

A simple microwave kit to convert an X-band ESR spectrometer for K-band operation

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Abstract. A kit for the conversion of an X-band ESR spectrometer for K-band operation is described and its performance has been tested with *p*-benzosemiquinone, vanadyl acetyl acetonate and 2, 5-ditertiary butyl semiquinone. The easy conversion of the X-band spectrometer to K-band operation enables us to distinguish between magnetic field dependent and field independent parameters. A varactor harmonic generator is used as a frequency doubler driving the fundamental power from the existing X-band source to give a power output at K-band without having to modify the electronic circuit; the modification of the microwave circuitry is proposed.

Keywords. ESR; K-band instrumentation; X-band spectrometer; microwave circuitry; varactor harmonic generator.

1. Introduction

It is advantageous to perform ESR studies at two different frequencies especially when one wishes to distinguish between magnetic field-dependent and field-independent spectral parameters. At high frequencies it is easier to distinguish between spectra of two species with different *g*-values. However, having two ESR spectrometers operating at two different frequencies is an expensive proposition and herein we report successful attempt to develop a kit for converting X-band ESR spectrometer to K-band operation by the use of varactor harmonic generators. The designed conversion kit enables one to operate the spectrometers at double the frequency without any change in the conventional electronics and the microwave sources. One has to ensure that the magnet and its power supply are capable of operation at twice the magnetic fields employed for X-band studies.

At the outset the design and the performance of the varactor harmonic generators used in our studies is discussed. The conversion kits developed for a 100 kHz field modulated X-band ESR spectrometer and for an X-band superheterodyne ESR spectrometer are described. The performance of these two spectrometers at K-band are also presented.

2. Design of varactor harmonic generator

The Sylvania diode type 5246 B was utilized as the varactor harmonic generator. Two types of mounts, the cross wave guide and the tapered transition mount were designed following the procedures described by Trivedi and Kodali (1971) and Mahapatra and

Pillai (1970). The final design adopted by us for both these types are given in figures 1 and 2 (Bhagat 1976). The results of the performance tests on the two types are given in figures 3 and 4. The maximum percentage conversion of 10% achieved in both types is much lower than the manufacturer's specification of 20% for the diode. We attribute this shortfall due to poor machining of the parts with the dimensions not being within allowed tolerances. When the work was started, the Sylvania diodes

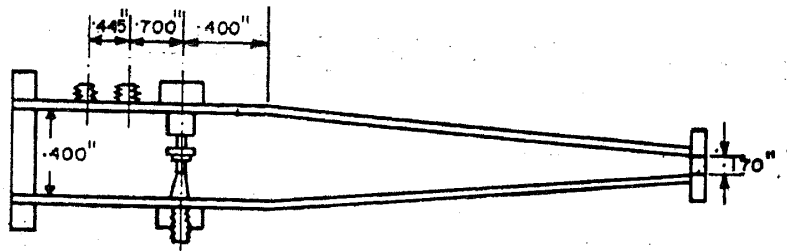


Figure 1. Cross section of tapered transition type of varactor mount.

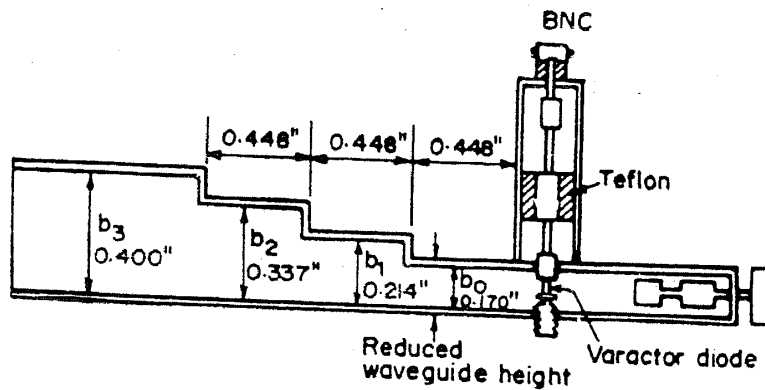


Figure 2. Cross section of homogeneous quarter wave transformer filter cum varactor diode mount.

