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VOC reduction at Raffineria di Milazzo's SRU complex–Part 1

Based on new environmental regulations focusing on volatile organic compounds (VOCs) at the emissions point of a refinery's sulfur recovery unit (SRU), Raffineria di Milazzo–a JV between Eni and Kuwait Petroleum Italia–carried out troubleshooting activities to identify and implement suitable actions to minimize the concentration of VOCs at the emissions point of the SRU complex.

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Many definitions of VOCs exist in scientific literature and technical references. In this article, VOCs refer to the sum of the contributions of methane and non-methane VOCs (C_1 and C_1 +). The technological arrangement of Raffineria di Milazzo's SRU complex ensures high sulfur recovery performance.

This article shares a troubleshooting case study (analysis and related solutions) as a support reference when facing similar environmental topics.

Raffineria di Milazzo's SRU complex

Raffineria di Milazzo's sulfur complex arrangement consists of three SRUs. Each SRU includes a Claus section (with two catalytic stages), a tail gas treatment section [utilizing the Shell Offgas Treating (SCOT) process or a derivatives technology] and a final conversion section (incinerators). The incinerators' tail gas outlets are combined into a common stack.

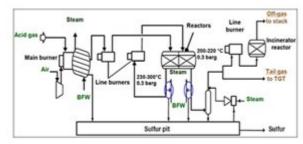
Theoretical analyses, combined with analytical activities on each SRU section, enabled refinery personnel to identify the key factors of VOCs present in the complex. Part 1 of this article details troubleshooting pathways for reducing VOCs. This includes identifying inlet streams that have a relevant impact on VOC content at the emissions point. The removal of VOCs from these streams has reduced VOC content by an order of magnitude.

Part 2 of this article (to be published in the April issue) will provide the steps taken to reach this order of magnitude reduction in VOCs (the VOC target achieved from these troubleshooting techniques was $< 5 \text{ mg/Nm}^3$).

Products from oil and gas processing (such as naphtha, gasoil and LPG) must be desulfurized to minimize sulfur oxide (SOx) emissions from internal combustion engines. Different refinery processes (including sweeting and hydrotreating, among others) have been developed to achieve more restrictive limits on residual sulfur in refined products. The extracted sulfur from refined products must be recovered as elemental sulfur. This is possible through the Claus process (and the tail gas treatment processes associated with it), where 99.9% of the sulfur is retrieved in liquid form, which is then used in the chemical industry (e.g., sulfuric acid production and the vulcanization of tires) or farming (especially as fertilizer).

The sulfur recovery process

The Claus process consists of partially combusting the hydrogen sulfide (H_2S) -rich gas stream (with one-third of the stoichiometric quantity of air) and then reacting the resulting sulfur dioxide (SO_2) and unburnt H_2S in the presence of an activated alumina catalyst to produce elemental sulfur. As shown in **FIG. 1**, the Claus unit consists of a reaction furnace, followed by a series of converters and condensers, where:



(/media/18219/falzone-fig-01.jpg)

FIG. 1. Simplified process flow diagram of an SRU (Claus process).¹

- The partial combustion and a part of the sulfur formation (2H₂S + 2O₂--> SO₂ + S + 2H₂O) occurs in the primary reactor.
- The main Claus reaction for sulfur formation (2H₂S + SO₂--> 3S + 2H₂O) takes place in the different catalytic converters.
- Liquid elementary sulfur is collected from the various condensers in a common pit.

Side reaction effects also occur that produce carbonyl sulfide (COS) and carbon disulfide (CS_2), which can create problems in the operations of Claus plants, since they cannot be easily converted to elemental sulfur and carbon dioxide (CO_2).¹ The high water content and temperatures involved should be adequate to hydrolyze both COS and CS_2 ; however, these slow reactions are generally quenched in a waste heat exchanger before completion–higher temperatures and longer residence times aid the hydrolysis of these species. In split-flow plants, maximizing the bypass fraction (toward the absolute maximum of two-thirds) will provide improvements in both residence time and temperature. The same effect can be obtained with the use of oxygen enrichment technologies (e.g., with the OxyClaus process) or by reducing the feedrate to the plant. However, it is possible to provide the first Claus catalytic converter with a layer of catalyst [based on titanium dioxide (TiO_2)] to maximize the hydrolysis of COS and CS_2 , as shown in the following reactions:

 $COS + H_2O --> CO_2 + H_2S$ $CS_2 + 2H_2O --> CO_2 + 2H_2S$

Since these hydrolysis reactions only occur to a sufficient degree at temperature above 315°C (600°F) and the only catalyst bed that can be operated at such a high temperature is the first, this is, therefore, the only chance to hydrolyze the COS and CS_2 .²

The SRU is characterized by its sulfur recovery efficiency, which is calculated as the fraction of sulfur in the feed that is recovered as a liquid sulfur stream and routed to the sulfur collection pits.

