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PRODUCTION OF GASEOUS TRACER I_2 FROM THE SODIUM IODIDE SALT

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ABSTRACT

Found in the nature in form different, the iodine has been used in diverse works in the area of the industry and health. The iodine is very unstable and volatile in the ambient temperature and the I_2 is one of the diverse gaseous forms found. In this work was developed methodology for production of gaseous tracer from the sodium iodide (NaI) 0,1 M marked with ^{123}I. The synthesis was processed with in chlorine acid (HCl) 1M and sodium iodate salt ($NaIO_3$). The production of gas I_2 initially was carried through in unit of glass with the inert material and the purpose was to study the kinetic of reaction. The synthesis occurs in the reaction bottle and the produced gas is stored in the collect bottle that contains a starch solution (5 g/100 mL water). To determine the efficiency of production of gas I_2, analytic tests had been carried through, where the consumption of iodide ions of the bottle of reaction is measured. The optimization of production of the gaseous tracer was studied varying parameters as: concentration of iodide and iodate, concentration of acid and temperature. Then, the synthesis of the radiotracer was realized in the compact unit, being utilized as main reagent the salt radiated of sodium iodide, $Na^{123}I$. The transportation of elementary iodine was studied by a scintillation detector NaI (2 x 2)" placed in the reaction bottle. To acquire the data, the detector use a set of electronic modules for the acquisition of signals generated.

1. INTRODUCTION

In the commercial processes for extracting iodine from natural iodiferous brines, the brine, which in all cases so far know contains at most an extremely small percentage of iodine, is treated with a suitable oxidizing agent, e.g. chlorine, to liberate the iodine in the elemental state. A direct separation from the aqueous medium of the minute amounts of free iodine present by the usual method of settling and filtration is not possible, owing to the fact that such amounts of iodine are considerably below the limit of solubility thereof in the aqueous medium. It is necessary, therefore, to vaporize the iodine by steaming or blowing out with air, and to recover the vapors by absorption in a suitable liquid medium, usually in an aqueous alkali, or by adsorption upon a suitable solid medium such as active carbon or charcoal. In the case of absorption of iodine in an aqueous alkali, the iodine is obtained in chemically combined form as a mixture of alkali metal iodine and iodate, while in the last mentioned case the iodine may be recovered from the charcoal by leaching with a hot aqueous alkali, whereby owing to the reducing action of the charcoal a solution of iodide may be obtained which is substantially free from iodate. The iodine is recovered in any case, therefore, not as

the pure element, but in chemically combined form as the alkali metal iodide or a mixture of the iodide and iodate [1, 2, 3].

2. METHOD USED

The radiotracer technique is based fundamentally on experimental curves that are measured from the entry stimulus. This procedure is called stimulus-response technique, where the introduction of the tracer, through an injection system, is classified as the stimulus and the exit of the system the responses, through the use of radiation detectors [4, 5]. The production of the radiotracer elementary iodine is of great interest in the petroleum industry and also in the health.

In the tracer laboratory at the Institute of Nuclear Engineering – IEN two studies had been made from iodide ion, the first one was with the inert salt (NaI) and the last with the radioactive salt ($Na^{123}I$). A distillation system was used to synthesize the I_2 gas from the inert salt of NaI, as shown in figure 1. For the radioactive salt a compact unit of gas production was used, as shown in figure 2.

