Novel Approaches to Signal Acquisition and Accumulation in FFC-NMR experiments

D. Canina, A. Galkin, S. Sykora and G.M. Ferrante

Stelar s.r.l. via E.Fermi,4 27035 Mede (PV)Italy - info@stelar.it

FFC problem to be tackled: field noise and sensitivity

Among the particularities of FFC-NMR relaxometry is the fact that it is based on frequent and very fast (~1 ms) switching of the main magnetic field B between several values differing by several orders of magnitude (0 to 1 T). This implies extreme dynamics of the magnet system which, while essential [1], makes the field somewhat unstable (noisy). Further field reproducibility problems arise from the thermal and mechanical stresses on the magnet, incurred by very high power dissipation (up to 15 kW) during the switching intervals. In the present system, field noise is of the order of 50 ppm and, unfortunately, cannot be improved by any of the traditional methods used with static electromagnet All this deteriorates signal coherence between successive scans and thus reduces the efficiency of the data accumulation process (FID shortening). The link between field stability and reproducibility and the achievable sensibility is of considerable importance because low S/N ratios still limit many potential applications of FFC NMR relaxometry.

Apart from signal coherence and stability between successive scans, one must in fact consider also the sensitivity achievable in a single scan (S/N ratio). In any NMR method, this parameter depends on the factors appearing in the well-known formula shown on the right [2]. In FFC, since the field B is not constant, the S/N ratio depends on all the field values applied during the field cycle, namely the polarization field B_p, the relaxation field B_r and the acquisition field B_a

$$S/N \propto B_0 \sqrt{\frac{h Q V_S}{K_B T}} \sqrt{\frac{n_0}{\Delta n}}$$

It can be shown, for example, that in the case of the basic pre-polarized sequence, the signal is proportional to B_p*B_a when the relaxation period τ is zero while, when $\tau >> T_1$, the signal is proportional to B_r*B_a. While the first factor in these expressions varies (B_p or B_r), the proportionality to B_a is always present. In order to maximize the S/N ratio, it is therefore advantageous to use always the largest possible acquisition field B_a and the corresponding operating frequency. Considering that the field strengths in FFC experiments are rather low, however, sensitivity is always a difficult issue in particular when measuring relaxation profiles of nuclides with low γ and/or low abundance, such as ^{2}D, ^{7}Li, ^{17}O, ^{13}C, etc.

A single-board NMR console

The problems described above had been addressed by Stelar in different ways, such as the development of FFC magnets with ever higher maximum fields (higher initial polarization), better cooling systems (improved field stability), higher acquisition frequencies (improved probe sensitivity), solenoid probes (better sensitivity for small-volume samples), novel null-biased sequences [3] (reduced noise propagation in data evaluation algorithms) and, last but not least, novel signal acquisition and accumulation methods implemented on the new SOC (system-on-chip) PCI board (is showns in Fig.1) described in this poster and its twin [4].

The board mounts a 12-bit Analog-to-Digital Converter (ADC) which allows sampling rates of up to 200 M samples/second and thus makes possible direct sampling of the input signals up to 90 MHz. The output of the ADC is divided into two data streams (odd and even) updated at half the sampling rate which is compatible with the top speeds of the digital receiver implemented within the FPGA. The latter includes a dual digital down-converter using two Direct Digital Synthesizers (DDS), followed by a re-combiner of the two streams, CIC filters (decimation ratios from 4 to 16383) and FIR filters (16 taps/18 bit coefficients). The down-converted and filtered in-phase and out-of-phase digital signals are then sampled by a data accumulation module described below which stores the accumulated data in four large SRAM chips. Being fully digital, the receiver automatically eliminates a number of analog-receiver artifacts such as DC offsets and quadrature amplitude and phase misadjustment (main functional blocks of the digital receiver are shown in Fig.2) .

