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Simple Data Acquisition System for Proton-Enhanced Nuclear Induction Spectroscopy

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A simple inexpensive data acquisition system is described which permits accumulation of 1024 digital points in <3 msec. This is useful for proton-enhanced nuclear induction spectroscopy where excessive accumulation time causes extraneous expenditure of rf power during multiple cross-polarization experiments. A brief example of the operation is presented.

An important requirement for multiple cross-polarization versions of proton-enhanced nuclear induction spectroscopy (*1*) is the rapid and efficient accumulation of data between cross-polarizations. That this is more than just a question of elegance and is of crucial technical importance can be made clear by considering carefully the process in question; sensitivity enhancement is effected by transfer of nuclear magnetic polarization between an abundant nuclear species I and a dilute species S under observation, with repeated cycles of cross-polarization, observation, and data accumulation. In the most convenient form of this multiple cross-polarization approach, the I spins are strongly irradiated to induce spin-locking during the cross-polarization step, and spin-decoupling during observation to produce high-resolution S spectra. Normally, the irradiation must continue during the accumulation of the S signal, and if this is a time-consuming process, an unnecessary expenditure of high-power rf ensues, causing problems of heat dissipation, limiting the range of I systems which can be studied, and reducing the sensitivity enhancement. This vitiates the whole basis of these experiments and the inherent advantages over the indirect detection schemes (*2*, *3*).

We have employed in our spectrometer (*4*) an exceedingly simple data acquisition system, utilizing a Biomation 802 transient recorder, interfaced to a PDP8/e minicomputer, which meets the timing requirements of these experiments with no additional hardware. The system is simple and quite inexpensive, yet very flexible, and has proved useful for studies of high-sensitivity ^{13}C NMR (*5*) and coherent transients in solids (*6*). Figure 1 depicts the system schematically, together with a simplified timing diagram. The acquisition is tailored to a single-channel phase detector and 1024 points. An IOT command issued by the computer determines options such as permit or refuse "break requests," select memory quadrant, and store or add to memory for accumulation.

Data break transfers are initiated after a "record" cycle is completed by the Biomation 802, which brings the record line low. The interface then asserts "output command"

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until all word transfers are completed. Following this, the Biomation raises the "flag" line up indicating that word "O" is available. Both address and data information are stored in the interface and "break request" is transmitted to the computer. After the data break is complete and the first word has been transferred to the computer, "word

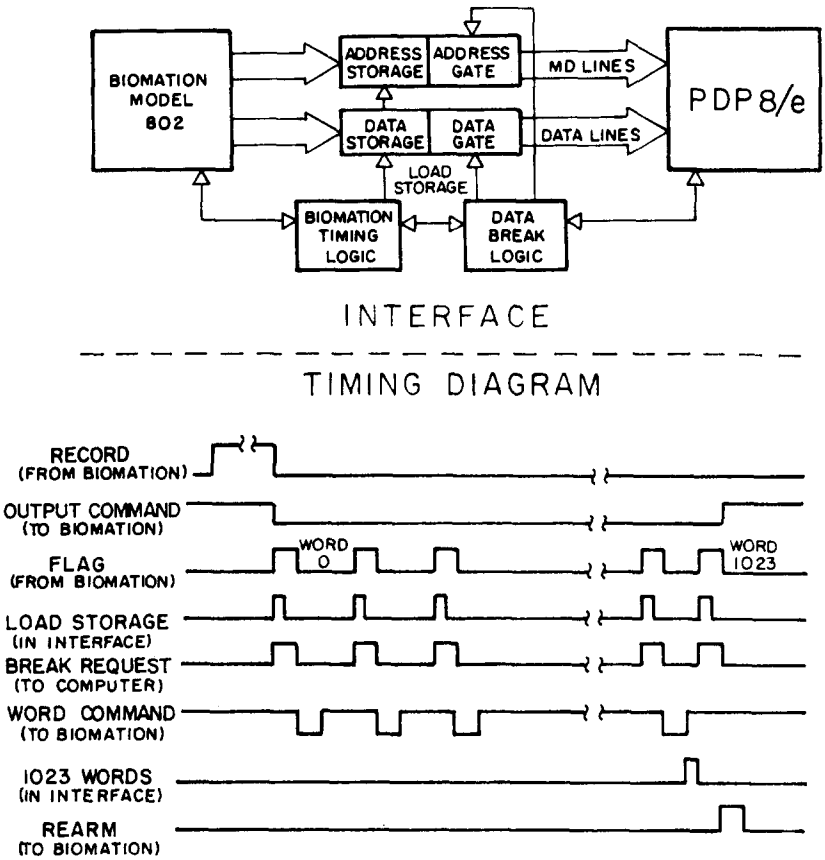

FIG. 1. Simplified schematic diagrams of interface for data acquisition and accumulation employing "data break" and corresponding timing pulses.

command" is asserted to the Biomation. When the next word is ready, the "flag" line goes up and the data break sequence is repeated. This continues until the Biomation is unloaded, which is detected by decoding address 1023. The "rearm" signal then signifies that the recorder is ready for the next cycle. The total transfer and accumulation takes <3 msec, which is normally absorbed in the cross-polarization time, so no additional time is added and no extraneous rf power is expended. The interface utilizes commercially available MSI circuits and is incorporated on a single PDP8/e circuit board.

