# HOW RECYCLED PET PRODUCT QUALITY AND PRODUCT SAFETY RISKS CAN BE CONTROLLED

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## EXECUTIVE SUMMARY

The importance of recycled Polyethylene Terephthalate (PET) is growing strongly. In Europe, the total demand for recycled PET (rPET) is steadily increasing, from about 31% in 2015 (1,110 kton/year) to about 43% by 2022 (1,520 kton/year). During the stages of PET production, product use and recycling, the PET product quality can deteriorate significantly by:

- Thermal degradation that affects the mechanical and chemical performance.
- The presence of solid contaminants.
- The presence of substances that affect the taste (limonene) and hazardous substances (benzene).
- Discolouration that affects the PET product appearance in visible applications.

Therefore, cost-effective processing and obtaining the right and safe rPET quality is essential for PET recyclers, PET packaging manufacturers and brand owners. This paper deals with insights into how to control the right rPET quality and improve product safety risks. This can be done by the right set of chemical analysis techniques. The purpose of doing analyses is to create more insights with respect to the company's business needs, cost savings and the regulations. Over 65 years, the laboratory of EMMTEC services (EMMTEC) has provided essential information to meet these business needs an d the regulation requirements fast & efficiently. This paper explains in-depth how:

- Substances such as limonene, benzene & acetaldehyde are frequently present in rPET. These substances can be detected by Headspace Gas Chromatography (GC) techniques. Typical limonene, benzene & acetaldehyde concentrations in rPET detected by EMMTEC are 0.1, 0.05 and 0.5 mg/kg (ppm), respectively.
- The presence of solid particle contaminants arising in rPET can be detected by the unique EMMTEC Partisol analysis. Typically, EMMTEC detects about 10 to 11 times more particle contaminants in rPET compared to virgin PET.
- The polymer quality can be controlled by Intrinsic Viscosity (IV) analysis. Typical IVs for rPET bottle flakes measured by EMMTEC range from 0.50-0.80 dL/g.
- rPET becomes greyer and/or yellower and can be detected by colour measurements.

These analysis techniques can reveal vital information for PET recyclers, packaging manufacturers and brand owners, such as about:

- Monitoring and optimising the quality of polymer products in relation to the applied PET recycling and packaging processing conditions.
- Assessing, monitoring, and optimising the concentrations of (potential) hazardous and substances that affect the taste in rPET.
- Cost savings by selecting the right rPET batches that minimise waste during production.
- Minimising the presence of particle contaminants, resulting in less haziness in transparent (amorphous) PET or in less waste during stretching in foil production.

## INTRODUCTION: BACKGROUND INFORMATION ON POLYETHYLENE TEREPHTHALATE (PET)

Polyethylene Terephthalate is the chemical name for a polymer better known as "PET" or polyester. It is a bulk polymer that can be created by the polycondensation of Terephthalic Acid (TPA) and Ethylene Glycol (EG). PET can also be polymerised by the polycondensation of Dimethyl Terephthalic (DMT) and EG. This polymer was first patented in 1941 by DuPont Du Nemours (USA). Polyester or PET is known by different trade names such as Mylar, Diolen, Decron, Lavsan, Terylene and Recron. PET is mainly applied in packaging and textiles. The majority of the world's PET production is for synthetic fibers (in excess of 60%), with bottle production accounting for about 30% of global demand [1].

### OVERVIEW OF PET PROPERTIES

PET is basically a semi-crystalline polymer with a glass transition temperature of 70°C and a high melting temperature of 250°C. The stiff polymer chains in the PET polymer imparts high mechanical strength, toughness and fatigue resistance up to 150-175°C as well as good chemical, hydrolytic and solvent resistance. The PET microstructure can be modified: the addition of (purified) isophthalic acid (PIA or IPA) suppresses the crystalline solidification behaviour and clear, amorphous PET can be obtained. Amorphous PET is widely used in transparent bottle and tray packaging applications. An important property to characterise the degree of polymerisation of PET is the intrinsic viscosity (IV). The longer the polymer chains, the more entanglements between chains and therefore the higher the IV. The average chain length of a particular batch of resin can be controlled during polycondensation or solid-state polymerisation (SSP). The PET application is highly dependent on the IV value. Table 1 provides an overview of the IV range for different applications [1].