The run-time control of all aspects of an NMR experiment, including the RF pulse sequence, all receiver settings, accumulation modes and, in fact, of all the instruments' hardware is achieved by means of a 128 bit/20 ns sequencer sub-processor described in detail in the twin poster [4].

Apart from the DDS generators which make part of the receiver, there are other two digital RF sources routed to three RF output connectors. Of these, two pass through digitally controlled attenuators permitting, for example, the generation of profiled pulses. All the on board DDS RF generators feature 32 bit frequency and 16 bits phase control, 14 bits outputs for Digital Analog Converters and the capability to run synchronously.

The board is interfaced to a PCI bus and runs under the control of a Host Computer which, however, does not need to be of the strictly real-time class since the board is capable of autonomous operation of even very complex acquisition procedures.

Finally, the board incorporates also 10 buffered TTL inputs for safety interlocks and external events sensing, all capable of generating Host interrupts as well as immediate, pre-programmed responses of the pulser/sequencer. Other features worth mentioning are a large number of buffered TTL output ports and the capability to host an extension board for special/prototyping purposes.

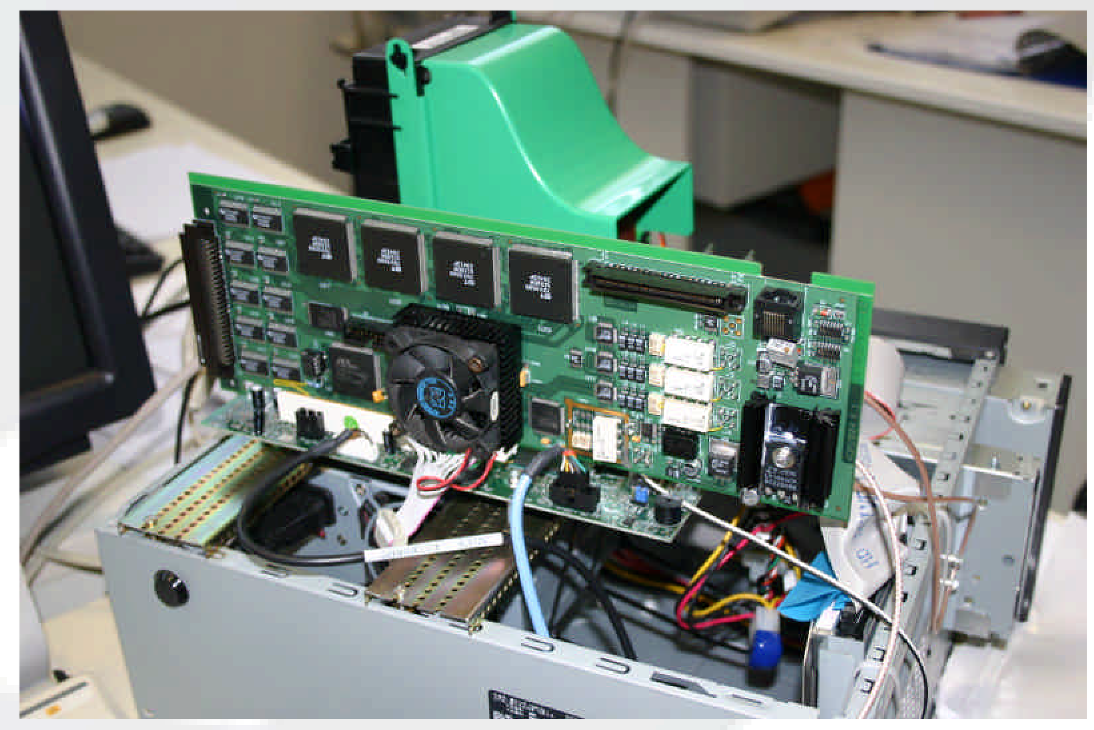


Fig.1

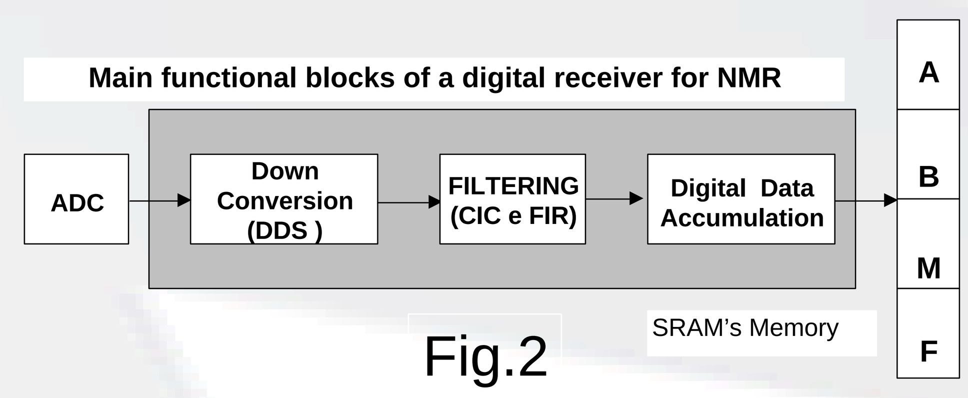


Fig.2

Run-time control of LF filters

In the quest for improving S/N ratios, we have implemented a run-time control of the whole filter block (CIC and FIR), whose settings need no longer be static during the whole experiment, but can be switched at any moment by the pulser among up to 64 pre-programmed settings.

The FIDs of the oil-stained Perspex are shown in Figures 3 and 4. It is a typical case of heterogeneous sample where, with traditional hardware, we would have to optimize filter width for the slowly decaying mobile part, distorting heavily the fast decaying solid portion or, alternatively, keep a large bandwidth suitable for the solid at the price of a needlessly large noise in the liquid (Figure 3).

The hardware of the new board gives us the possibility to combine the desirable features of both situations while avoiding the necessity to run all experiments twice. The FID shown in Figure 4 uses two filters with different setting. The pulser is programmed to select the output data of large bandwidth filter in the first portion of the FID and the data generated by narrow bandwidth filter in the second portion. Simultaneously, it changes the sampling rate and thus optimizes the FID accumulation memory usage