The traces in Fig. 2 depict the operation of the system on a prototype sample of solid adamantane, and provide a visual illustration of the process of rapid accumulation and sensitivity enhancement. Details appear in the caption. The final signal, for 20 cross-polarizations, was acquired in 0.4 sec and further enhancement was limited only by

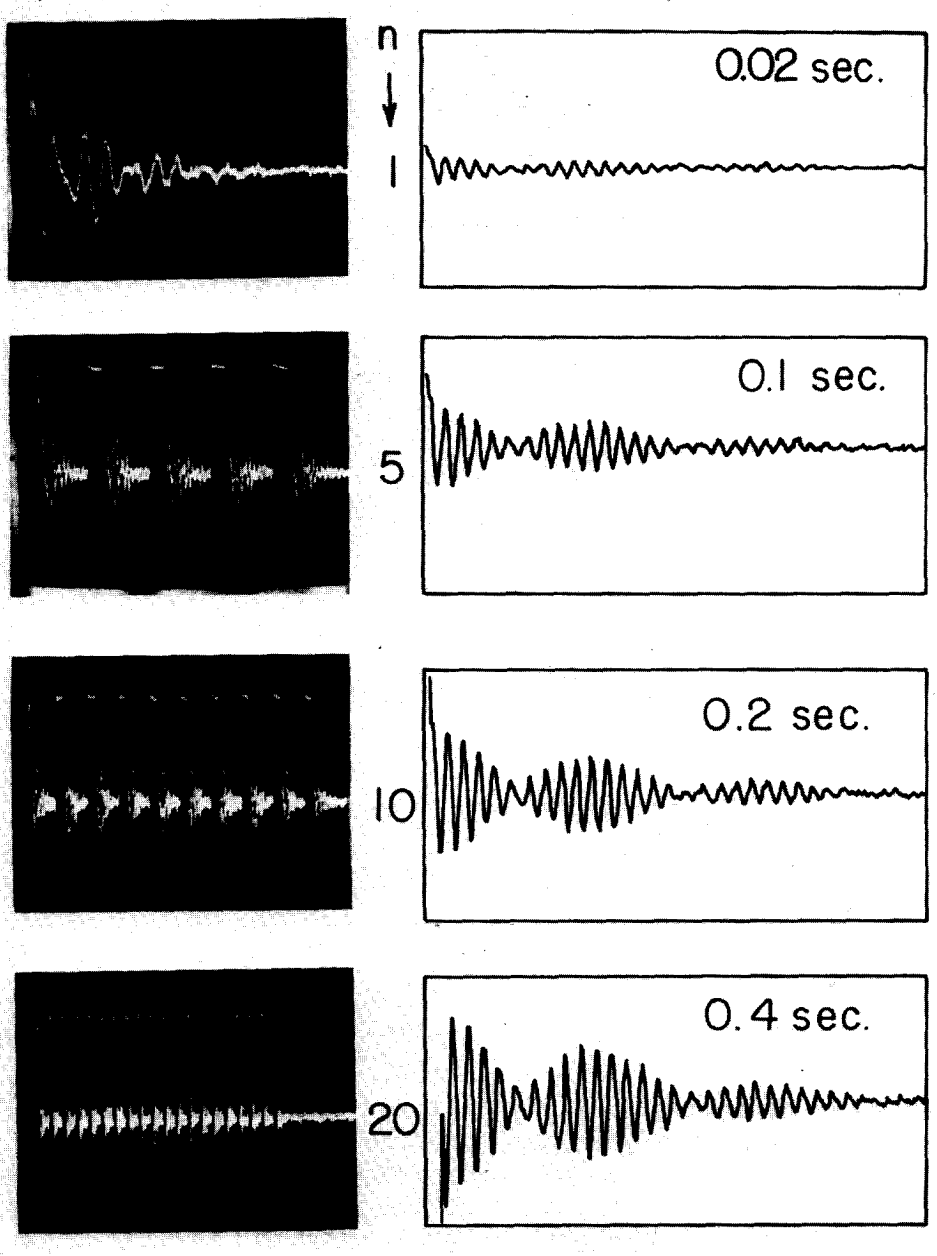

FIG. 2. ^{13}C NMR in solid adamantane. The oscilloscope photographs on the left depict the output of the ^{13}C phase detector for various numbers (n) of $^{1}H \rightarrow {}^{13}C$ cross-polarization steps in a proton-enhanced NMR experiment. The positive pulses are from rf receiver blocking during the cross-polarization, followed by proton-decoupled free induction decays. It is these which are rapidly transferred and accumulated by the computer. The cross-polarization pulses are 5 msec long. The traces on the right show the accumulated signals and acquisition times corresponding to the number of cross-polarizations on the left. These were recorded further off resonance.

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