



NMR APPLICATIONS NOTE NUMBER 4

Automatic Measurement and Storage of Inversion Recovery T_1 Measurements

The measurement of T_1 , the spin-lattice relaxation time has been of interest to chemists in recent years because of its relationship to the rotational correlation time.¹ One of the more common methods for the measurement of T_1 is the inversion recovery pulse sequence, first described by Vold, Waugh, Klein and Phelps.² This sequence has also been called the Partially Relaxed Fourier Transform technique by Allerhand.¹ This PRFT sequence has also been shown to be of some use in the analysis of spectra containing overlapping lines having different relaxation times.³

The inversion recovery sequence is performed using a pulse sequence often abbreviated as $[180 - \tau - 90 - (\text{sample}) - T]_n$ where n is the number of scans to be signal averaged, T is the recovery time of the system, usually at least 5 times the longest T_1 , and τ is a variable delay time between the two rf pulses.

The variable delay τ is chosen so that it starts out much shorter than the shortest T_1 in the system and several increments are chosen throughout the range. The longest τ is much longer than the longest T_1 in the system, usually, so that this τ - value approaches the recovery time T . This last value is usually called τ_∞ .

During this experiment, spectra are accumulated in the signal averager after each 90° pulse and after sufficient scans have been obtained, the averaged free-induction decay is stored on magnetic disk for later analysis. When $\tau \ll T_1$, the resulting spectrum will show a completely inverted peak, when $\tau = T_1 \ln 2$ the spectrum will show a null, and when $\tau \gg T_1$, a normal upright peak will be produced. The values of T_1 for each line in the decoupled carbon spectrum can then be determined by least squares analysis of the lines in the spectra where it has been shown that the amplitude of a line in any given spectrum is given by

$$A = A_0 [1 - 2 \exp (-\tau/T_1)]$$

Least squares analysis on this set of spectra will produce a set of quite reliable T_1 - values from these spectra.

The Nicolet Inversion-Recovery Package

The Nicolet system is composed of the NIC-293 General Controller so configured as to produce a set of two pulses and two delays, one of which is varied between experiments by the software, and the NIC-294 disk system.

