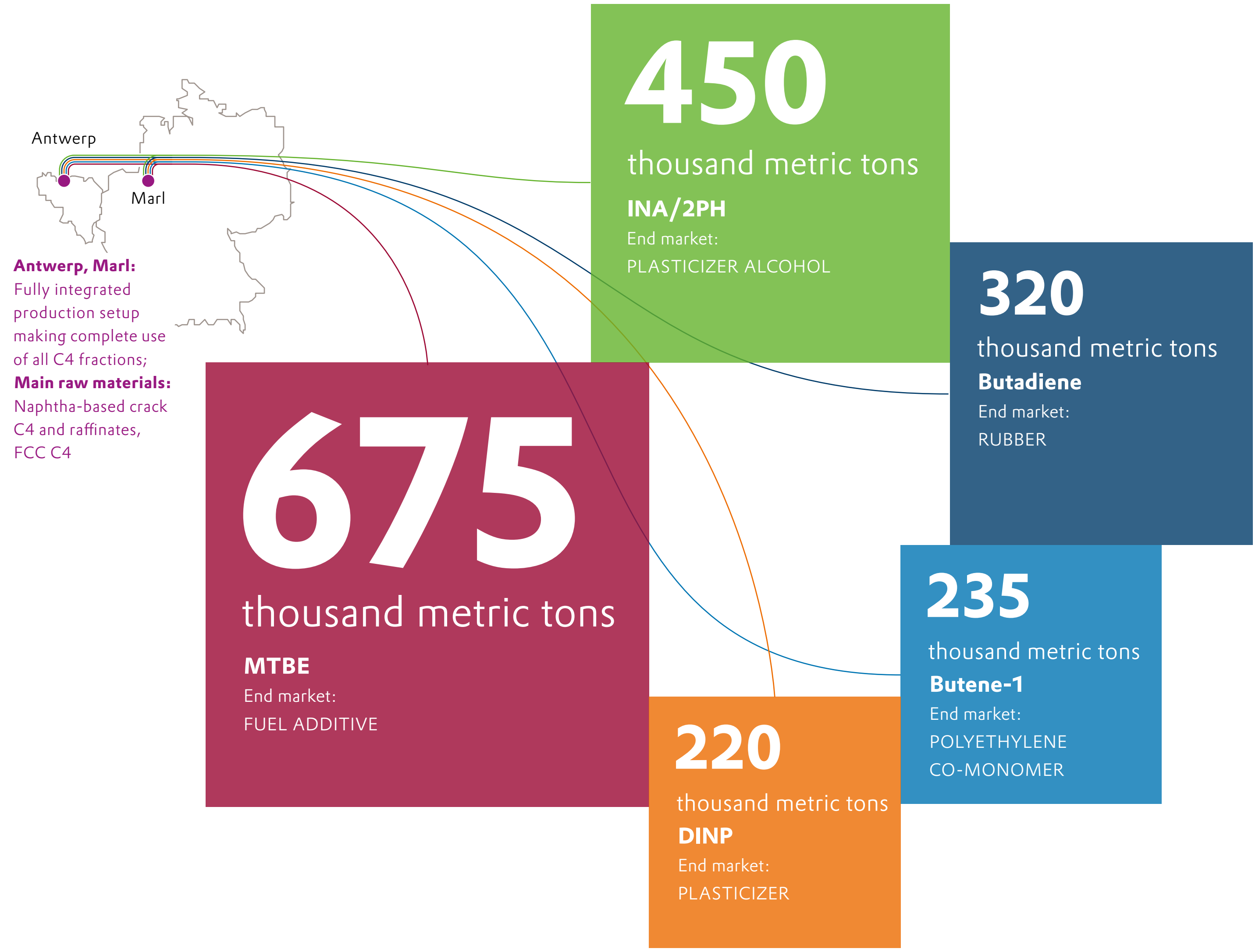

THE NEW C4 FORMULA

Evonik's Performance Intermediates Business Line produces thousands of metric tons of in-demand industrial chemicals every day. Thanks to a special purification process, the Group is the only company worldwide that can use a very special material—FCC-C4—from refineries as feedstock for this production. The researchers who developed the process have now been awarded the Evonik Innovation Prize for their work

TEXT **FRANK FRICK**

The new FCC-C4 purification plant in the Marl Chemical Park

A substantial part of production in Marl, Evonik's largest location, is based on the formula C4. It stands for liquid gases, the molecules of which contain four carbon atoms. These liquid gases are refined more and more, via a series of steps, at the C4 facilities in Marl and similarly at the Antwerp location. The products of these processes include MTBE (methyl tert-butyl ether), which is used as an antiknock additive to increase the octane value of gasoline and thus improve combustion, DINP (diisononyl phthalate), a plasticizer that turns brittle PVC into a flexible material, and butadiene, which is processed into synthetic rubber for car tires. All of the C4 facilities are connected closely together by material and energy flows. Even the byproducts of a process can be used as raw materials in other facilities, and the waste heat from one process step can supply the energy needed for another.

