below 250 ^{o}C and is removed at higher temperatures. The control of the water flow through the heat sink regulates the heat transfer from the 900 ^{o}C section to the isothermal section. The water flow through the system is controlled by a water flowmeter (RMA Rate-Master, Dwyer Instruments, Inc.). The meter controls flow from 2 gal/hr to 24 gal/hr. Appropriate water flow settings have been determined for each isothermal temperature.

3.4. THE RECLUSTER COMPONENTS

The volatile species, after leaving the chromatography column, enter the recluster chamber where they are attached to KCl aerosols in nitrogen to be transported through a capillary to the detection system. The recluster unit consists of the recluster connecting piece, the recluster gas distributor, the recluster chamber and the recluster end piece (Fig. 4).

The KCl aerosols for the N_2/KCl gas jet system are generated by sublimation of crystalline KCl at approximately 650 ^{o}C inside a quartz tube. Nitrogen gas, at the rate of 1.5 liters per minute, is used to sweep the aerosols out of the tube and into a 'presorter' capillary, where the larger aerosol particles are allowed to settle out. The presorted aerosols are transported directly into the recluster gas distributor via a 4.8 mm i.d. polypropylene tube. The recluster gas distributor is a 9.0 cm long stainless steel cylindrical piece with a 6.5 cm o.d.. The recluster gas exits the distributor through four 2.4 mm i.d. polypropylene capillaries and is introduced to the recluster unit through four

inlets on the recluster connecting piece. The stainless steel recluster connecting piece, illustrated in Fig. 4, connects the Inconel jacket to the recluster unit. The recluster chamber is a 30.5 cm long cylindrical piece of 2.5 mm thick Pyrex glass with a 6.7 cm i.d.. The species entering the recluster chamber through the tip of the quartz chromatography column are collected on the aerosols in nitrogen. The activity laden aerosols are then swept out of the chamber into a 2.4 mm i.d. polypropylene capillary from the center of the recluster end piece and transported to the detection system. The recluster unit operates at negative 2 pounds per square inch of pressure.

3.5. THE GAS FLOW SYSTEM

During an experiment two types of measurements are performed. The activity laden aerosols which exit the target system are either injected into the chromatography system, where chromatography is performed before transport to the detection system, or the chromatography system is bypassed and the activities are sent directly to the detection system where a yield check is performed. A yield check is simply a measurement of the amount of activity from specific nuclides under study, without chemical separation. A comparison of the activities from a yield check measurement and a chemical separation measurement gives the relative yield of a species. In order to perform the above two measurements, it is crucial to maintain the uninterrupted flow of gases in the system in order to maintain the temperature profiles in the quartz column and to maintain a constant gas-jet efficiency.

The controlled flow of gases in HEVI is obtained through several mass flow controllers and two direction control switches. Fig. 5 shows a schematic of the controlled flow of gas through the entire system. The two direction control switches are made out of Lucite with dimensions of 9.0 x 9.0 x 3.5 cm. The first direction control switch enables the aerosols to bypass the chromatography oven to perform a yield check measurement. At the same time, pure helium is directed into the chromatography column at a rate of 2 l/min, to maintain the temperature profiles. The second switch enables the aerosols to either enter the detection system or bypass the detection system and proceed directly to waste. All corrosive gases are neutralized by a scrubber system as shown in Fig. 5.

3.6. DETECTION SYSTEMS

Two detection systems have been used to detect either alpha/SF activities or gamma ray activities. To detect alpha/SF activities, our horizontal rotating wheel system [11], the MG, can be used. The MG is located approximately 10 m away from the gas system. The 25.4 cm radius horizontal wheel of the MG has 80 equally spaced collection positions about its circumference. A steel ring with a 0.63-cm i.d. hole, which is covered with a 40+15 $\mu g/cm^2$ film of polypropylene, is placed in each collection position. Activity laden aerosols are deposited onto the polypropylene film. The wheel is stepped at the appropriate time intervals so as to move the foils consecutively from the collection site into position between pairs of passivated ion-implanted planar silicon detectors (PIPS). Because of the corrosive nature of the reactive gases used, PIPS detectors are required for this experiment since they are chemically inert. Six pairs of detectors are used to measure the kinetic energies of fission fragments and alpha particles. The efficiency for detection of alpha particles in a given detector is 30%. Since each detection station consists of two detectors, one above and the other below the wheel, the efficiency for the detection of alpha particles is about 60% and that for coincident fission fragments is about 30%.

For detection of species with characteristic gamma or X-ray activities, an intrinsic Ge detector is used. The activity laden KCl aerosols are collected on a 2.54 cm diameter glass fiber filter inside a Lucite collection site in front of a Ge detector (Fig. 6). The glass fiber filter is supported by a glass frit. The glass fiber filter is then surveyed for gamma and X-ray activity. After each experiment the glass fiber filter is replaced.

