



## METHODS FOR CONTROL OF SOME CRITICAL SITUATIONS AFFERENT OF A $^{13}\text{C}$ 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  $^{13}\text{C}$  Carbon 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. Carbon isotopes can nowadays be separated using many methods, one of them being the cryogenic distillation of carbon monoxide in Isotope Separation Column. This research is intended to simulate a distributed control system of critical situations that may arise in the operation of the distillation column. In these simulations, a wireless communication network is used, to transmit data. We considered as study case, two critical situations that may arise due to increasing pressure: increasing the pressure inside the column and increasing the pressure in the vacuum cover of the column.

**Key words:** critical situation, isotop separation, distributed system

### 1. INTRODUCTION

Carbon represents the fourth most abundant chemical element in the world, having two stable and one radioactive isotope, [1]. The  $^{13}\text{C}$  Carbon isotope, 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, [4]. Carbon isotopes can nowadays be separated using many methods, one of them being the cryogenic distillation of carbon monoxide in Isotope Separation Column.

The major issue with  $^{13}\text{C}$  comes from the difficulty of obtaining it and raising its natural fraction. Carbon isotopes can nowadays be separated using many methods, one of them being the cryogenic distillation of carbon monoxide, [4].

Very few aspects regarding the construction and operating conditions of such cryogenic plants are known today, and even less information is available as far as the separation process modeling and control are concerned.

However, the efficient control of the carbon monoxide distillation process represents a necessity for large-scale  $^{13}\text{C}$  production.

### 2. SEPARATION COLOMN'S CONTROL PROBLEMS

Referring to a classic distillation process, several models for carbon isotope separation have been proposed, some based on mass, component and energy balance equations, some on the nonlinear wave theory or the Cohen equations.

However, even though these models capture the essence of the isotope separation process, they can generally be used to determine in each moment and column-coordinate the concentration of the useful isotope, thus describing and modeling the isotope fraction distribution as a function of time and as a function of the column dimension, but not for control purposes

The column, with the simplified scheme, separates the carbon isotopes based on the cryogenic distillation of pure carbon monoxide, which is fed at a constant flow rate as a gas through the feeding system, [2].

At extremely low temperatures (about  $-192^\circ\text{C}$ ), the vapor pressure ( $P_1$ ) of ( $^{12}\text{C}^{16}\text{O}$ ) is greater than the pressure ( $P_2$ ) of ( $^{13}\text{C}^{16}\text{O}$ ) and the separation coefficient is:

$$\frac{P_1^o}{P_2^o} = \alpha \approx 1.007 \quad (1)$$

Due to the very-low operating temperature, an efficient thermal isolation vacuum jacket is necessary. Since the “elementary separation ratio”, [4] is very close to unity in order to raise the (<sup>13</sup>C) isotope concentration up to a desired level, a permanent countercurrent of the liquid-gaseous phases of the carbon monoxide is created by the main elements of the equipment: the boiler in the bottom-side of the column and the condenser in the top-side.

The gaseous carbon monoxide upstream (from boiler and from the feeding system) condenses on the “coldwall” of the condenser, cooled with liquid nitrogen by atmospheric pressure and falls, in small drops, downstream to the electrically heated boiler, [14].

The (<sup>13</sup>C) isotope, slightly heavier than the predominant (<sup>12</sup>C) isotope, accumulates in liquid phase and will be extracted as end product at the bottom of the column, while the (<sup>12</sup>C) component accumulates in vapors phase and will be extracted as waste at the top of the column. The column operates with two zones: the stripping zone, from the feeding point to the top of the column and the enriching (rectifying) zone, in its lower part, [7].

The characteristics of the carbon cryogenic separation column, presented in what follows, suggest the necessity of modern control strategies.

The counter current cryogenic distillation column related is a highly complex plant, nonlinear, multivariable, with large time constants that overcomplicate the control solutions. The control strategy is to maintain the column operation parameters constant, by eliminating the effects of the disturbances while keeping the overall system stable, despite the uncertainties that may arise, [6].

The main parameters that need to be monitor and control 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.

-The electrical power supplied to the boiler. 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.

