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ONE-LINE GAS PHASE SEPARATION PROCEDURE
WITH RHENIUM**

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WITH RHENIUM**

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*Rhenium Separation / Nielsbohrium (Element 107) / Thermochromatography /
On-Line Gas Phase Separation*

Abstract

As a basis for future heavy element experiments off-line tracer studies with rhenium have been carried out to model the chemical behavior of nielsbohrium (element 107) in thermochromatographic separations with oxygen as a reactive gas. A specific version of the On-Line Gas Phase Separation Apparatus, OLGA, has been developed to perform on-line chemical separations of short-lived products from heavy ion nuclear reactions, and to enable α -spectroscopic measurements of the separated products after condensation on thin foils.

1. Introduction

Radiochemical separation methods have been applied in various fields of heavy element research and gas-phase separations have been a powerful tool [1]. Chemical properties of elements up to atomic number 105 (hahnium) have been studied in aqueous phase [2,3,4] and in the gas phase [5,6,7,8] (for more complete reviews see e.g. Ref. 9,10, and 11), and searches for new, heavy and superheavy elements produced in nuclear reactions have been carried out with radiochemical methods; see e.g. Ref. 12 and 13 and references therein. The unexpected observation of predominant α -decay for isotopes of element 108 [14,15], including the even-even isotope ^{264}Hs , has initiated a number of theoretical calculations which predict enhanced nuclear stability in the region of atomic number $Z = 109$ and neutron number $N = 162$ [16,17,18]. These findings and the observation of long half-lives for the newly found most neutron-rich isotopes of element 103 [19] have revived attempts to synthesize and investigate more neutron-rich nuclides bridging the gap between the presently known nuclei and the predicted island of spherically stable nuclei at $Z = 114$. Most recently a Dubna-Livermore collaboration, using a gas-filled separator, succeeded in finding, in this transition region, two new, presumably long-lived isotopes of element 106 with mass number 265 and 266 [20].

So far a large number of radiochemical investigations performed in the region of transactinide elements have severely been hampered by a technical deficiency. Instead of measuring the nuclear decay of the separated product to obtain a direct information on the chemical behavior in these experiments only grand-daughter or grand-grand-daughter products in α -decay chains [21] or tracks from spontaneous fission (sf) decay [5,6,22] have been measured. These detection techniques provide only indirect evidence, and additional assumptions are needed for the interpretation of the observables. On the other hand, in many of these experiments especially on-line gas phase separations have proven to be very powerful radiochemical tools for fast and efficient separations. To explore the possibility of further heavy element studies

thermochromatographic separation schemes for elements 106 through 110 based upon the volatilities of the oxides or hydroxides have been developed [23,24,25], and also in one experiment applied in a search for new isotopes [22].

In the first part of this paper we report on off-line thermochromatographic parameter studies with rhenium tracers as a model to find the best conditions for an on-line separation procedure of element 107, nielsbohrium (Ns). In its general properties Ns, located in group 7 of the Periodic Table of the Elements as Eka-Re, is believed to chemically behave similar to rhenium. Theoretical calculations [18,26] indicate that yet unknown isotopes of element 107 with neutron numbers close to $N = 162$ may have half-lives long enough for detection after fast chemical separations.

In the second part we introduce the On-Line Gas Phase Chemistry Apparatus, OLGA. It was built for a fast on-line thermochromatographic separation and the immediate, direct detection of the separated heavy elements by α -spectroscopy. The combination of the rapidity and efficiency of the chemical method with the advantages of using modern spectroscopic techniques for nuclear decay measurements will be discussed specifically on the basis of a preparation for an element 107 chemistry. The present version of this set-up has been developed and applied in the reaction $^{16}\text{O} + ^{254}\text{Es}$ as a realistic test for an extensive search for a new isotope of element 107 [27].

