

**REVAMPING OF THE NEVINNOMYSSK AZOT**  
**METHANOL SYNTHESIS CONVERTER.**  
**THE FIRST APPLICATION**  
**OF THE CASALE 'IMC' DESIGN**

*By P. L. Golosnichenko and S. M. Kononov*  
*NEVINNOMYSSK AZOT,*  
*Russia*  
*and*  
*E. Filippi*  
*METHANOL CASALE S.A.,*  
*Lugano, Switzerland*  
*and*  
*N. Ringer*  
*SUED-CHEMIE AG*



presented at the  
**WORLD METHANOL CONFERENCE**  
Phoenix, Arizona, USA \* 10-12 November 2003

## 1) FOREWORD

Nevinnomyssk Azot operates a 320 MTD methanol plant in its manufacturing complex at Nevinnomyssk, Russia.

This plant uses as feedstock the off-gas of an acetylene cracker, added with a stream of hydrogen coming from an acetic acid plant.

The methanol plant has been designed in early 1970's by the company GIAP. The methanol synthesis converter had four adiabatic beds and intermediate cooling by quench, lozenge type.

The operating pressure before revamping was of 53 bar a maximum, and, by consequence, the pressure vessel is a fairly large size, i.e. almost 4m ID by about 8 m TL.

The total catalyst volume installed before revamping was about 50 m<sup>3</sup>.

In the year 2000 Nevinnominsk Azot decided to revamp the converter with the aim of increasing its capacity and reducing the energy consumption.

Methanol Casale S.A. was inquired for this revamping project and at the end of 2000 it was awarded the contract.

## **2) THE REVAMPING PROJECT**

The revamping project had two aims: the reduction in energy consumption and a capacity increase.

The Energy consumption reduction was related to the circulation power consumption, to be reduced by 2300 kW and the recovery of reaction heat by raising LP steam, the expected value of which was 12.2 t/h of steam.

The capacity increase goal to be reached was a capacity of 425 MTD, when the necessary synthesis gas would be available.

Therefore, due to a lack of gas, the converter had to run at the beginning at 324 MTD, and later on up to 425 MTD, when the necessary gas was available.

To take full advantage of the new converter internals the synthesis loop revamping project included the revamping of the synthesis gas compressor and of the circulator and, as mentioned above, the addition of a waste heat boiler downstream the converter to recover the reaction heat. The circulator revamping was necessary in order to reduce the recycle ratio down to the new value allowed by the new converter internals. The steam raised in the new boiler was to be used in the distillation column.

The scope of work of Methanol Casale included the license, engineering and supply of the new converter internals and catalyst, and the new instrumentation related to the new internals, the specification of the new recycle wheel and waste heat boiler, and the detailed engineering of the piping modification.

Nevinnominsk Azot provided all the remaining materials, i.e. the revamping of the syngas and recycle compressor, the waste heat boiler the piping and all necessary bulk material, and the manpower for the installation.

### **3) THE NEW METHANOL CONVERTER INTERNALS**

The converter internals provided for the revamping are of a completely new design, applied in this occasion for the first time in the industry.

The new internals are of pseudo-isothermal type, where the cooling fluid is the fresh feed gas to the reactor, which is preheated before entering the catalyst bed. This feature is of course not new, having been used by Casale in hundreds of ammonia and methanol converters in the past.

What is new in this design are a number of mechanical and process features:

- The heat transfer surface immersed in the catalyst is not made with tubes, but with plates
- The temperature in the catalyst bed is controlled not only at the bed inlet, but also along the catalyst bed
- The revamping is made 'in situ', i.e. the existing converter vessel has been re-used, untouched, and the new internals have been inserted, pre-fabricated, through the existing top manhole, and assembled inside the vessel.

It is therefore the first time in which an adiabatic quench methanol converter is transformed to a pseudo-isothermal one with an 'in situ' modification. In addition it was the first time a heat transfer surface different from tubes was used, having very innovative characteristics of catalyst bed temperature control.

This new design has been named 'CASALE IMC', i.e. CASALE Isothermal Methanol Converter.

#### **3.1) The Plate Exchangers**

Plate heat exchangers have been in use since a long time in the petrochemical industry, there are many manufacturers producing plates and their production technology is advanced and well established.

The plates are normally produced with a fully automated process, with little or no manual intervention. The production process starts normally from a coil of the desired metal sheet, which goes through a sequence of automatic operation like welding, inflation or printing, assembling and quality control.

