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Is Quantitative CD-XPS of Plasma Deposited Organic Coatings a Valid Analytical 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, 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.
A previous comparative study where an oxygen plasma modified polypropylene was analysed by 6 different laboratories across Germany highlighted a significant dispersion of results amongst laboratories performing similar procedures [1]. The present contribution presents further efforts towards a valid analytical procedure. Different formulas used for calculating functional group concentration are first described and discussed. In a validation study arrays of commercially available functional polymers and organic molecules have been submitted to derivatization procedures in order to investigate potential undesirable side reactions and to evaluate their potential use as test samples. Valid test samples are a pre-requisite for a development of standardized CDXPS procedures in the future. Reaction kinetics is investigated for different experimental configurations for both test samples and nitrogen rich plasma polymerized films. Finally, a series of common identified pitfalls with pratical CDXPS are described alongside their workarounds.
[1] T. Gross, F. Pippig , B. Merz, R. Merz, U. Vohrer, R. Mix, H. Steffen, W. Bremser, W.E.S. Unger. *Plasma Process. Polym.* 7 (2010) 494-503.

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