*Table 1. Overview of IV ranges for different PET applications*

| Type of grade | Applications | IV range (dL/g) |
|---|---|---|
| Fiber grade | Textile fibers | 0.40-0.70 |
| | Industrial fibers | 0.72-0.98 |
| Film grade | BoPET (biaxially-oriented PET film) | 0.60-0.70 |
| | Sheets (for thermoforming) | 0.70-1.00 |
| Bottle grade | Water bottles | 0.70-0.78 |
| | Carbonated soft drink bottles | 0.78-0.85 |
| Monofilaments | Brushes, fishing lines, filtration sieves | 1.00-2.00 |

The IV is an important PET property to control the processing conditions of the recycling or polycondensation processes or the polymer quality of the end application. Rapid IV routine testing can support PET polymer manufacturers, PET recyclers or PET packaging makers to control and adjust their raw material selection, process conditions and final product quality. As an example, typical IVs for rPET bottle flakes measured by EMMTEC range from 0.50-0.80 dg/L.

### THE IMPORTANCE OF rPET IS INCREASING SIGNIFICANTLY

The PET market is highly dynamic nowadays. Consumer awareness about plastic waste is shifting. PET collection, separation and refund schemes are gaining ground and the use of single-use disposables is being increasingly restricted by EU regulations. These trends both result in an increasing supply and demand volumes of recycled PET (rPET) in the market. The expectation is that the total rPET volume in Europe will increase from about 1,110 kton/year in 2015 to about 1,520 kton/year by 2022 [2]. The demand for virgin PET in Europe is expected to remain pretty stable at around 3,520 kton/year [2]. Therefore, the importance of rPET in the total PET demand in Europe is steadily increasing from about 31% in 2015 to about 43% by 2022.

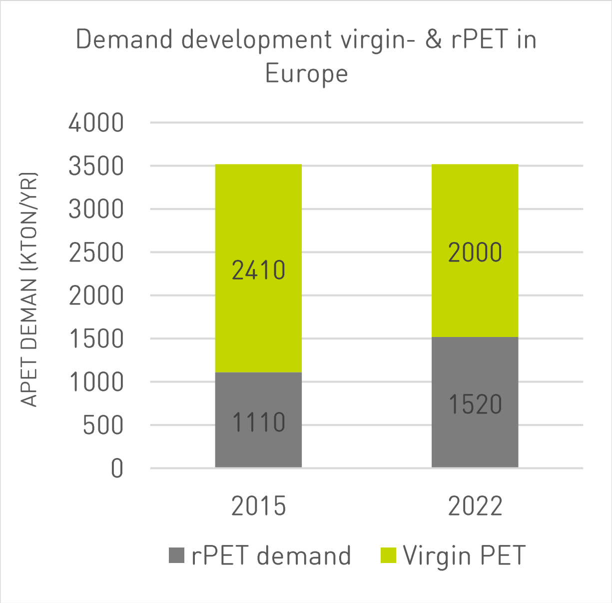


*Figure 1. Development in demand for virgin and rPET in Europe from 2015-2022.*

## (RECYCLED) PET IS PRONE TO DEGRADATION OR CONTAMINANTS UPTAKE

During PET production, processing or recycling, thermal degradation can cause a breakdown of the polymer microstructure that could involve the main chain backbone, polymer side chains or chemical (end)groups. Crosslinking can result in the formation of gel or so-called "fish eyes". In addition, several decomposed substances can occur, such as:

- Acetaldehyde
- Ethylene
- Benzene
- Biphenyl
- CO
- CO2

Therefore, thermal degradation can result in poor processability characteristics, the presence of undesired substances and poor material performance. In addition, during mechanical recycling of PET, particle contaminations can remain behind in the recycled PET because they are not completely removed during the sorting and separation processes. Contaminants can originate from dust, paper, glass, metals, organics or from other immiscible polymer blends such as polyethylene, polypropylene, or polystyrene. PET discoloration can also occur due to particle contamination. As a result, transparent PET can become hazier. Discolouration is due to the formation of various chromophoric systems after or during thermal treatment at elevated temperatures. Usually, recycled PET becomes greyer and/or yellower. Haziness is caused by an internal, random scattering of light, which is determined by crystallinity, optical defects and the dispersion of the aforementioned contaminant particles. Discolouration and haziness become a problem when the optical and brand image requirements of the PET polymer are high, such as in visible packaging applications.

PET degradation or contaminants uptake can also occur during the product service life. An example is PET textiles. When PET fibers are exposed to UV light and alkaline based detergents during frequent laundry cycles, physical and chemical attacks, respectively, can result in degraded polymer chains in PET textiles. When PET packaging contains food or beverages specific substances, such as limonene, can migrate into the walls of the PET packaging during storage. Consequently, the PET will become contaminated and is a subject of study to avoid potential hazardous human health effects.