The approach is that of a mass production, where, once the production parameters have been set and tested, all the elements are identical, minimizing the possibility of defects, and with a very effective impact on costs.

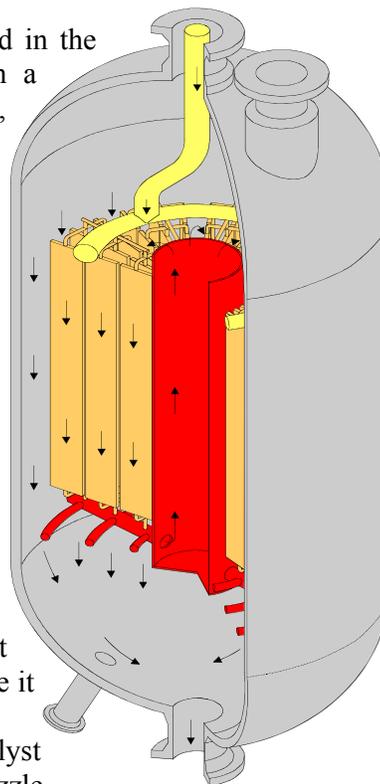
The plate's geometry is very flexible, allowing the design of converters of any size.

In the METHANOL CASALE design the plates are immersed in the catalyst bed, and the cooling fluid is flowing inside the plates.

Figure 1 illustrates the gas flow in the Nevinnominsk Azot converter after revamping.

As it can be seen, the plates are immersed in the catalyst bed, the cooling gas entering from a nozzle on the top is distributed to the plates, inside the plates the gas flows downward, i.e. co-current with the gas in the catalyst bed, and after having been preheated it is collected to a central raiser pipe, from where it reaches the top of the catalyst bed. There the gas flows downward in the catalyst bed, and then exits from the bottom central nozzle. On top of the cooled catalyst bed, there is a small adiabatic layer of catalyst, to give an initial boost to the gas temperature.

As it can be seen the plates are immersed in the catalyst bed, the cooling gas enters from a nozzle on the top and is distributed to the plates. Inside the plates the gas flows downward and after having been preheated it is collected to a central raiser pipe, from where it reaches the top of the catalyst bed. There the gas flows downward in the catalyst bed, and then exits from the bottom central nozzle.



**FIGURE 1**

### **3.2) Mechanical Advantages**

The main features of this type of construction are that:

- It does not have a tube sheet, therefore it is possible to perform ‘In Situ’ revamping.
- The catalyst bed is continuous, supported by a bottom layer of inert material, therefore it can be easily loaded from the top, and unloaded from the bottom through drop-out pipes, minimizing the downtime for catalyst replacement.
- The plates are connected in modules (groups of plates) and each module can pass through the manhole. The central raiser pipe is large enough to be accessed by a man, assuring in this way an easy access to all part of the reactor, allowing the replacement or repair of any item that could fail during the operations, i.e. the pressure vessel, the plates or the internal pipes.
- Thanks to the absence of a tube sheet and to the automated production of the plates, the overall construction is very cost effective.

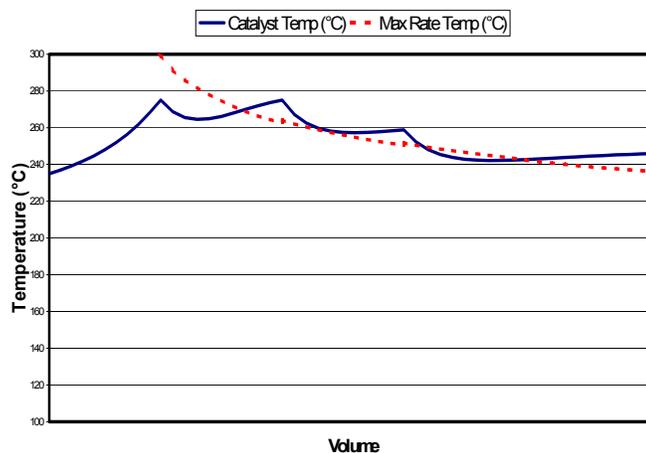
### 3.3) Process Features

As mentioned, the new internals have temperature control of the catalyst bed not only at its inlet, but also along the bed. In this way it is possible to control the temperature profile in the catalyst bed so that the catalyst mass can work as close as possible to the line of maximum reaction rate or to any other profile desired by the operator.

This feature consists in the control of the amount of cooling fluid fed to the different part of the plates, so that it is possible to modify the temperature profile in the catalyst bed adapting to the specific needs, i.e. fresh catalyst or spent catalyst conditions or other requirements, as if it was a quench type converter.

