

## APPLICATION OF THE COMPUTER MONITORING AND CONTROL SYSTEM TO AN ISOTOPE SEPARATION COLUMN

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Abstract: The chemical processes developed in a separation plant are very complex and many details are not yet known in totality. The Labview project is an important step forward for the correlation of the theoretical scientific research results with the concrete information acquired from the experimental plant, and also in the analysis and better understanding of the process operation. The paper presents a short description of the column monitoring and control system for main variables that ensure an efficient and safe column operation.

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### 1. INTRODUCTION

The field of application for the ( $^{13}\text{C}$ ) isotope of carbon is broad: chemical engineering, pharmaceutical production, biology, medicine, etc. The “minor” isotope ( $^{13}\text{C}$ ) is a valuable tracer in chemical, biological and environmental science. It is extremely useful in medical diagnostic investigation of various body organs. On the other hand, the “waste” from the column, CO with high-purity in ( $^{12}\text{C}$ ) is used in electronics technology to produce synthetic diamond crystals with 50% better thermal conductivity than that of the “usual” synthetic crystals.

A method to produce various substances with high-concentration of these isotope is based on the cryogenic distillation of carbon-monoxide, with a natural abundance of ( $^{13}\text{C}$ ) of 1.1% ( $^{12}\text{C}^{16}\text{O} \Rightarrow 98.9\%$ ;  $^{13}\text{C}^{16}\text{O} \Rightarrow 1.1\%$ ). If distillation temperature is decreased under 85°K, the vapor pressure for ( $^{12}\text{C}^{16}\text{O}$ ) is greater than the pressure for ( $^{13}\text{C}^{16}\text{O}$ ), so that the isotope ( $^{13}\text{C}$ ) accumulates in the liquid phase, while the concentration of ( $^{13}\text{C}$ ) in gaseous phase decrease (“stripping” process). In a static steady-state liquid-gas contact, the accumulation of the isotope of interest is very small, the concentration rising with

the “elementary separation ratio” ( $\alpha$ ), very close to the unity:  $\alpha \approx 1.011$ . In order to rise the ( $^{13}\text{C}$ ) isotope concentration up to a desired level, a permanent counter-current of the liquid-gaseous phases is achieved in a dedicated equipment: the packed isotope separation column.

### 2. THE ( $^{13}\text{C}$ ) ISOTOPE SEPARATION COLUMN

The column (C) is a steel packed pipe, figure 1, feed with pure gaseous carbon-monoxide by the flow-rate ( $Q_{in}$ ) and ( $^{13}\text{C}$ ) isotope concentration ( $N_f$ ),  $N_f = N_o = 1.11\%$ . The gaseous carbon monoxide with lower ( $^{13}\text{C}$ )-concentration is evacuated as “waste” at the top side, by flow-rate ( $Q_{out}$ ) and the concentration ( $N_w$ ). The efficient thermal isolation of the column is based on the external vacuum jacket (Radoi, 1999).

The liquid-gas permanent counter-current is provided by the two important elements: an electrically heated boiler (B) in the column base and a condenser (K), cooled with liquid nitrogen, at the column top side. The liquid nitrogen level ( $h_c$ ) in condenser and the liquid carbon-monoxide in boiler ( $h_b$ ) must be maintained at a constant level.

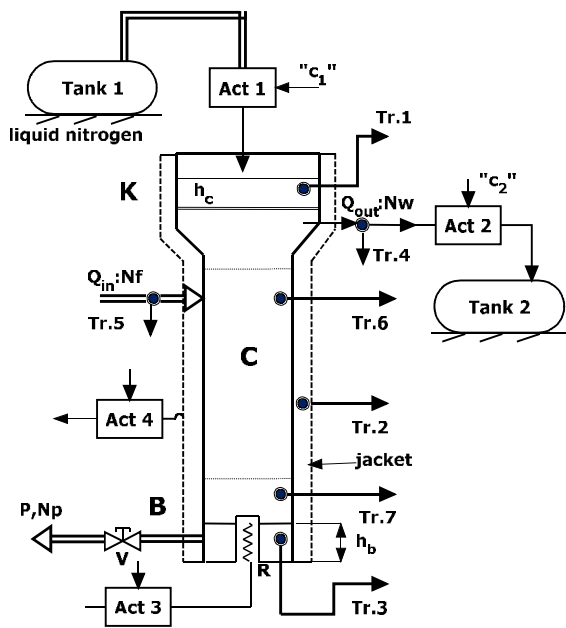


Fig. 1. Separation process scheme

The isotope of interest ( $^{13}\text{C}$ ), accumulated in the liquid carbon-monoxide, is withdrawn as final product at the flow rate ( $P$ ) and concentration ( $N_p$ ).

