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Quantitative Chemical Determination of Organic Layers using CDXPS: A Standard Experimental Procedure ?

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Functionalised organic layers prepared by plasma or analogous processes have complex surface chemistries. The problem of elucidating a specific surface chemistry, a determining parameter for the properties of the material, is an extensively studied subject. One of the preponderant methods for acquiring such information is chemical derivatisation XPS (CDXPS), a technique initially developed more than 20 ago. A typical procedure consists in a gas-surface reaction with a readily quantifiable chemical marker reacting selectively with one functional group. In this contribution, the focus is made on the most widely used CDXPS procedures, namely the quantifications of primary amines and alcohols with 4-trifluoromethyl benzaldehyde (TFBA) and trifluoroacetic anhydride (TFAA), respectively.

While these procedures have been used for a long time by various laboratories around the world, the use of slightly varying protocols is acknowledged, making for a difficult comparison of results. Here, we present our efforts to address this situation by providing a rigorous, yet simple, protocol for a reliable analysis of the above mentioned functional groups. We first present a comparative study where an oxygen plasma modified polypropylene was analysed by 6 different laboratories across Germany using both in-house and "standard" protocols [1]. The dispersion of the results informs us on the expected exactitude of the method in its present state of use. During the course of our study, many specific shortcomings and pitfalls were identified and are described here in details. Finally, we describe the preparation of reliable standard samples and their use for obtaining a precise quantification of functional groups in plasma modified and plasma polymerised materials.

[1] T. Gross, F. Pippig, B. Merz, R. Merz, U. Vohrer, R. Mix, H. Steffen, W. Bremser, W.E.S. Unger. Plasma Process. Polym. In press.

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