## SOME RELEVANT CONTAMINANTS IN RECYCLED PET PACKAGING APPLICATIONS

In cases where PET is recycled and applied for food packaging purposes, intense cleaning and purification recycling technologies are required to meet all food contact safety regulations. In the European Union, all materials used in contact with food must comply with the general requirements of the European Framework Regulation (EC) No. 1935/2004.9. For polymer materials, more specific requirements are described in the European Plastics Regulation (EU) No. 10/2011. According to the latter regulation, the overall migration limit of 10 mg/dm2 of food contact surface area must be met. In addition, specific migration limits (SMLs) stated in the European Plastics Regulation (EU) No. 10/2011 must also be met. Relevant are the SMLs for PET monomers and degradation products: acetaldehyde, ethylene glycol, terephthalic acid and isophthalic acid of 6, 30, 7.5 and 5 mg/L, respectively [3].

The migration of non-intentionally added substances (NIAS) must also comply with the safety requirements of Article 3 of the European

Framework Regulation (EC) No. 1935/2004 [3]. NIAS classifies substances that are regarded as carcinogenic, mutagenic and or toxic to reproduction (CMR) and as non-CMR. For substances classified as non-CMR, such as limonene, the detection limit of the substance can be higher than CMR substances, such as benzene. Therefore, from a product safety point of view, benzene is an important substance. Below, we discuss some frequently detected substances, such as limonene, acetaldehyde and benzene, in more detail.

## LIMONENE

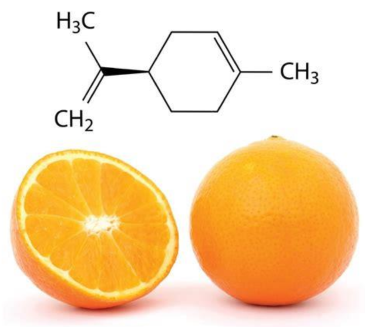


*Figure 2. Molecule structure of limonene*

Limonene is a common dietary supplement and fragrance ingredient for cosmetic products [4]. It is also applied as a solvent for cleaning purposes, such as for the removal of adhesives or oil removal from machine parts [6]. When carbonated soft drinks or orange juices containing limonene are stored in PET bottles, limonene can migrate into the PET packaging. In a European rPET screening study [5], a maximum concentration of about 20 parts per million (ppm) of limonene was found in conventionally recycled PET. And 98% of all examined rPET flake samples showed a limonene concentration of below 10 ppm. The average value of limonene in conventionally recycled PET flakes was determined to be 2.9 ppm = 2,900 parts per billion (ppb) [5]. Figure 3 shows a graph in which the limonene concentration in PET was detected by means of Headspace Gas Chromatography (GC) analysis.

The taste threshold for limonene in water is 35 ppb. To a achieve this minimum concentration of migration levels of limonene, the concentration of limonene in the PET packaging must be at least 100 ppm at 20oC and at least 40 ppm at 40oC, both for 1-year storage [3]. As the maximum limonene concentration detected in rPET amounts to 20 ppm, it is not likely that the taste of the water is affected by limonene once the water is bottled in rPET material. The taste threshold of limonene can hardly be achieved from rPET bottles. However, limonene is detectable in very low concentrations in nearly every post-consumer rPET sample. Therefore, limonene can be used as an effective indicator to assess the cleaning and purification efficiency of the rPET recycling processes.

The applied headspace GC/FID method is a suitable method for such a routine check of limonene in production. Typical limonene concentrations in rPET measured by EMMTEC are in the range of 0.1 mg/kg (ppm) = 100 ppb. This concentration is lower than the average limonene concentration in rPET (2.9 ppm), but exceeds the taste threshold concentration (35 ppb). In virgin PET, EMMTEC normally doesn’t detect any limonene.

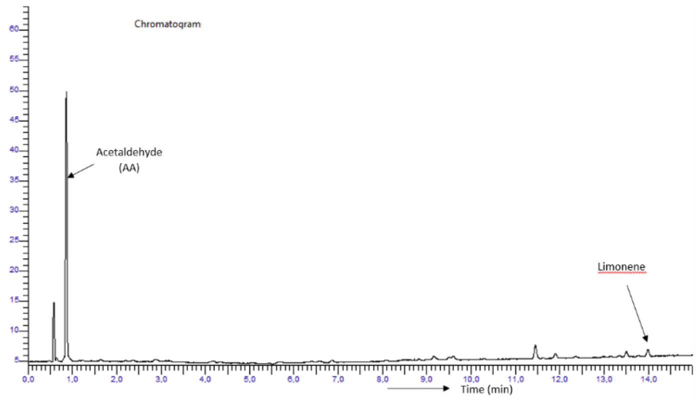


*Figure 3. Headspace GC graph for Acetaldehyde (AA) & limonene; measured by EMMTEC.*