2. Experimental

The electronic ground state configuration of the free neutral atom of element 107, Ns, was calculated [28] to be $5f^{14} 6d^5 7s^2$ which is analogous to the rhenium configuration of $4f^{14} 5d^5 6s^2$. Nielsbohrium is expected to be a member of group 7 of the Periodic Table of the Elements and should exhibit chemical properties similar to rhenium and its compounds [29,30]. Therefore we have used tracer activities of Re to model the chemical behavior of oxo- and hydroxo compounds of nielsbohrium in the gas phase. The thermochromatographic behavior of volatile Re-compounds in the presence of

various amounts of oxygen and water, and its application to chemical separations and selective isolation of rhenium from several irradiated targets, has been the topic of many investigations [31-37]. There has also already been one experimental approach to develop a continuous thermochromatographic separation of rhenium in preparation for an element 107 chemistry [24], and to apply it to a search for spontaneous fission activities in the reaction of ^{249}Bk with ^{22}Ne [22].

2.1. Off-Line Parameter Studies with Rhenium Tracer

Tracer activities of $^{186,188}\text{Re}$ were frequently produced by irradiation of a 1 mg sample of NH_4ReO_4 or ReO_3 for six hours with a flux of 7×10^{11} neutrons $\text{s}^{-1} \text{cm}^{-2}$ at the Mainz Triga Reactor. After irradiation the samples were dissolved in dilute HNO_3 . An aliquot part was transferred into a quartz crucible and was slowly evaporated to dryness with a heat lamp under a stream of Ar gas to avoid losses from volatile oxides.

Carrier free activity of $^{182,184}\text{Re}$ has been obtained by an irradiation of a 0.1 mm thick W-target with 30 MeV protons at the compact cyclotron at Karlsruhe. After irradiation small parts of the W-target ($\sim 10 \text{ mm}^2$) were heated to 1000°C in a flow of oxygen at a flow rate of 6 l/h for about one hour in a quartz apparatus. Volatile Re-oxides were condensed from this oxygen stream onto 8 mg/cm^2 Au-foils mounted in a water-cooled, Ni-coated holder. Pieces of about 10 mm^2 of this Au-foil with carrier-free Re activity were put into a quartz crucible before loading the activity into the thermochromatographic column.

The set-up for the off-line thermochromatographic studies is shown in Fig. 1 together with a typical temperature distribution measured with a thermocouple along the inside of the quartz tube while the carrier gas was flowing. On the side of the gas inlet the quartz tube had a removable fitting to insert the crucible with the tracer activities into the cold region of the tube. This fitting had feed-throughs for a thermocouple and a steel rod which was used to push the crucible into the hot region

of the tube to start the chemical separation. The region of furnace number one and two was used to evaporate volatile species and the temperature was kept above 1000 °C. For the separation in the open quartz tube with an inner diameter of 3 mm a desired temperature gradient was achieved along the independently adjustable furnaces number three through six. To avoid inhomogeneities in the temperature gradient the quartz tube was inserted into a compound tube which consisted of a stainless steel tube inside a copper tube which again was inside a ceramic tube, see insert in Fig. 1. To obtain temperatures below 100 °C at the end of the column the last section of the tube was cooled by refrigerated ethanol. At the outlet of the quartz tube the gas flow impinged onto the surface of a replaceable metal disc kept as low as -30 °C. The most volatile species were caught from the carrier gas in a cooled charcoal trap. The gas flow through the system was maintained by a gas pressure between 1.1 and 1.2 bar at the inlet and a vacuum pump at the outlet. A modular gas flow control system consisting mainly of four mass flow meters and control valves coupled to a minicomputer, was used to adjust the flow rates and the mixing ratios of various gases. Drying of the gases was obtained by a commercially available drying unit with a specified gas purity of less than 0.5 ppm H₂O. To adjust a desired H₂O content in the reaction gas a valve was opened to connect a H₂O containing bottle to the gas inlet into the chromatographic tube. At this point a moisture meter (Shaw, type SHA-TR) was used to monitor the water content in the range between 0.5 and 1000 ppm. Before a chemical separation was started a quartz crucible with the Re activity was placed into the cool section of the quartz tube, the system was closed again and it was waited for the entire set-up to reach stable conditions regarding temperature gradient, flow rate, and purity of the gases. As the start of the chemistry we took the time when the boat was pushed into the hot section of the quartz tube. To stop the separation the furnaces were turned off and opened up for rapid cooling. At the same time the flow of the reactive component in the gases, oxygen or water, was stopped and only the flow of the inert carrier gas, helium or nitrogen was continued. After some time for cooling the quartz tube was taken out and was positioned in front of a lead collimator with a slit

