Nuclear Magnetic Resonance (NMR)

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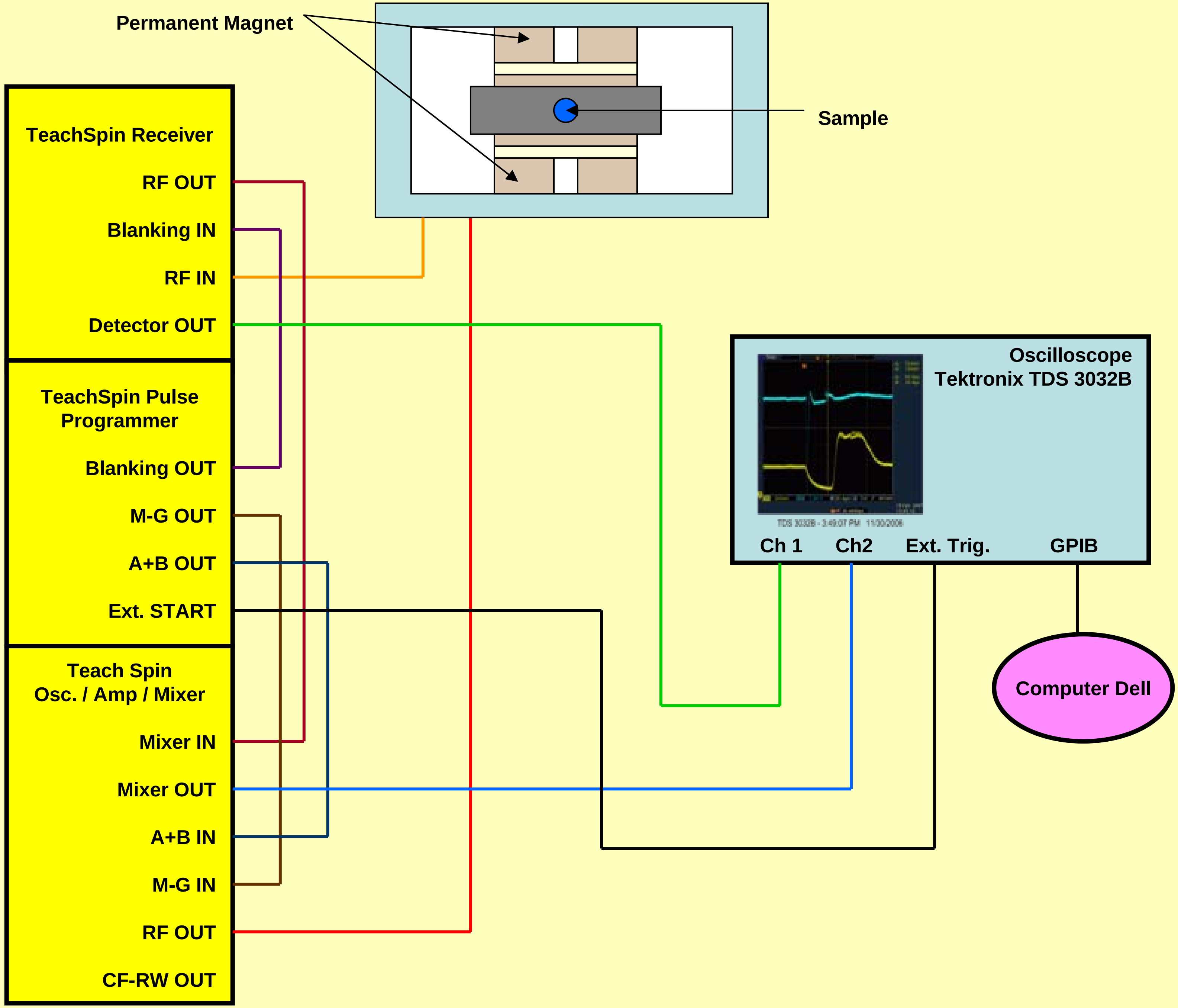
The **N**uclear **M**agnetic **R**esonance Spectroscopy (NMR) is one of the most important spectroscopic methods to explore the structure and dynamic of molecules especially in organic chemistry and biochemistry. Suitable subjects for this method are materials with odd nucleon numbers in their nucleus. All nuclei that contain odd numbers of protons or neutrons have an intrinsic magnetic moment and angular momentum. The most commonly measured nuclei are hydrogen-1 (the most receptive isotope at natural abundance) and carbon-13, although nuclei from isotopes of many other elements (e.g. ^{15}N, ^{14}N, ^{19}F, ^{31}P, ^{17}O, ^{29}Si, ^{10}B, ^{11}B, ^{23}Na, ^{35}Cl, ^{195}Pt) can also be observed. NMR resonant frequencies for a particular substance are directly proportional to the strength of the applied magnetic field, in accordance with the equation for the Larmor precession frequency. In an external magnetic field the magnetic momentum of the nucleus gets aligned according to quantum mechanical rules and the nuclear Eigenstates split in energy. The energy difference between these states is proportional to the magnetic field strength and depends also on the so-called gyromagnetic ratio. Transitions between those states can be resonantly induced by application of electromagnetic radiation of the appropriate frequency. One observes a phenomenum called relaxation which describes several processes by which nuclear magnetization prepared in a non-equilibrium state return to the equilibrium distribution. In other words, relaxation describes how fast spins "forget" the direction in which they are oriented. The rates of this spin relaxation can be measured in both spectroscopy and imaging applications. One observes: A. Longitudinal Relaxation or Spin-Lattice-Relaxation (M_z) characterized by the relax.-time T_1 and B. Transversal Relaxation or Spin-Spin-Relaxation (M_x and M_y) characterized by T_2, the spin-spin relaxation time T_2.