The sulfur recovery efficiency of an SRU closely depends on the number of Claus reactors that are operated in series in the SRU chain, as shown in **TABLE 1**. As illustrated in this table, the sulfur recovery efficiency can reach 98% through the Claus process. To reach a higher sulfur recovery efficiency (theoretically to 99.99%), a tail gas treatment unit (TGTU) or cleanup procedure must be associated with the Claus process. Many processes for TGTUs have been developed to enhance the recovery of sulfur compounds from natural gas and/or refinery sources. According to the principles applied, the most frequently operated TGTU processes can be broadly divided into the following four categories:

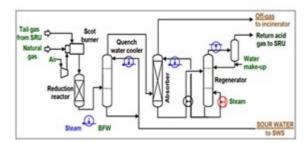
No. of Claus reactors	No. of datasets	Sulfur recovery efficiency, % H ₂ S recovered		
		Observed range	Median	Typical
1	N/A	N/A	N/A	90
2	87	92.4-97.8	963	95-96.5
3	27	96-98.4	97	96.5-98

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- Direct oxidation to sulfur (such as through a PROClaus or SUPERCLAUS process)
- Continuation of the Claus reaction (such as through cold bed adsorption or through Clauspol, Sulfreen or Maxisulf processes)
- Reduction to H2S and then recovery of sulfur from it [utilizing the SCOT process, high Claus ratio (HCR), FLEXSORB technology, reduction-absorption-recycle (RAR), a bacterial sulfate reduction (BSR) process, the LO-CAT sulfur recovery solution, or the Mobil Oil Direct Oxidation Process (MODOP)]
- Oxidation to SO2 and then recovery of sulfur from it (using the Wellman-Lord or LabSorb processes).

In this article, the authors will focus on the SCOT process and its derivative HCR process, since these are the only two technologies associated with the Claus units at the Raffineria di Milazzo.

As shown in **FIG. 2**, the concepts underlying the H2S scrubbing processes in the SCOT unit are:



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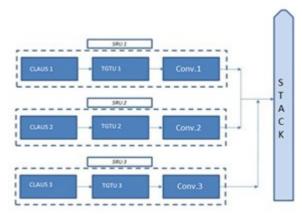
FIG. 2. Simplified process flow diagram of a tail-gas SCOT unit.¹

- Hydrogenation and hydrolysis of all sulfur compounds to H2S by passing them through a cobaltmolybdenum catalyst at 300°C (600°F) with the addition of a reducing gas
- Absorption of H2S by an amine solution (either a generic amine or a specialty amine)
- Regeneration of the amine solution and the recycling of H2S to the upfront Claus reaction furnace.

The clean tail gas (outlet of the amine adsorber) reaches an incinerator section (as illustrated in **FIGS**. **1** and **2**) where the residual H2S is converted to SO2 before the emissions point. The incinerator section can be catalytic or thermal. In other words, the catalytic incinerator consists of a heater [to heat the clean tail gas to the proper reaction temperature of $280^{\circ}C-350^{\circ}C$ ($536^{\circ}F-662^{\circ}F$)] and a fixed-bed reactor where H2S is selectively oxidated into SO2. In a thermal incinerator, the tail gas is heated to a very high temperature [> 720°C (> 1,328°F)] in oxidant conditions (proper air excess) to guarantee a complete conversion of the residual H2S to SO2.

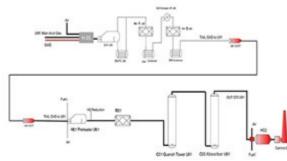
Sulfur recovery at Raffineria di Milazzo

Three different SRUs are dedicated to sulfur recovery at Raffineria di Milazzo. Each unit comprises a Claus section, a TGTU section and a final conversion section (**FIG. 3**). Every Claus section has two catalytic Claus reactors. The TGTU sections utilize the SCOT process (SRU1 and SRU2) and HCR (SRU3). The final conversion section includes catalytic incinerators for SRU2 and SRU3, along with a thermal incinerator for SRU1. **FIGS. 4**, **5** and **6** show the process flow diagrams of SRU1, SRU2 and SRU3, respectively.