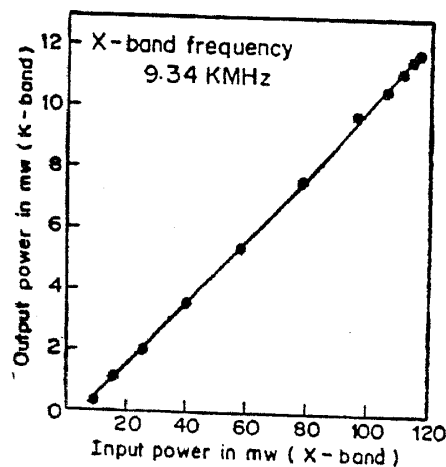


Figure 3(a). Plot of output power vs input power for taper transition design of the varactor mount.

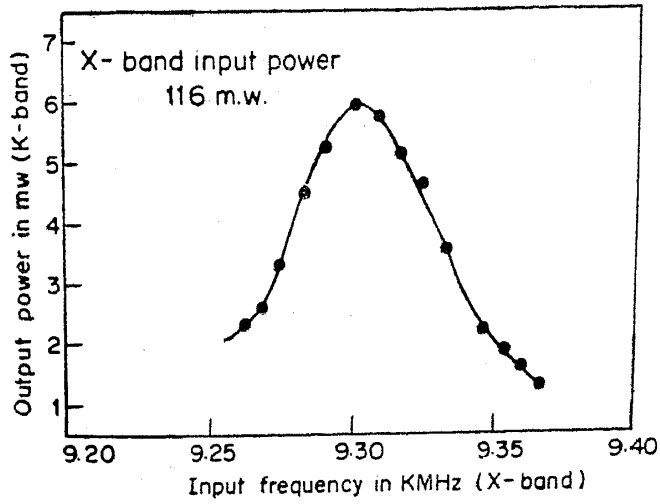


Figure 3(b). Plot of output power vs input frequency for taper transition design of the varactor diode mount

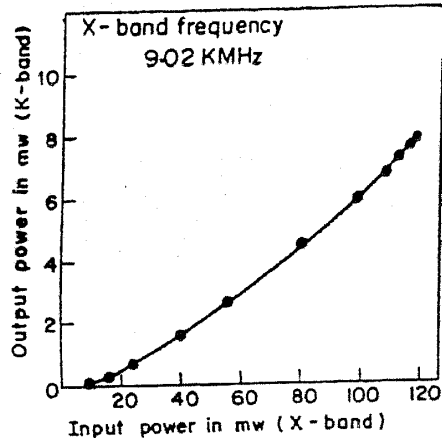


Figure 4(a). Plot of output power vs input power for cross guide design of the varactor diode mount.

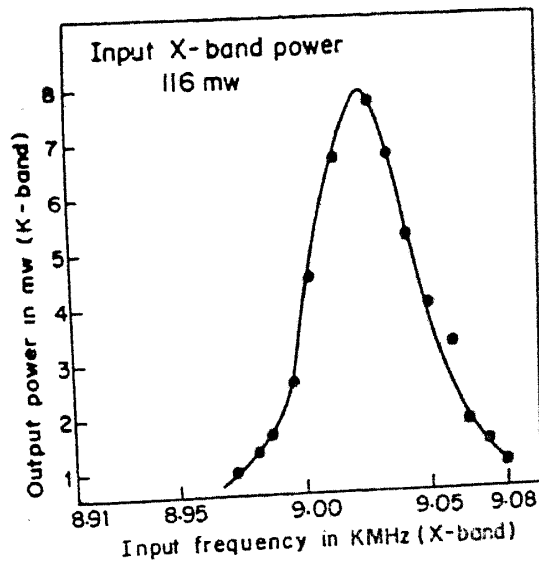


Figure 4(b). Plot of output power vs input frequency for cross guide design of the varactor mount.

with an efficiency of 20% were the best available; however at present step recovery diodes with 80 to 90% efficiency for doubler operation are available and we should expect reports of better performances than what we have been able to achieve. However, it is pertinent to point out that this work was undertaken only to prove the feasibility of this approach and demonstrate the ease with which it could be carried out.