The isotope separation process may be controlled by measuring the main variables given in table 1.

Table 1. Main variable definitions

No	Variable name	Measurement device, sensor	Device index	Obs.
1	Liquid nitrogen level ( $h_c$ )	Resistive level transducer	Tr.1	-
2	Vacuum level	Dedicated electric device	Tr.2	-
3	Liquid carbon-monoxide level ( $h_b$ )	Capacitive level transducer	Tr.3	-
4	Output (waste) carbon-monoxide flow-rate ( $Q_{out}$ )	Rotameter	Tr.4	Only as indicator
5	Input (feeding) carbon-monoxide flow-rate ( $Q_{in}$ )	Rotameter	Tr.5	Only as indicator
6	Top-side column internal pressure	Pressure gage	Tr.6	Only as indicator
7	Bottom-side column internal pressure	Pressure gage	Tr.7	Only as indicator

In order to use a minimal number of data acquisition converters, the output transducer signals are scaled as DC voltages in the range of (0÷10 V).

Some of the indicated variables may be controlled using proper actuators, given in table 2.

Table 2. Actuator definition

No	Directly controlled variable	Name and type of actuator	Input of variable	Obs.
1	Liquid nitrogen level ( $h_c$ )	Special electro valve (Act 1)	Voltage: TTL	Logic signal
2	Waste out-flow rate ( $Q_{out}$ )	Volumetric pump (Act 2)	Continuous DC voltage -5÷+5V	-
3	Vacuum level	Dedicated two-stage vacuum pumps	Voltage: TTL	Logic signal
4	Electric dissipated power $W_{el}$	DC voltage controller	Continuous DC voltage 0÷10V	-
5	Feeding in-flow rate ( $Q_{in}$ )	Fine-valve	-	Manual control

### 3. THE MONITORING SYSTEM

The Labview project is an important step forward for the correlation of the theoretical scientific research results with the concrete information acquired from the experimental plant, and also in the analysis and better understanding of the process operation.

The front panel of the program has numerical and graphical indicators, buttons for available options, alarms to warn the user that a problem appeared in the process. A sample of this front panel is presented in figure 2.

The data acquisition and transmission is based on the modules series I-7000 built by ICP CON-producer (\*\* ICP-DAS Literature). The application is based on the modules:

- 1) I-7017R: an 8-channel voltage and current input module with high voltage overload protection, 240Vrms, added. It has 3000V DC intra-module isolation; 24-bit sigma-delta ADC to provide excellent accuracy: +/- 0.1%; input impedance 2M Ohms; input power +10 to +30 V DC; power consumption 1.3 W.
- 2) I-7024: a 4-channel analog output module that supports bipolar voltage output. It accepts input power supply in the range +10 to +30 V DC; power consumption 2.4 W; accuracy: +/- 0.1%; 14-bit resolution; 3000V DC intra-module isolation.