Two main methods are applied in NMR-spectroscopy, 1. The continuous wave spectroscopy (CW-NMR) and 2. The pulsed NMR. Further, one applies Fourier-transforms to obtain spectra in the time and the frequency domain.



Permanent Magnet
Sample
TeachSpin Receiver
RF OUT
Blanking IN
RF IN
Detector OUT
TeachSpin Pulse Programmer
Blanking OUT
M-G OUT
A+B OUT
Ext. START
Teach Spin Osc. / Amp / Mixer
Mixer IN
Mixer OUT
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Oscilloscope Tektronix TDS 3032B
Ch 1
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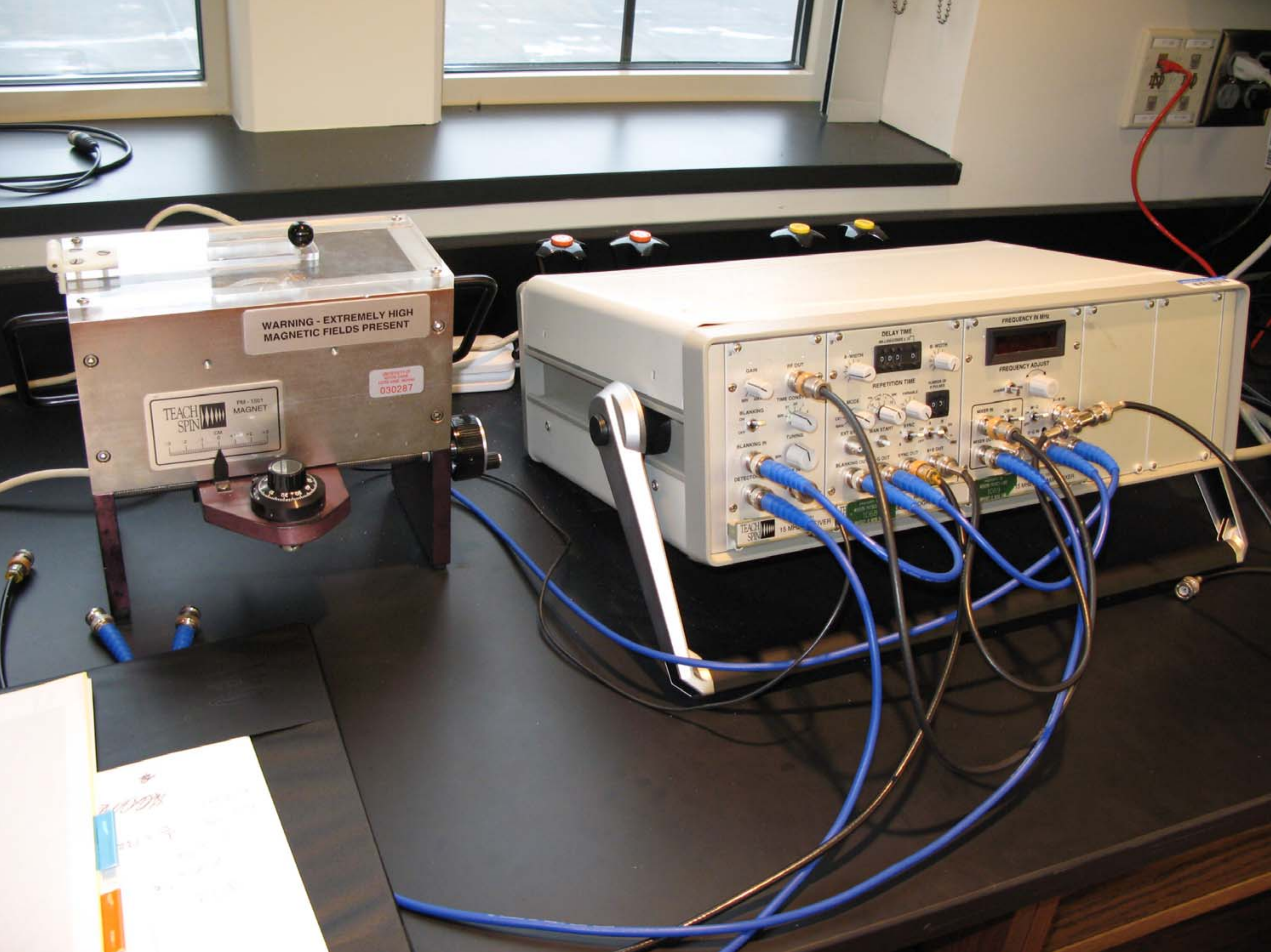
GPIB
Computer Dell