length of 50 mm to measure the distribution of the Re activity along the column by γ -ray spectroscopy with a Ge-detector and standard electronics.

We have studied the influence of the following parameters on the formation of different compounds of Re-oxides and their condensation temperature in the quartz tube: helium or nitrogen as an inert carrier gas, the oxygen content between 5 and 30 % (volume), the water content between 20 and 1000 ppm, the flow rate of the gas between 0.75 and 1.5 l/min, and the time of separation between 1 and 30 min.

From these studies we obtained the following as the most important information:

1. Below a water content of 200 ppm, in the following referred to as dry, in the chromatogram only one component at about 420 °C condensation temperature, T_C , was present.
2. In a water containing, 'humid' atmosphere well above 200 ppm two additional components with varying amounts of about 50% of the total intensity were present. The deposition temperature for one component was at about -20 °C and it was always found in the charcoal trap.
3. The position of the peaks and their relative intensities are very insensitive to changes of all the other parameters (time, flow rate, O_2 content) within the investigated range.
4. Within one minute, the shortest time used in off-line experiments, $\geq 95\%$ of the Re was evaporated from the crucible.

A typical chromatogram together with the temperature gradient along the column is given in Fig. 2 for a flow ($v = 1.5$ l/min) of dry helium with 30% oxygen added and an experimental time of 1 min. The observation of one peak at a deposition temperature of about 420 °C, in our experiment is in good agreement with other experiments performed under slightly different conditions [24,33,36].

2.2. The On-Line Gas Phase Chemistry Apparatus **OLGA**

In a series of earlier experiments chemical separation techniques in the gas phase have been applied to investigate the production and chemical behavior of elements 104 and 105 [5,6], and also to search for a new isotope of element 107 [22]. These experiments have relied on spontaneous fission track detection inside the chromatographic column, which unfortunately is a very unspecific detection technique. As we decided to overcome this drawback we had to use a separation procedure which allowed only for the desired species to pass through the chromatographic column, and to be deposited outside the column on thin films for α -spectroscopy. Such a set-up has originally been developed for the search for superheavy elements in the ^{48}Ca on ^{248}Cm reaction [38,39], and it was named **On-Line Gas Phase Chemistry Apparatus, OLGA**. The set-up mainly consists of a gas supply and transport system, the thermochromatographic oven with column, and a detection system which was directly coupled to the exit of the chromatographic tube. This makes spectroscopic studies with no further delay after the chemical separation possible, and it is especially well suited for studies of very short lived radionuclides. The further developed version named **OLGA II** [40] is a slower version with certain advantages concerning the use of corrosive gases. A schematic view of the main parts of **OLGA** is shown in Fig. 3.

In on-line experiments the nuclear reaction products are transported from the beam line to the chromatographic column with a $\text{He}(\text{KCl})$ - cluster jet. The end of the He jet capillary, typically a 1.5 ± 0.5 mm inner diameter polyethylene or teflon tubing, is flanged to a glass ceramic tube with the same inner diameter in such a way that no losses of activity occur by avoiding any edges. This tube is then inserted into the chromatographic quartz tube and it ends a few millimeters in front of a quartz wool plug at a position with a typical oven temperature of 1050°C . All KCl clusters are destroyed at such a temperature and the nuclear reaction products are released. Through a T-joint at the closed inlet of the chromatographic column an adjustable flow of a reactive gas, e.g. oxygen, can be fed into the quartz tube and will mix with the He-jet