This design therefore presents the same operating flexibility of a quench converter, but with the high efficiency of an ideal pseudo-isothermal design with indirect cooling .

Figure 2 illustrates the calculated temperature profile that can be achieved in the catalyst bed in a co-current configuration similar to the one in Nevinnominsk Azot converter.



**FIGURE 2**

The process features of this design can then achieve these advantages:

- The possibility to obtain the desired temperature profile in the catalyst, therefore achieving the optimal temperature profile in conditions different from the design ones, and that best suite the plant operations.

In practical terms this means that in a revamping project, such as the Nevinomyssk Azot one, where an adiabatic bed quench-type converter is transformed 'in situ' to the Casale IMC, it is possible to achieve a reduction in energy consumption by reducing the recycle ratio and/or increase the carbon efficiency of the system by reducing the loop purge.

An additional advantage is that normally the catalyst volume required is smaller than in the pre-revamping situation, reducing then the cost of catalyst replacement.

#### 4.) THE NEVINNOMYSSK AZOT EXPERIENCE

The new internals have been installed in the converter during the turn-around of October 2002, and the converter is now in service since late November 2002.



The new internals have been installed without particular difficulties, and the catalyst loading has revealed to be easy and quick, thanks to the internals design, achieving high densities and good uniformity.

Thanks to the presence of un-used thermocouples nozzles at the bottom of the catalyst bed, it has been possible to take samples of catalyst there, at the end of the loading, which showed that no damage at all was suffered by the catalyst because of the loading.

After loading the catalyst reduction went smoothly, and, thanks to the removal of heat operated by the plates, which was particularly easy and effective, with no peak temperatures or hot spots. During this operation the perfect uniformity of temperature achieved in the catalyst bed in all directions was already evident, indicating that the gas distribution in the plates and in the catalyst bed was optimal.

The converter went on stream without any problem, and it immediately confirmed the uniformity of temperatures in the catalyst mass, and also the docility with which it could be operated using the temperature control in the plates.

At the beginning of the operations the converter had to run with a stoichiometric number lower than the design, due to external problems, i.e. as low as 2.1 at the inlet, instead of the design value of 3.5. Nevertheless the converter could be operated without showing any tendency to hot spots formation, and allowing adapting the catalyst bed temperature profile to the new conditions.

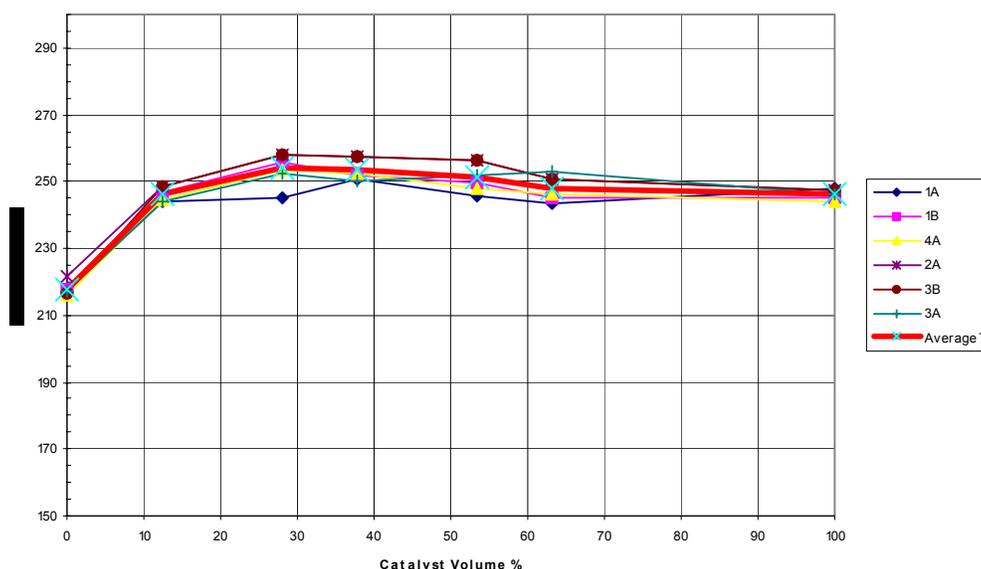
After the outside problems were fixed the converter could run as per design, and the test run was made achieving, and exceeding, the guaranteed values.

