

## **Amplitude Modulation Method for Producing Sidebands in NMR Experiments**

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urements show that  $grad(H^2)$  [Eq. (6)] can be calculated from macroscopic measurements.

The lines of flow, as shown in Fig. 2, were observed by placing an iron cylinder within a glass cell and applying a uniform magnetic field. The cell was filled with a suspension of polystyrene lattices of 25 to 50  $\mu$  in diameter in 3% potassium chloride solution. The cell was placed in the optical path of the Unitron model U-11 microscope and the trajectories of the particles upon application of the field were traced on the viewing screen. The results of these observations are redrawn in Fig. 3. The calculated lines of flow (Fig. 2) are in agreement with the observed lines of flow.

The chief advantages of this design are the production of large values of  $grad(H^2)$ , and the calculation rather than the measurement of the gradient.

This design is particularly applicable for the measurement of the magnetic susceptibility of individual particles by taking measurements along the axis of  $H_0$ . Only a small error would arise if the particle under consideration was not exactly on this axis. However, as  $grad(H^2)$  varies considerably with r, the force on the particle must be obtained by integration of the gradient along the flow line which the particle followed.

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## Amplitude Modulation Method for Producing Sidebands in NMR Experiments\*

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The use of amplitude modulation for the generation of sidebands offers certain advantages in NMR experiments, particularly when relatively large modulation frequencies (say above 1 kc) are required. For the Varian 40- and 60-Mc high resolution spectrometers, minor circuit modifications are described by means of which usable sideband signals are produced for modulation frequencies up to 70 kc, with amplitude reasonably constant from 200 cps to 40 kc.

TWO of the methods now commonly in use for the generation of sidebands in nuclear magnetic resonance (NMR) experiments, namely field modulation and frequency modulation of the rf generator, share a certain disadvantage: The peak deviation  $\Delta\omega$  or  $\Delta H$ , as the case may be, must be increased in proportion to the modulation frequency  $\omega_m$  if the sideband amplitude is to be kept constant. The amplitude of the first pair of sidebands is proportional to the Bessel function  $J_1(x)$  where x is  $\Delta\omega/\omega_m$  for the case of frequency modulation, or  $\gamma\Delta H/\omega_m$  for the case of field modulation.

As a result, the field modulation method fails when it becomes impractical to achieve sufficiently large field deviations to produce adequate sideband amplitude. This occurs at about 1000 cps with the Varian V4310C and V4311 spectrometers. On the other hand, with frequency modulation, serious errors may result from shifting of the carrier frequency due to the use of the large peak deviations needed at high modulation frequencies. For example, with

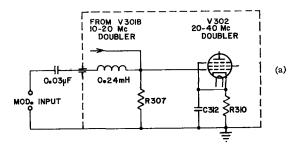
the Varian V4311, we have observed shifts of nearly 100 cps in the apparent position of a fluorine resonance as the amplitude of the modulation voltage was changed, and similar shifts were measured with a counter. Under these conditions, large errors may result when frequency separations are measured by the sideband method.

Comparable errors can arise even with systems designed to produce amplitude modulation, if the modulation is introduced at the oscillator stage. With the V4310C, for example, appreciable shifts in carrier frequency can result from the use of high modulation amplitudes.

To overcome these difficulties, we have employed pure amplitude modulation of the rf generator for the measurement of splittings over a very wide range of modulation frequencies, with excellent results. The circuit changes made in the rf generators of the V4310C and V4311 are shown in Fig. 1. The modulation is introduced at the grid of a multiplier stage, at a point well isolated from the oscillator.

In the case of the V4310C, no alterations are required in the receiver circuits. Sideband signals are detected by

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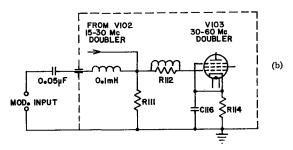


Fig. 1. Portions of rf generator circuits showing modifications for producing amplitude modulation in Varian NMR spectrometers (a) model V4310C, 40 Mc and (b) model V4311, 56.4 and 60 Mc. In both (a) and (b), the added components consist of a 2300-pF feedthrough capacitor in addition to the components whose values are shown.

virtue of the fact that the leakage necessarily also acquires modulation sidebands, so that a dc output is produced by signals at a sideband frequency.

While the V4311 can also be used without further change, the sideband amplitude varies markedly with frequency as shown by the dashed line of Fig. 2. The drop-off with decreasing frequency was corrected by making several changes to improve the operation of the phase-sensitive detector in this application: (1) For proper detection of sideband signals, the reference voltage must bear modula-

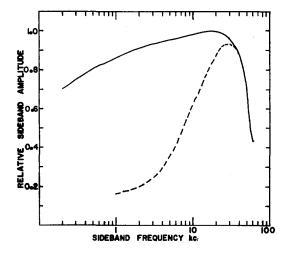


Fig. 2. Dependence of sideband amplitude on modulation frequency for the V4311. The results obtained before and after modifying the detector circuit are given by the dotted and solid lines, respectively.

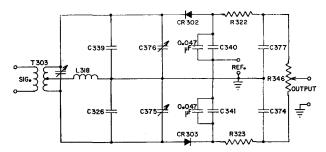


Fig. 3. Phase-sensitive detector circuit of the V4311, showing changes made to improve sideband response.

tion. Since the AVC system of the reference voltage amplifier suppresses modulation below about 200 cps, a 0.1-µF capacitor was added in parallel with C217 to lower this cutoff to about 20 cps. (2) Ringing in the reference voltage output circuit at high modulation levels was suppressed by placing a 3900- $\Omega$  composition resistor in parallel with the reference output jack (J103). (3) For efficient functioning of the detector, the diode load capacitors must hold their charge over a full period of the modulation cycle. The 0.047-µF capacitors shown in Fig. 3 were added for this purpose. With capacitors of this size, the response time is still determined primarily by the filter circuit (not shown) following the detector.

With these changes, the variation in sideband amplitude is greatly reduced, as shown by the solid line of Fig. 2. From about 200 cps to over 40 kc, the sideband amplitude changes so little that it is not necessary to readjust the modulation voltage when changing the frequency.1

As expected for pure amplitude modulation, no change due to modulation is detectable in the frequency as measured by a counter except when the modulation is so deep that cycles are missed by the counter. Frequency splittings in NMR spectra have been measured by this method with excellent reproducibility. Consistent results are obtained even with a very large degree of modulation, since the carrier frequency remains strictly constant.

Amplitude modulation should also work well in spin decoupling experiments following schemes otherwise similar to those of Itoh and Sato<sup>2</sup> and Kaiser<sup>3</sup> in which the transmitter voltage is modulated, the carrier serving to produce spin decoupling while a sideband is used for observation of resonances. The advantage as compared with frequency modulation would be the greatly reduced dependence of sideband amplitude on modulation frequency.

<sup>&</sup>lt;sup>1</sup> Upper and lower sideband signals have somewhat different amplitudes and show, in some cases, a dispersion-mode component when the reference phase is adjusted to give a pure absorption-mode line for the carrier signal. These effects are thought to result from a difference in the frequency response for the signal and reference channels and might be eliminated by realignment of the i.f. circuits. They have caused no difficulty in practice.

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