Modelling aspects regarding the control in $^{13}$C isotope separation column

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Abstract. Carbon represents the fourth most abundant chemical element in the world, having two stable and one radioactive isotope. The $^{13}$C isotopes, with a natural abundance of 1.1%, plays an important role in numerous applications, such as the study of human metabolism changes, molecular structure studies, non-invasive respiratory tests, Alzheimer tests, air pollution and global warming effects on plants [9]
A manufacturing control system manages the internal logistics in a production system and determines the routings of product instances, the assignment of workers and components, the starting of the processes on not-yet-finished product instances. Manufacturing control does not control the manufacturing processes themselves, but has to cope with the consequences of the processing results (e.g. the routing of products to a repair station). In this research it was fulfilled some UML (Unified Modelling Language) diagrams for modelling the C$^{13}$ Isotope Separation column, implement in STARUML program. Being a critical process and needing a good control and supervising, the critical parameters in the column, temperature and pressure was control using some PLC (Programmable logic controller) and it was made some graphic analyze for this to observe some critical situation than can affect the separation process.
The main parameters that need to be control are:
- The liquid nitrogen (N2) level in the condenser.
- The electrical power supplied to the boiler.
- The vacuum pressure.

1. Introduction
Stable isotope applications include highly varied fields. In physics, stable isotopes are mainly used as materials mono-isotopic, pure or as a feedstock for pure radioisotopes using nuclear reactions. In this field, isotopes are particularly important for the study of nuclear constants, properties isotopes, and structure and energy characteristics of the molecule. In chemistry, stable isotopes are used to study the reactions of exchange, being used as tracers in chemical reactions. In geology, the data from the analysis of isotope concentrations are used to study fields, various archaeological finds dating, determining the temperature of sedimentation. Stable isotopes are found, while applications in biochemistry, biology and medicine, MRI (magnetic resonance imaging) research pathways and distribution dynamics of infectious microbes in the body components live organs of renovation study, to investigate human metabolism.

The utility of these stable isotopes of particular importance for a wide range of applications is overshadowed by one main drawback, namely the very high costs of production. For example,
glycogen syntheses study using nuclear magnetic resonance requires about 70 g of glucose labeled with $^{13}\text{C}$.

The isotopic composition of the mixture is usually expressed using fractions of atomic isotope extracted, the ratio between the number of atoms of the isotope extracted and the total number of atoms of the element. In analysis of the separation plant is used relative concentration is defined like:

$$C = \frac{c}{1-c}$$

(1)

where C is the relative concentration, and c atomic fractions from isotope [1].

One of the important factors that affect the separation size, distillation time and cost is the proportion of the condensate is returned to the column as reflux. This ratio is measured by the ratio of reflux. It is expressed by the ratio between the number of moles of reflux liquid / time unit and the number of moles of the liquid product / unit of time or of the number of moles of reflux and the number of moles of vapor that rises through the column, both expressed in the same units. A high reflux ration means better separation and higher time distillation for a given amount of the mixture.

During any period of time, a certain proportion of liquid introduced into the plant, exist in the column like reflux or vapor. This quantity is known as the holdup. It is important in the discontinuous distillation (batch) because limits the percentage of charge which can be distilled and has no effect on the accuracy of the separation. For any process of distillation, pressure of vapor is a basic concept. Each liquid evaporate in a vacuum space until its vapor pressure reaches a size feature, called vapor pressure. It is fixed for any liquid at a given temperature increases with temperature and varies from one liquid to another. The vapor pressure is an equilibrium value in the sense that if the vapor pressure is higher or lower than the feature will cause condensation and evaporation of the liquid until the balance will be restored.

In carbon isotopes, $^{12}\text{C}$ isotope is predominantly in natural concentration of about 98.89%, while the isotope "hard" $^{13}\text{C}$ is in its natural state with an abundance of only 1.11%. Isotope separation relies on the fact that at very low temperatures (~ -192 °C) carbon monoxide with $^{12}\text{C}$ isotope has a higher vapor pressure than carbon monoxide containing the $^{13}\text{C}$ isotope [2]. The separation is achieved while maintaining a constant temperature, the temperature at which coexist both the gas phase of carbon monoxide ($^{12}\text{C}$), and the liquid $^{13}\text{C}$, the two compounds being "distilled" like typical industry distilling process [9]: the gas phase will be eliminated, while the liquid phase - containing carbon monoxide with a higher concentration of $^{13}\text{C}$ isotope - is extracted as the final product. The main factor that indicates the carbon isotope separation is "separation coefficient" $\alpha$ [9], given the vapor pressure ratio of the two compounds:

$$\alpha = \frac{p^{12}\text{CO}}{p^{13}\text{CO}}$$

(2)

where $\alpha=1.0069$ at -191.3 °C temperature [71], $p^{12}\text{CO}$ vapor pressure of $^{12}\text{CO}$ and $p^{13}\text{CO}$ vapor pressure of $^{13}\text{CO}$[1]

2. Model of the column $^{13}\text{C}$ isotope separation column

Each gas has a temperature of liquefaction according to a given pressure. Increase or decrease its pressure, change the temperature of liquefaction / boiling transforming all liquid in gas or vice versa.

The distillation column of CO for separating $^{13}\text{C}$ isotope works slightly below atmospheric pressure.
The pressure in the column is measured at various locations throughout the column. It is measured and monitored as the top column (in the condenser) and the bottom of the column (the heater).

