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Implication of particle-by-particle characterization of nanoparticle dispersions for in vitro testing

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Over past decades, nano-bio research has essentially been focused on studying the underlying mechanisms of nano-bio interactions, their potential nano-environment health and safety implications (Nanotoxicology) as well as utilizing this knowledge for designing better therapeutics (Nanomedicine). In vitro assessment of nanoparticles (NP) and nano-formulations is the most common testing platform for evaluating biological responses, which requires prior NP dispersion, stabilization, and characterization in cell culture media. Dispersion inefficiencies and active aggregation of particles often result in polydisperse size distributions. Accurate characterization of important properties of such polydisperse distributions (size distribution, effective density, charge, mobility, aggregation kinetics, etc.) is critical for understanding differences in the effective dose delivered to cells as a function of time and dispersion conditions, as well as for nano–bio interactions. Here, standardized dispersion and dosimetry platform was used for preparing NP dispersions and estimating the delivered dose to cells based on dispersion characterization input from dynamic light scattering (DLS) and tunable resistive pulse sensing (TRPS), for a set of 8 distinct nanomaterials. The TRPS technology offers higher resolution and sensitivity compared to DLS and provides unique insights into NP size distribution and concentration, as well as particle behavior and morphology in complex culture media. The overall relative in vitro toxicity ranking for 8 NP types changed considerably, matching notably better the *in vivo* inflammation data ($R^2 = 0.97$ versus 0.64). These findings further reinforce the need to reanalyze and reinterpret *in vitro* data in the light of dispersion and dosimetry considerations (or lack thereof) and to adopt these standardized protocols for future *in vitro* assessment studies.

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Developing a qNMR method for purity profiling of crack-cocaine samples seized by police forces in northeastern Brazil

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Illicit use of Crack-cocaine has become a major health issue in Brazil which is currently a large consumer of world's cocaine according to UN statistics. The present work describes the development of a 1H-qNMR method to assay crack cocaine samples seized by police forces in northeastern Brazil. Samples were weighted and dissolved in D2O acidified with DCl. The internal standard used was the sodium salt of 3-(trimethylsilyl)-2, 2, 3, 3,-D-4 propionic acid (TSP-D4). The same samples assayed with the 1H-qNMR method were submitted to analysis by a validated quantification method based on reversed-phase high performance liquid chromatography with diode array detection for comparison of the cocaine content. Also, a GC/MS method was used for confirmation of the impurities detected with the NMR method. The results revealed that the average content of cocaine in the samples was above 70% (w/w). There was a large variability in the purity of samples as determined by both methods (5.2-89.1%, w/w for 1H-qNMR and from 4.9 to 93.8% w/w for the HPLC method). The best correlation between the HPLC and the 1H-qNMR values was obtained when using the integration values of the 3.6 ppm singlet signal corresponding to methyl ester protons (r^2=0.83). The main impurity detected in the 1H-NMR spectra of the samples was phenacetin, which was found in 84% of the samples. This adulterant was confirmed by GC/MS analysis. The developed 1H-qNMR can be regarded as a simple, convenient and fast screening method to assist in the purity profiling of crack cocaine samples.

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