3. Conversion of X-band spectrometers to K-band

3.1. Sample cavity

The heart of the spectrometer is the resonant cavity where the paramagnetic sample is placed and we shall discuss the design and fabrication of the K-band reflection cavity. We employed a TE_{102} mode cavity which was made from a rectangular K-band wave guide ($0.42'' \times 0.70''$) by adjusting its length to be resonant at 18.6 kMHz. The cavity was closed at one end with a flat section and coupled at the other end to the spectrometer through a specially designed iris similar to the irises used in Varian ESR spectrometer, the coupling being adjusted with the help of a teflon stub carrying a gold plated brass piece at its end.

One of the difficult procedures when 100 kHz field modulation is used is the design of the inside walls of the cavity that have a thickness larger than the skin depth at microwave frequencies but smaller than the skin depth at 100 kHz. This difficulty is usually overcome by making cutouts in the broad faces of the rectangular cavity and closing them with ceramic side plates which are gold plated to the appropriate thickness. We found that the copper clad printed circuit boards are excellent substitutes for the gold plated ceramic side walls by carrying out tests on the X-band sample cavity of our 100 kHz field modulated ESR spectrometer. The Q 's obtained were reasonable and the amplitude of field modulation at 100 kHz at the sample was altered only slightly. We therefore adopted a similar procedure for our K-band rectangular cavity by providing cutouts on the broad sides and closing them with highly polished section made from copper clad printed circuit boards. The Helmholtz coils for modulation are placed on the insulated side of the boards. Such cavities were found adequate for normal use but for obtaining high Q 's and therefore greater sensitivity cavities were milled from brass pieces maintaining high precision in the dimensions.

3.2. Conversion of 100 kHz field modulated ESR spectrometer

The conversion kit consisted of the harmonic generator, K-band microwave components obtained from manufacturers and the sample cavity. In figure 5, which shows the block diagram of the converted spectrometer, the components inside the box with dotted outlines form the K-band conversion kit. The X-band slide screw tuner serves a useful purpose for matching the source to the varactor harmonic generator. The output of the doubler is fed into the K-band sample cavity which has provisions for 100 kHz field modulation. The reflected power from the cavity is detected by the K-band detector placed in the third arm of the circulator. A part of the detector output is used for the AFC locking of the X-band klystron while the

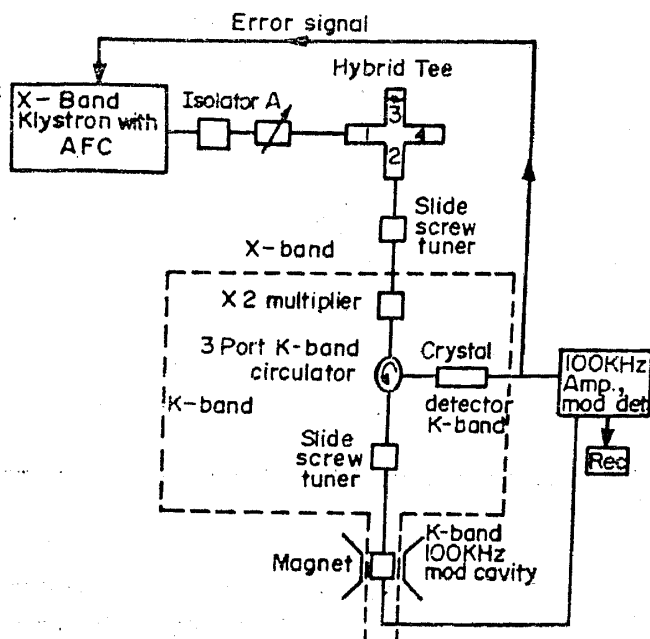


Figure 5. 100 kHz modulation K-band spectrometer.

other is used for detecting the signal by the 100 kHz lock-in-amplifier (Varian type V-4560) which again is an integral part of the 100 kHz field modulated X-band ESR spectrometer. When X-band power fed into the harmonic generator was 116 mw, the K-band power output of the harmonic generator was 11.8 mw.