Fig.3

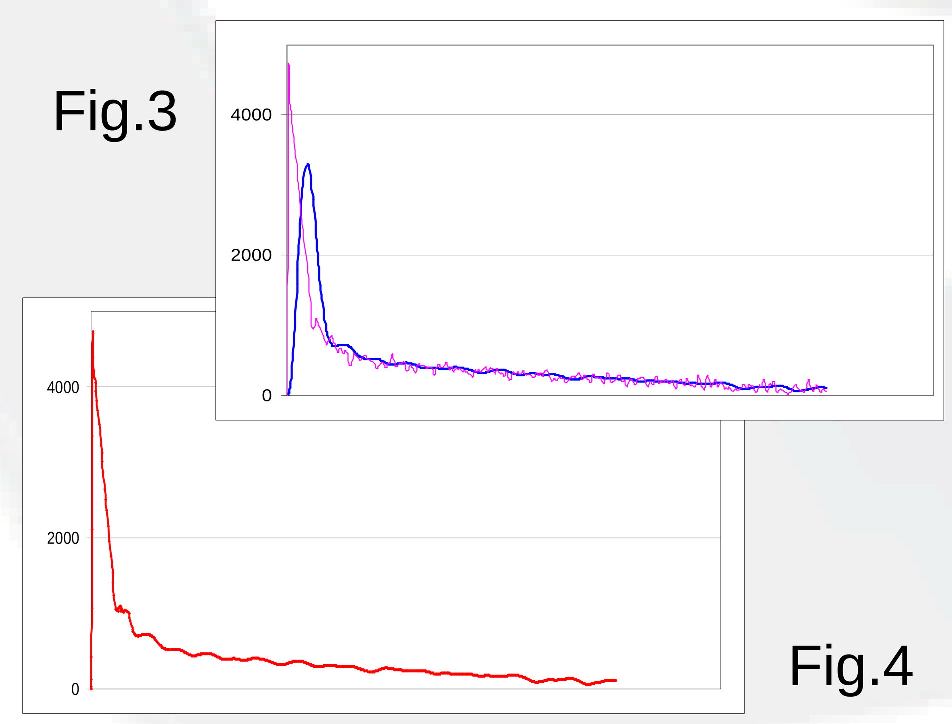