4. TEMPERATURE PROFILES

Fig. 7 illustrates temperature profiles inside the quartz chromatography column for each of the thirteen isothermal temperature settings from 50 ^{o}C to 650 ^{o}C in 50 ^{o}C increments. To obtain these profiles, a thermocouple was inserted inside the column and pulled back in 1 cm increments from the column tip to the quartz wool plug and the temperature at each point was recorded. The flow of gases through the system was regulated to produce exact experimental gas flow conditions. As previously mentioned, the heat sink was removed at temperatures above 250 ^{o}C. This is the cause of the sudden increase in temperature observed in Fig. 7 at the region from 5 cm through 15 cm between the 250 ^{o}C setting and all higher temperature settings. The length of the isothermal section part in the chromatography column is about 15 cm at 50 ^{o}C settings,

approximately 40 cm at 100 °C through 250 °C settings, and approximately 30 cm at 300 °C and all isothermal temperatures above. The term 'isothermal' implies T±5 °C. The greater lengths of HEVI's isothermal sections are a major improvement over OLGA II [7], whose isothermal lengths are 20 cm at 700 °C, 18 cm at 600 °C, 16 cm at 400 °C and 500 °C, 15 cm at 300 °C, 11 cm at 200 °C and only 6 cm at 100 °C. This improvement should lead to more distinct separations.

5. PRELIMINARY RESULTS

Preliminary experiments were performed with ^{167}Hf which was produced at the LBL 88-Inch Cyclotron by the ^{Nat}Sm(^{20}Ne,xn) reaction. The beam intensity was typically on the order of 20 pnA. The 315.2 KeV gamma ray emitted in the decay of this isotope was used to calculate its relative yield at different temperature settings. As previously explained, relative yields are calculated by the comparison of a yield check experiment and a chromatography experiment. HBr gas was used as brominating agent. The results from this experiment are illustrated in Fig. 8. For comparison a plot of similar experiments performed with OLGA II using ^{167}Ta has been included. HBr gas along with BBr_3 was used as brominating agents in the OLGA II experiments. The rise in relative yield from a minimum to a maximum, in the HEVI experiment, occurs over temperature range of 50 °C. The same rise in relative yield occurs over a 150° temperature range in OLGA II. The reason for this difference is the much longer isothermal lengths in HEVI.

A Monte Carlo simulation of the yield curves has been developed based on the model proposed by Zvara [12]. In this microscopic model the downstream migration of a sample molecule is described as a rather small number of some effective random displacements, and sequences of adsorption/desorption events. As illustrated in Fig. 8, this model agrees quite well with the results obtained.

6. ACKNOWLEDGEMENTS

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FIGURE CAPTIONS

1. Illustration of the Heavy Element Volatility Instrument (HEVI). Shown here is the side view of the chromatography and recluster segments.
2. Split shell furnace (Teco F-6-1000-H-1.5-1V-SSL) designed for operation up to a maximum temperature of 1000 ^{o}C.
3. Horizontal cross-section view of the graphite/ceramic arrangement inside the Inconel jacket. High density graphite is used for its excellent heat conductivity properties and ceramic is used for its good heat insulation properties. This arrangement insures uniform isothermal temperature profiles in the quartz chromatography column.
4. Illustration of the recluster components. The recluster unit consists of the recluster connecting piece, which connects the Inconel jacket to the recluster unit, the recluster gas distributor, which distributes the recluster gas equally to the four inlets on the connecting piece, the recluster chamber and the recluster end piece.
5. Illustration of the gas flow schematic of the system. The flow of gases in the system is controlled through several mass flow controllers and two direction control switches.
6. Illustration of the Lucite collection site and detector arrangement used for detection of gamma and X-ray activities. The activity laden aerosols are collected on the glass fiber filter 4.5 cm from the Ge detector. The filter paper is replaced after each experiment.
7. Illustration of temperature profiles inside the quartz chromatography column for each of the thirteen isothermal temperature settings from 50 ^{o}C to 650 ^{o}C in 50 ^{o}C steps. The sudden increase in temperature observed at the region from 5 cm through 15 cm between the 250 ^{o}C settings and all higher temperature settings is due to the removal of the heat sink.
8. Comparison of experimental results obtained form HEVI, OLGA II, and calculations from a theoretical Monte Carlo computer program. A better separation is observed for HEVI in comparison to OLGA II due to longer isothermal lengths. The results from the Monte Carlo simulation of the yield curves agrees quite well with experimental findings.

