A Microwave Fourier Transform Spectrometer in the Frequency Range of 2 to 4 GHz and its Performance

C. Gerke and H. Dreizler

Abteilung Chemische Physik im Institut für Physikalische Chemie der Universität Kiel,
Bundesrepublik Deutschland

Z. Naturforsch. **47a**, 1058–1062 (1992); received July 29, 1992

We present a microwave Fourier transform spectrometer with a coaxial cell in the range from 2 to 4 GHz. The performance is demonstrated by some examples showing the sensitivity and resolution.

Introduction

In the recent years we published different set-ups for microwave Fourier transform (MWFT) spectrometers in the region from 4 to 40 GHz [1–9] using standard waveguides as sample cells. We proved that MWFT spectroscopy [10] is a powerful tool to investigate rotational spectra as the sensitivity, resolution, and accuracy of the spectrometers are higher than that of Stark spectroscopy [11]. We investigated many molecules. Most of the results were published in Zeitschrift für Naturforschung and Journal of Molecular Spectroscopy from 1979 on.

As the hyperfine structure (hfs) produced by nuclear quadrupole coupling in rotational spectra can be investigated with the most precise results in transitions with low angular momentum quantum numbers J, we decided to extend the range of our spectrometers to frequencies lower than 4 GHz. Especially heavy molecules have low J transitions in that region.

It would have been possible to extend the waveguide technique, but three waveguides R 14 (1.14–1.73 GHz with cross section 16.5 × 8.2 cm), R 32 (2.69–3.95 GHz with 7.2 × 3.4 cm), and R 22 (1.72–2.61 GHz with 10.9 × 5.4 cm) should be used to work in the fundamental mode H_{10} only, which proved necessary in former work. Only with bridge type spectrometers [12] it was possible to use oversized waveguides.

The large cross section of the waveguides (R 22, 59 cm^2) would have asked for travelling wave tube amplifiers (TWTA) with an output of the order of 500 W to produce a power density of approximately 10 W/cm^2. Besides the high costs it is presently not possible to find PIN switches and other parts tolerating this high power.

So we decided to use a coaxial sample cell and coaxial MW parts in our new set-up.

Experimental set-up

In Fig. 1 we present the experimental set-up for the frequency region from 2 to 4 GHz. We intend to follow the same lines for the adjacent lower frequency region.

As the frequency is rather low, we use a synthesizer 1 as a master source, which is by 10 MHz referenced to the normal frequency and time station DCF 77 Mainflingen by the receiver 53 and regulated quartz oscillator 54. All other frequencies of the spectrometer are also referenced to 54. The output frequency of 1 is doubled, 2, band limited, 3, amplified, 4, and divided, 5, to provide the local oscillator frequency v, and after a frequency shift by a single sideband modulator 8 driven by 160 MHz also the polarizing frequency $v + 160$ MHz or $v - 60$ MHz. This method follows a similar set-up [8]. The polarizing frequency is, by the PIN switch 7, only present when it is necessary to produce the polarizing MW pulses. This reduces interferences. The isolator 6 reduces reflections of 7, especially when it is closed.

The conversion loss of 8 and the insertion loss of the following components 13 to 20 are compensated, and the necessary power for driving the TWTA 21 is provided by the amplifier 12 and adjusted by the attenuator 11. Although the single sideband modulator 8 suppresses the carrier and unwanted sidebands by 15 dB it is useful to insert a digitally driven YIG bandpass filter 13 to avoid polarization with unwanted

---

Reprint requests to Prof. Dr. Helmut Dreizler, Institut für Physikalische Chemie der Christian-Albrechts-Universität, Olshausenstr. 40–60, W-2300 Kiel, F.R.G. Fax 04 31/8 80-14 16.

0932-0784 / 92 / 1000-1058 $ 01.30/0. – Please order a reprint rather than making your own copy.