WARNING - EXTREMELY HIGH
MAGNETIC FIELDS PRESENT
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MAGNET
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Nuclear Magnetic Resonance (NMR) : Required Knowledge

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- Principles of
- Principles of
- What ?
- What?
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- Comp
- Principles of

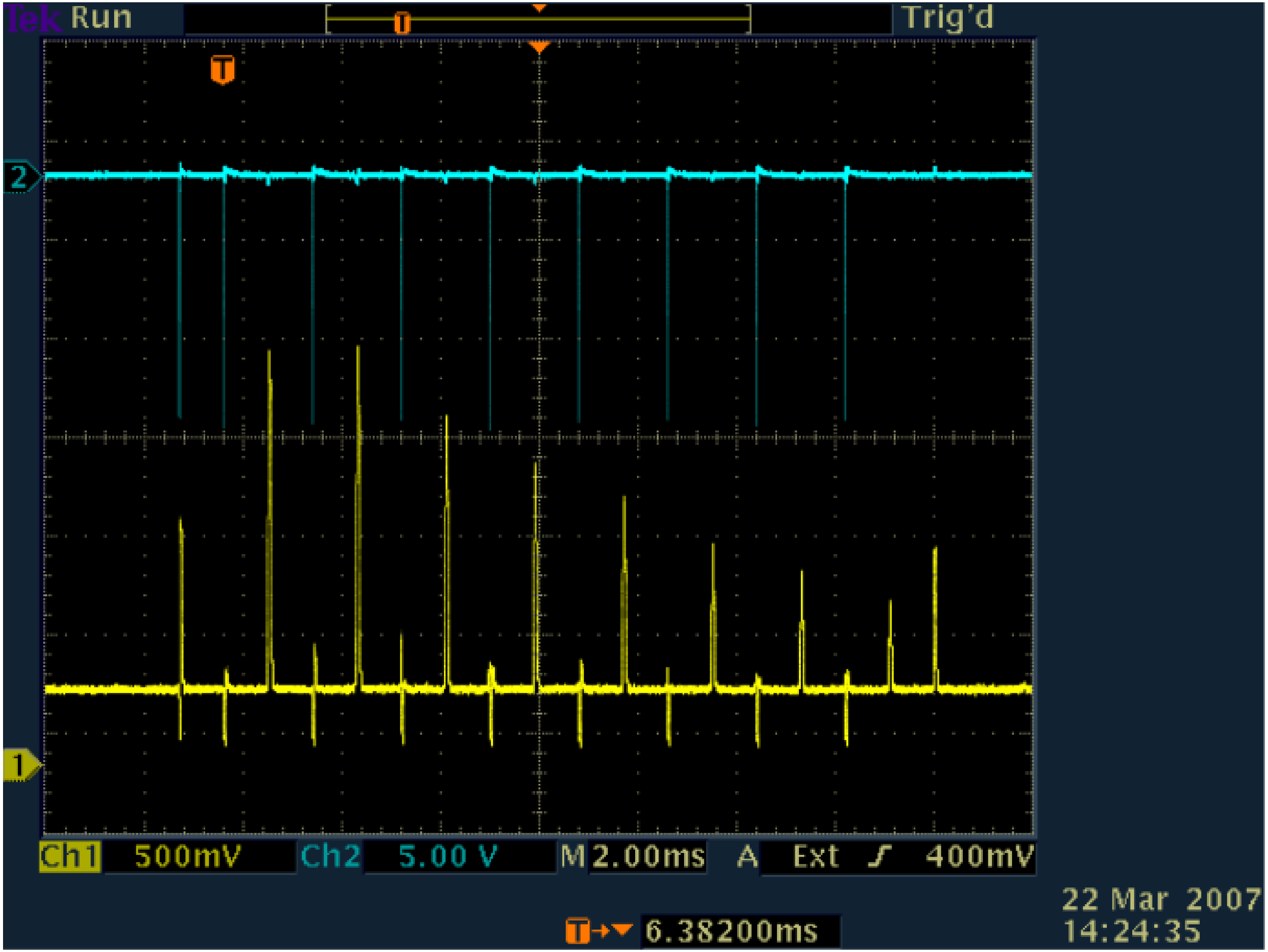
Nuclear Magnetic Resonance (NMR) : Tasks and Goals