## ACETALDEHYDE

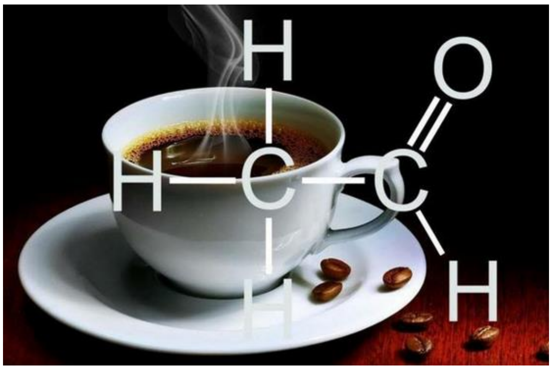

*Figure 4. Molecule structure of Acetaldehyde (AA).*

Acetaldehyde is a colourless, volatile substance with a fruity odour. It is naturally formed in coffee, bread and some fruits. In human bodies, it is also produced by the partial oxidation of ethanol by the liver enzyme alcohol dehydrogenase and is a contributing cause of hangovers after alcohol consumption [6]. Figures 3 and 5 show two graphs in which the acetaldehyde concentration in PET is detected by means of Headspace GC analysis. Typical acetaldehyde concentrations measured by EMMTEC in rPET are in the range of 0.05 mg/kg = 50ppb.

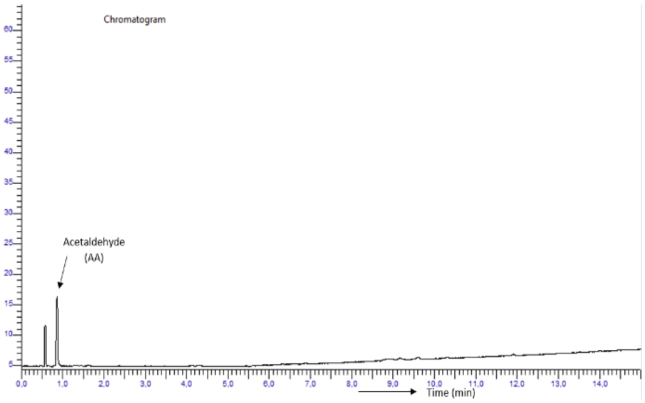


*Figure 5. Headspace GC graph for Acetaldehyde (AA); measured by EMMTEC.*

Acetaldehyde can be obtained by thermal degradation of PET. The formation of acetaldehyde is favoured by exposing PET to temperatures above 250°C, high processing pressures, extruder speeds (excessive shear flow raises the temperature) and/or long barrel residence times. The first stage of thermal degradation is a random so-called beta-scission of the in-chain ester linkage resulting in the formation of a vinyl ester and carboxyl end groups, see figure 6. Transesterification of the vinyl ester then occurs to obtain vinyl alcohol, which is rapidly transformed to acetaldehyde. By the way, the conversion from active hydroxyl and carboxyl end groups into vinyl esters reduces the reactivity of the re-polycondensation of (r)PET or by means of solid-state polymerisation [7]. As a result, the recyclability of repeatedly recycled PET is likely to be reduced using these technologies.