The figures achieved during test run are summarized in the following table

		<b>GUARANTEE</b>	<b>TEST RUN DATA</b>
<b>Pressure at converter outlet</b>	Bar a	46	46
<b>Production rate, refined MeOH</b>	MTD	321	357
<b>Converter pressure drop</b>	bar	1.5	1.3

The temperature control also ensures the lowest by-product formation, which was in fact well below the expected value, and this will also ensure a longer catalyst life.

The present temperature profile in the catalyst bed, at present, is the following:



As it can be seen the temperature profile is very smooth, with very little dispersion of the temperature value around the average.

The operating conditions corresponding to the above temperatures are: a load of 375 MTD of refined methanol, the stoichiometric number is of 3.34, the heat recovered in the downstream new waste heat boiler allows the production of 15.4 t/h of steam against a design of 12.2 t/h.

The catalyst volume loaded was less than 45 m<sup>3</sup>, against about 50 m<sup>3</sup> used before revamping.

The converter capacity is limited at present by the availability of synthesis gas, and could well be further increased. The peak temperature in the reactor never exceeded 265 °C, even when the gas had a 2.1 stoichiometric number..

The revamping has allowed Nevinnomysk Azot to reduce the energy consumption in the circulator by 2300 kW by reducing the circulation rate from 14 to 7, while the steam import has been reduced by 15.4 ton/h. The carbon efficiency has also increased from 81% before revamping to 93 % at present.

As it can be seen all the parameters are very satisfactory, and the overall project has been a success, allowing Nevinnomysk Azot to reach and even exceed the target of reduction in energy consumption, while the production rate has been increased already by more than 15 %, and there is plenty of room in the synthesis loop to further increase it when more synthesis gas will be available.

## 5.) METHANOL SYNTHESIS CATALYST

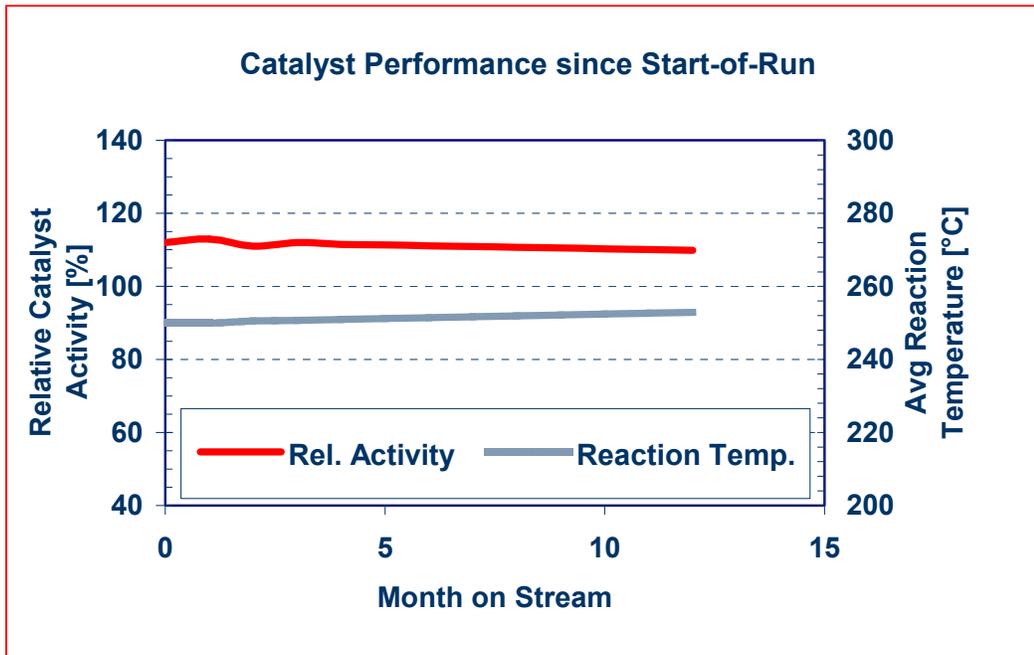
The catalyst used for this project was supplied by Sud-Chemie. The isothermal converter requires a catalyst with good low temperature activity, high stability and resistance to thermal and hydrothermal sintering.

Süd-Chemie manufactures different types of methanol synthesis catalysts to provide the best match for each application, and the catalyst applied in this case was the new C79-7GL developed to match the requirements of a CO rich synthesis gas as well as a CO<sub>2</sub> rich synthesis gas. The key is smaller copper crystallites, higher copper surface area and optimised copper dispersion. This newly developed C79-7GL methanol synthesis catalyst exhibits many technical advantages:

- > 20 % higher activity
- better low temperature activity
- long life
- low amount of impurities in crude methanol
- higher crushing strength
- low shrinkage after reduction
- high tolerance to poisons.