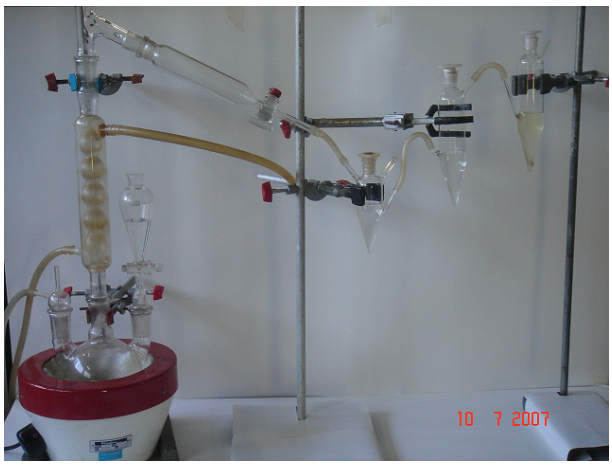

Figure 1. System of production of gas I_2

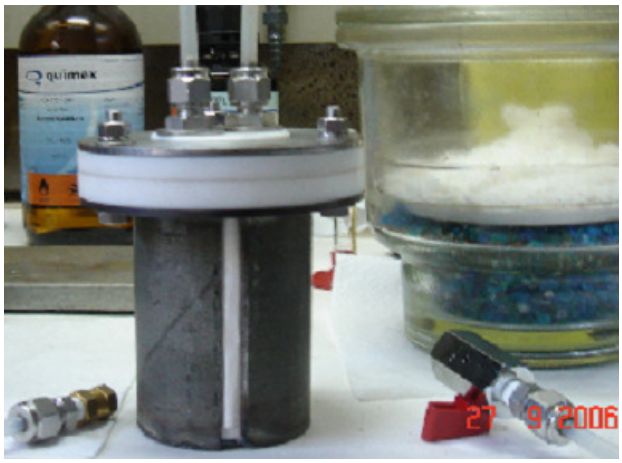

Figure 2. Lateral sight of the compact unit of gas

Among the several synthesis of production of the gas I_2, the synthesis below is more favorable in terms of cost, security and to the laboratory conditions. With a mixture of iodide

and iodate simple acidification with chloride acid, will result in precipitating iodine, and if the molecular proportion is in the ratio of five iodides for one iodate, such precipitation by acidification will be quantitative, in accordance with the typical equation:

$$5NaI_{(s)} + NaIO_{3(s)} + 6HCl \longleftrightarrow 3I_{2(g)}\uparrow + 6NaCl_{(aq)} + 3H_2O_{(l)} \quad (1)$$

If there is a deficiency either of iodate or of iodide as required by the equation, this may be made up by suitable addition to the solution prior to acidification [6].

For the test with the inert salt it was evaluated the efficiency of production of elementary iodine of reaction 1. It was necessary the development of methodology for measure of concentration of iodide contained in the solution. The employed procedure was the titration by the Farjans method, with $AgNO_3$ as titrant, whose concentration is known with precision and it reacts quantitively with a known solution that contains the iodide ion to be determined [6]. The titrant $AgNO_3$ was standardized, to a 0.0877 M concentration, and the eosin was chosen as the acid indicator.

Now, for the test with the radioactive salt was chosen as radiotracer the ^{123}I ($T_{1/2}$ = 12,9 h e E_γ = 0,159 MeV), in the solid form NaI. The salt was produced way cyclotron at the Institute of Nuclear Engineering (IEN), resulting in an activity of about 27,75 MBq. The extraction of radioactive elementary iodine was registered for one NaI (2 x 2)" scintillation detectors as seen in figure 3. It was shield by lead walls with 5 cm of thickness, where the collimator had an opening of 0,5 cm of diameter.

3. DISCUSSION OF RESULTS

Initially the synthesis was carried through with the inert salt and after studied the behavior and the efficiency of the reaction, the salt was radiated. The necessary quantity of volumes for the execution of the synthesis was calculated, through the stoichiometric calculation of the chemical equation 1. The synthesis was processed with 2 mL of chlorine acid (HCl) 1M, 2 mL of sodium iodide (NaI) 0,1 M and 0,112 g of sodium iodate salt ($NaIO_3$). Being that, first the acid is mixed with the $NaIO_3$, immediately afterwards the NaI is dripped on the mixture that it is being agitated. When the NaI enters in contact with the mixture, has an instantaneous reaction, in which are formed elementary iodine crystals (black color) and also I_2 gas (violet color), as shown in figure 3.

Figure 3. Formation of elementary iodine crystals and also I_2 gas

Depending on the concentration of the NaI it has a greater or minor formation of the precipitated one. The solution is yellowish indicating that it had the formation of a complex of the NaI with the I_2, indicated for NaI_3^-. This chemical species is steady to the ambient temperature and makes with that the I_2 is fixed. The increase of the temperature has contributed in the release of the gas and also in the kinetic one of the reaction. After produced, the iodine cannot be launched in the atmosphere. Thus a way was developed to hold back it. The produced gas is stored in the collect bottle that contains a starch solution (5 g/100 mL water). Another employed test was the observation in the color of the starch that reacts with the I_2, how many more gas is produced, more saturated is the starch solution.