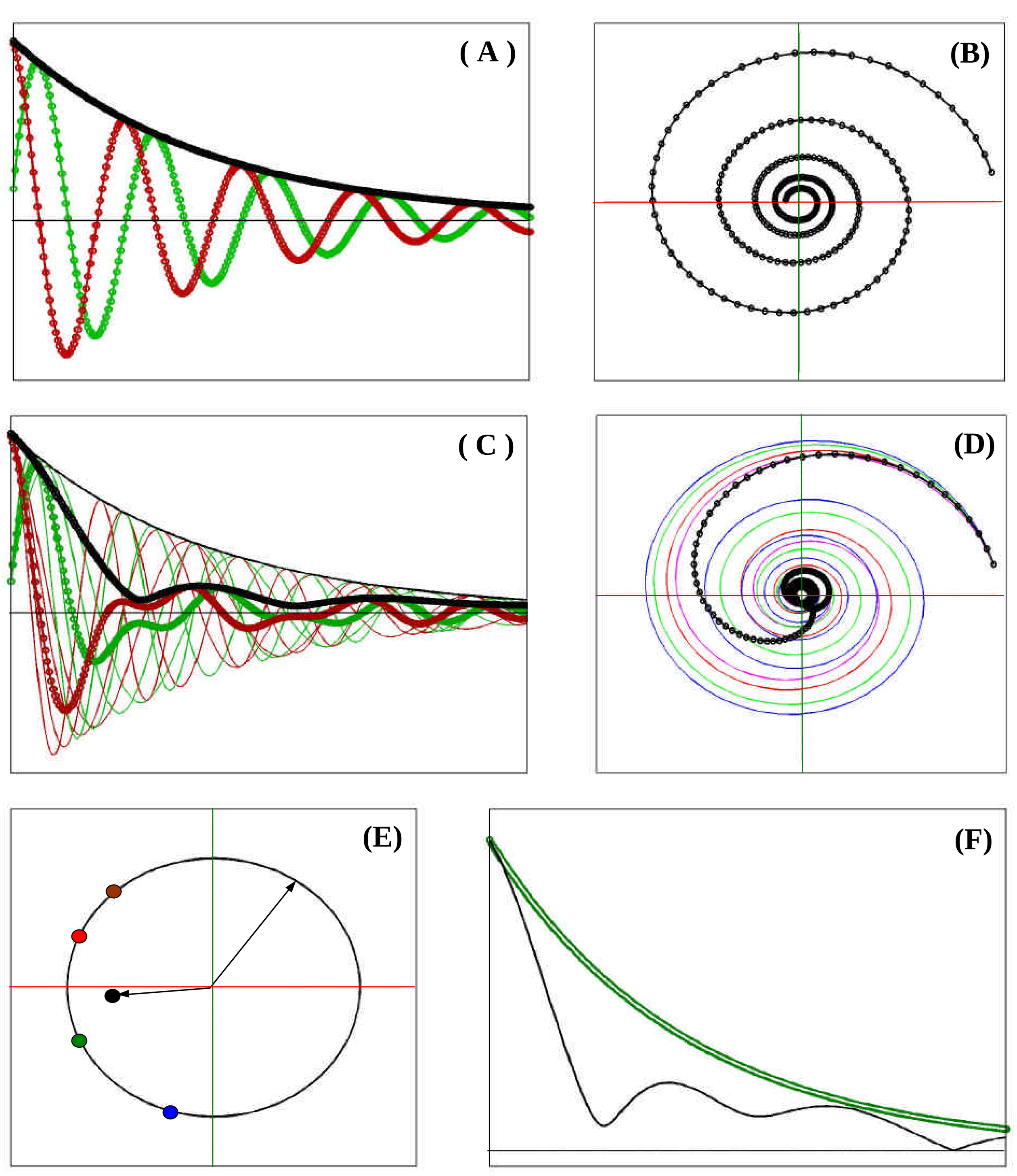


