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Low cost, compact, two-channel NMR spectrometer for CP-DNP

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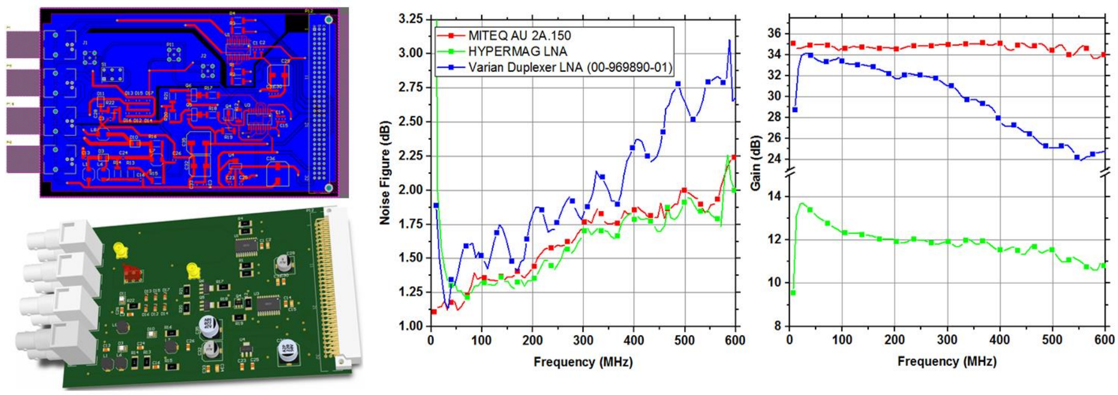
Hyperpolarization by dissolution dynamic nuclear polarization (dDNP) has become a leading method to boost sensitivity of low gamma nuclei (e.g. ^{13}C, ^{6}Li, ^{15}N, etc.) in magnetic resonance experiments, typically up to four orders of magnitude[1]. Nevertheless, the microwave driven polarization transfer from electron spins to the aforementioned nuclear spins is a slow process, especially at high field (6.7 T)[2]. It has recently been demonstrated that cross-polarization (CP) from protons to low gamma nuclei is beneficial to improve both the build-up time and maximum achievable polarization of the later[3]. Monitoring of polarization build-up in the solid state at lower frequencies can be achieved with simple low power, single channel NMR spectrometers with low cost. To the best of our knowledge, CP has so far been implemented using dedicated high-resolution spectrometers. We herein present a 10-450 MHz, two-channel, compact, low-cost solution based on a benchtop spectrometer (Kea[2]; Magritek, New Zealand), a power amplifier (TwinPulse 400, Tomco, Australia) and a home-built duplexer. The improved duplexer allows for experimental flexibility and faster switching between transmitting and receiving modes. All valuable features for hardware demanding NMR sequences, such as CP.

Fabricated on an FR-4 Euroboard PCB, the duplexer installs directly into the Kea[2] console (Figure 1a) and operates between 10 to 450 MHz. The module accommodates three independently controlled TTL outputs (for power amplifier blanking, microwave gating[3] and a spare). The duplexer switching mechanism consists of a $\lambda/4$ cable, PIN diodes (MAP504-1072T; MACOM, USA) and three pairs of crossed 1N4148 diodes thus reducing the dead time $< 2\mu s$. The preamplifier consists of a single stage low noise amplifier (LNA). Two candidate LNAs were tested (Figure 1b and 1c), AU-2A-150 (Miteq, USA) and a HYPERMAG-developed GaAs LNA and benchmarked against the performance of an INOVA preamplifier (Part No. 01-9104-08; Varian Inc, USA). The selected LNAs provide a maximally flat response with a noise figure < 2 dB. Low gain in the HYPERMAG LNA permits the cascade of the Kea[2] variable RX amplifier module without saturating the spectrometer's analog-to-digital converter.

The NMR spectrometer was tested at 400 MHz on a liquid sample against an Agilent Direct Drive console. Similar performance was obtained. Likewise, similar performance to standard NMR consoles for solid state NMR detection was obtained. The two channels allow simultaneous pulsing on two nuclei and may therefore be suitable for certain advanced sequences that does not require large memory or fast update rates. A $^{1}H/^{13}C$ CP sequence was implemented and tested with similar performance as an Agilent Direct Drive console.



(a) (b) (c)

Figure (1a): *Duplexer layout and module (160 mm x 100 mm) with 8 BNC connectors (XMETER (IN), LNA (OUT), Probe (IN/OUT), $\lambda/4$ (IN/OUT), 3 TTL (OUT)).* ***(1b, 1c)****: Measured noise figure and gain of MITEQ AU-2A-150, HYPERMAG and INOVA duplexer LNAs using E4440A spectrometer calibrated with 346B noise source (Keysight Technologies, USA).*

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