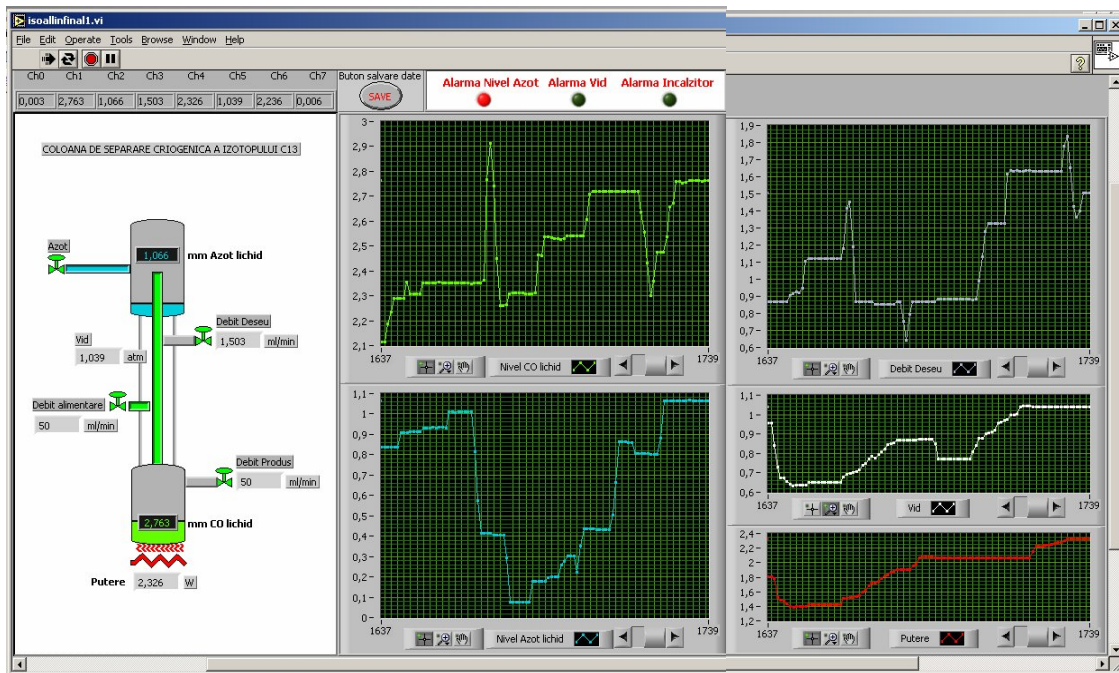


Fig. 2. Front panel of the monitoring application

- 3) I-7044: a 4-channel isolated digital input and 8-channel isolated digital output module. The input type is Sink or Source, Isolated channel with common power or ground and the output type is Open Collector, Isolated channel with common power with 30 V maximum output voltage. The input power voltage range is +10 to +30 V DC and the power consumption is 1W.

On the front panel are shown the voltage values read from the network data acquisition module I-7017R, part of the ICP CON family of network data acquisition and control modules, providing analog-to-digital, digital-to-analog, digital input/output, timer/counter and other functions. Communication between the modules and the host is in ASCII format via an RS-485 bi-directional serial bus standard, as shown in the figure 3.

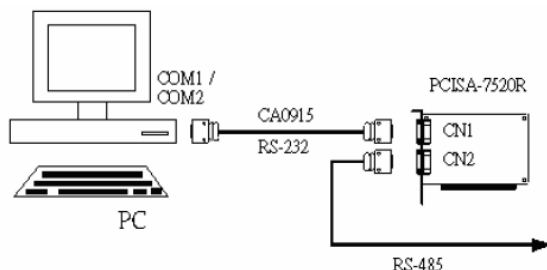


Fig. 3. Communication between the modules and the host

The user can supervise the normal progress of the plant and detect an atypical value read by the sensors. Another way to signal the user that an error occurred

is the alarm system. The alarm system is activated when the limit level, previously established for the correct development of the process, is surpassed.

The instantaneous values for the variables of the process, obtained through the sensor's calibration for this parameters, are shown using the numerical indicators in engineering units and graphical from, on the real plant diagram. The charts show the time variance of the parameter's values of the process. The sampling period (freely selected) was 60 seconds and the chart history length is 1000000 samples.

The data from the process can be saved in Excel by pushing the "Save" button and typing the name and path of a file. The file has columns for the sample number, date, time and the input values read for each parameter.

#### 4. THE CONTROL SYSTEM

In accord to the requested control performance specifications, a broad range of control methods are recommended. The actual paper present two simple versions, implemented in the computer monitoring and control system.

In the figure 4 is presented the simplified diagram of the liquid nitrogen level control in condenser (K). The data acquisition module (DAM-1) converts the transducer output voltage into a digital size for the host computer. The control algorithm, implemented in a discrete form in computer, generates the control signal to the logic output module (LOM-1). It controls a solid-state relay (SSR) which supplies the electrovalve coil.

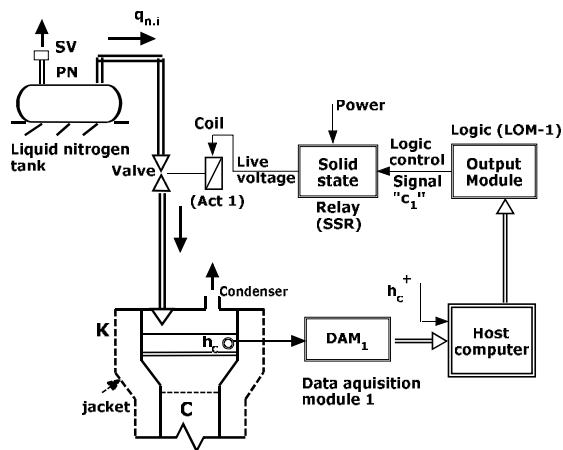


Fig. 4. Liquid nitrogen level ( $h_c$ ) control diagram

The control algorithm is a bang-bang type with hysteresis, figure 5, where ( $h_c^*$ ) is the set-point and ( $2\Delta h$ ) is the hysteresis band (Dorf, 2006).