Fig.4



Novel data accumulation methods (Digital Data Accumulation)

To combat the field stability and reproducibility problems mentioned above, we have implemented a number of novel data accumulation modes, some of which differ substantially from the usual averaging of the Cartesian components of the complex signal. Thus, for example, it is possible to average - in separate buffers - the magnitudes and phases at each FID point and thus remove most of the field instability effects and extend the usable portion of the FID (see the next paragraph). It is even possible to average simultaneously the two Cartesian coordinates as well as the magnitude-phase pairs in four distinct buffers.

Accumulation of FFC-NMR signals in polar coordinates

In a perfectly stable field, an off-resonance quadrature FID looks like the one shown in **(A)** and is perfectly reproducible. Here the red points indicate the in-phase Cartesian component u and the green ones the out-phase component v, while the black ones denote the computed magnitude m of the complex signal. The DISPA plot in **(B)** shows the same data in the complex plane (u,v), with the dots corresponding to the evenly distributed sampling moments.

When the field is not stable, the FID's are not reproducible and data accumulated in the standard way (i.e., summing up the Cartesian components) are dramatically compromised, as shown in **(C)** and in the corresponding DISPA plot **(D)** showing four scans at varying offsets and, in bold, the result of the accumulation. Only a tiny fraction of the FID is usable for signal-intensity estimates if one wants to keep the offset-induced errors below 1%.

What happens is best evidenced in **(E)** where the four colored dots show the position of the complex signal point #32 of the four FID's. Since all four points were taken at the same time t after the excitation pulse, they all have the same amplitude but, because of the unstable offsets, a quite different phase. The amplitude, in fact, is insensitive to phase and depends only upon t and T2*. Averaging the Cartesian coordinates of the four points results in a data point represented by the black dot, which is physically almost meaningless.

Averaging separately the polar coordinates (magnitudes and phases) leads to a correct magnitude estimate. This is shown in **(F)** where the thick green line corresponds to the computed averaged amplitudes of the four FID's. The overlaid white line indicates the ideal amplitude decay (the same as the black trace in A), while the bumpy black line is the result of Cartesian-components averaging (the same as the black trace in C). The averaged magnitudes obtained in this way are valid for the whole duration of the FID's which, in liquids, amounts to a precision-improvement factor of up to 100 (with their very short decays, solids are little affected by these effects).

Exploitation of the phase averages is at present hindered by the lack of suitable averaging algorithms for circular distributions (there is work in progress). Overcoming this obstacle, one should be able to recover an accumulated signal with correct amplitude, corresponding to an average and constant offset value. A great advantage of the new board design is that all future improvement of mathematical algorithms can be implemented by reprogramming its FPGA with no need to redesign the whole board.

1) Ferrante G., Sykora S., *Technical Aspects of Fast Field Cycling*, in Adv.Inorg.Chem., Ed.Rudi van Eldik, Vol.**57**,p.405-470,(2004).
2) Rainer Kimmich, Esteban Anoardo , *Field-cycling NMR relaxometry* in Progress in Nuclear Magnetic Resonance Spectroscopy 44 (2004) 257–320 .
3) Stanislav Sýkora - Gianni Ferrante , *Null-Biased Fast-FieldCycling NMR Sequences, poster at 4th Conf.FC Relaxometry –Torino-2005*
4) A.Galkin, D.Canina, S.Sykora, G.M.Ferrante , *Novel Approaches to the Timing of FFC-NMR experiments – poster at 4th Conf.FC Relaxometry –Torino-2005*