3.3. Performance tests of the 100 kHz field modulated K-band spectrometer

We have tested the performance of the converted spectrometer in the following manner: (a) By checking the sensitivity with a standard solution sample we found it poorer than is expected for a K-band spectrometer taking into account various considerations like the enhanced sensitivity due to the frequency dependence of sensitivity, lower filling factor for a solution sample, smaller sample, lower power, etc.

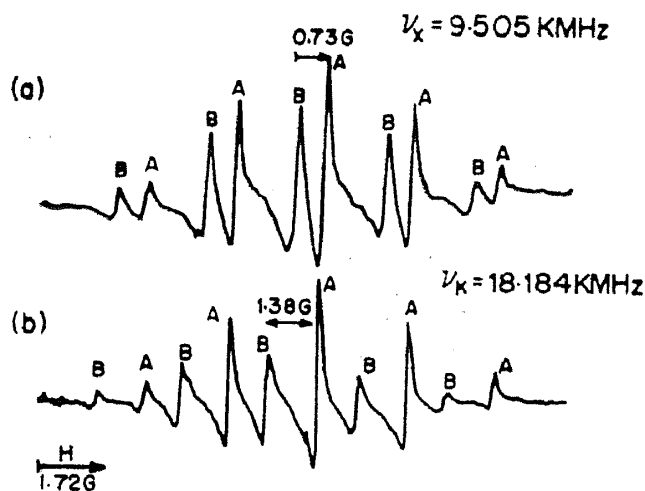


Figure 6. ESR spectrum of PBSQ in (A) ethanol & (B) DMSO.
 (a) X-band;
 (b) K-band.

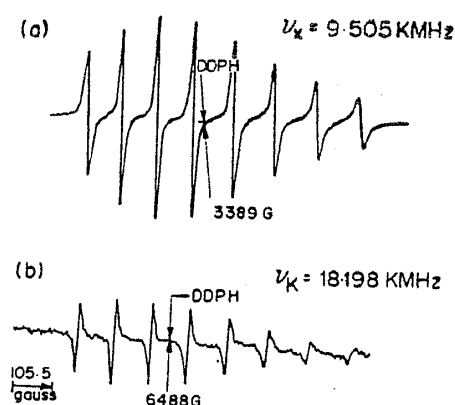


Figure 7. ESR spectrum of vanadyl acetylacetonate in chloroform at (a) X-band and (b) K-band at 295° K.

The main reason for the poor sensitivity is believed to be the poor Q of our sample cavity (around 1500) which we are attempting to improve by better design and machining. (b) We have recorded the ESR spectra of a solution having two different species with different g -values. Figure 6 is the ESR spectra of parabenzosemiquinone ions (PBSQ) in ethanol and dimethylsulphoxide (DMSO) at K-band and X-band frequencies. The spectra were taken with the two different solutions placed simultaneously in concentric sample tubes in the cavity. The semiquinone has different spectra with different g -values in the different solvents. The separation between the central hyperfine lines of PBSQ in ethanol and DMSO is 0.70 ± 0.02 Gauss at X-band. The separation should be proportional to the frequency and the calculated separation at K-band is 1.40 ± 0.04 gauss at K-band, while we measured 1.38 ± 0.04 Gauss. (c) The second order correction to the hyperfine separation should be less as we operate at higher frequencies. This is illustrated in figure 7 wherein we compare the ESR spectra at X-band and K-band of vanadyl acetyl acetonate in chloroform. The magnetic field scale is the same for both the spectra and the second order effect is clearly more pronounced at X-band. Another interesting observations can be made on the lines of minimum line width. It is the third and fourth line from the low field side that are narrower than observed in the X-band case, and at K-band, the second and third are the narrower ones.