gas in front of the quartz wool plug. Non-volatile species, like the heavy actinides and their oxides, will be retained on the quartz wool while volatile products like rhenium oxides will move downstream the open chromatographic quartz column which has an inner diameter of 3 mm. The quartz tube ends in a nozzle at the end of the oven such that the temperature along the column can be controlled up to the very end of the quartz tube. The oven is a specially designed and built apparatus (Heraeus, Hanau) with five independently controllable heating sections to obtain a desired and smooth temperature gradient along the column. The oven consists of an inner and outer tube made of Alsint-99.7 (Haldenwanger, Berlin), and a filling of a ceramic powder and Kaowool as an isolator in between. The inner tube supports on the outside the heating wires, carries on the inside the quartz chromatographic tube, and has six 1.5 mm diameter small channels along the entire length of the tube to position 1 mm thick Ni-CrNi thermocouples. The signals from the thermocouples were fed into an automated control unit, which kept the temperature gradient along the important inner part of the oven on preset values by varying the times in the heating cycles. A typical temperature gradient, as tested with Re tracers, and found to presumably be the optimum for an element 107 experiment, is shown in Fig. 3. The oven system was attached to a vacuum counting chamber such that the end of the quartz tube was positioned at a distance of 3-4 mm from a thin (0.67 mg/cm^2) Ni-catcher foil coated with $50 \text{ } \mu\text{g/cm}^2$ Ta for a slightly better adsorption of highly volatile rhenium compounds. The most important advantage of a thin metal catcher foil compared to the most commonly used thin plastic foil is the wide range of temperatures, from far below 0°C up to even a few hundred degrees of Celsius, under which the catcher can be operated. The range of thicknesses between 0.6 and 1.0 mg/cm^2 Ni has been found to present an optimum regarding to the mechanical stability of the foil and the energy loss of an α -particle or fission fragment passing through the foil toward a backward detector. Foils of 0.5 mg/cm^2 and below frequently broke under routine operation for the size used in these experiments. The Ni(Ta)-foils were glued to aluminum washers with an inner diameter of 13 mm and were mounted on the perimeter of a rotating stainless steel wheel with 64 positions available. The still hot carrier gas from the

chromatography tube together with volatile reaction products were impinging on one side of the thin Ni(Ta) foil. A controlled flow of He gas cooled to liquid nitrogen temperature was impinging from the opposite side onto the foil not only to cool it to a temperature of 10 °C and below but also to aerodynamically stabilize the thin foil which would have been blown away with the gas flow from one side only. In addition to pumping the entire counting chamber with a roots pump, the deposition region, which was designed as an additional small chamber, has been pumped separately. The remaining pressure was below 0.5 mbar with a He content of about 90%; conditions very well suitable for α -spectroscopy.

The catcher wheel was rotated from outside the chamber through a vacuum feed-through with a stepping motor equipped with a reduction gear to provide a position resolution of 10.000 steps per 360°. Constant current power units were used for the motor control. The control of the positioning was achieved by an opto-electronic shaft decoder which provides a 3-digit (0...999) BCD-coded position read by a micro-computer developed at GSI [41]. In a slightly modified version it was used to control an automated rapid chemistry apparatus [42,43]. The micro-computer, operated as a slave of the master computer responsible for data accumulation, compares the actual position with a preset value and controls the adjustment of the final position of the wheel. The time lapse between start of rotation, triggered from a time out of a preset counting time from the master computer, and the end of positioning was 590 ms on the average. The positioning was equal or better than ± 1 mm.