### 3. MODEL OF THE <sup>13</sup>C SEPARATION COLUMN

Experimental separation column can be configured in two ways: one - two columns of different diameters placed one within the other extension, and second way, one column with set diameter, [2].

The important parts of the column are:

- Condenser- C;
- Primary column C1;
- Final column C2;
- The boiler of Primary column B1;
- The boiler of Final column B2;
- Vacuum jacket, M;
- Aggregates vacuum, PVP (PD).

In the column, condenser (C) provides the reflux process to the top of the column with total condensation of carbon monoxide, using liquid nitrogen boiling at atmospheric pressure. Condenser is cylindrical and is provided with longitudinal fins. It is made of stainless steel, mono-block design, assembly of which is made by welding. Its length is 800 mm and a diameter of 200mm.

Fluid flows gravitational from the condenser C1 on the column packing in countercurrent with monoxide carbon gas. In the first column, part of the liquid is vaporized and sent to the condenser and the other part enters into column C2. At the base of the column, the boiler B2 ensures the reflux process of carbon monoxide liquid, [1].

Column C1 is 26mm diameter and 2500mm length. Feed point divides the column in two enrichment sections: one of 1000 (1500) mm and second one dilution section of 1500 (1000) mm. It is filled with spiral triangular 2x2x0.2mm stainless steel wire.

Column C2 is 16mm diameter and 4250mm length. It is filled with spiral triangular 1.8x1.8x0.2mm stainless steel wire (figure 1).

Boilers, B1 and B2 are heated by electrical resistance, which have a maximum power of 100W and 50W.

Separation column is the main component of an isotope distillation process, because in the column, using the contact between the two phases - the liquid and vapor, takes place the isotopic enrichment, [13].

The <sup>13</sup>C experimental separation column is connected in its upper part, with liquid nitrogen-cooled condenser at atmospheric pressure (77 K) and at base on the boiler, which ensure the vaporization of the liquid. It operates at a temperature of about 79 K.

Column height is 7062 mm and is made of stainless steel. We consider two columns: one with diameter of 16 mm and one with diameter of 26 mm, both the same height, [12].

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

The material equation for the distillation column is:

$$\frac{d(N \cdot x)}{dt} = F_a x_a - F_v y - F_b x \quad (2)$$

where: N is the number of moles of liquid from the column, and Fa, Fv, Fb are the molar flow of feeding at the top and base of the column.