Gas Chemistry Setup

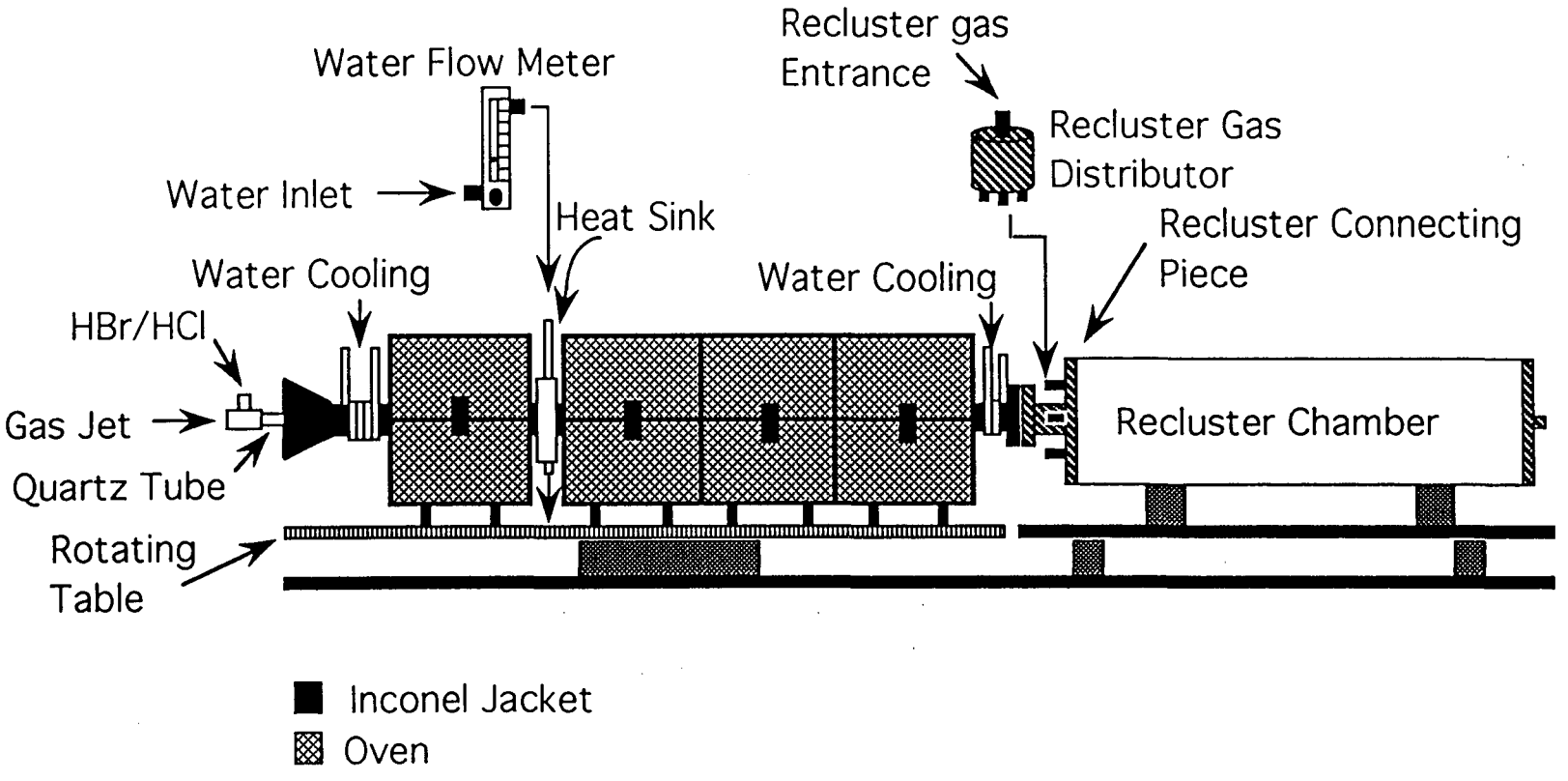

Fig. 1

Split Shell Oven

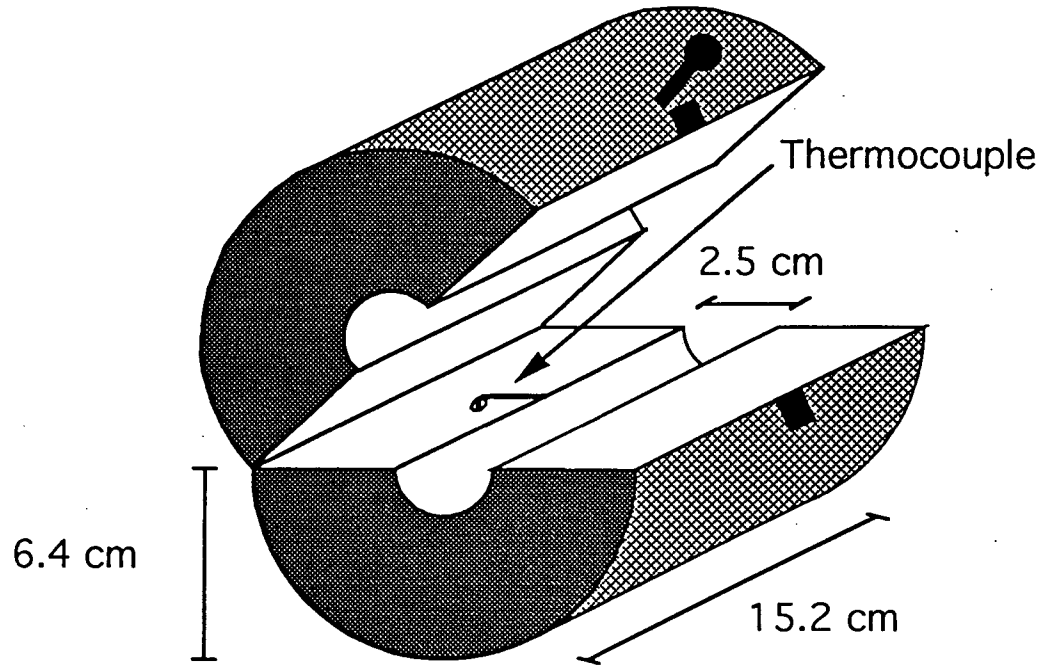

Fig. 2