Heater pressure must overcome the pressure drop due to the filling of the separation column such that:

$$P_{heater} = \Delta p + p_{condenser}$$  \hspace{1cm} (3)

where, $\Delta p = \sim10$ torr.

$\Delta p$ depends on the CO loading of the separation column (flow ratio of liquid CO cross-sectional area of the column).

A proper pressure for working in the separation column, there is a liquefaction temperature of CO. The gradient allowed the separation column is the maximum 2°C (from -192 °C, the liquefaction temperature of CO to -190°C).

Working in low temperatures (-192 °C), maximum gradient of 2 °C the temperature of the column is ensured by using one device for evacuate to high vacuum (10-3-10-4 tor).

If pressure in the system for some reason (disturbance flow, temperature) increase, then increase the boiling temperature of liquid CO, and causes vaporization of the CO in separation column. Thus further increase more the pressure in the column and is going in avalanche up to damage the plant.

Increasing the pressure above the threshold requires the discharge of CO gas from the column in a large volume tank, vacuum in advance, using a regulation loop of the pressure using an electromagnetic valve which opens at increased pressure.

When the CO liquid in case of damage or total shutdown of the plant, is evacuate total (plant temperature reaching ambient temperature), the condition of pressure in the column equal to atmospheric pressure is not accomplished.

The amounts of CO liquid (hold-up liquid) and CO gas (HOLD-Up Gas) determines the total amount of CO in normal circumstances from which it can determine the volume of container that will eliminate CO in damage case, forced stop or normal stop.

HOLD-up of liquid CO is larger than gas HOLD-up, the amount of CO gas resulting from formula:

$$Y = \frac{\text{Hold-up CO}_{\text{liquid}} \cdot 22.4}{28}$$  \hspace{1cm} (4)

where molecular weight is CO=12+16=28g=1 mol= 22.4l, [1].

CO ratio of the feed rate, in the section is constant for an optimal mode of CO distillation; retention of the column with CO liquid is constant.

In transient retention of the column CO liquid may increase if the column working at rates higher than those prescribed so can lead to flooding the column. When the entire column is flooded with CO liquid, CO gas bubbling through it, isotopic separation is compromised; the plant operator must stop, or try to get out from flooding by gradually reducing the flow of gas.

In case of flooding pressure increases continuously, the plant operator must of decreasing the flow of CO gas column (about 10-20%), and if $\Delta p$ does not decrease, it takes total shutdown of the plant.

The temperature of N2 from the condenser was monitoring using one application using PLC for record the values of the N2 temperature and was made an application which shows this value (figure 1).

In automated systems, PLCs represent the main part of the management and control of the process. Through the execution of a program stored in the program memory, programmable machine monitors the status of the process by the signals received from the input devices. By building the program, PLC determines actions on output devices. In the case of more complex processes it is possible to connect several PLCs from a PC [8,9, 10]. Automatic management of industrial processes to ensure both with general purpose PLCs type, and, also, with specialized PLCs type. Examples of
specialized PLCs for industrial processes may be: automation equipment for boilers and heating installations in buildings, control and signalling equipment for the detection and alarm, fire detection, driving pumping stations and water supply of communities, etc. The operative part orders send command to the system and to the operative information about evolution is collected under orders sent [8,9,10].

We can choose very easy the period of day and time and see the behavior of the condenser.

The main parameters that need to be analyzed are:

• The liquid nitrogen level in the condenser. The drop of the liquid nitrogen level below a critical value would lead to the impossibility of efficiently condensing the vapors upstream and thus would compromise the entire separation process (figure 1). Figure 1 shows the values of temperature sensor from liquid nitrogen in the condenser. This was record using dedicate PLC.

• The electrical power supplied to the heater. High variations would affect the separation by modifying the upward gaseous stream.

• The vacuum pressure. Variations in the vacuum pressure bring about the loss of the efficient thermal isolation and cause the increment of the inner column temperature.

To establish the mathematic model of the distillation process it is important to know the material and energetic equations for the distillation column [2][3].

![Figure 1. Some critical situations measure in condenser, [7].](image)

3. Model of the column $^{13}$C isotope separation column using UML (Unified Modeling Language) Diagrams

Use case diagram is used during the analysis phase of the column to identify the system functionality (figure 3, figure 4, and figure 5). It describes the interaction of people or external device with the system under design. It doesn't show much detail, but only summarizes some of the relationships between use cases, actors, and systems (figure 2) [4][5].

It was made some models for $^{13}$C isotope separation column using UML Diagrams. The aim for these models is to have a good and exact control and monitor to the isotope separation column and can proceed in a proper time if some critical situations appear.
In the work with anomaly detection that has been carried out so far, the main emphasis has been on monitoring the behaviour of the system (sequences of function calls, connections between machines and so on) rather than the data passed around the system \[1\][6].

**Figure 2.** Use cases diagram for $^{13}$C Isotope separation column.

**Figure 3.** Activity diagram for $^{13}$C Isotope separation column.

**Figure 4.** Classes diagram for $^{13}$C Isotope separation column.

**Figure 5.** Communication diagram for $^{13}$C Isotope separation column.

4. Conclusions

It was made some models for $^{13}$C isotope separation column using UML Diagrams. The aim for these models is to have a good and exact control and monitor to the isotope separation column and can proceed in a proper time if some critical situations appear.

It was implement a PLC at condenser level to control the temperature of the N2, and it was deeply monitor using TAG EVOLUTION.

PLC’ś are used increasingly more often in the industry. An application of those is temperature control of industrial furnaces (e.g. for drying of painted components) with electrically or gas heated.
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