

# Methanol: synthesis loop recycle

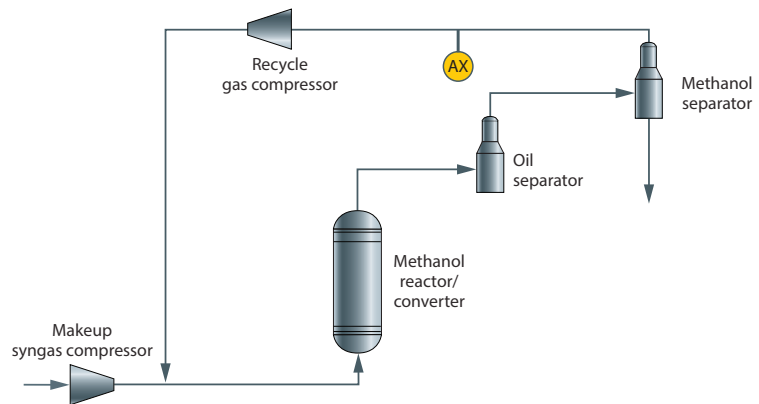


Figure 1: Typical methanol synthesis loop recycle measurement point\*

## Benefits at a glance

- Unique spectroscopic capability to measure all syngas components, including H<sub>2</sub> and N<sub>2</sub>
- Pipe-centric sampling and measurement at the sample tap
- Sample can often be returned to process, avoiding disposal to flare header
- Complete syngas speciation
- No valves, columns, or carrier gas
- No routine calibration
- No interference from moisture

The methanol synthesis loop includes the recycle stream from the methanol reactor in which unreacted H<sub>2</sub>, CO, CO<sub>2</sub> are recovered. The methanol produced in the reactor is condensed in the methanol separator and diverted to the raw methanol tank, which is purified using a single- or multi-stage distillation process, depending on the intended end use of the final product (i.e. sufficient for methanol-to-olefins (MTO) or further refined to meet AA/IMPCA specifications). The overhead from the methanol separator contains the off-gas from the reactor, which also includes small amounts of methanol vapor. The synthesis loop recycle gas is blended with the make-up syngas stream to re-establish the modulus M target in the blended stream.

## Measurement of recycled syngas

The Raman Rxn5 analyzer is a measurement solution for the recycled syngas stream. A typical Raman spectrum and stream composition for the recycle gas stream is shown in Figure 2. Note the simplicity and complete speciation and baseline separation of individual spectral peaks, including H<sub>2</sub>, N<sub>2</sub>, and CH<sub>3</sub>OH, in the Raman spectrum. As the Raman Rxn5 is essentially transparent to

moisture, any residual moisture in the sample does not interfere with the analysis as long as it does not condense. Regardless of moisture content, the analysis results are provided on a dry basis. The Raman Rxn5 also uses a normalized analysis which makes it very robust against pressure and temperature changes as well as any slow fouling that may occur.

## Reliability issues with traditional methods for recycled syngas analysis

Recycled syngas is often analyzed using process gas chromatography (GC) or mass spectrometry (MS). Both technologies require transporting and conditioning the sample at both the sample tap and at the sample conditioning panel close to the analyzer. Due to low sample pressure, it is not possible to return the sample to process, so it must be sent to flare. Protecting the GC or MS analyzers from liquid carryover after the methanol separator becomes the main sampling system challenge, as this event can damage columns in a GC or the ionization chamber in an MS. The Rxn-30 probe used by the Raman Rxn5 cannot be damaged by liquid carryover or fouling.

\* See the general Methanol: production analytics overview

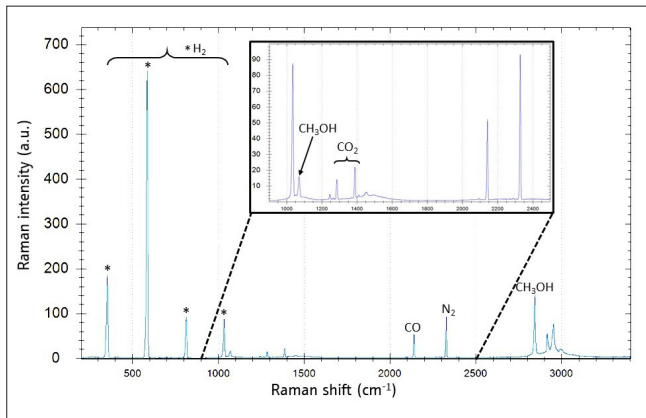


Figure 2. Raman spectrum of typical methanol recycle stream

### Solution: Raman Rxn5 analyzer with the methanol synthesis loop recycle stream method

In the case of relatively clean and dry streams like a natural gas feed, the Raman Rxn5 analyzer with an Rxn-30 probe allows for a wide range of sample pressure (70-800 psia typical) and sample temperature (-40 to 150 °C). The Rxn-30 probe can be easily integrated into sample conditioning systems to measure process streams at higher temperatures and pressures. The ability to measure at higher pressures often allows the sample to be returned to the process at a lower pressure sampling point - flaring of the returned sample is avoided. Sampling lag time is essentially zero, as no sample transport is required, increasing the speed of analysis.

The Raman Rxn5 analyzer for the methanol synthesis loop recycle stream contains the following per measurement point:

- Dedicated laser module
- Rxn-30 fiber optic probe
- Industrial hybrid electro-optical cable (up to 150 m long, customized to your plant requirements)
- Combined pressure and temperature sensor with cable (up to 150 m long, customized to your plant requirements)
- Dedicated methanol synthesis loop recycle method

Typical process conditions	P (barg)	T (°C)
At sample tap	39	40
At Rxn-30 probe	39	55

Typical stream composition					
Component	Range (Mol%)	Normal (Mol%)	Precision (Mol%) k=2	Cal gas (Mol%)	Precision (Mol%) k=2
Hydrogen	35-65	51.1	0.03	50	0.03
Nitrogen	15-35	26	0.03	26	0.03
Carbon monoxide	5-20	7.7	0.01	8	0.02
Carbon dioxide	0-10	4	0.02	4	0.02
Methane	0-2	0.3	0.01	1	0.01
Methanol	5-20	10.9	0.01	11	0.01

Table 1: Typical process conditions and stream composition

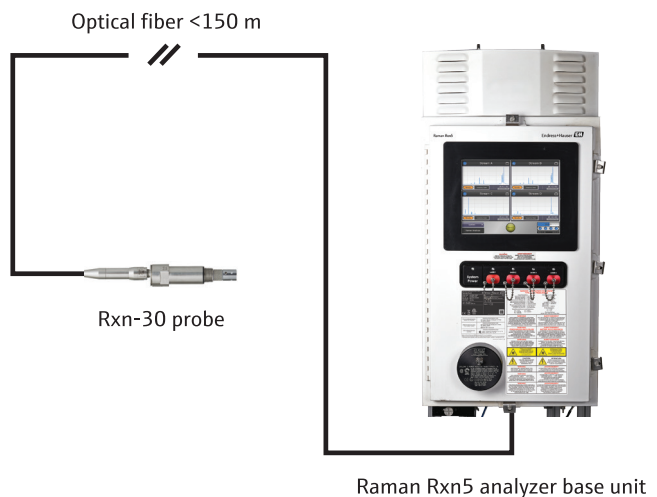


Figure 3: Recommended system configuration