3.1. Synthesis via inert salt.

For the synthesis with the inert salt the consumption of present iodide in the reaction vase was studied through of titration. With an automatic pipette, they had been removed, to each 15 minutes, samples with volume of 0,5 mL, diluted for 20 mL of water and placed in test tube. In each sample, 1 mL of the indicator eosin was added. After using a calibrated burette to add the titrant, it is possible to determine the exact amount that has been consumed when the endpoint is reached. The solution changed of color, passing of the color orange for violet.

The first sample of the reaction vase was collected as soon as the synthesis was initiated, being the initial concentration of ions iodide of 0,1 M. The synthesis was initiated to a temperature of 27 ºC (ambient temperature) e, immediately afterwards, the temperature was raised for 40 ºC. After 15 minutes (stabilization of the temperature in the reaction vase), a new aliquot was collected, where the concentration of ions iodide was of 0,05 M, indicating that half of ions I^- had been transformed into the I_2 gas. When the temperature was increased around 60 ºC, a new aliquot was collected and the concentration, determined (production around 34%). This temperature was kept around 30 minutes and figure 4 show that did not occur consumption of ions I^-, as it is observed by the formation of a small level in the concentration curve. The temperature was raised up to 80 ºC and three aliquot had been collected, in intervals of 15 minutes. A decrease in the concentration of I^- was observed, indicating new production I_2 gas. Later the temperature it was kept constant and the consumption of ions iodide continued to occur more slowly until reaching a constant value (240 minutes of synthesis), indicating that the amount of reagents in the way does not favor more the production of the I_2 gas. The total efficiency of the production of I_2 in the reaction was around 90%.

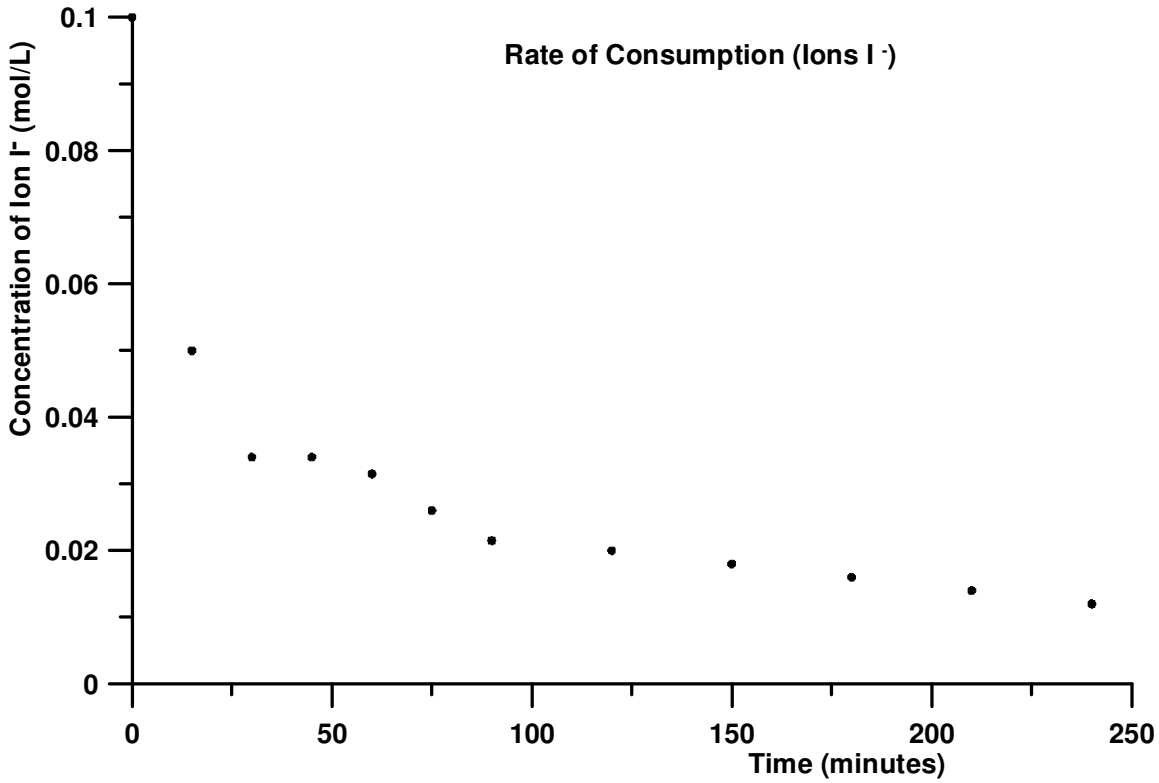

Figure 4. Chemical analysis indicating the rate of consumption of iodide.