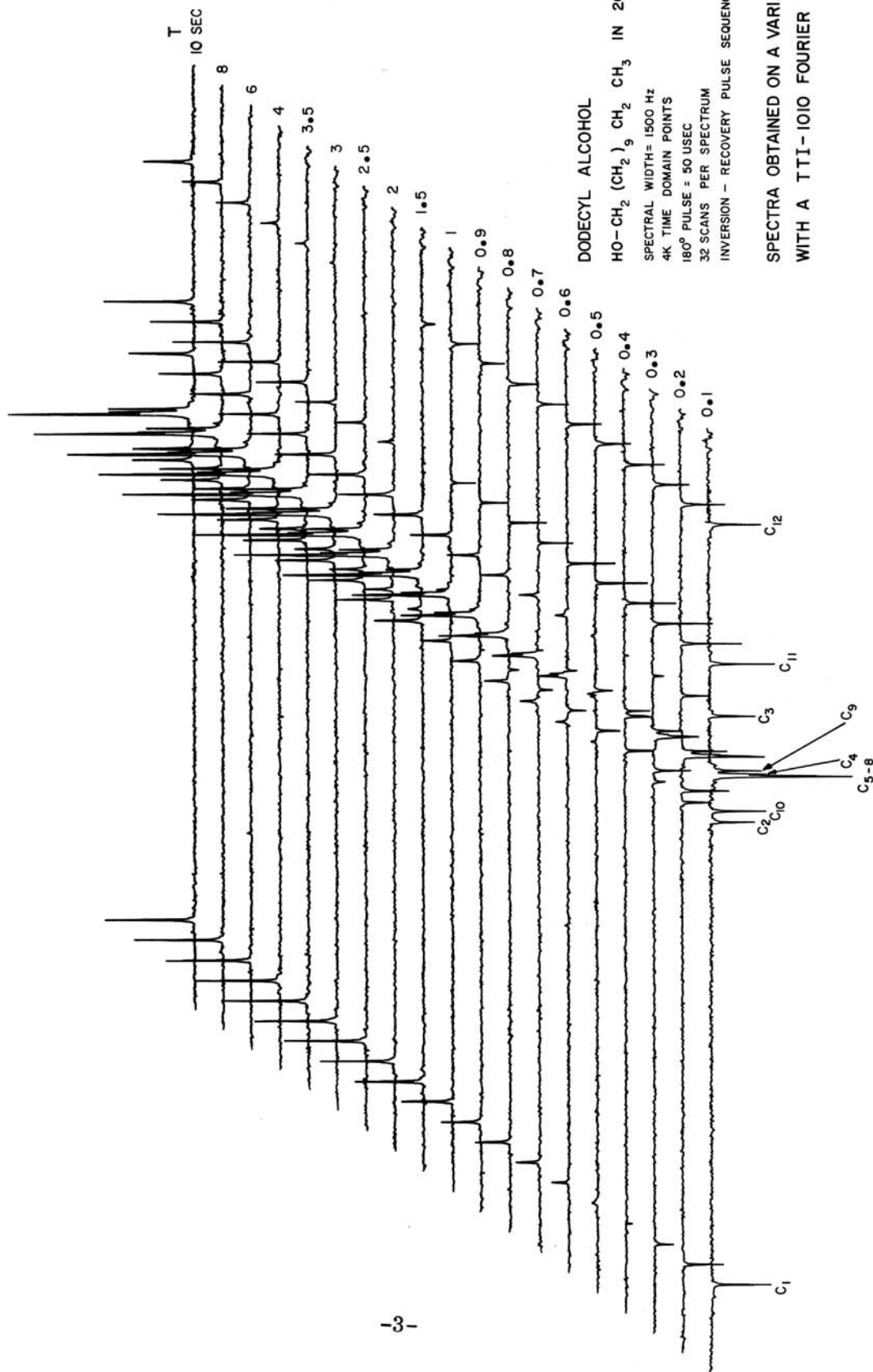
During the actual experiment, a list of τ - values is entered in tabular form with a maximum of 30 different values in a single list. The pulse widths for an optimum 180 and 90° pulse are determined and entered and the total delay time between scans, T, is entered. Then a file name is given to the program for disk storage of the accumulated data at each τ - value and the entire data acquisition proceeds automatically from there on. The GO command begins by clearing memory and loading the 293 timing modules with the desired pulses and delay times. The edge of the 90° pulse triggers data acquisition in the 1080 and after the number of scans set on the Autostop counter have been taken, the data is stored on disk. The τ - value is then changed, memory zeroed and new data acquired. When all the scans at all τ - values have been obtained and stored the program awaits further instructions.

In this case, the commands are to process the data using the parameters set into the FT-Nmr program for resolution or sensitivity enhancement, Fourier transformation and phase correction, to view successive scans on the CRT, and to produce a stacked isometric plot such as was shown in the original paper on the subject.²

This approach frees the user from two conventional worries: first, that the data will be lost if the pen skips while plotting and second, that the data will not be as useful as desired because of an incorrect choice in the exponential or trapezoidal window parameters. In the Nicolet system, the data are always stored on the disk for recall, and no data are lost by operator error.

The spectrum shown on the following page was obtained using this program on an XL-100 with a TTI-100 Fourier Accessory. The technical data are given in the figure. The spectra were provided to Nicolet by its affiliate, Transform Technology, Inc.

Those customers interested in performing this experiment on XL-100's should contact Transform Technology, and those who wish to perform it on other spectrometers along with a 1080 and disk system should contact Nicolet directly. Copies of this program are provided free of charge to all Nicolet customers having 293 Controllers and Disk systems.



DODECYL ALCOHOL

$\text{HO}-\text{CH}_2(\text{CH}_2)_9\text{CH}_3$ IN $20\% \text{CDCl}_3$

SPECTRAL WIDTH = 1500 Hz

4K TIME DOMAIN POINTS

180° PULSE = 50 USEC

32 SCANS PER SPECTRUM

INVERSION - RECOVERY PULSE SEQUENCE ... $T-180^\circ-T-90^\circ$ - SAMPLE ...

SPECTRA OBTAINED ON A VARIAN XL-100

WITH A TTI-1010 FOURIER TRANSFORM ACCESSORY

References

1. A. Allerhand, D. Doddrell, V. Glushko, D. W. Cochran, E. Wenkert, P. J. Lawson and F. Gurd, J. Am. Chem. Soc. 93, 544 (1971).
2. R. L. Vold, J. S. Waugh, M. P. Klein, and D. E. Phelps, J. Chem. Phys. 48, 3831 (1968).
3. A. Allerhand and D. Doddrell, J. Am. Chem. Soc. 63, 2777 (1971).