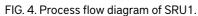


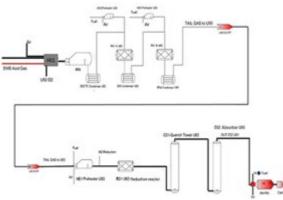
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FIG. 3. Block diagram of Raffineria di Milazzo's SRU complex.



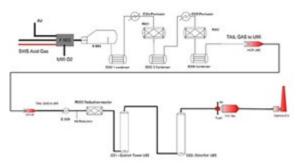
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(/media/18224/falzone-fig-05.jpg)

FIG. 5. Process flow diagram of SRU2.



(/media/18225/falzone-fig-06.jpg)

FIG. 6. Process flow diagram of SRU3.

The main differences between the three SRUs are:

- 1. **Claus thermal section:** In SRU1 and SRU2, only the once-through arrangement can be adopted. In SRU2 and SRU3, the oxygen enrichment arrangement can be employed, while the split-flow configuration can be used in SRU3.
- 2. **Catalytic Claus preheating:** In SRU1, a partial bypass of the first condenser ensures a proper inlet temperature in the first Claus reactor. A steam heat exchanger is employed to warm up the stream at the inlet of the second Claus reactor. In SRU2, two furnaces preheat the process stream for the first and second Claus reactors. In SRU3, two steam heat exchangers are used to heat the process stream before entering the Claus reactor inlet.
- 3. **Reduction reactor preheating:** Both the SRU1 and SRU2 tail gas streams are heated in a furnace before the inlet of the TGTU reduction reactor. In SRU3, the tail gas heating is performed with a steam heat exchanger.
- 4. **TGTU H2S adsorbing column:** In both TGTU1 and TGTU2, the H₂S absorbers are a trayed column with a head gas-liquid separator. TGTU3's H₂S adsorber is a packed-bed column without a head gas-liquid separator (a demister was installed prior to the outlet of cleaned gas).
- 5. **Final conversion section:** SRU2 and SRU3 both contain a catalytic incinerator. In SRU1, a thermal incinerator has been installed.

TROUBLESHOOTING ACTIVITIES

According to European Commission (EC) Directive 1999/13/EC (Solvent Emissions Directive), VOCs are functionally defined as organic compounds having, at 293.15 K (20°C), a vapor pressure of 0.01 kPa or more, or having a corresponding volatility under conditions of use.

The aim of this work is to identify the sources of VOCs at Raffineria di Milazzo and to minimize their concentrations in the SRU complex's stack to adhere to new environmental constraints (i.e., $20 \text{ mgC}_{eq}/\text{Nm}^3$).

However, it is important to specify that the design of each SRU plant aims to maximize the sulfur recovery yield (see the "Sulfur recovery process" section in this article). In the design of the SRU complex, minimizing the concentration of VOCs at the outlets of the SRUs was not considered. For this reason, a preliminary theoretical analysis to identify all possible sources of VOCs was carried out, followed by several analytical activities.

Theoretical considerations

The first step was to identify all possible sources of VOCs in the refinery's SRU complex. Analysis considerations included:

