Liquid-State Polarization of 30% through Photo-Induced Non-Persistent Radicals on 13C Pyruvic Acid

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In the present work, thanks to a thorough
investigation of the radical generation process and distribution of the photo-induced paramagnetic centers inside the DNP samples, we provide a robust protocol to enhance the efficiency and performances of the UV-radical technique.

A new sample irradiation setup was designed to work in a moist-free environment (see Fig 1). The set-up provides a fast generation of the UV-induced radicals thanks to a high power (20 W/cm²) broad-band UV-source (Dymax BlueWave 75) shining light on the tail of a quartz cold finger placed inside a reflecting aluminum chamber. On a sample containing a mixture of PA:H₂O 1:1 (v/v) we were able to induce more than 50 mM of radicals in about 5 min, thus doubling the maximum yield and reducing the irradiation time by a factor of 30 compared to the current state of the art [5, 6].

The distribution of the paramagnetic centers inside the DNP samples was investigated via ESR by changing the radical precursor (PA) concentration, in a solvent mixture of glycerol:water 60:40 (GW64), and the dimension of the frozen pellets. Taking into account the strong absorption of the UV-light while travelling through the spherical frozen samples, a radicals’ distribution profile with exponential dumping ($\sim e^{-x/d}$) from the surface to the core of the pellet was assumed and verified experimentally (see Fig 2). The attenuation parameter $d$ increased from about 0.1 mm to 3 mm by decreasing the PA concentration from 90% to 10% of the total sample volume.

DNP wise the best compromise between PA concentration and homogeneity of the radicals’ distribution was found to be a mixture of PA:GW64 1:1 (v/v). In this sample $^{13}$C nuclei polarized up to 30% in about 1 h working at 6.7 T and 1.1 K. These values represent a clear improvement compared to the current state of the art making the UV-radical technique useful for the most recent demanding metabolic imaging applications.

Reference