Seven pairs of 300-mm² SB-detectors were mounted along the perimeter of the wheel such that the angle between the collection position and the first detector pair and the angle between successive detectors was 45°. Provisions were made to mount an additional annular detector at the position where the activity is deposited. Measurements were performed with such a detector in experiments where no chemical separations were performed and only the total activity was deposited on the wheel [44]. The detection efficiency was determined from a calibration source to be 24% for each detector on one side and 17% for the individual detectors on the opposite

side of the catcher wheel, which gives a total efficiency for α -decay of 41% and 82% for a fission. This exceeds the efficiency as obtained with a fission track method [22].

3. Results and Discussion

To test the on-line separation of nielsbohrium in the OLGA in a first step off-line separations of ^{183}Re deposited on a Au-foil were carried out mainly to find the best temperature gradient to let this rhenium oxide compound pass through the column, which in the former experiments were deposited at 420°C, and to measure the deposition yield on the Ni (Ta) catcher foil. These experiments were performed with a mixture of 70% nitrogen and 30% oxygen with less than 200 ppm H_2O at a flow rate of 1.5 l/min. In five minutes long separations 88% of the total activity were found by γ -spectroscopy on the cooled ($T \leq 10^\circ\text{C}$) Ni(Ta)-foil when a temperature gradient as given in Fig.3 was applied to the quartz column.

In a next step $^{176-178}\text{Re}$ was produced in a $^{16}\text{O} + ^{165}\text{Ho}$ nuclear reaction, and was transported to the OLGA in a He(KCl)-cluster jet for an on-line separation [27]. After adding 21% of oxygen a total flow rate of 0.95 l/min of a gas with a H_2O content of equal or less than 100 ppm was obtained. The total activity (100%) of $^{176-178}\text{Re}$ produced in the nuclear reaction was obtained by a γ -spectroscopic measurement of a 1.7 mg/cm² Al-catcher foil placed behind the target which was removed after a 10 min irradiation. The transport efficiency of the He(KCl) jet to the OLGA was determined in one experiment [27] to 56% by a catch of the activity on a filter paper. An equally high chemical yield was found for the 5.7 min and 14 min half-life Re activity on the Ni(Ta) catcher foil in OLGA after it passed through the thermochromatographic column. This indicates that no substantial losses occur in the separation and condensation part of the experiment. With typical transport times of less than five seconds in a He jet [27], and even shorter separation times in a hot thermochromatographic column for highly volatile species, see e.g. Ref. 24, the set-up as it is described above, should allow to study heavy element isotopes with half-lives

down to a few seconds. The chemical separation should result a significant reduction of interfering background activity, and, in combination with α -spectroscopy, should provide an unambiguous identification of the observed species. The direct condensation of the separated products on thin foils, allowing measurements with high detection efficiencies, and the total kinetic energy determination of fission fragments, are important advantages of this version compared to OLGA II [40]. Especially for very short lived radionuclides OLGA has a higher efficiency than OLGA II because there exists no time delaying step like the reclustering and transport of separated products to the moving tape system [40]. An additional feature, the possibility of the measurement of genetic links through mother-daughter α - α -correlations in the OLGA system is very well suited to search for new isotopes of heavy elements, in this specific case of element 107 [27].

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Figure Captions

Fig.1. Experimental set-up for tests with tracer activities in off-line thermochromatographic separations. The upper part shows the gas supply and the oven system. The composition of a special tube which carries the quartz separation column is shown in the upper right part. A typical temperature gradient along the inside of the quartz column is shown in the lower part.

Fig.2. Chromatogram of rhenium oxide (solid line) in a flow ($v = 1.5$ l/min) of dry ($\text{H}_2\text{O} < 20$ ppm) helium/oxygen mixture with 30% O_2 . The separation time was 1 min. The temperature distribution (dashed line) along the column is also given.

Fig.3. Experimental set-up of the **On-Line Gas Phase Chemistry Apparatus, OLGA**. The oven system with the five segments and a typical temperature distribution is shown on the left part. The oven is attached to a vacuum chamber with a rotating catcher wheel and surface barrier detectors, shown on the right side.

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Fig. 1

Fig. 2

Fig. 3