- The contamination of feedstock: Acid gas to the SRUs comes from the amine regeneration section and sour water stripping (SWS) offgas. A physiological absorption (especially in amine treating units) and stripping of light hydrocarbons are inevitable. In the thermal reactor, if the VOCs are not completely oxidated, a breakthrough occurs in the downstream sections of the SRUs.
- 2. The contamination of the hydrogen stream in TGTUs: A hydrogen stream is fed to the inlet of the reduction reactor at the TGTUs. This stream guarantees a proper concentration of hydrogen (3 mol%–5 mol%) inside the reactor for all operative conditions (e.g., standard operations, startup phases, shutdown phases). In TGTU3, this stream is high-purity hydrogen (> 99.5 mol%) and comes from the refinery's hydrogen network [the outlet of pressure swing adsorption (PSA) units]. Conversely, in TGTU1 and TGTU2, the hydrogen stream comes from the catalytic reforming unit. This stream has a variable hydrogen concentration of 58%–82%, with light hydrocarbons (mainly C1–C3) as the remainder. Therefore, this stream can be considered as a contributor of VOCs at the inlet of the SRU complex.
- 3. **Online preheating furnaces at the Claus section:** In SRU2, the two furnaces are used for preheating the gas before it enters the Claus catalytic reactors. The fuel employed is refinery fuel gas. These furnaces work at stoichiometric conditions (to avoid excess air to the catalytic Claus reactors), so fuel composition fluctuations could result in incomplete fuel combustion and a slip of VOCs.
- 4. **Online preheating furnaces in the TGTU section:** As described in the "Sulfur recovery at Raffineria di Milazzo" section of this article, in TGTU1 and TGTU2, a furnace is used to heat the tail gas before it reaches the reduction reactor inlet. The fuel employed here is refinery fuel gas. These furnaces work at sub-stoichiometric conditions to avoid oxygen breakthrough in the reduction reactor. It is important to ensure that no VOC slip occurs at the furnaces' burners.
- 5. **Online preheating furnaces at the final conversion section:** In the final conversion section, a furnace is used to heat the tail gas to a proper temperature for the catalytic incinerators (in SRU2 and SRU3) or to direct oxidation in the thermal incinerator (SRU1). These furnaces work in excess air conditions, so a VOC slip should be unlikely. However, a combustion check must be conducted.
- 6. **TGTU amine adsorber section:** In TGTUs, an amine absorbing column is used to reduce the H_2S concentration in the tail gas before the final conversion section. The amine circuit (absorbers and regeneration section) can suffer hydrocarbon contamination. During absorbing phases, hydrocarbons can be transferred in the amine solution. If they are not stripped during the regeneration phases, they are released in the absorption column and exit as a gas. Another phenomenon that can occur in the amine absorption column is amine solution carryover. If, in the gas outlet section, there is not efficient gas-liquid separation, the amine is transported by the gas to the incinerator, where it decomposes into ammonia and light hydrocarbons (VOCs).
- 7. **VOC generation reactions:** All reaction sections must be analyzed to eliminate the possible generation of VOCs due to chemical reactions.

Analytical troubleshooting activities

The theoretical analysis conducted identified the primary possible sources of VOCs, which had to be investigated through a field measurement. The following is a brief description of the analytical setup used, along with the troubleshooting steps.

Description of the analytical setup for the detection of VOCs. UNI EN 12619:2013 was used to measure the concentration of VOCs (as total organic carbon) in the streams of Raffineria di Milazzo's SRU complex. This method determines the mass concentration of total gaseous organic carbon through a flame ionization detector (FID). The measuring principle of the FID is summarized in **FIG. 7**.

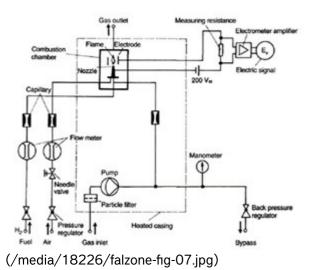


FIG. 7. Diagram of an FID.⁴

The hydrogen flame burns out of a metal nozzle, which simultaneously represents the negative electrode of an ionization chamber. The positive counter-electrode is fixed above the flame. Direct voltage is applied between the two electrodes. The ion current is measured as a voltage drop above the resistor. The measuring gas is added to the burning gas shortly before entering the burner nozzle. The air required for combustion flows in through a ring slot around the burner nozzle.

For stable measuring conditions, it is essential that all gases–combustion gas, combustion air and measuring gas–are conducted into the flame in a constant volume flow. For this, all gas flows are conducted via capillaries. Constant pressure before the capillaries ensures a constant flow. Sensitive pressure regulators for gas combustion and air are used to achieve this fine-tuning. The measuring gas is pumped past the capillary in the bypass in a high-volume flow. Pressure is kept constant by the backpressure regulator so that a constant partial flow reaches the flame via the capillary. To avoid condensation of the hydrocarbons to be measured, all instruments must be heated to 150°C–200°C (65.5°F–93°F). Heating includes the particle filter and the measuring gas pump. In most cases, particularly with cold exhaust gases, a heated sampling line is also used for measuring gas sampling to the measuring instrument.