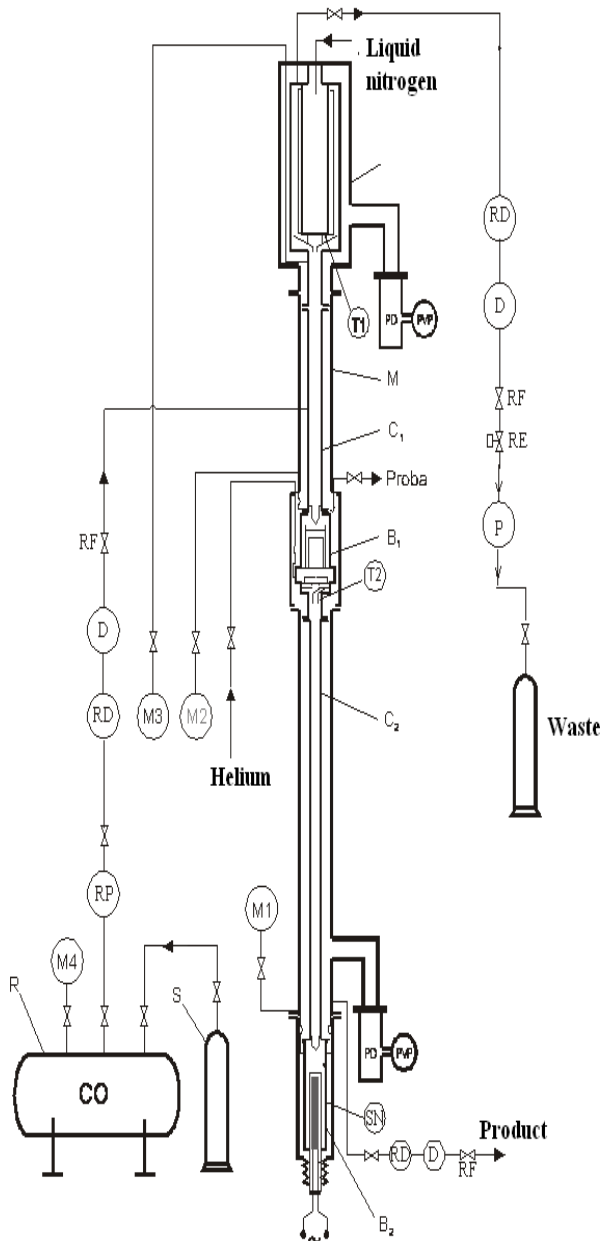


Fig. 1. Separation column, [1]

Energy balance equation for separation column will determine the enthalpy of the liquid and vapor in the system and is:

$$\frac{d(N\lambda)}{dt} = F_a \lambda_a - F_v \lambda_v - F_B \lambda - a_p + Q_{ag} \quad (3)$$

Abstract design of the distillation column can be representing like in figure 2.

The first part includes the heat exchanger and the base of the column.

The second part includes the area median of the column, and the third is the top of the column with liquid nitrogen condenser.

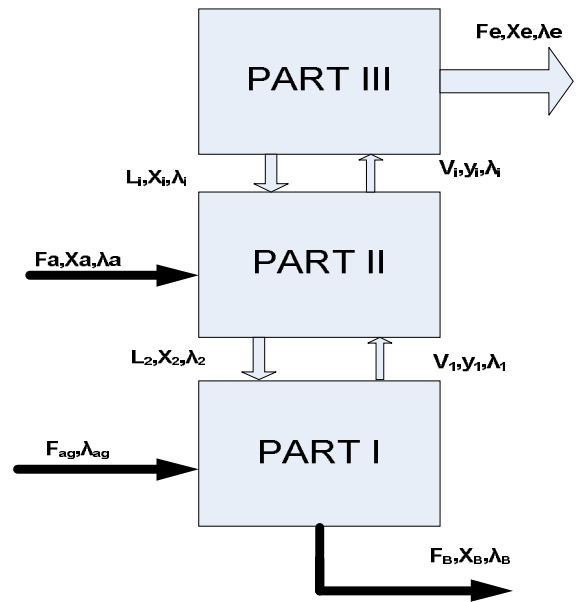


Fig. 2. Abstract design of the distillation column

### 3. PRESENTING THE PROCESS AND THE APPLICATION OF SIMULATION

This research is intended to simulate a distributed control system of critical situations that may arise in the operation of the distillation column. In these simulations, a wireless communication network is used, to transmit data. We considered as study case, two critical situations that may arise due to increased pressure:

- a) increasing the pressure inside the column;
- b) increasing the pressure in the vacuum cover of the column .

In the first case it is necessary the action of an electrovalve that will open pipe, connecting the column and a vacuum container, resulting in pressure drop in the column to the reference value.

To automatically adjust the electrovalve's position and implicit the flow rate of pipe on which it is acting, it is used a PI controller.

In the second case it is necessary the consecutive and in parallel startup of two vacuum pumps. For proper operation of the distillation column is required thermal insulation from the outdoor environment. Thermal insulation is achieved by ensuring a high vacuum values in column casing. On the occurrence of an alarm that signals the increase of pressure in casing of column, first of all is turned on high vacuum pump. If pressure in casing does not drop for a specific time, in parallel with the first, the second pump is turned on, [5].

The model of distributed simulation system has two components at 20 m distance from each other, both being represented by a block True Time kernel (real time nucleus) and a battery of real time. A node sensor / actuator driven of time sampled the exit

process and transmit the samples through network to node of controller. Control function implemented in this node calculates the control signal and sends the result back to the node sensor / actuator, where is applied to process (Figure 3).