Graphite/Ceramic Arrangement Inside the Inconel Jacket

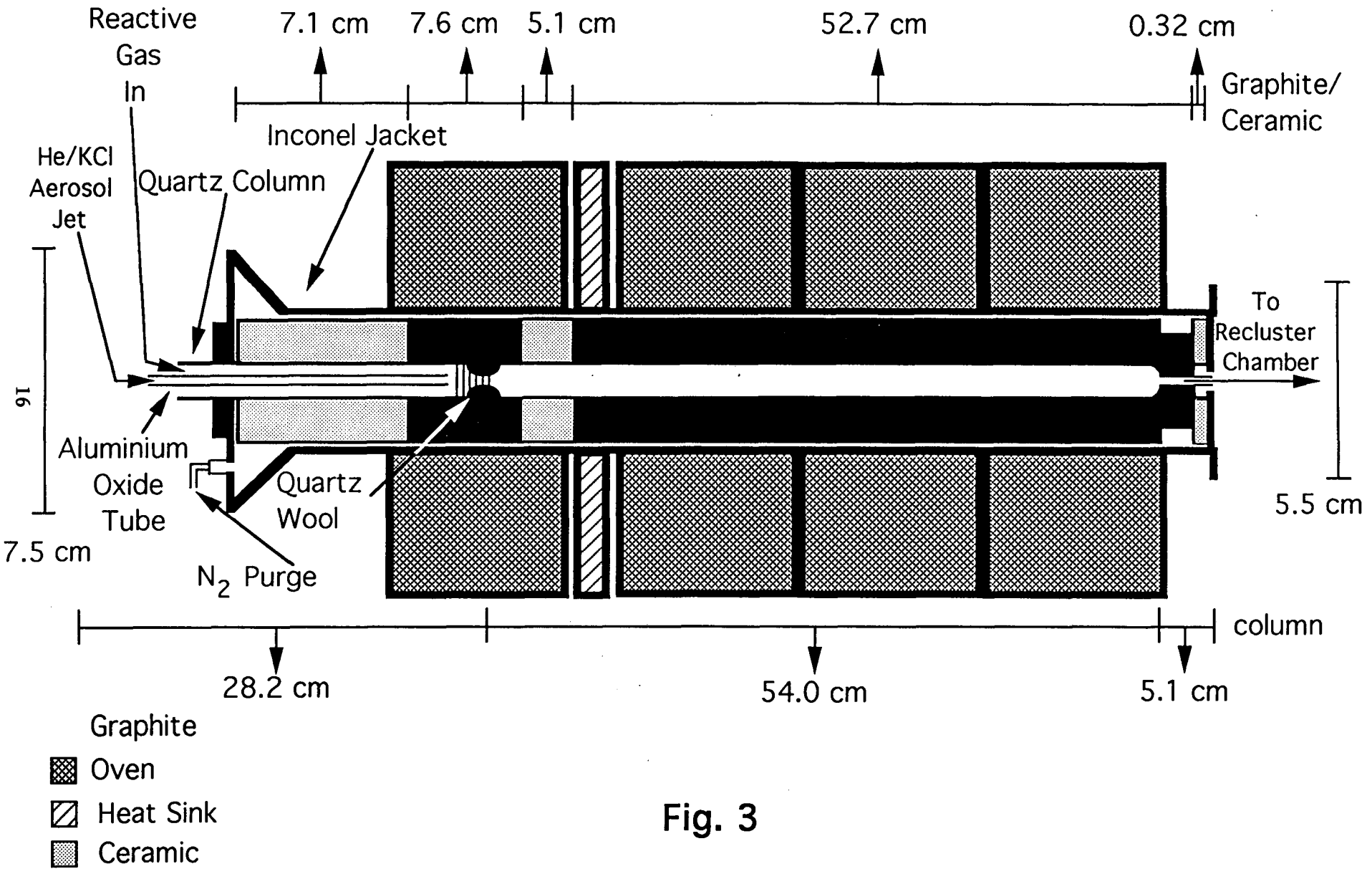

Fig. 3

Recluster Components

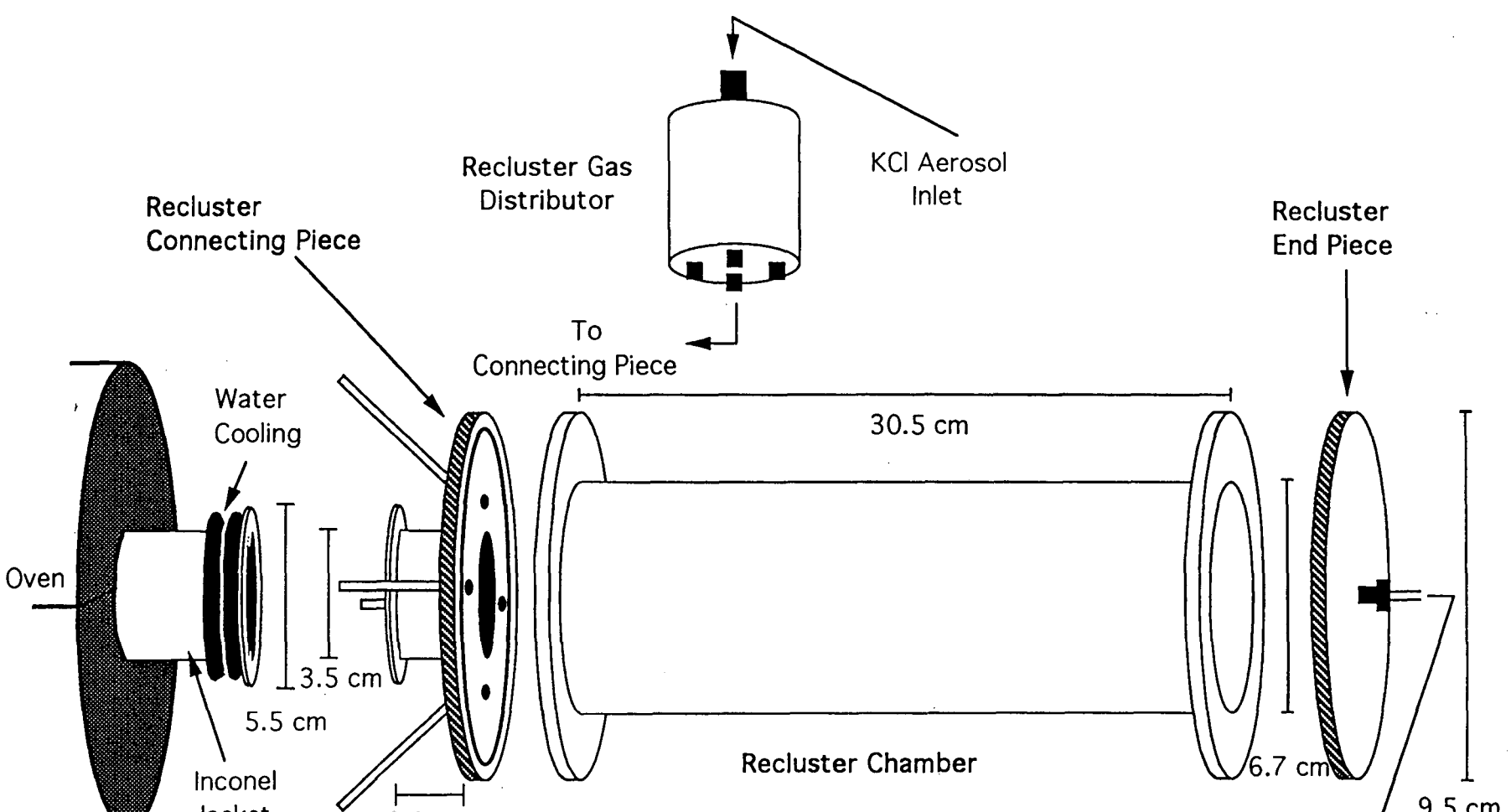