3.2. Reaction via radioactive salt

Two tests had been carried through in the compact unit, the first one were placed a concentration of NaI 0,075 M and the last was carried through with a concentration of NaI 0.1 M. The response curve of detector, figure 5 and 6, represents the production and transportation of the I_2 gas. Each curve was normalized and the initial temperature was around 32 ºC. Variations were observed in both response curves for the total time of the synthesis, these oscillations are due to the intense movement of I_2 cloud in the reaction vase.

The first synthesis occurred after the time of acquisition of 35 minutes, having been the interval each counting of 1 second. It was observed that it did not have removal of radioactive material, because did not have inclination of the curve. After of 20 minutes of synthesis the temperature was increased for 90 ºC, after 10 mL of acid 1 M had been added and exactly thus it did not have variation in the format of the curve. The confirmation was through the starch solution, therefore no variation in the color did not occur.

After observed that it did not have extraction of I_2, then the concentration of NaI was increased for 0,1 M. The contour conditions had been the same ones used in the last test.

As presented in figure 6, around 25 minutes, the temperature was approximately in 70ºC, and the first injection of the I_2 in the collect bottle occurs. So that the extraction is maximum was passed air compressed in a period of 5 minutes and after close the valve. In the response curve was observed a great removal of I_2, verified through the great inclination of the curve. The unit again was pressurized and in approximately t = 40 minutes, a new injection was effected, being registered a new removal of radioactive material, due to the increase in temperature for 81 ºC. Between t = 50 minutes and t = 100 minutes, occur a great variation in

the consumption of the material and the temperature oscillated between 81 ºC and 85 ºC. After 100 minutes it was not observed no removal of the gas I_2. The variations in temperature showed that the material was removed from the reaction vase and the total efficiency of the extraction of I_2 was of around 83,3 %.