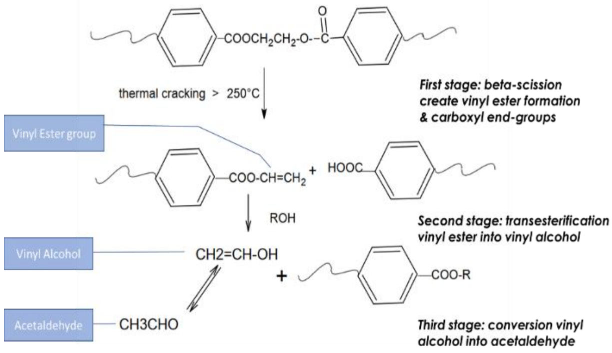


*Figure 6. Acetaldehyde formation by thermal degradation of PET.*

Once acetaldehyde is formed, some acetaldehyde is present in the wall thickness of a PET container, tray or bottle. Acetaldehyde can subsequently migrate into the product stored inside the PET packaging. This will change the taste and aroma of the stored food or beverage. The effect on taste is not such a problem for non-consumables (such as shampoo), for fruit juices (which already contain acetaldehyde), or for drinks with strong tastes, such as soft drinks. The taste of these beverages and foods masks the taste of acetaldehyde. However, this is different in the case of bottled potable water. The already very low acetaldehyde concentrations (10–20 ppb in the water), can create off-tasting water, which makes an acetaldehyde limit desirable. From a human health perspective, no serious cases of acute intoxication have been recorded. Acetaldehyde naturally breaks down in the human body. However, acetaldehyde is considered to be carcinogenic in humans [6]. Therefore, it is essential to minimise acetaldehyde concentrations in PET. Consequently, it is important that PET polymer manufacturers, PET recyclers and PET packaging makers strictly control and monitor the acetaldehyde concentrations in PET materials and packaging products.

## BENZENE

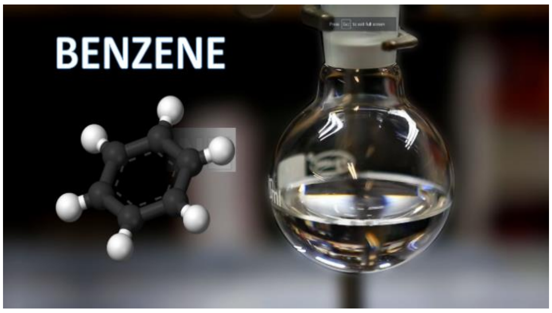


*Figure 7. Molecule structure of benzene.*

Benzene is a colourless organic chemical compound with the molecular formula C6H6. The benzene molecule is composed of six carbon atoms joined in a planar ring with one hydrogen atom attached to each. As it contains only carbon and hydrogen atoms in a ring shape, benzene is classed as an (aromatic) hydrocarbon.

It has been demonstrated that benzene can originate from pyrolysis products in both virgin PET and rPET. However, in rPET a wide variety of contaminants can be present that induce the conversion of benzene. These contaminants could include PVC polymer remnants from sorting inefficiencies, PVC-based inks and carbon black. In addition, traces of benzoic acid, juice and detergent components could be converted into benzene at elevated temperatures [8]. In the case of PVC contamination in rPET, the PVC dehydrochlorinates at elevated temperatures to form hydrochloric acid. It is suggested that this acid could catalyse the rearrangement of terephthalic groups into benzene and carbon dioxide. It is also believed that hydrochloric acid, originated from PVC contaminants, is the most likely catalyst that converts PET into benzene when exposed to elevated temperatures [8]. Figure 8 shows a graph in which the benzene concentration in rPET is detected by means of Headspace GC analysis. Typical benzene concentrations in rPET, measured by EMMTEC, are in the range of 0.05 mg/kg = 50 ppb. Benzene concentrations are not or are hardly detected by EMMTEC in virgin PET.

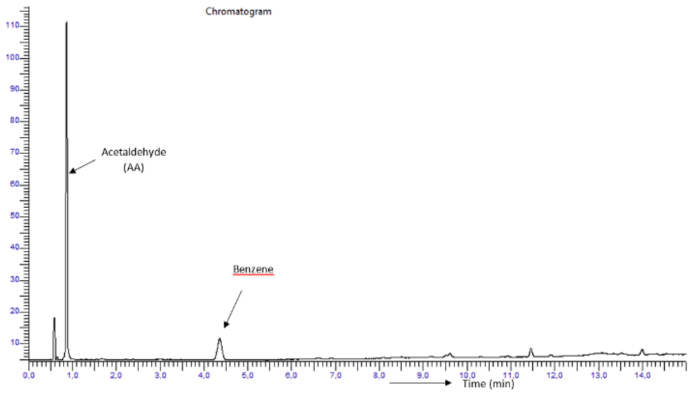


*Figure 8. Headspace GC graph for Acetaldehyde (AA) & Benzene; measured by EMMTEC.*

# ANALYSES AT EMMTEC LABORATORY

## THE ANALYSIS OF THE INTRINSIC VISCOSITY (IV): THE UBBELOHDE VISCOSIMETER

The laboratory of EMMTEC measure the IV by means of an Ubbelohde viscosimeter. Basically, an Ubbelohde viscosimeter is constructed by means of an U-shaped piece of glassware with a reservoir on one side and a measuring bulb with a capillary on the other. The temperature is strictly controlled. A liquid is introduced into two bulbs and then sucked through the capillary and measuring bulb. The liquid is allowed to travel back through the measuring bulb and the time it takes for the liquid to pass through two calibrated marks is a measure of the viscosity [9]. Figure 9, shows an overview of the measuring principle.