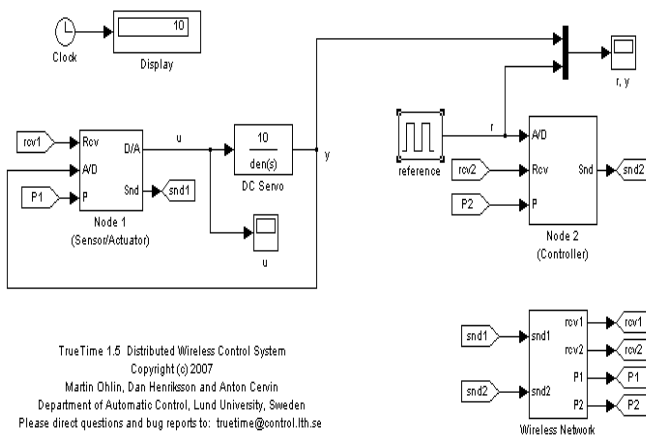


Fig. 3. Simulation scheme of distributed control system for critical situations afferent to column

WLAN network type is set. Network speed is also set to 800000bits / s. The use of Wireless networks permits a significant reduction of data transmission infrastructure inside the facility is located (Figure 4). In both cases (critical situations) alarm signal will be considered as having logical value (when the column is 0 operation is carried out in the normal range and when it has the value 1 appears critical situation). Both critical situations mentioned above will be treated in two ways, namely by using appropriate execution elements in both open loop and closed loop.

As reference to the DC output signals of the process, when working in automatic mode, signals of gear type are used, with appropriate value for each case. In all simulations it will be followed the execution elements reaction and time course of signals generated by these both to the appearance and disappearance of alarm signal.

#### 4. SIMULATION RESULTS

In all simulations will follow the reaction of the actuators and time evolution of the generated signals to the appearance and disappearance of the alarm signal.

The simulation model contains two nodes of the distributed system at 20 meters from each other. Both of which are represented by a block True Time Kernel (Kernel real time) [8], and a real-time battery. A node sensor / actuator driven by time sample the outputs and send the samples over the network to the controller node.

Control function implemented in this node calculates the control signal and sends the result back to the node sensor / actuator, which is applied to the process.

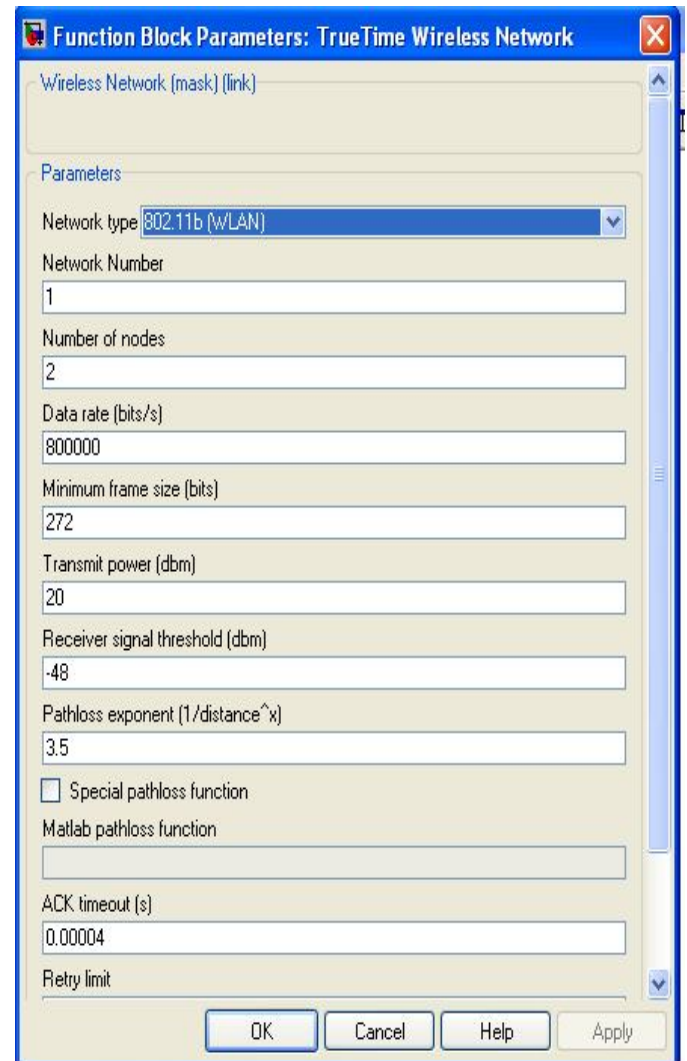


Fig. 4. The parameters of the Wireless network

In Figure 5 it is show the time evolution of the output signal of the electro-valve that allows opening the pipe vacuum container. At appearance of the alarm signal was applied an constant input signal voltage equal to 10V to the electro-valve entrance,. At disappearance of the alarm signal, the electro-vakve power was restored to 0V.