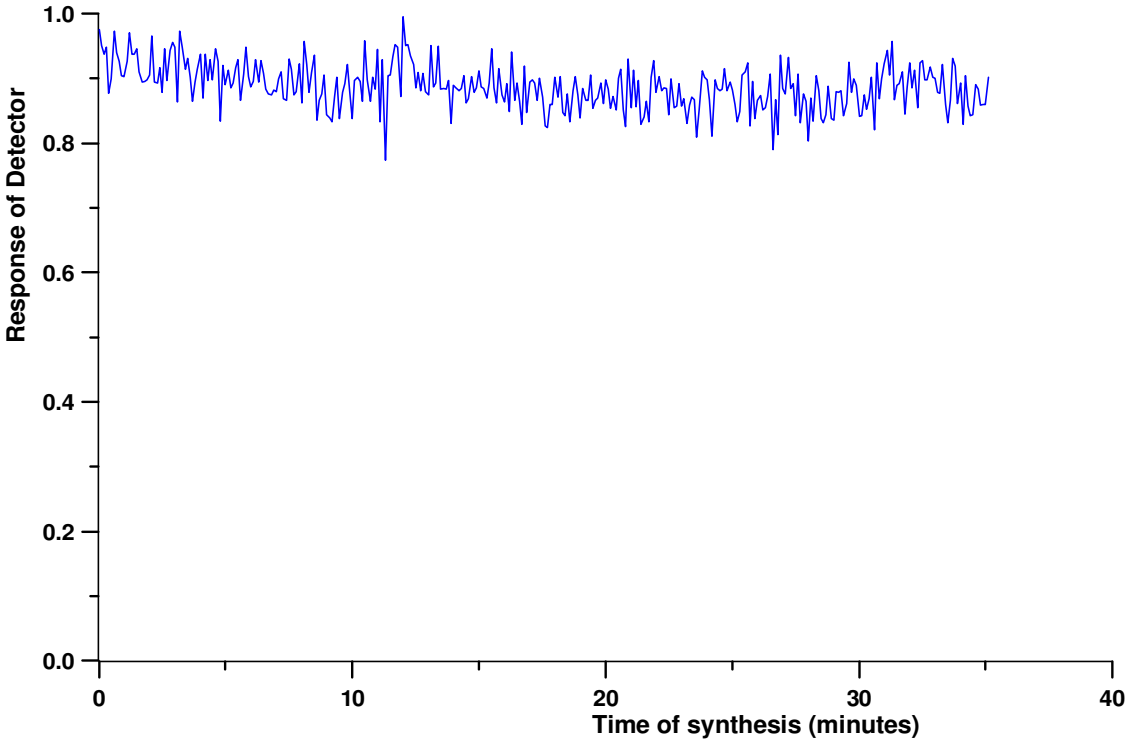

Figure 5. Response curve of detector for first test (concentration of NaI 0,075 M).

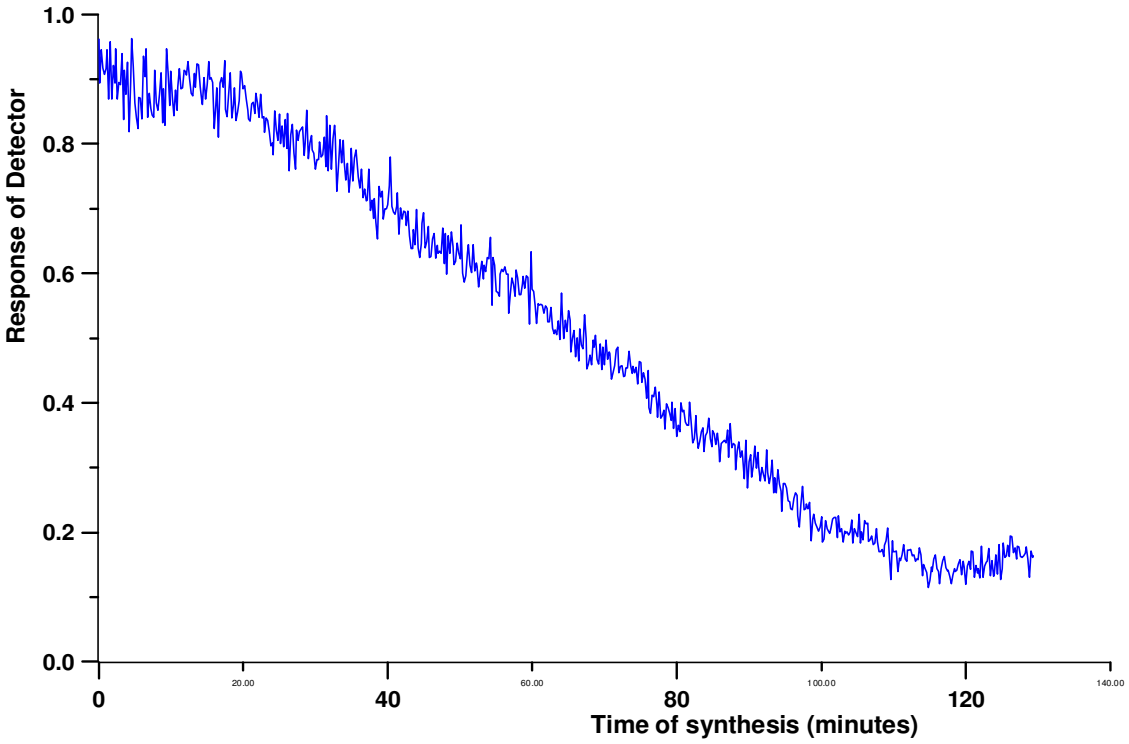

Figure 6. Response curve of detector for last test (concentration of NaI 0,1 M).

3. CONCLUSIONS

In accordance with the reactions of the synthesis, one can conclude that a number of factors influence and others no influence the production of the I_2 gas, such as:

- Temperature – increase in production is directly dependent on the increase in temperature;
- The quantity of reagents used:
- And the excess of chloride acid, for those reactions, it does not favor the removal of the gas I_2.

Some of the advantages of the process, such as the fact that the reagents are easily acquired on the local market at a low cost, and that the radioactive tracer can be used in diverse works in the area of the industry and health due to its ease of production, identification and measurement.

ACKNOWLEDGMENTS

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