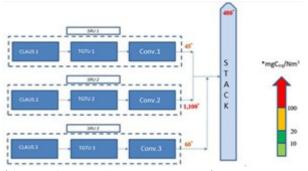
Hydrocarbon compounds are oxidized in the flame, with ions being formed as an intermediate product. In a certain range of the accelerating voltage, the strength of the ionization current is in first approximation directly proportional to the amount of carbon atoms of the burned substance. Therefore, an FID basically responds to all hydrocarbons and measures their total sum. Corresponding to the number of carbon atoms, larger molecules with many carbon atoms produce a higher signal than smaller molecules with a small number of carbon atoms. Ionization energy does not only stem from the flame's energy, but primarily from the oxidation energy of the carbon. Accordingly, partially oxidized hydrocarbons provide a weak detector signal, and completely oxidized hydrocarbons provide no signal at all (e.g., CO and CO2 are not detected).⁵

The FID analyzer employed for this work was equipped with an oxidation catalyst to oxidate all hydrocarbon species, excluding methane. In this way, it was possible to switch the gas sample to the catalyst to also include a methane concentration measurement. It is worth noting that the H2S concentration was not negligible in many of the analyzed streams. In these cases, an additional caustic solution neutralization step was necessary to avoid possible instrumentation damage.

Analytical troubleshooting: Starting point. During troubleshooting, several VOC concentration measurements were carried out through the analytical setup described above. First, a VOC concentration was measured at the outlet of the SRUs and the stack to determine if some differences could be detected. Second, the refinery's SRU complex was divided into clusters, and the VOC

concentrations were measured at the inlet and outlet of the clusters to identify the possible VOC sources. For each source identified, a proper action was implemented to minimize or eliminate the source.

FIG. 8 reports VOC concentration as mgCeq/Nm3 at the outlet of each final conversion section and at the stack before starting troubleshooting activities. The measurement of the concentration of VOCs was corrected to the reference condition of 3% O2, according to UNI EN 12619:2013. **TABLE 2** summarizes the configuration of the refinery's SRU complex at this starting point.



(/media/18227/falzone-fig-08.jpg)

FIG. 8. Concentration of VOCs at the outlet of the SRUs and the stack at the starting point.

Unit	Split-flow configuration	O ₃ enrichment amangement	SWS offgas feed	Load (based on liquid sulfur production)
SRUT	-		No	55%
SRU2	-	Yes	Yes	65N
SRU3	No	Yes	Yes	60%

(/media/18229/falzone-table-02.jpg)

Some considerations can be deducted from this starting point. These included the following:

- 1. **The highest value of VOC concentration is at the outlet of SRU2:** This was expected because, theoretically, SRU2 had the largest number of possible VOC sources (see the "Theoretical considerations" section).
- 2. **The lowest value of VOC concentration is at the outlet of SRU1:** The presence of a thermal incinerator should ensure the removal of many VOCs. However, a relevant concentration of VOCs was measured, so contamination sources had to be checked (see the "Theoretical considerations" section of this article).
- 3. Although theoretically possible, VOC sources are lowest in SRU3: A relevant concentration value was found at the outlet stream of the final conversion section.

Ultimately, the VOC concentration in any outlet stream of the SRUs does not reach the expected target of < 20 mgCeq/Nm3, and the VOC concentration at the stack is well above it.

Analytical troubleshooting Step 1: Contamination of the hydrogen stream in the TGTUs. In the "Theoretical considerations" section, the contamination due to the hydrogen stream feed at the inlet of the reduction reactor was highlighted. For TGTU1 and TGTU2, the hydrogen stream comes from the catalytic reforming unit. The composition of this stream varies with the catalyst's lifecycle (**TABLE 3**). In particular, the hydrogen concentration decreases during the catalyst's lifecycle, while the hydrocarbon concentration increases. Consequently, the hydrogen stream to TGTU1 and TGTU2 must increase during the reforming catalyst's lifecycle to ensure a constant hydrogen concentration at the reduction reactor. The hydrogen stream is a definite source of VOCs, but it also increases with the reforming catalyst's lifecycle.