- Set-up
- Produce
- Set-up
- Determine
- Determine
- Determine energy
- Determine the

- Measure energy
- Determine
- Compare energy

WARNINGS

- Be careful.
- Shut down
- Never touch
- Remove source after measurement



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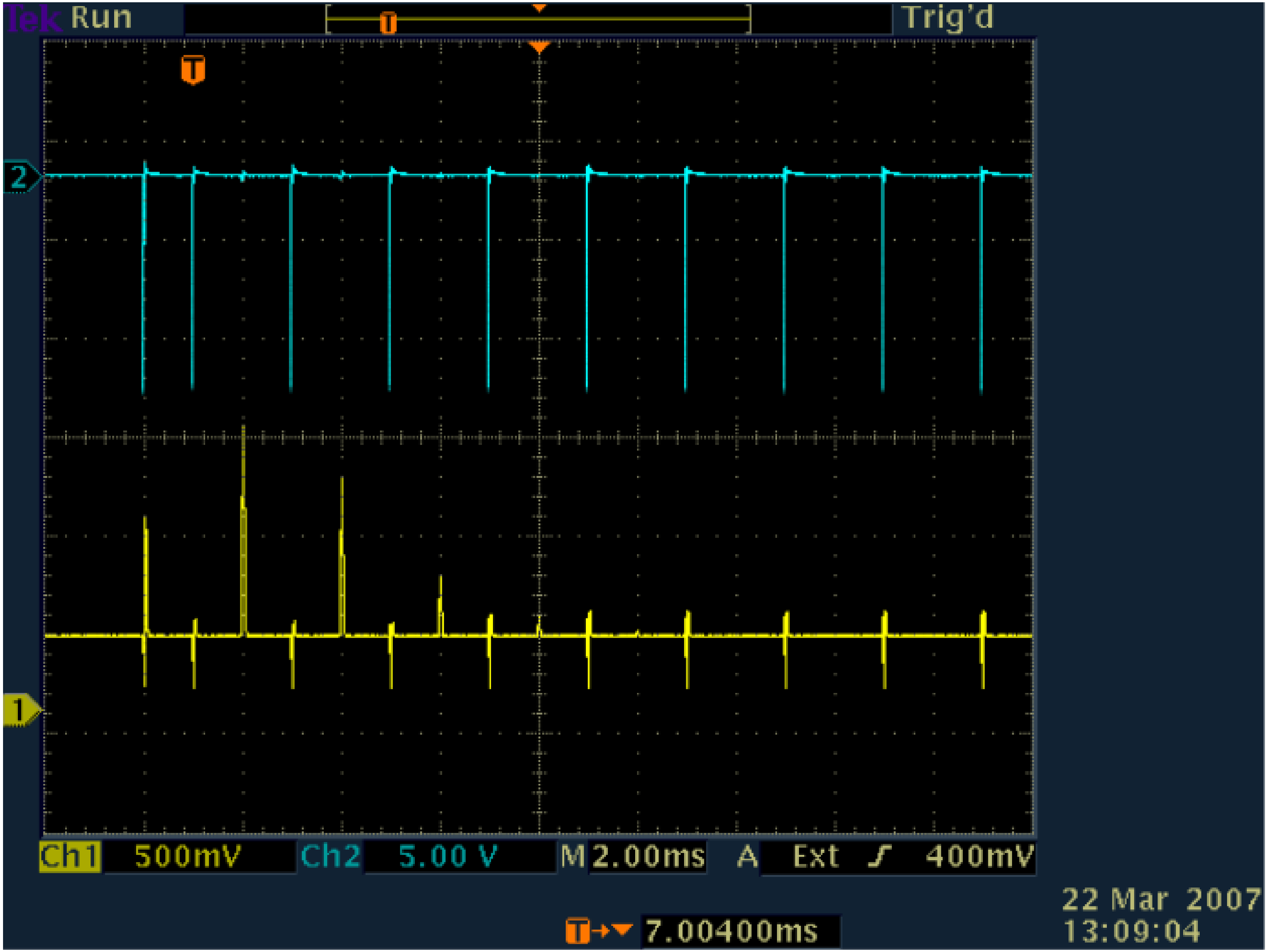
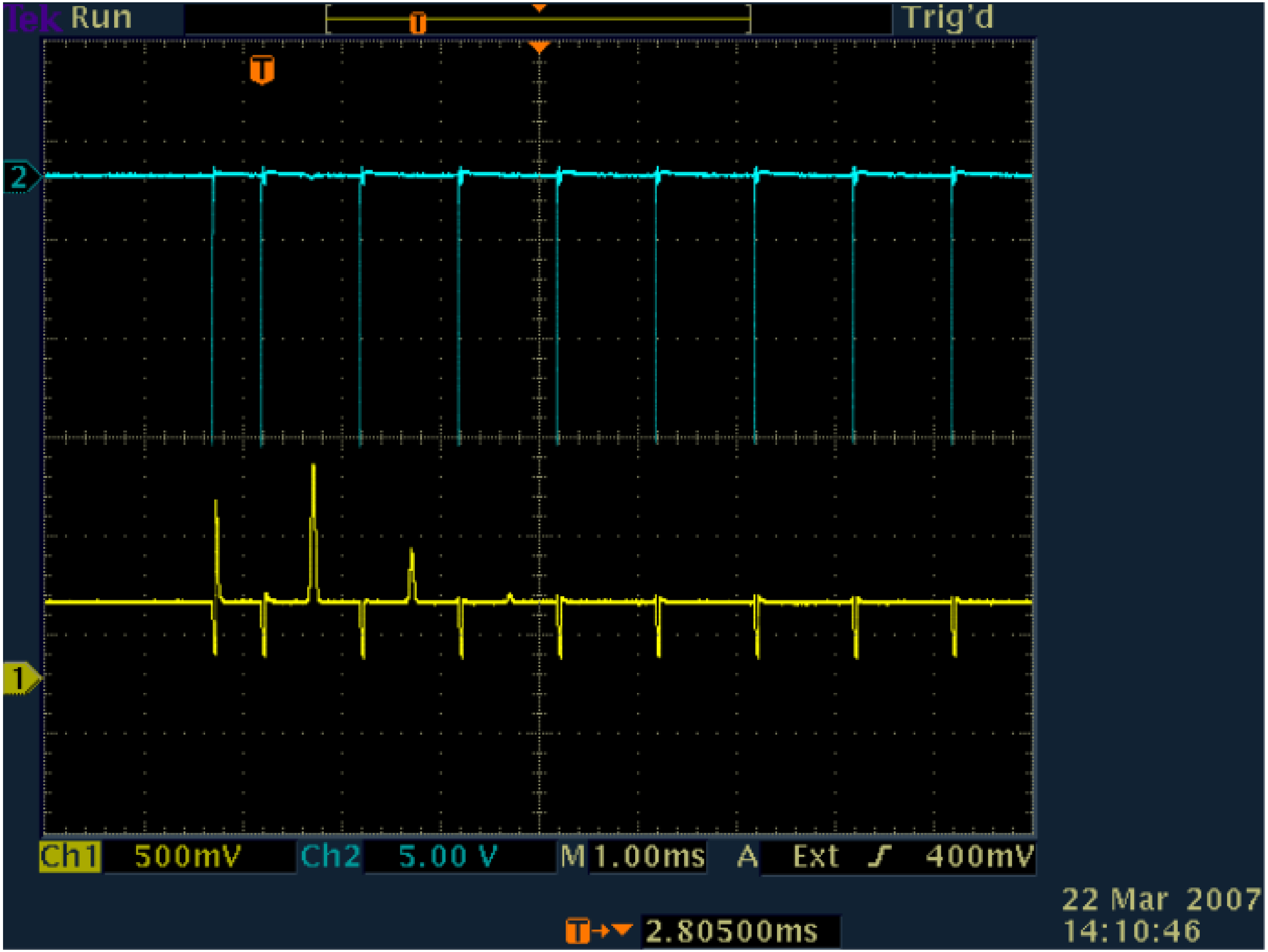