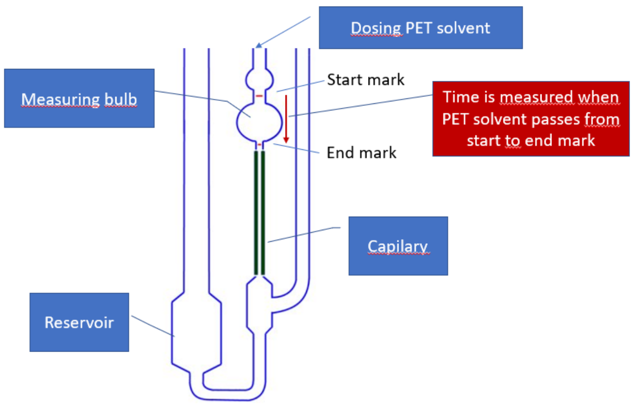


*Figure 9. Schematic overview of the Ubbelohde viscosimeter measurement principle.*

*Figure 10. Ubbelohde viscosimeters at EMMTEC.*

The IV measurement is carried out on a solution of the polymer sample in a suitable solvent. Each IV test requires at least 100 grams of solid PET granulates or flakes per sample. The suitability of the solvent is dependent on the PET microstructure. Amorphous PET from bottles or trays can be effectively dissolved in m-cresol in accordance with EMMTEC's own standard AP 332 [10]. For example, semi-crystalline PET from fibers can be dissolved in dichloroacetic acid (DCA) in accordance with EMMTEC's own standard AP 337 [10]. The intrinsic viscosity of the material is derived by the extrapolation of the relative viscosity to zero concentration, which is measured in decilitre per gram (dL/g).

## THE ANALYSIS OF LIMONENE, BENZENE, ACETALDEHYDE: HEADSPACE GAS CHROMATOGRAPHY

Headspace Gas chromatography (GC) is a type of chromatography used in analytical chemistry for separating and analysing compounds that can be vaporised without decomposition. A Headspace gas chromatograph uses a flow-through narrow tube known as the column, through which different chemical constituents of a sample pass in a gas stream (carrier gas, mobile phase) at different rates depending on their various chemical and physical properties and their interaction with a specific column filling, called the stationary phase. As the chemicals exit the end of the column, they are electronically detected and identified. The function of the stationary phase in the column is to separate different components, causing each one to exit the column at a different time (retention time). Other parameters that can be used to alter the order or time of retention are the carrier gas flow rate, column length and the temperature. In the case of Headspace, the headspace gas is directly injected onto a gas chromatographic column for separation and analysis. In this process, only the most volatile (most readily existing as a vapor) substances make it to the column. Figure 11 shows an overview of the measuring principle. Typical uses of GC include testing the purity of a particular substance or separating the different components of a mixture [11].

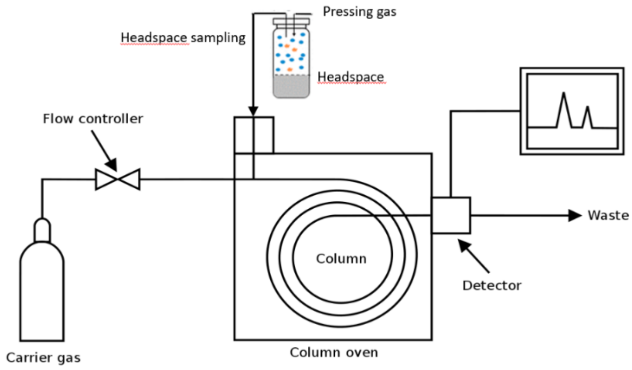


*Figure 11. Overview of the Headspace GC measuring principle.*

*Figure 12. Headspace GC at EMMTEC.*

## THE ANALYSIS OF PARTICLE CONTAMINATIONS: PARTISOL

The Partisol Dirt Vision method is based on the detection of dirt particles and other contaminants in a solvent. This is a laboratory technique where the rPET flakes, foil or yarn samples are dissolved in a very strong solvent with a very low refractive index. The low refractive index results in a large contrast between non-dissolved particles (for example PE, PP, PVC, glue) and the background, ensuring particle detection with a high sensitivity: The lower detection limit of the particles is about 3 micrometres. The Partisol analysis results in a histogram with a particle size distribution of the contaminants. Typically, EMMTEC detects about 11,000 particles in 10,000 measuring images in rPET samples. In virgin PET, we typically detect about 1,500 particles in 10,000 measuring images. That is significantly lower than in rPET!