3.4. Conversion of the superheterodyne spectrometer

The description of our superheterodyne ESR spectrometer operating at X-band is found in literature (Lingam *et al* 1972). It consists of two klystrons operating with a frequency difference of 60 MHz to provide the IF of 60 MHz. If we wish not to change the conventional electronic set up including the IF amplifiers, the two klystrons have to be operated 30 MHz apart so that on doubling both the signal and local oscillator frequencies to K-band, an IF of 60 MHz is obtained. It is obvious that in our set up we need two microwave doublers. Figure 8 is the block-diagram of the converted set up. Here the K-band microwave conversion kit is found to the right of the two dotted lines and the conventional electronic part remains totally unchanged. The same cavity that was designed for the 100 kHz field modulated spectrometer was utilized for our preliminary tests; however no 100 kHz field modu-

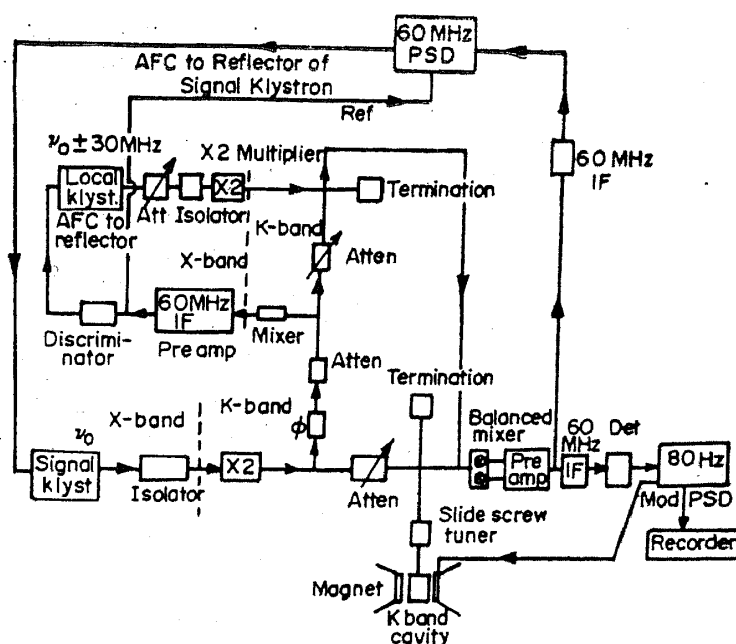


Figure 8. K-band superheterodyne spectrometer.

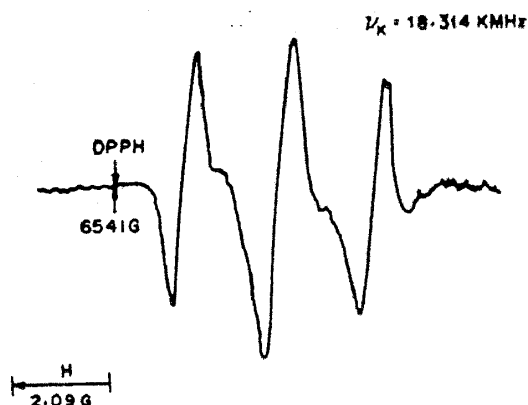


Figure 9. ESR spectrum of 2, 5-di-tertiary butyl semiquinone in ethanol using superheterodyne K-band spectrometer.

lation is employed. Instead an 80 Hz field modulation was provided with the help of Helmholtz coils mounted on the pole faces of the magnet.

The X-band power in the signal arm was about 150 mw and the K-band power available after the doubler was 15.1 mw. The local oscillator arm had an X-band power of 50 mw and K-band power was 4 mw. Even under these low power observations the stability and the sensitivity of the entire system including the frequency stability of the klystrons was found to be very good. The performance was tested with standard samples, and Figure 9 is the ESR spectrum of a free radical solution (about 10^{-3} M) commonly employed in our laboratory for performance tests.

4. Conclusion

This report has demonstrated the relative ease with which operational frequencies of ESR spectrometer could be changed with a very small investment. For low power

operations this is ideally suitable; however, with step recovery diodes capable of 80-90% conversions as reported in recent literature the available microwave power at higher frequencies generated by the multiplier technique may be quite high. It may even be possible to treble the frequencies and operate near 30 GHz with an available power of about 10-20 mw utilizing these techniques.

The availability of such a kit to enable observation of ESR spectra at two different frequencies, simplicity and economy of operation, should be of great boon to solid state studies where very often it is necessary to identify spectral lines as belonging to one centre or different centres for e.g. in radiation damage studies. Such distinctions are usually made from the expected difference in the behaviour of lines of two species when temperature or microwave power is changed. Similarly in chemical reactions the ease of availability of spectra at two different frequencies would enhance the utility of ESR spectral studies.

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