CuSO4 - .25 M Multipulse e decay



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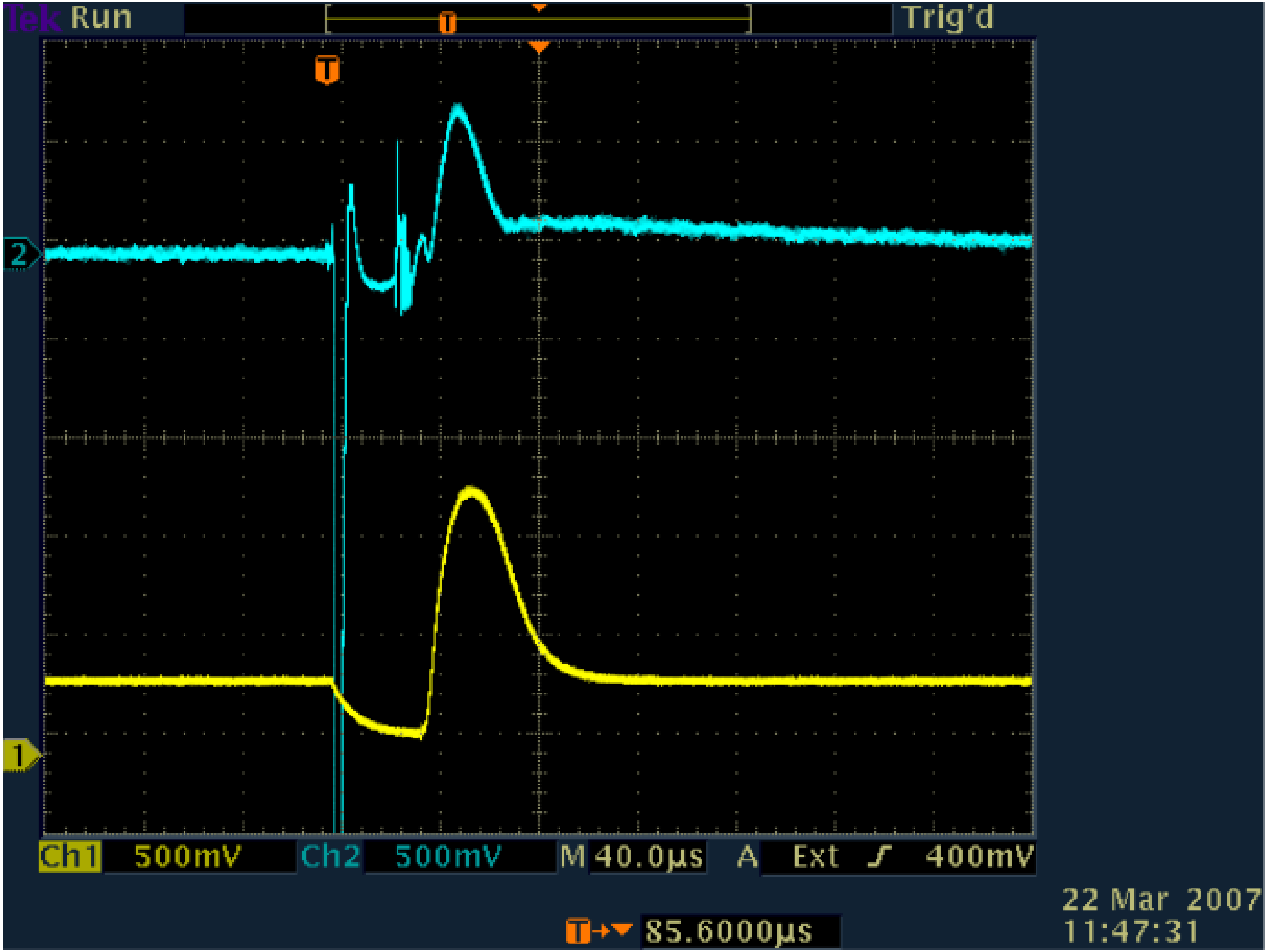
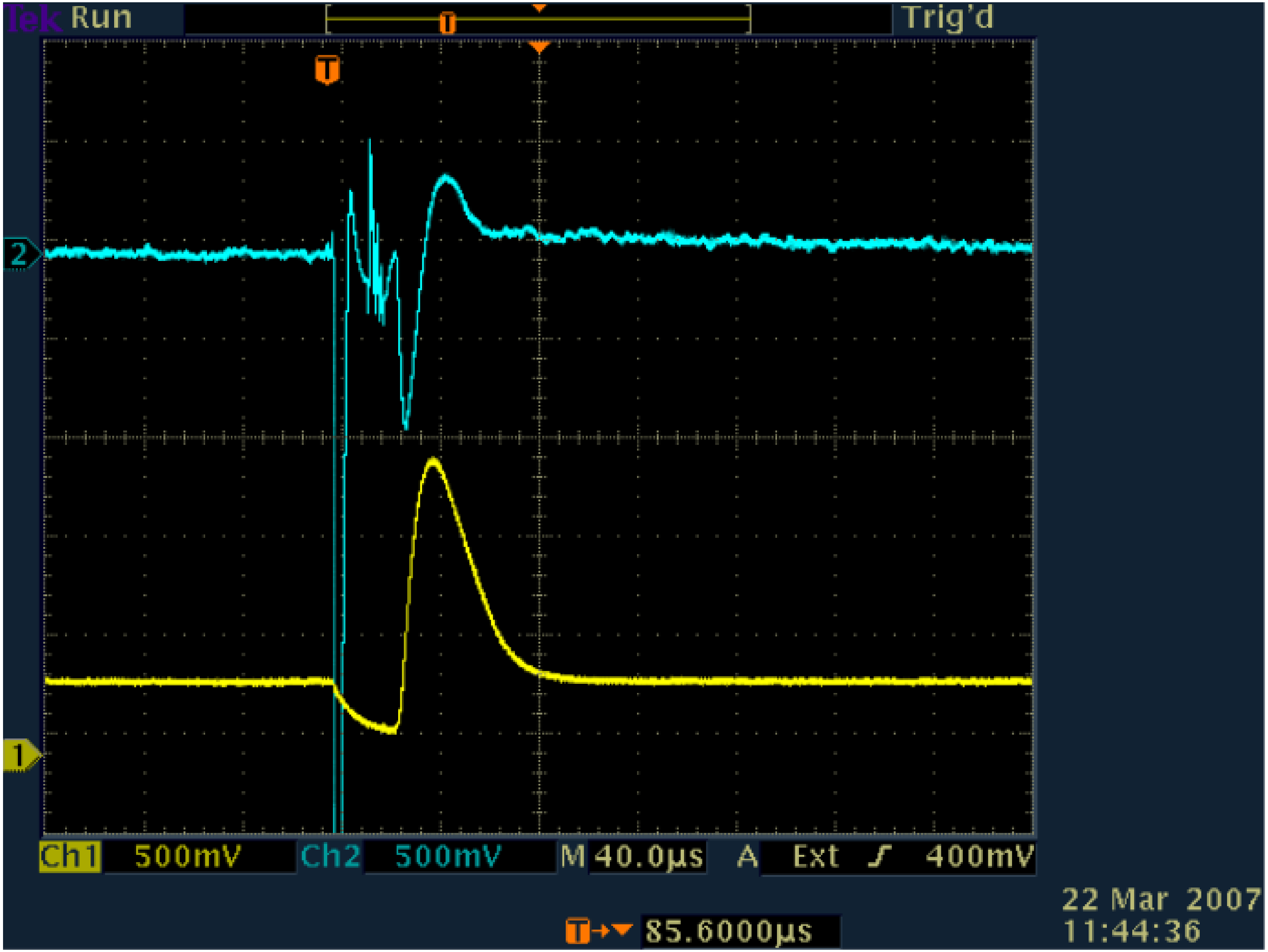
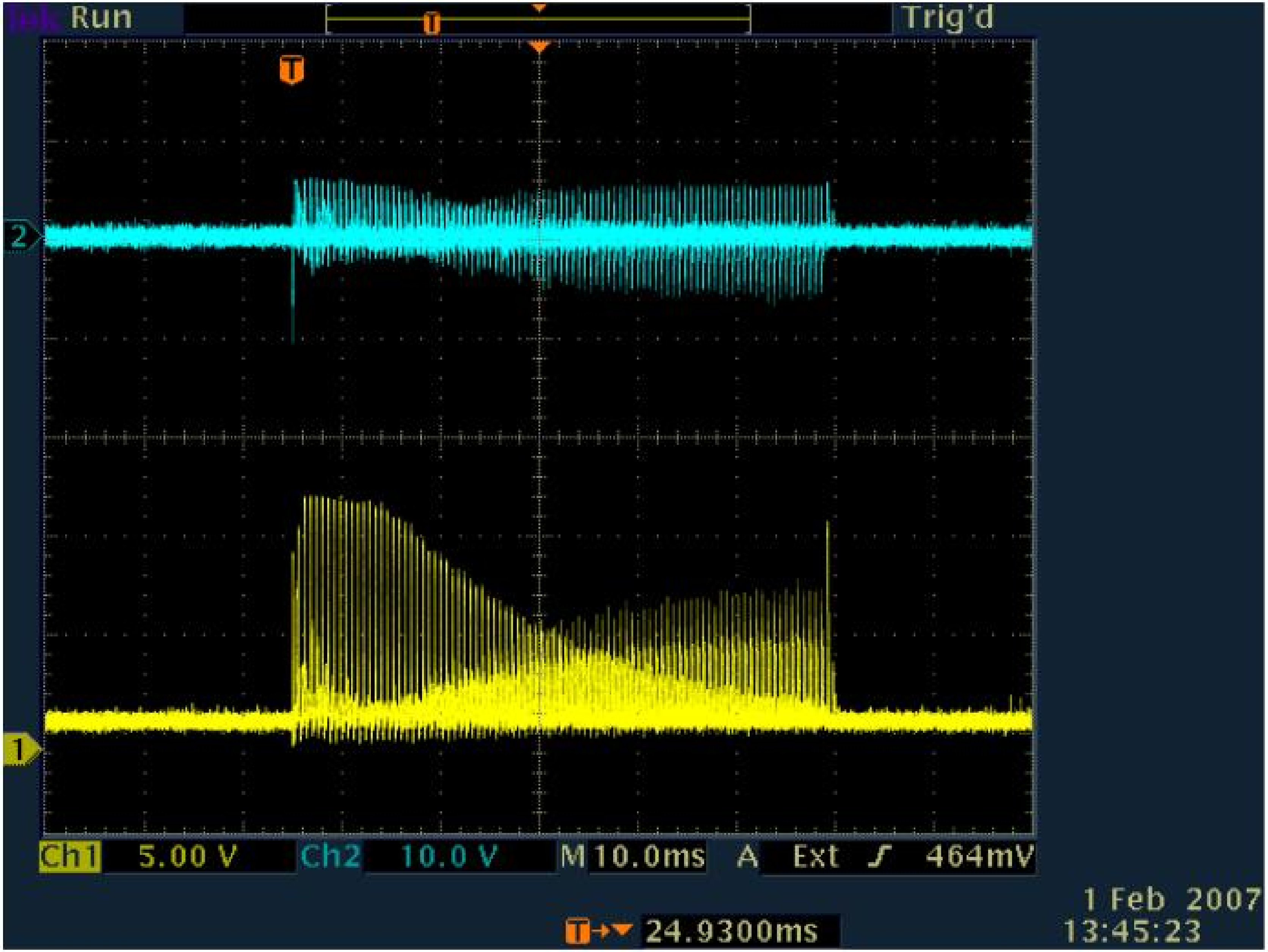