Fig. 4

Gas Flow Schematic

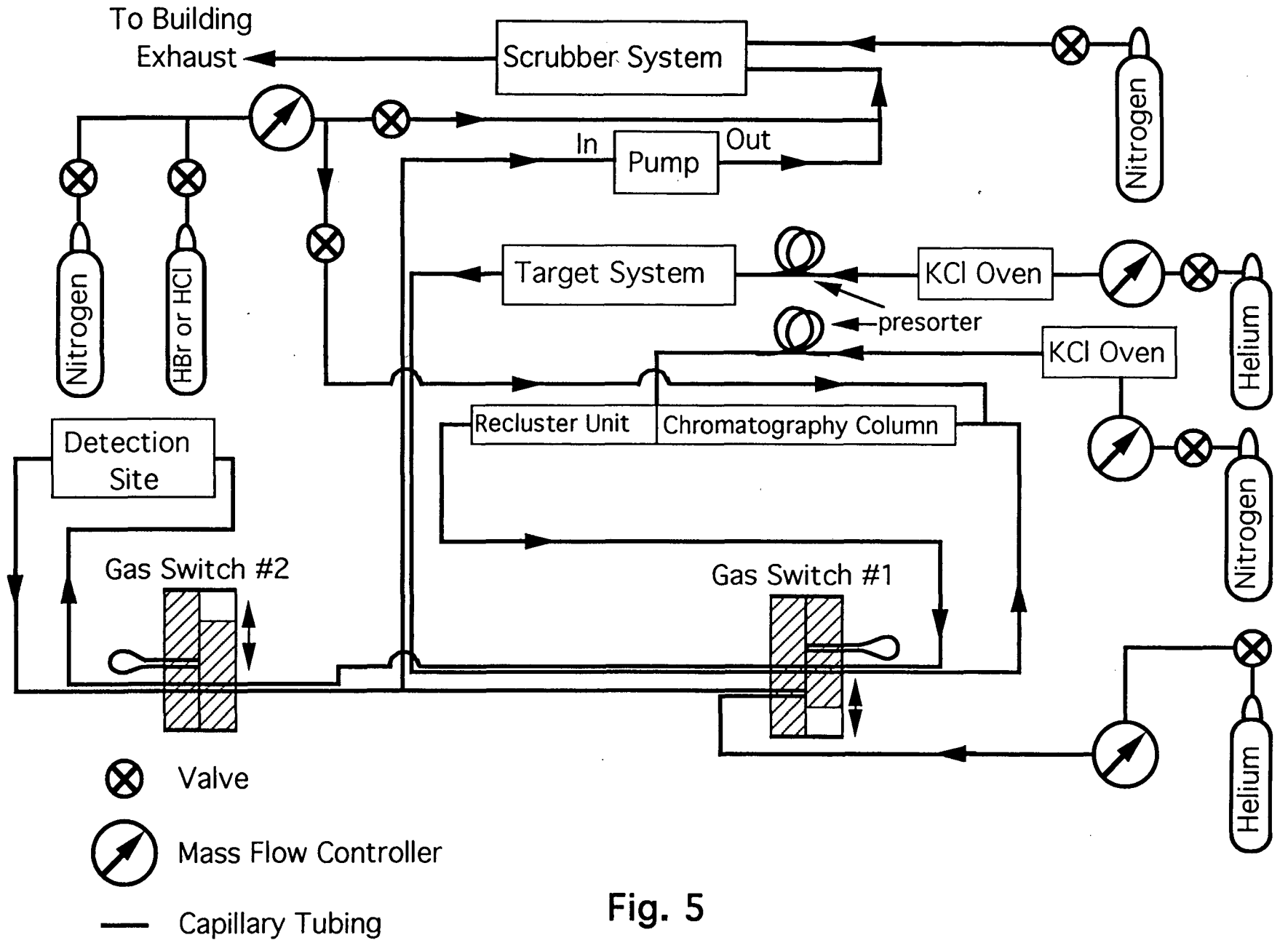

Fig. 5

Lucite Collection Site

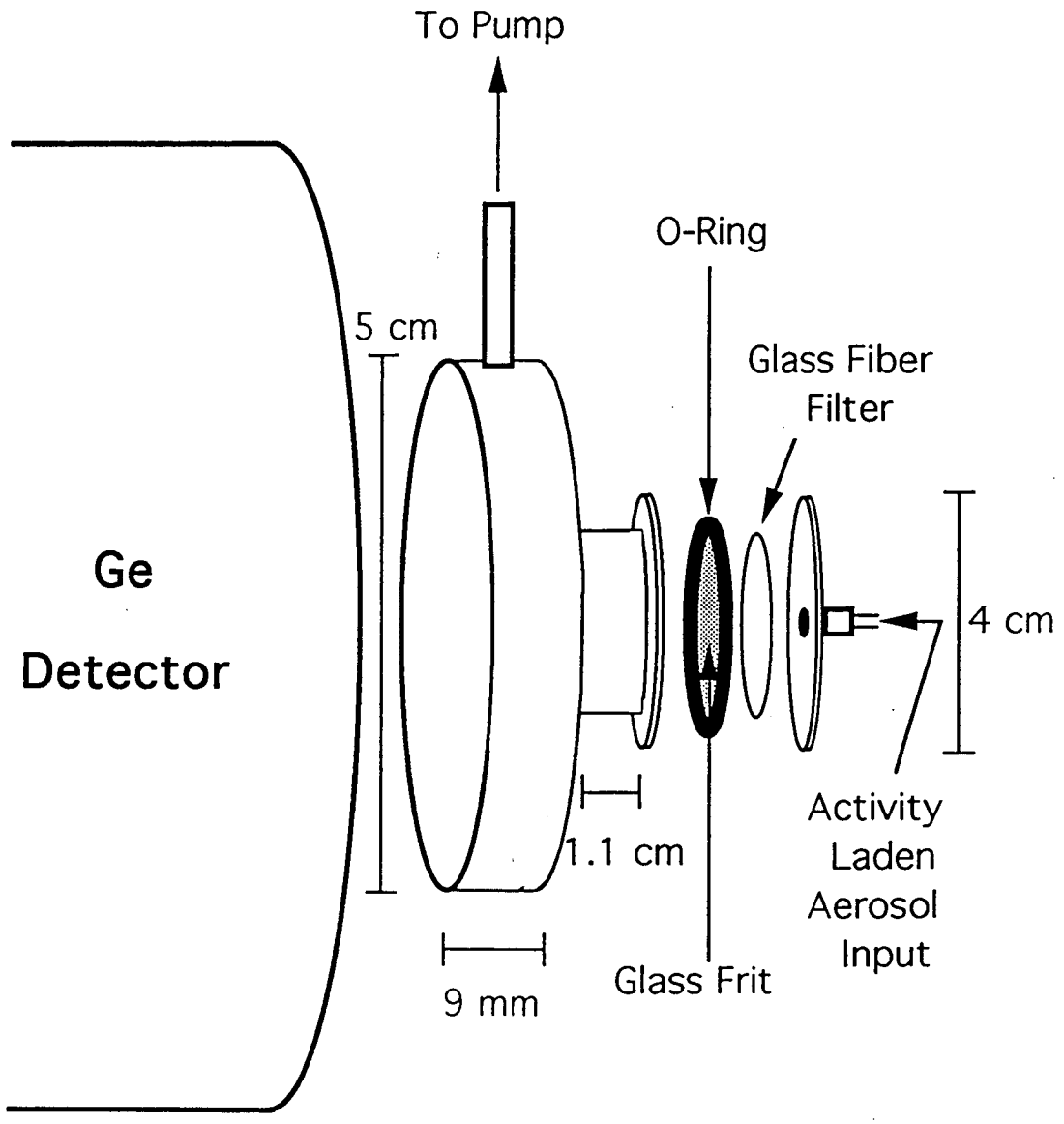