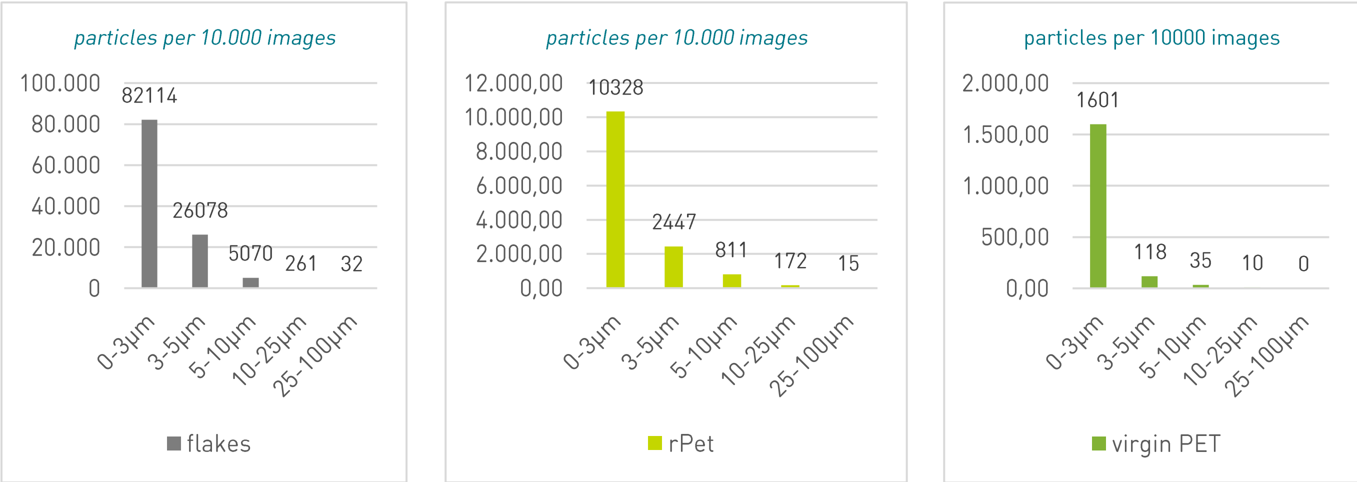


*Figure 13. Typical histogram graphs for PET flakes, rPET and virgin PET.*

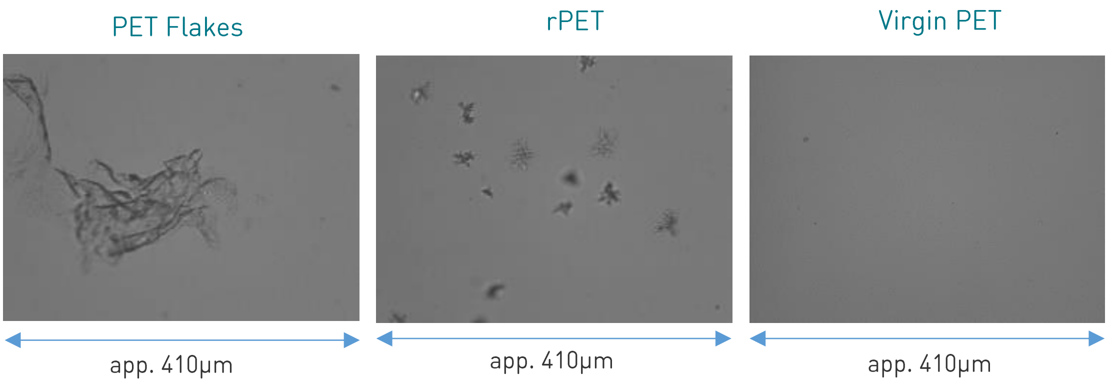


*Figure 14. Partisol images for PET flakes, rPET and virgin PET*

## OVERVIEW OF THE DISCUSSED ANALYSIS TECHNIQUES

Table 2 shows the characteristics of the EMMTEC IV, GC and Partisol analysis techniques.