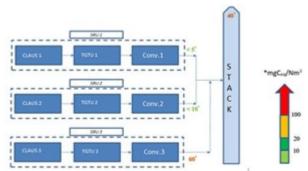
Compound	Start of run, vol%	End of run, vol%	Starting point, vol%
Hydrogen	82.0	58.76	78.17
Methane	939	19.38	10.33
Ethane	5.08	11.75	6.39
Propane	2.41	6.81	3.18
i-Butane	0.61	1.58	0.78
n-Butane	0.42	1.43	0.75
i-Pentane	0.13	0.46	0.26
n-Pentane	0.05	0.23	0.14

(/media/18234/falzone-table-03.jpg)

Considering the catalytic reforming hydrogen composition at the starting point and its rate to TGTU2 (15 kg/hr-20 kg/hr), it is easy to calculate the theoretical hydrocarbon contamination at SRU2 due to this source: 12 kg/hr-16 kg/hr. Neither the reduction TGTU2 reactor nor the catalytic incinerator can guarantee hydrocarbon oxidation. Therefore, most of the hydrocarbons entering with the hydrogen reduction stream will reach the outlet of SRU2. Considering that the SRU2 outlet volumetric rate at the starting point was 10,000 Nm3/hr, it is easy to estimate the expected VOC concentration at the SRU2 outlet (Eq. 1):

 $X_{VOCs,SRU2}$ =(12/10,000) x 10⁶ (mg/Nm³) ≈1,200 (mg/Nm³) (Eq. 1)

The order of magnitude obtained from the estimation through Eq. 1 is the same as the value measured with the FID (**FIG. 8**). Consequently, a new system to feed a high-purity hydrogen stream (99.5% mol) was built in place of the catalytic reforming hydrogen stream for SRU1 and SRU2. In this way, the refinery's SRUs employ a high-purity hydrogen reduction stream for the TGTUs. **FIG. 9** shows the VOC concentration after Step 1 was completed. This included:



(/media/18228/falzone-fig-09.jpg)

FIG. 9. VOC concentration at the outlet of the SRUs and the stack after Step 1.

- A reduction in VOC concentration by an order of two magnitude at the SRU2 outlet stream. From the theoretical consideration and calculation, the catalytic reforming hydrogen stream was identified as the "main cause" for the VOC contamination in this unit.
- Through these analytical results, the other possible sources for SRU2 have been excluded. Regarding the first bullet point in the "theoretical considerations" section of this article, some measurements were carried out at the Claus section, and no VOCs were detected at the outlet of the thermal reactor–this is in accordance with literature.2 In addition, the burning of the furnaces (refer to bullet points 3–5 in the "Theoretical considerations" section) has been verified, and no hydrocarbon slip was found.
- A reduction in the VOC concentration by an order of one magnitude at the SRU1 outlet stream. Also, in this unit, the primary contributor of VOCs at the outlet stream was the catalytic reforming hydrogen stream. As previously mentioned, the SRU1 final conversion unit comprises a thermal incinerator where high temperatures [720°C-750°C (1,328°F-1,382°F)] are reached and oxidant conditions (3% O2-9% O2) are present. A thermal incinerator produces a non-selective oxidation, so it oxidates all the compounds that can take part in these reactions. However, the design of the thermal incinerator was carried out to oxidate the H2S slip from TGTU1 and not for the elimination

of VOCs. Therefore, the complete elimination of VOCs did not occur. It was necessary to replace the catalytic reforming hydrogen stream with a pure hydrogen stream in TGTU1 to reach an acceptable reduction of VOCs in the unit's outlet stream.

A reduction of one order of magnitude of the VOC concentration at the stack was achieved. However, it did not represent an acceptable value (the target was < 20 mgCeq/Nm3). For this reason, further investigation into SRU3 was necessary. Although this unit has the lowest number of theoretical VOC sources, its outlet stream had the highest VOC concentration.

Takeaways

Troubleshooting was performed to reduce VOCs at Raffineria di Milazzo's SRU complex's stack. A theoretical analysis to identify VOC sources in each SRU was conducted, and a series of measurements at different SRU streams was carried out to detect the most relevant ones. This analysis was carried out through a proper analytical setup based on FID.

Initially, the stack's VOC concentration was 480 mgCeq/Nm3, with different contributions by each SRU. The highest VOC concentration was detected at the SRU2 outlet stream (1,100 mgCeq/Nm3)–a hydrogen stream from the catalytic reforming unit was fed to TGTU1's and TGTU2's reduction reactors. A relevant VOC rate entered the system due to the low hydrogen purity of this stream (58 mol%–82 mol%). The existing stream was replaced with a high-purity hydrogen stream (> 99.5 mol%), which led to a reduction in VOC concentration by an order of magnitude of one at the outlet streams of SRU1 and SRU2 (< 5 mgCeq/Nm3 and < 10 mgCeq/Nm3, respectively), and, in turn, at the stack (40 mgCeq/Nm3).

Part 2 of this article will appear in the April issue of Hydrocarbon Processing. HP

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