Fig. 6

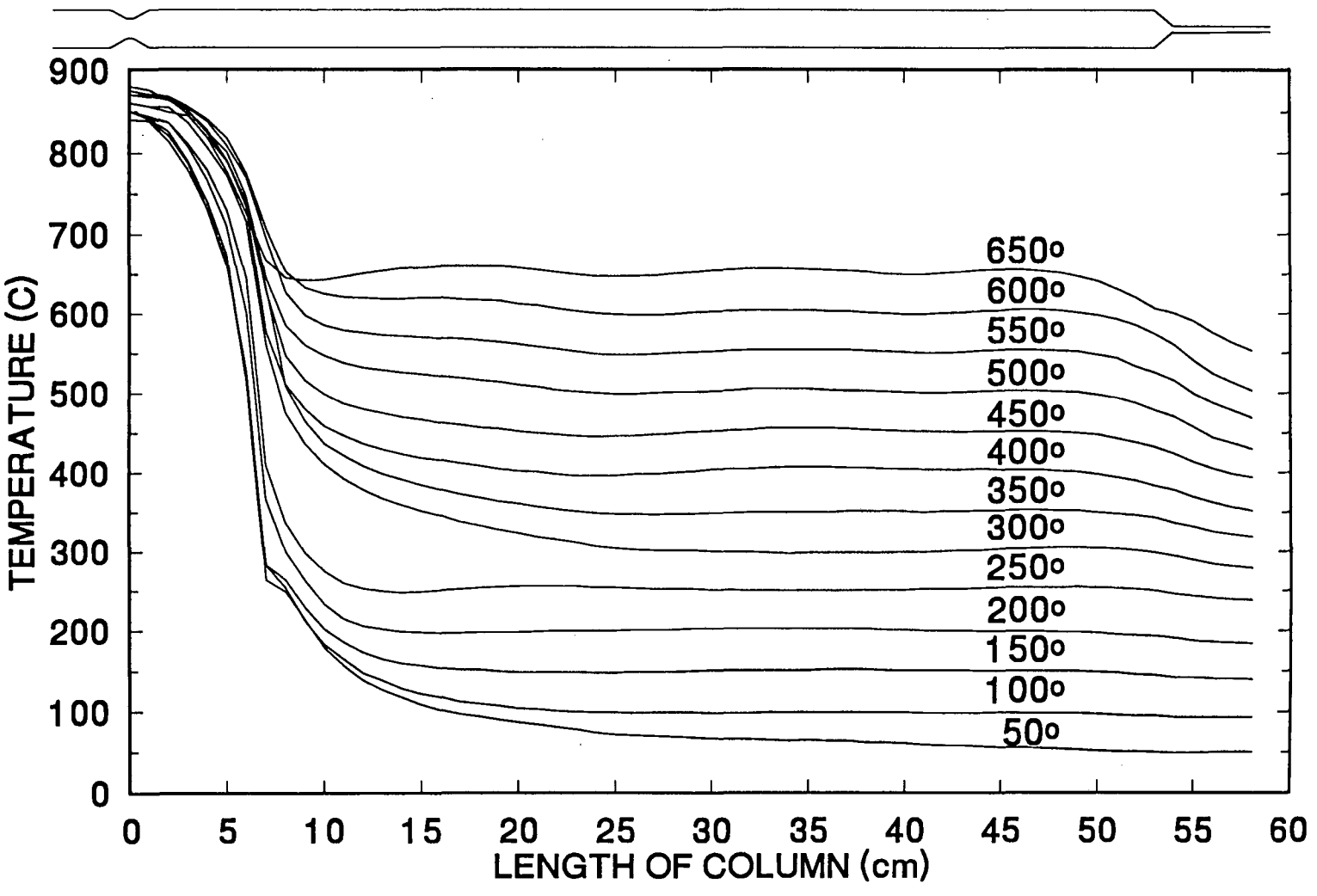
TEMPERATURE PROFILES INSIDE THE QUARTZ COLUMN
TEMPERATURE (C)
900
800
700
600
500
400
300
200
100
0
650o
600o
550o
500o
450o
400o
350o
300o
250o
200o
150o
100o
50o
0
5
10
15
20
25
30
35
40
45
50
55
60
LENGTH OF COLUMN (cm)