*Case 1. Simulation the opening position of the electro-valve (1) in open loop (Figure 5).*

In figure 5, the output signal generated by the execution element was expressed as a percentage (the maximum being 100% clear). The Figure shows that the automatic reduction of the pressure in the column is working properly; the reaction of the electro-valve is in accordance with the alarm signal evolution.

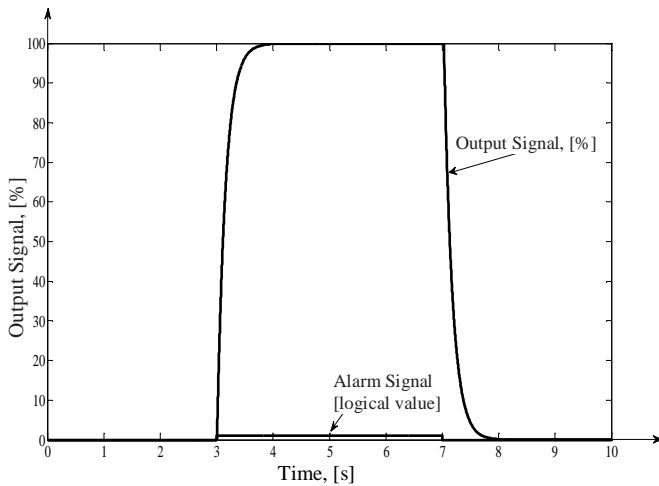


Fig. 5. Reaction of the electrovalve to the appearance/disappearance of an alarm signal

*Case 2. Simulation the opening position of the electro-valve (1) in closed loop (Figure 6).*

This case is useful when you want to partially open the pipe between the separation column and vacuum recipient. Figure 6 is the corresponding simulation when electro-valve is brought to a degree of openness of 50% (set as reference).

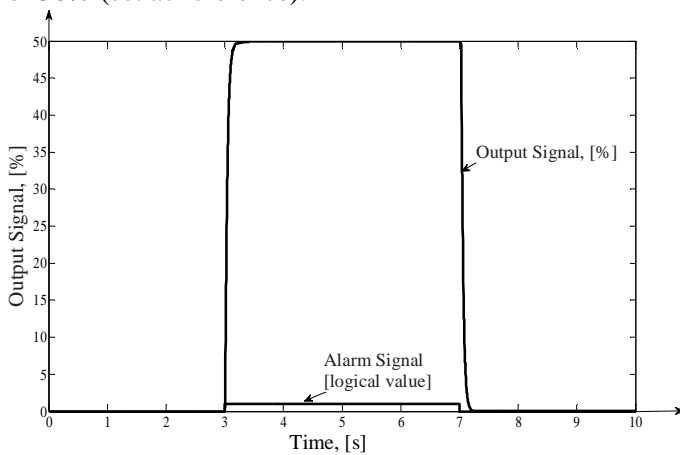


Fig. 6. Reaction of the closed loop electrovalve to the appearance/disappearance of an alarm signal

Figure 6 shows that the system of automatic management of critical situations works correctly, moreover, the used of the regulator involve the electro-valve reaction was much faster (can be obtain more easilly the references values during the appearance and disappearance of the alarm signal).

Using wireless networks requires a discussion of the data transmission rate. Thus, serving a system that is critical in the operation of the column (for its safety system) is necessary to ensure the correct transmission of data between the various components of the application.

Simulation shows in figure 5 was performed using the settings shown in figure 4. When using the same parameters except the data transmission rate will be fixed at the value of 106 700 bits / s, the simulation

under the same conditions as in the case of figure 4, we get (figure 7).