CuSO4 - .1 Molar - Pi - FID Signal



CuSO4 - .1 Molar - Pi/2 - FID Signal



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Isotopes with odd nucleon numbers used for NMR

Many chemical elements can be used for NMR analysis :

- ^{1}H, the most commonly used, very useful. Highly abundant, the most sensitive nucleus apart from tritium. Narrow chemical shift, but sharp signals. In particular, the ^{1}H signal is that used in magnetic resonance imaging.
- ^{2}H, commonly used in the form of deuterated solvents to avoid interference of solvents in measurement of ^{1}H. Rarely used in NMR measurents themselves, due to low resolution and low sensitivity; mainly utilized in determining of effectiveness of chemical deuteration.
- ^{3}He, very sensitive. Low percentage in natural helium, has to be enriched. Used mainly in studies of endohedral fullerenes.
- ^{13}C, commonly used. Low percentage in natural carbon, therefore spectrum acquisition takes a long time. Frequently used for labeling of compounds in synthetic and metabolic studies. Has low sensitivity and wide chemical shift, yields sharp signals. Low percentage makes it useful by preventing spin-spin couplings and makes the spectrum appear less crowded.
- ^{15}N, relatively commonly used. Can be used for labeling compounds. Nucleus very insensitive but yields sharp signals. Low percentage in natural nitrogen together with low sensitivity requires high concentrations or expensive isotope enrichment.
- ^{14}N, medium sensitivity nucleus with wide chemical shift. Its large quadrupole moment interferes in acquisition of high resolution spectra, limiting usefulness to smaller molecules.
- ^{19}F, relatively commonly measured. Sensitive, yields sharp signals, has wide chemical shift.
- ^{31}P, 100% of natural phosphorus. Medium sensitivity, wide chemical shift range, yields sharp lines. Used in biochemical studies.
- ^{17}O, low sensitivity and very low natural abundance.
- ^{10}B, lower sensitivity than ^{11}B. Use quartz tubes, as borosilicate glass interferes with measurement.
- ^{11}B, more sensitive than ^{10}B, yields sharper signals. Use quartz tubes, as borosilicate glass interferes with measurement.
- ^{35}Cl and ^{37}Cl, broad signal. ^{35}Cl significantly more sensitive, preferred over ^{37}Cl despite its slightly broader signal. Organic chlorides yield very broad signals, use limited to inorganic and ionic chlorides and very small organic molecules.
- ^{43}Ca, used in biochemistry to study calcium binding to DNA, proteins, etc. Moderately sensitive, very low natural abundance, has to be enriched.
- ^{195}Pt, used in studies of catalysts and complexes.
- Other nuclei, usually used in the studies of their complexes and chemical binding, or to detect presence of the element : ^{6}Li, ^{7}Li, ^{9}Be, ^{21}Ne, ^{23}Na, ^{25}Mg, ^{27}Al, ^{29}Si, ^{33}S, ^{39}K, ^{40}K, ^{41}K, ^{45}Sc, ^{47}Ti, ^{49}Ti, ^{50}V, ^{51}V, ^{53}Cr, ^{55}Mn, ^{57}Fe, ^{59}Co, ^{61}Ni, ^{63}Cu, ^{65}Cu, ^{67}Zn, ^{69}Ga, ^{71}Ga, ^{73}Ge, ^{77}Se, ^{81}Br, ^{87}Rb, ^{87}Sr, ^{95}Mo, ^{109}Ag, ^{113}Cd, ^{125}Te, ^{127}I, ^{133}Cs, ^{135}Ba, ^{137}Ba, ^{139}La, ^{183}W, ^{199}Hg.