

# APPLICATION NOTE

## Grinding Polymers for Qualitative and Quantitative Analyses

With kind permission of the Federal Institute for Materials Research and Testing (BAM)

In the application discussed here, the 6750 Freezer/Mill<sup>®</sup> was used for grinding compact synthetics (pellets, webbing fabric, textiles, etc.) as well as polymer yarns, fleece material, filaments (polypropylene (PP), polyethylene or polyester). Grinding was required for accurate qualitative and quantitative analysis of additives contained in synthetic materials (e.g. antioxidants using HPLC and INOC-Test). The Freezer/Mill is ideal for this application, providing the required particle size reduction in a relatively short time period.

### Example: PP fleece materials

The material is first cut into approximately 2mm squares. The grinding vials are then filled half way with sample; then ground as per the program below:

Pre-cooling : 15 min  
Run : 1.5 min  
Cycle cool : 2 min  
Cycles : 5  
Rate : 10Hz

For the analysis discussed here, the method above was found to be the optimum grinding program for PP fleece materials. The yield is high (80% - 90%) and during the short grinding times the material does not warm enough to lose its brittleness or decompose. The number of grinding cycles is dependent on the quantity of sample placed in the vial and the desired final particle size. In order to exclude particles that are too coarse the ground fleece material can be sieved, for example passed through a 500µm sieve.

### A) Sample Preparation for HPLC analysis of additives

Approximately 2.0g of the ground material is weighed into a 125ml flask to an accuracy of 1mg and 40ml toluene is added. The sample is brought to a boil with constant stirring. After approximately 30 minutes the material has completely dissolved, the solution is

than slowly cooled to 50°C. In order to precipitate the polymer, 40ml of methanol is added with further stirring. The mixture is then cooled to room temperature and filtered through a Nutsche (suction) filter. The filtrate is then passed through a 0.45µm filter. The stabilizers necessary for analysis are now in solution and ready for oxidation and HPLC separation.

### B) Material analysis: INOC-Test

Depending on the stabilizer content, about 200 to 400mg of sample material and 10.0mg of AIBN (initiator) are weighed accurately into the reaction vessel. 10.0ml of Cumol is then added with a pipette. The vessel is connected to the oxidation apparatus (see fig.) The Cumol efficiently extracts the stabilizer contained in the pulverized fleece material permitting AIBN oxidation. The INOC Test measures time dependent oxygen consumption during the reaction. From the data obtained, a diagram is generated enabling visual evaluation of the stabilizer content in the samples.<sup>1)</sup>

### Conclusion

The 6750 Freezer/Mill efficiently reduces the particle size of samples that are difficult to grind without thermal degradation of the material, for example fleece materials to a particle size of less than 500µm. Sample preparation time is drastically reduced to less than 15 minutes due to the increased brittleness of the samples at these very low temperatures.



Application Note SP007:  
**Grinding/  
Homogenization**

Apparatus:  
**Freezer/Mill<sup>®</sup>  
6750 and 6850**

Application:  
**Polymer Yarns  
and Fleece  
Materials**

# APPLICATION NOTE

## Grinding Polymers for Qualitative and Quantitative Analyses

203 Norcross Avenue, Metuchen, NJ 08840

Tel: 732.549.7144 Fax: 732.906.2492

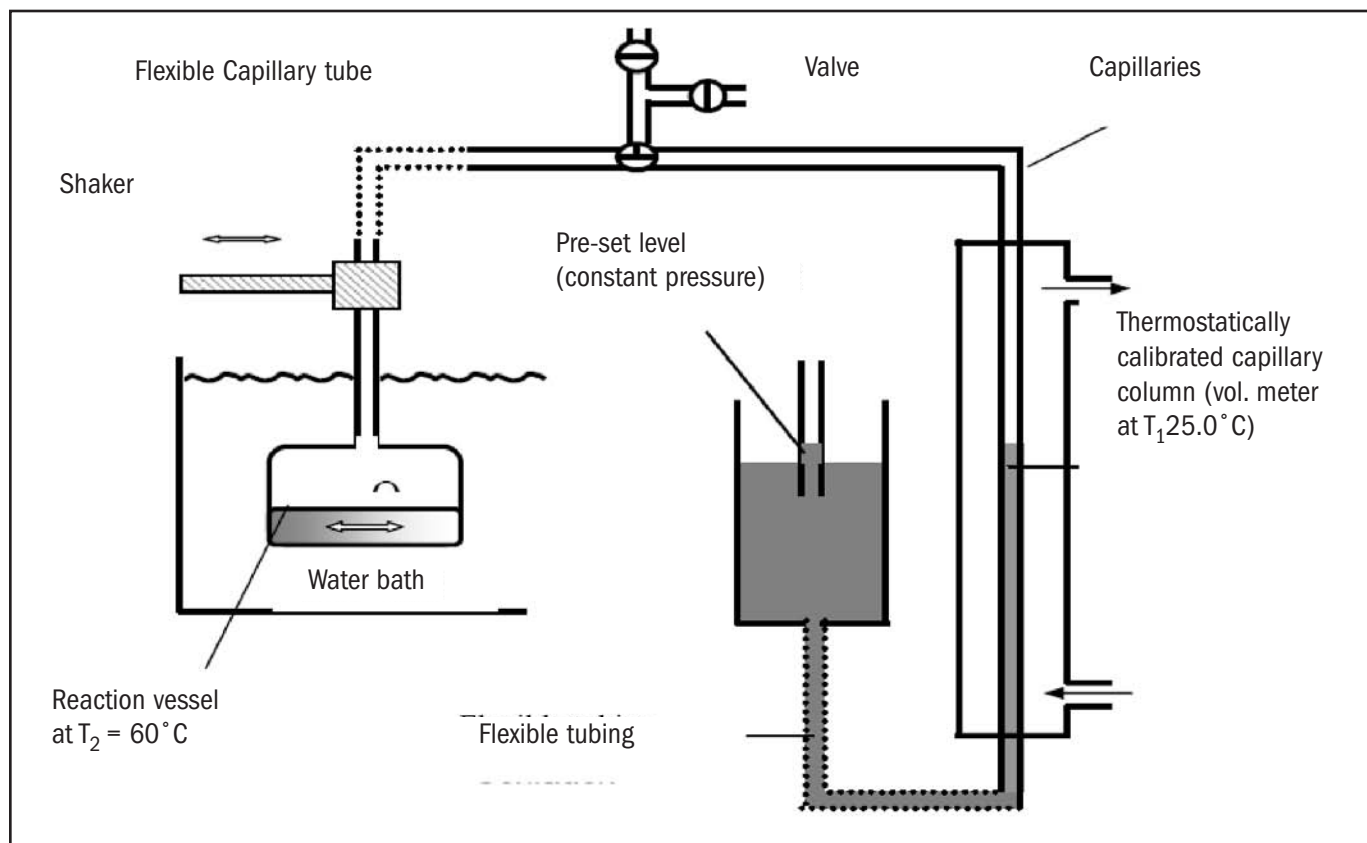
800.522.7739 (800.LAB.SPEX)

E-mail: sampleprep@spexcsp.com

**Online Ordering**

www.spexcsp.com

### Apparatus:



<sup>1)</sup> (H. F. Schroeder, E. B. Zeynalov, H. Bahr H, and T. Rybak, POLYMERS & POLYMER COMPOSITES, 2002, 10 (1), 73-82: "Analyzing the Content of Antioxidants in PP Materials")