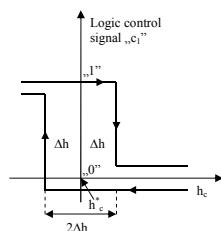


Fig. 5. Static diagram of relay control:

The logic control signal (" $c_1$ ") is given by the equation:

$$"c_1" = \begin{cases} "1" & \text{for } h_c < (h_c^* + \Delta h) \text{ and } \dot{h}_c > 0 \\ "0" & \text{for } h_c > (h_c^* + \Delta h) \text{ and } \dot{h}_c > 0 \\ "0" & \text{for } h_c > (h_c^* - \Delta h) \text{ and } \dot{h}_c < 0 \\ "1" & \text{for } h_c < (h_c^* - \Delta h) \text{ and } \dot{h}_c < 0 \end{cases} \quad (1)$$

The liquid nitrogen level ( $h_c$ ) is the result of the balance between the inflow rate ( $q_{n,i}$ ) and the evaporation outflow rate. If the valve-coil is energized (by the solid state relay), the inflow ( $q_{n,i}$ ) surrounds the evaporation flow rate and the level ( $h_c$ ) rises. For " $c_1$ "=0, the level ( $h_c$ ) decreases. The limits of variation for  $h_c(t)$  are given by the process particularities and the value of ( $\Delta h$ ).

The second example illustrates the control of the liquid carbon monoxide level ( $h_b$ ) in boiler for  $h_c \approx ct.$  and  $W_{el} = ct.$  The disturbance is the inflow deviation  $\Delta Q_{in}(t)$  and the manipulated variable is the "waste" outflow  $Q_{out}(t)$ .

The simplified level control system is illustrated in the figure 6. The output voltage of the transducers (Tr3) controls the data acquisition module (DAM-2). The control algorithm is in this case a continuous one in the PD form (Dorf, 2006):

$$C(s) = V_R \left( 1 + \frac{\tau_d s}{T_N s + 1} \right); \tau_d \gg T_N \quad (2)$$

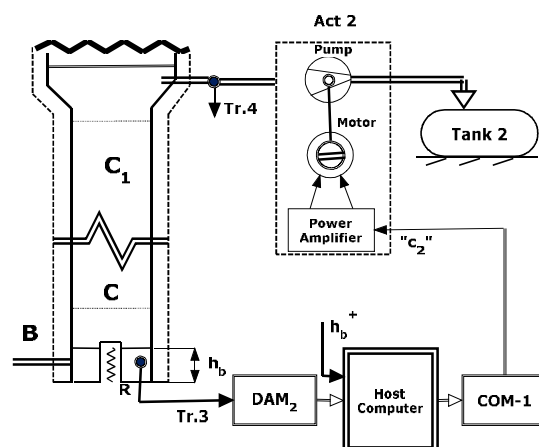


Fig. 6. Liquid carbon monoxide level control diagram

The output continuous module (COM-1) converts the digital controller output in an analogic form " $c_2$ ", which activate the power amplifier (PA) of the actuator (Act 2), in order to change in a proper manner the outflow  $Q_{out}(t)$ .

## 5. CONCLUSIONS

A short description of the column monitoring and control system for main variables that ensure an efficient and safe column operation is presented in the present paper. The monitoring system follows the main variables of column: liquid carbon monoxide level in boiler ( $h_b$ ), liquid nitrogen level in condenser ( $h_c$ ), vacuum jacket pressure, "waste" outflow carbon monoxide rate, electrical power dissipated in boiler. The control system contains the liquid nitrogen level control in condenser (using a bang-bang type controller with hysteresis) and the liquid carbon monoxide level ( $h_b$ ) control in boiler. In the actual stage, the control functions are analyzed only by simulation. As possible results of the monitoring functions can be mentioned the record of the column operation history, a deeper understanding of the separation process and the possibility for an optimal column control as a future task.

## REFERENCES

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