Fig. 7

Results

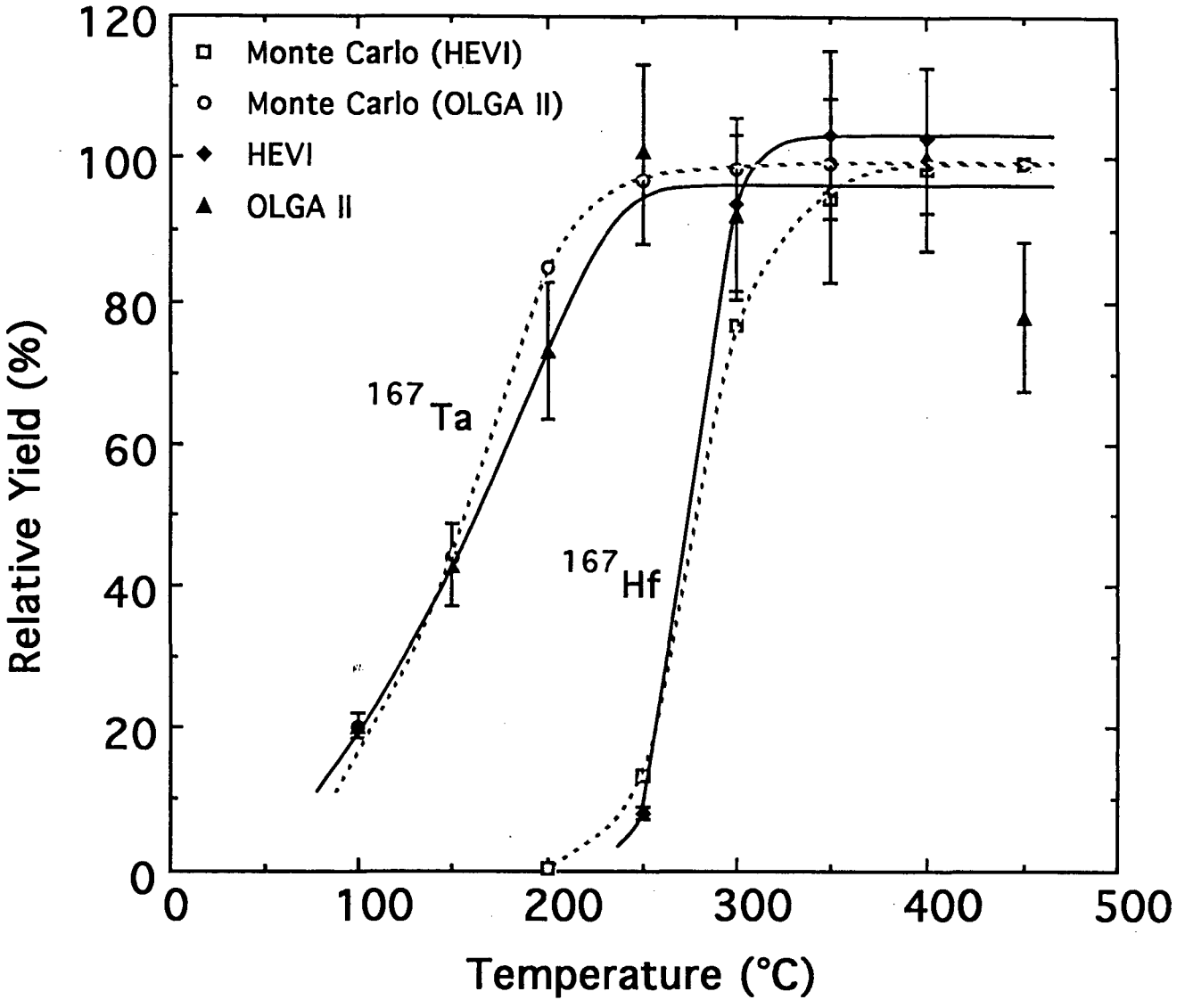

Fig. 8

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