For decades, the production network in Marl has been using crude C4—an unprocessed stream of C4 from petrochemical plants that thermally decompose ("crack") crude oil fractions in steam or naphta crackers. The petrochemical industry uses these methods to obtain chemicals such as ethylene and propylene, as well as the crude C4 stream.

VALUED RAW MATERIAL

But the supply of crude C4 is not infinite. Thanks to the growth of the market for the secondary products, more and more competitors are taking an interest in this raw material flow. At the same time, the number of steam crackers in Europe has stagnated—as has the supply of crude C4.

Oil and gas extraction from non-conventional sources by means of fracking processes is booming in the USA, but the material extracted has a different composition from that of traditional crude oil. As a result, if the fracking material is then processed in crackers, the result is smaller quantities of lower-quality crude C4.

Increasing demand for C4-based industrial chemicals but stagnating availability of the raw material stream—Performance Intermediates recognized this dilemma ten years ago and started watching out for alternative C4 raw materials. During this time, the Business Line came across fluid catalytic cracking (FCC), a process used by refineries to catalytically crack high-boiling petroleum crude fractions into fuel components. As in a steam cracker, the resulting material stream is mainly made up of compounds containing four carbon atoms.

But there is one substantial difference in the composition: Crude C4 contains a maximum of five percent n-butane and isobutane, while FCC-C4 contains more than 25 percent of these compounds. Because these two alkanes contain exclusively single bonds, they are relatively unreactive and are difficult to process into valuable chemicals. "That would be a clear disadvantage of FCC-C4 compared to the established crude C4 as a feedstock for the Marl production network," explains Dr. Markus Winterberg, who was the project leader at Process Development and later took on responsibility for its implementation.

POISON FOR THE CATALYSTS

A further negative aspect of the FCC-C4 stream quickly became apparent in the laboratory. It poisons the catalysts used in the plants of the Marl network. That means the catalysts, which are essential for the highly efficient and economic functioning of the production processes, would become less effective within a matter of hours.

The analytical experts from Evonik had tested new detectors and developed appropriate methods of gas chromatography that use →

Dr. Markus Winterberg
was the project leader at Process Development

Dr. Stephan Peitz
was responsible for carrying out the laboratory experiments

a different operating principle than the standard methods. After that approach was used, the reason for poor catalyst performance became clear. The FCC-C4 mixture contained more than 50 unwanted nitrogen and sulfur components. The new detector for nitrogen compounds is so sensitive that it can measure concentrations as low as 50 ppb (parts per billion)—that corresponds to 50 grams of nitrogen compounds in 1,000 metric tons of FCC-C4 mixture.

That laid the foundation for developing a concept to separate these components. Firstly, the boiling points and other thermodynamic properties of all of the components were determined. The data obtained about the material properties was fed into computer simulations. That made it possible to investigate which unwanted components could be relatively easily separated from the FCC-C4 flow by means of distillation.

SIMULATING PRODUCTION PROCESSES

"In the next step, we concentrated on the remaining components, which we couldn't remove by means of distillation," explains Dr. Stephan Peitz, who was responsible for carrying out the laboratory experiments as part of the process development. This step took place in experimental reactors with a maximum length of three meters and a maximum diameter of three centimeters.

The researchers fed synthetic streams of C4 into these experimental reactors—synthetic because they had added varying amounts of the impurities that they had previously discovered in the FCC-C4. "These contaminated streams of C4 enabled us to simulate and test important processes from the production network to determine how the impurities affect the catalysts and processes," says Peitz. In this way, the researchers collected information about whether and in what amounts undesired components are tolerated by the production network.

IN-HOUSE DEVELOPMENT INSTEAD OF SOLUTIONS OFF THE PEG

Sample analyses, computer simulations, and investigations of the catalytic processes were carried out in parallel and iteratively, rather than in series. Otherwise the development of an effective and economic purification process for the FCC-C4 stream within a few years would not have been possible. The original idea, to buy in an industrial-scale purification process, soon had to be given up. The purification processes available on the market either failed to adequately separate the catalyst poisons, or they were uneconomical because they used too much energy or produced too much waste.

And that's why the researchers from Performance Intermediates worked together with colleagues from Process Technology, Engineering, and Production to develop an in-house process. Its most obvious feature—and now its trademark—is the 90-meter-high distillation column in the Marl Chemical Park. The column separates isobutane and other volatile components from the FCC-C4 stream. Another process part removes sulfur-bearing impurities by chemically transforming them into higher-boiling components that can then also be removed by distillation. Additional purification stages are based on adsorption beds—large-area layers of materials that bind the catalyst poisons.

The complete purification plant went into operation in 2015 and since then has been processing several hundred metric tons of FCC-C4 per day. "Evonik is thus the only chemical company capable of utilizing this C4 stream as a feedstock for the production of specialty chemicals," says Winterberg. For this achievement, the development team was awarded the Group's own Innovation Prize at the end of 2017. The facility not only reduces the integrated production network's dependence on crude C4 but also enables an increase in the production capacities for the C4-based chemicals, which are in high demand. The process is so successful that the purification facility is currently being expanded. By mid-2018, Performance Intermediates intends to substantially increase the amount of FCC-C4 that can be prepared by the newly developed purification process and further consolidate its position as a technological leader.