

Chapter 2

Pulsed NMR and NQR Spectrometers

2-1. Pulsed NMR Spectrometer

A homemade spectrometer was used for the measurement of the ^1H spin-lattice relaxation time ($T_{1\text{H}}$). This spectrometer was constructed by assembling the following components: An Anritsu MG3601 frequency synthesizer, a Thamway A57-4702 wide-band power amplifier, a JEOL JES-CC2 electromagnet, a Matec Model 251 (45-90 MHz) preamplifier, an R&K A-520 main amplifier, Thamway Model P10-6601, P10-6602 and P10-6701 phase shifters, an R&K Model M12CA phase sensitive detector, an Iwatsu DS-9121 storage scope, a Tektronix TM 502A DC amplifier, a homemade pulse programmer, a homemade pulse gate, and a home made RF probe head. The composition of the pulsed NMR spectrometer is shown in Figure 2-1.

In a temperature range 85-300 K, the sample temperature was controlled by a CHNO SU10-2121 LNN temperature controller and measured by a digital multimeter with a chromel-P-constantan thermocouple within ± 1 K. Nitrogen gas flow from a vessel of liquid nitrogen was used for cooling the sample in a temperature was range 85-300 K. In a temperature range 30-85 K, the sample temperature was controlled by using an OXFORD ITC500 temperature controller, an OXFORD CF1200 cryostat, an

OXFORD VC300 helium flow meter, and an OXFORD GF3 helium pump, and measured by a digital multimeter with gold-0.07 atomic percent iron vs KP thermocouple within ± 0.5 K. Helium gas flow from a vessel of liquid helium was used for cooling the sample in a temperature range 30-85 K.

T_{1H} was determined by the saturation recovery method using the $\pi/2$ ($-\tau$ - $\pi/2$)⁸ - τ - $\pi/2$ pulse sequence, where a $\pi/2$ pulse width of *ca.* 3.5 μ s and τ of 1 ms were employed, and a Larmor frequency of 54.3 MHz was used.

For the ¹H NMR T_{1H} measurement, polycrystalline samples were sealed in glass ampoules under a dry nitrogen or a helium atmosphere.

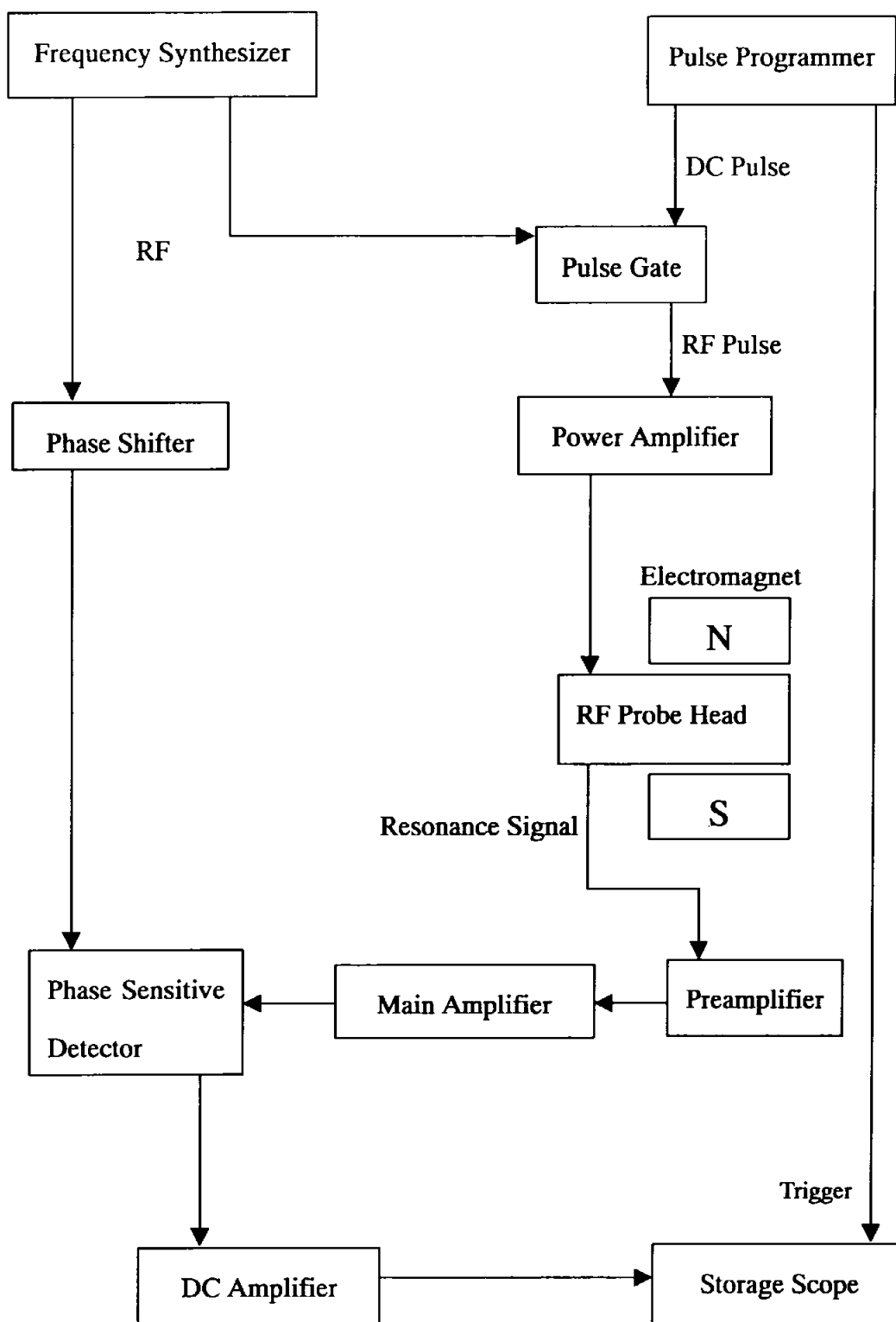


Figure 2-1. The composition of the pulsed NMR spectrometer.