*Table 2. Overview of the characteristics of IV, GC and Partisol analysis techniques*

| Technique | Material | Standard | Detection Range | Lower Detection limit | Delivery time (working days)[1] |
|---|---|---|---|---|---|
| IV | Amorphous PET[2] | AP 332 (m-cresol)[4] | 0.10-2.60 | - | 1-2 |
| | Crystalline PET [2] | AP 337 (DCA) [4] | 0.40-2.50 | - | 1-2 |
| Headspace GC | Limonene | see [5] | - | 0.03 ppm | 5-10 |
| | Acetaldehyde | ASTM F2013/10 | - | 0.1 ppm | 5-10 |
| | Benzene | see [5] | - | 0.03 ppm [3] | 5-10 |
| Partisol | PET polymer [2] | AM 350 [6] | 3 µm-100µm | - | 5-10 |

1. *From the day that EMMTEC received the sample(s) at the laboratory.*
2. *Can be delivered in granulate, flakes, tray or preforms; the samples can or will be grinded prior to analysis.*
3. *ppm = parts per million = mg/kg.*
4. *Own standard as described in ref [8].*
5. *For the determination of Limonene and Benzene, the same method is used as for Acetaldehyde ASTM F2013/10.*
6. *Own standard.*

EMMTEC can perform many more analysis techniques. For a complete overview, see appendix 1.

## HOW THE RESULTS OF OUR ANALYSES CAN BE TRANSLATED INTO INSIGHTFUL INFORMATION

The purpose of doing analyses is to create more insights with respect to your business needs. The laboratory of EMMTEC provides essential information to polymer and packaging producers, recyclers, and brand owners for the purposes of:

- Monitoring the quality of polymer products in relation to production conditions.
- Reducing and managing risks related to product safety.
- Setting up accurate Technical Data Sheets and material Safety Data Sheets
- Accelerating and providing additional insights in new process and product developments.
- Checking compliance with regulations concerning the environment, food, process, and product safety.

Based on the data and insights, EMMTEC contributes to efficient production, optimum product safety and quality assurance, and the acceleration of new product or process developments. Depending on the analysis technique, insights into different information can be obtained.

## INTRINSIC VISCOSITY INSIGHTS

The IV gives insight in the polymer weight distribution of the PET polymer. It is an important measure to assess and control the polymer quality for polymer producers, packaging companies and PET recyclers. The higher the IV, the more demanding requirements the PET can meet in the intended application. In addition, the higher the IV of PET reduces the susceptibility for environmental stress corrosion.

## VOLATILE COMPONENTS IN HEADSPACE GAS CHROMATOGRAPHY INSIGHTS

Headspace GC provides insights in volatile components present in virgin or rPET. It is important from a product safety and taste point of view. In addition, by measuring acetaldehyde and benzene, we can obtain insights in the degree of thermal degradation of the (r)PET and pro-active measures could be taken to avoid thermal degradation as much as possible.

## PARTISOL INSIGHTS

Partisol is a good analysis method that provides an insight into the contamination of all kinds of raw materials, additives, and products. Partisol has been proven to be a useful tool to:

- Improve the yarn or other production processes and to avoid unnecessary waste.
- Assess the relation between haziness of transparent (amorphous) PET and the presence of contaminants. A linear correlation has been observed between the particle contamination and the haziness of (transparent) rPET preforms and bottles.
- Optimise the production and handling of flakes and chips. The performance of Partisol analysis is recommended when the amount of waste material during yarn or packaging production is to be reduced and/or more cost control is required. This is based on our experiences with repeated Partisol measurements of rPET raw materials for spinning or packaging manufacturing purposes. We believe this is a great and cost saving insight.

## EMMTEC AT A GLANCE

EMMTEC helps companies run their business processes efficiently and effectively. We do so at the EMMTEC Industry & Business Park in Emmen, at other sites in the Netherlands and in foreign countries. As a company with more than 65 years of experience and some 350 enthusiastic employees, we supply utilities and services in the fields of engineering, laboratory analysis and logistics. We provide innovative products and services that satisfy your requirements across a broad spectrum, varying from power generation to water treatment, tank cleaning to warehousing and process optimisation to laboratory analysis. Solutions that make your company stronger, smarter and more efficient.

## OUR COMPENTENCIES

EMMTEC is dependable, solution-oriented and applies its knowledge and expertise in a practical manner. You can always rely on us and always benefit from a solution that works for you. How do we approach this? Professionally, inquisitively, proactively and with ambition. And obviously, with a great deal of expertise!

## DO YOU WANT TO KNOW MORE ABOUT OUR SERVICES FOR YOUR SPECIFIC ISSUES? CONTACT US!

### Your contact persons

Rick ten Velde
Accountmanager
Mobile: +31 (0) 6 1286 2512
Email: rick.tenvelde@emmtec.nl

Jan Willem Slijkoord MSc
Business Development Manager
Mobile: +31 (0) 6 53 90 40 36
Email: jan-willem.slijkoord@emmtec.nl

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