The controller used is given in figure 9. Reaction signal can be generated by a transducer mounted on the pipe flow acting the electro-valve, flow rate being proportional to the degree of openness of the actuating unit (electro-valve).

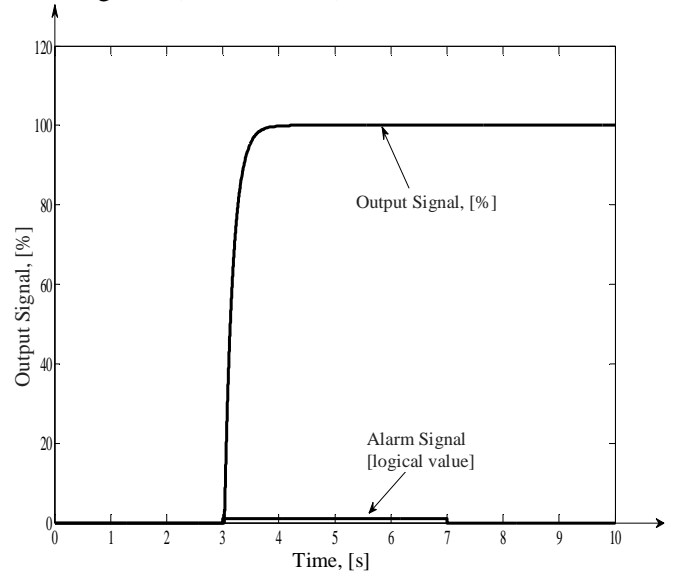


Fig. 7. The electro-valve reaction to the appearance/disappearance of an alarm signal, using different data transmission rate

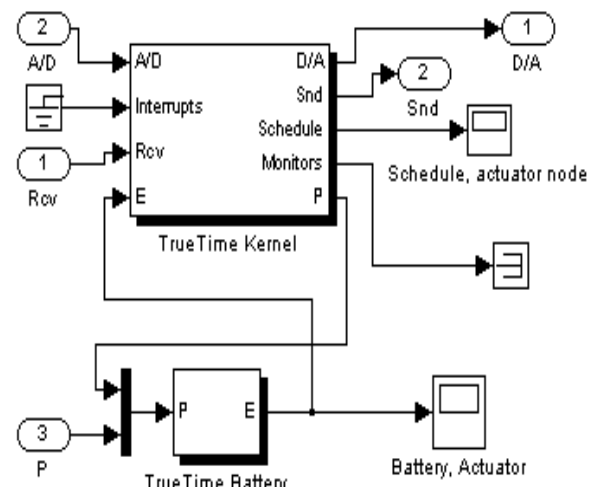


Fig. 8. Sensor/Actuator Structure

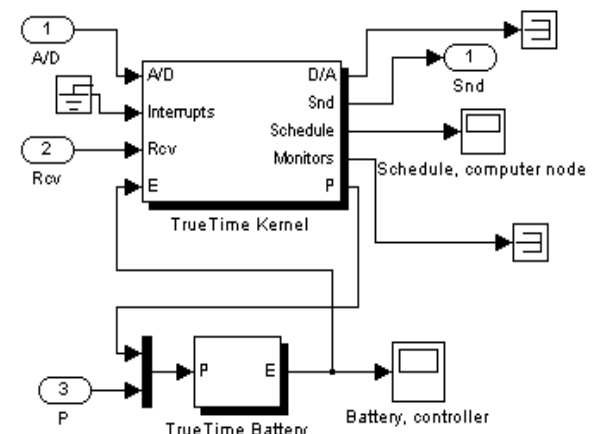


Fig. 9. The controller structure

## 5. CONCLUSION

In this research we create and simulate some models of distributed control system for some critical situations that may arise in the operation of the distillation column.

We analyze the behavior of the distributed control system using the wireless connection and electrovalve for the appearance/disappearance of an alarm signal in the distillation column.

This simulation was made to detect and solve some mission critical in isotopic carbon distillation processes that can appear during the process.

As a future work we propose to implement this model on experimental cryogenic separation of CO system.

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