2-2. Pulsed NQR Spectrometer

A homemade pulsed NQR spectrometer was employed for the measurement of the ^{35}Cl NQR frequency and spin-lattice relaxation time (T_{1Q}). This spectrometer was constructed with an Anritsu MG3601A frequency synthesizer, a Thamway N210-126C pulse programmer, an R&K A-520-S pulse gate, an R&K A8520-RS wide-band power amplifier, a Matec Model 251 (20-50 MHz) preamplifier, an R&K A-520 main amplifier, a Thamway Model P10-6704 phase shifter, an R&K Model M12CA phase sensitive detector, a Tektronix TM502A DC amplifier, an Iwatsu DS-9121 storage scope, and a homemade RF probe head. The composition of the pulsed NQR spectrometer is shown in Figure 2-2.

The sample temperature was controlled by a CHNO SU10-2121 LNN temperature controller and measured by a digital multimeter with a chromel-P-constantan thermocouple within ± 1 K. Nitrogen gas flow from a vessel of liquid nitrogen was used for cooling the sample.

The ^{35}Cl NQR frequency was determined from the FID shape after a $\pi/2$ pulse, and the T_{1Q} was determined by the inversion recovery method using the $\pi - \tau - \pi/2$ pulse sequence, where a π pulse width of *ca.* 20 μs and a $\pi/2$ pulse of *ca.* 10 μs were employed.

For the ^{35}Cl NQR frequency and T_{1Q} measurements, samples were sealed in glass ampoules under a dry nitrogen atmosphere

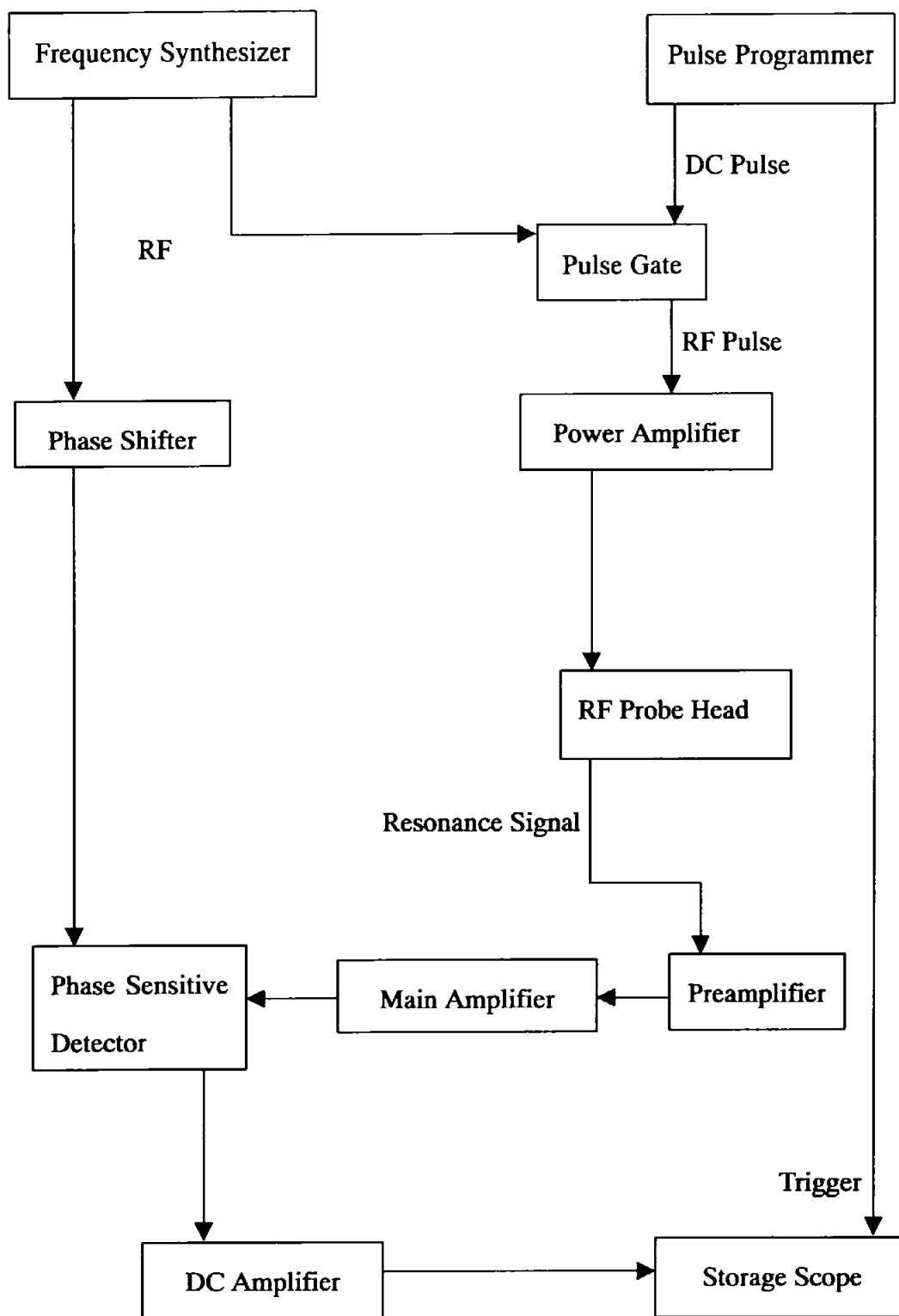


Figure 2-2. The composition of the pulsed NQR spectrometer.