DETERMINATION OF THE MOLECULAR MASS DISTRIBUTION BY TOF-MS: INTERLABORATORY COMPARISON
BY MOLEDI-TOF-MS: INTERLABORATORY COMPARISON
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Preparation

A series of laser desorption ionization time-of-flight mass spectrometry (MALDI-TOF-MS) measurements were performed by 11 laboratories as part of a round-robin interlaboratory comparison study to determine the molecular mass distribution (MMD) of a synthetic polystyrene (PS) polymer sample. The laboratories were asked to determine the MMD of the PS sample by means of laser desorption ionization mass spectrometry (LID-MS) by comparing the peak areas of the polymer sample with the peak areas of a known reference polymer. The peak areas were then used to calculate the MMD of the PS sample. The results of this study were used to evaluate the performance of the different laboratories and to determine the reproducibility of the MMD measurements.

Preparation of the Polymer Sample:
The polymer sample was prepared by the following procedure: A solution of PS in chloroform was evaporated to dryness and the residue was used as the sample for the MMD determination. The MMD was determined by laser desorption ionization mass spectrometry (LID-MS) using a pulsed laser to desorb the polymer and a time-of-flight mass spectrometer to measure the mass distribution of the polymer.

Results and Discussion:
The results of the MMD determination were compared to the known reference polymer samples. The MMDs determined by the different laboratories were found to be in good agreement with each other. The relative errors between the MMDs determined by the different laboratories were found to be less than 5%. This indicates that the MMD determination was performed accurately by all the laboratories.

Conclusion:
The interlaboratory comparison study has shown that the MMD determination by laser desorption ionization mass spectrometry (LID-MS) is a reliable method for determining the molecular mass distribution of polymers. The results demonstrate the reproducibility of the MMD determination and the accuracy of the different laboratories involved in the study.

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References:

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The results indicate that the variation among laboratories is much greater than the variation within laboratories.

When a matrix of 40-90 moisture with 2% salt as its sample preparation, at M, of 66.1% and at 2 M, of 47.3% was obtained. When the polystyrene sample was prepared with a dilution matrix, at M, of 62.1% and at 2 M, of 48.9% was obtained (figure 6). ANOVA tests were performed on the moisture to test variation between laboratories. The results compared, data from laboratories which ran 40-90 moisture well and dilution determination. The analysis shows that the matrix used and the sample preparation has no significant influence on the polystyrene mass distribution changed by MALDI.

Also compared the influence of linear or reboiler heat of the enthalpy in the polystyrene mass distribution. When the masts were removed, no difference in variance was noted between the present moisture level, and within the matrix. However, the ANOVA of the linear heat shows that the linear or reboiler heat effects the ratio of the distribution. The null hypothesis, that no difference exist between the test and the reboiler heat of analysis, is accepted for all of the tests except for those with representing the tails of the distribution. The data indicate the enthalpy made is more sensitive to the few mass species whereas the linear heat is more sensitive in the high mass species.

Conclusions

NIST Polyurethane and laboratory comparison of TOF MALDI MS using a polystyrene of about 7500 g/mol showed good agreement between all purifying laboratories, in general. Some differences were found for different laboratories in the range of hundreds of mass with the most significant differences noted at 2 M, of 48.9% and at 2 M, of (200-450) g.

These results were taken from the following test conditions used:

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