

Sample preparation of PET preforms for subsequent analytics

Acetaldehyde is commonly found in alcoholic beverages like wine, liqueur or whiskey. PET bottles, e.g. for mineral water, are another source of acetaldehyde. This is a serious problem for consumers as acetaldehyde is not a harmless chemical substance but classified as potentially carcinogenic.

In the beverage industry plastic bottles – or rather their preforms - are analyzed for their acetaldehyde content to prevent any health risk for consumers. The acetaldehyde must be extracted from the preforms prior to analysis. To ensure correct results, the bottles first have to be crushed and homogenized by laboratory mills. Care must be taken that the volatile substance acetaldehyde does not escape during the process due to heat build-up inside the grinding chamber. To preserve the acetaldehyde, part of the sample preparation is carried out under cryogenic conditions.

Suitable laboratory mills

First, the PET preforms are pre-cut in a **RETSCH Cutting Mill SM 300** using a 6 mm bottom sieve. This mill reduces the size of the samples by cutting and shearing effects produced by a rotor and stationary cutting bars in the grinding chamber. For pulverizing elastic and temperature-sensitive materials, the parallel section rotor with its 3 large blades is most suitable. Thanks to the powerful 3 kW motor and the resulting high torque, the SM 300 is able to cut 200 g preforms in only one minute to a mean particle size of 5 mm. Thermal effects, which would lead to loss of the volatile components, are easily prevented by adapting the cutting speed within a range from 700 rpm to 3000 rpm. Cleaning of the mill is a quick and easy process thanks to the fold-back hopper, push-fit rotor and smooth surfaces in the grinding chamber.



To further decrease the particle size of the elastic preforms to < 0.5 mm, liquid nitrogen is required to embrittle the sample and improve its breaking properties. Moreover, the cooling effect prevents the loss of the volatile acetaldehyde. For size reduction at cryogenic conditions, **RETSCH's CryoMill** is the perfect choice. The grinding jar of the CryoMill is continuously cooled by liquid nitrogen before and during the milling process thanks to the integrated cooling system of the CryoMill. Liquid nitrogen is supplied by an autofill system, ensuring temperature consistency at -196°C . Thus the user never gets into contact with LN_2 , making operation of the CryoMill efficient and safe.

After representative sample division, 6 g of the pre-cut preforms are filled into a 50 ml stainless steel grinding jar together with a 25 mm stainless steel grinding ball. The grinding process is carried out in intervals: 2 minutes grinding followed by 1 minute intermediate cooling. After milling for 4 minutes, (5 min including the cooling step), 87% of the sample has a particle size below 500 microns. The pulverized sample is now ready for extraction and subsequent spectroscopic analysis via color reaction (e.g. using MBTH or Schiff test).



*Cutting Mill SM 300 (left) and
CryoMill (right) from RETSCH*