**5.1) Catalyst Activity**

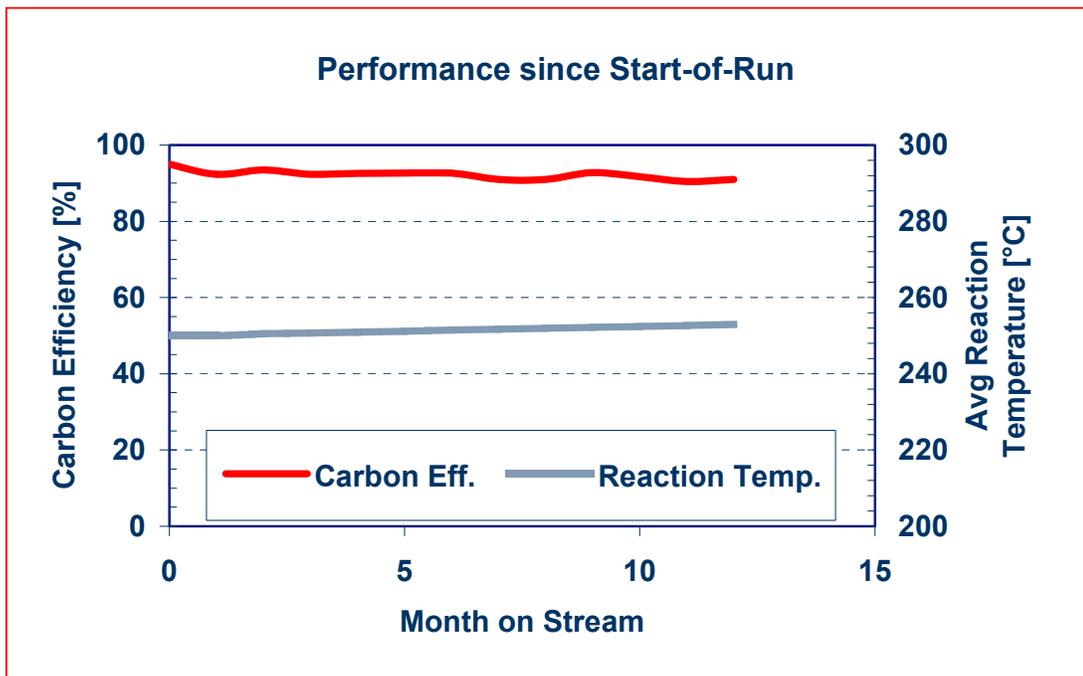
Based on performance data from the plant it turned out the actual catalyst performance exceeded the expectations. As shown in figure 1, the relative catalyst activity is more than 10 % higher than what was used when we projected the performance for the revamped converter.



**FIGURE 2 - Catalyst Performance**

During the test run, a production rate of 357 MTPD of refined methanol was achieved which also is well above the guaranteed figure of 321 MTPD.

Figure 2 below indicates carbon efficiency, which also could be greatly increased from low 80 % before the revamp to an average of 93 % after revamp.

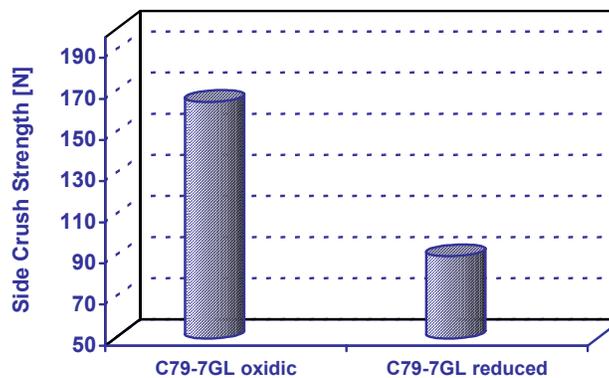


**FIGURE 3 - Carbon Efficiency**

**5.2) Pressure Drop**

Of utmost importance in an isothermal reactor is the high mechanical strength of the methanol synthesis catalyst. Figure 3 shows the crushing strength of C79-7GL in the oxidic and reduced state. Low crushing strength would lead to pressure drop increase and premature catalyst change-out.

**FIGURE 4 - Crushing Strength**



The pressure drop across the revamped methanol synthesis converter is lower than expected.