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Committee D20 on Plastics
Subcommittee D20.22 on Cellular Materials - Plastics and Elastomers

Research Report D20-1243

Interlaboratory Study to Establish Precision Statements for ASTM D7252, Standard Test Method for Polyurethane Raw Materials: Determination of Monomer and Isomers in Isocyanates

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To: tom.conti@bayerpolymers.com, FLOUTZW@BASF-Corp.com
cc:
Subject: Monomer/Isomers Round Robin

Tom and Bill, please forward this email and attachments to your participating labs.

You will soon be receiving the six samples and the derivatized standard for the monomer and isomers round robin The samples, numbered one to six, are of the types listed below.

PURMAC 1 - MI-50 (~100% monomer, ~50% 2,4' isomer)
PURMAC2 - Polymeric MDI (~42% monomer, ~4% 2,4' isomer)
PURMAC3 - Uretonimine-modified (~60% monomer, ~1% 2,4' isomer)
PURMAC4 - Prepolymer (~50% monomer, ~2.5% 2,4' isomer)
PURMAC5 - 20:80 PURMAC1:PURMAC2 Blend (by weight)
PURMAC6 - 40:60 PURMAC1:PURMAC2 Blend (by weight)

The standard was produced from monmer MI-30 containing the following isomer ratio (by GC/FID):

2,2' isomer - 1.66%
2,4' isomer - 29.79%
4,4' isomer - 68.55%

The standard was derivatized to the methyl urethane using dry methanol and was done this way to preserve its quality. The calculations in the attached method will compensate for the molecular weight difference between the iso and the carbamate.

The samples should be run in duplicate on two separate days to provide a total of four results (two pairs of duplicates). Note that the duplicates should be two different sample setups, not two injections of the same sample setup. It is also of interest to capture response factor data as well, so sample weights and area counts should also be recorded (if available). An Excel spreadsheet has been attached for convenience of recording the data.

Timing for running this round robin will depend on when the samples are received in your lab. If possible, try to run your samples the last week in September or the first week in October. This should allow time for the data to be tabulated for discussion at the Fall PURMAC meeting on October 20-21. Please send results to me (David_P_Mullen@HUNTSMAN.COM) when available.

Please contact me if you have any questions. Thanks for participating in this study!

-David Mullen
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1.0 SCOPE

1.1 This method is designed to determine the amount of diisocyanate and individual diiso isomers in Pure and Polymeric MDI, as well as isocyanates derived from these. Samples are derivatized with methanol and the diiso isomers are separated from the matrix by normal-phase liquid chromatography. Analytes are detected by UV absorption and are quantitated using an internal standard.

2.0 HEALTH AND SAFETY PRECAUTIONS

MDI is a skin and respiratory sensitizer. Wear proper protective clothing and gloves.

3.0 EQUIPMENT

3.1 High Performance Liquid Chromatograph (HPLC) consisting of:
3.1.1 Quaternary Pump
3.1.2 Heated Column Compartment
3.1.3 Variable Wavelength Detector
3.1.4 Autosampler (optional, samples can be injected manually)
3.1.5 Solvent Degasser (optional, but solvents must be degassed before use)

3.2 Suitable integrator/computer for data handling

3.3 250 mm x 4.6 mm x 5 µm Cyano HPLC column, such as Phenomenex Luna CN # 00G-4255-E0

3.4 Volumetric flask or pipette, 100 ml.

3.5 Sample vials with caps and crimper (suitable for autosampler, if used)

3.6 4 oz. bottle with cap

3.7 Calibrated analytical balance, 4-place