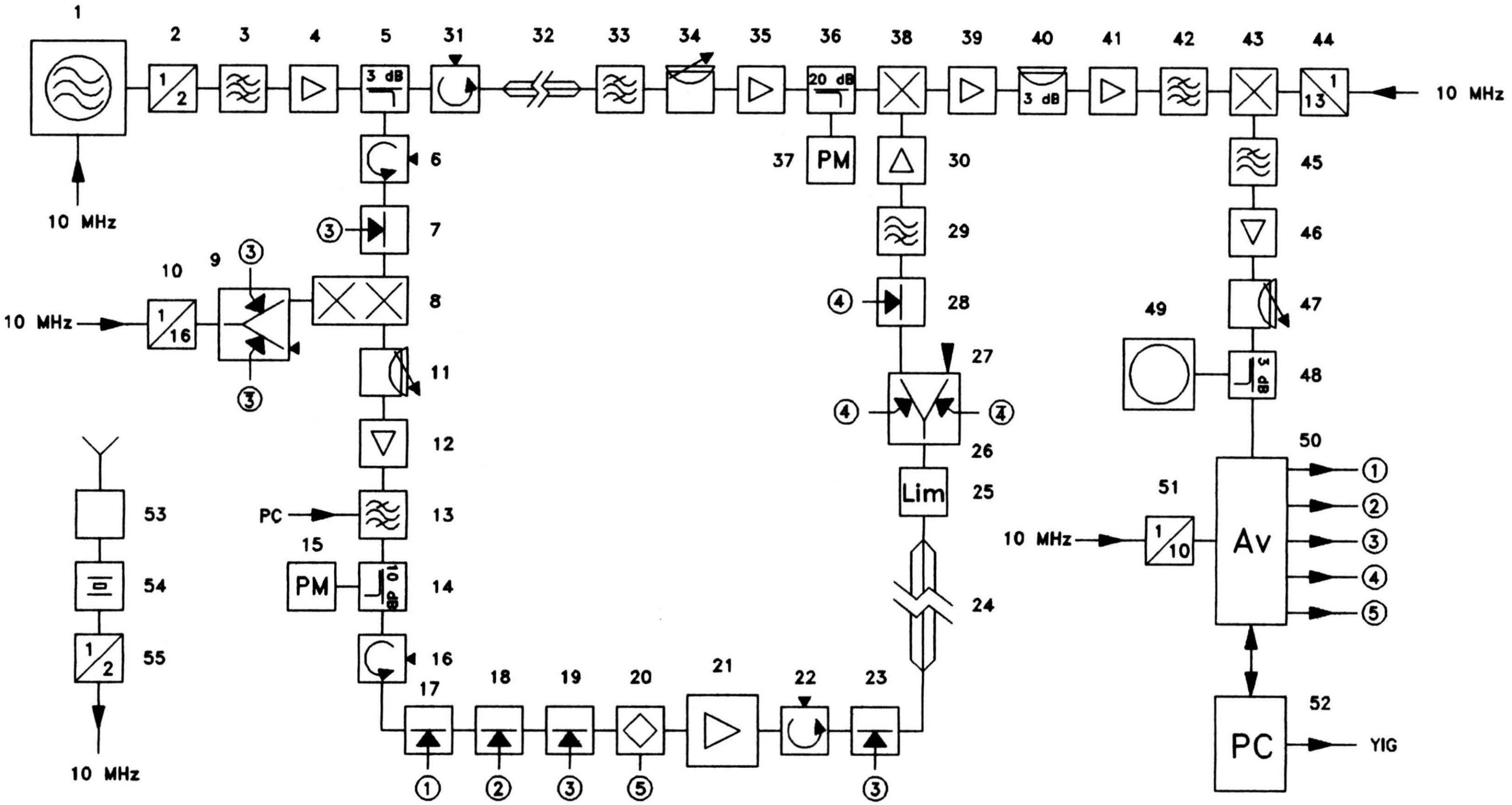

Fig. 1. Experimental set-up of a microwave Fourier transform spectrometer in the range from 2 to 4 GHz using coaxial cells.

1 synthesizer, Rohde & Schwarz SMPD 5 kHz–2720 MHz
2 frequency doubler, Avantek DRX 2075 M
3 bandpass filter, FSY Microwave WM 1950-4100-7-9 SS
4 amplifier, Trontech W40GA-2
5 power divider, Mini Circuits (MC) ZAPD-4
6 coax. isolator, Ferrite I8738
7 PIN switch, Hewlett-Packard (HP) 33144A with driver HP 33190B
8 single sideband mixer, RHG IRDS 1-4/160
9 SPDT PIN switch with TTL-driver, MC ZYSWA-2-50DR (optional)
10 frequency multiplier 10 to 160 MHz
11 variable attenuator, Alan 50CA4-20 PM
12 amplifier, Trontech P42GA
13 YIG filter with digital driver, Watkins Johnson 5272-004DA
14 directional coupler, MAC C 3204-10, 10 dB
15 power meter with sensor, HP 432A and 8478B
16 coax. isolator, T-Hi-Tech H20S40A2
17–19 see 7
20 double balanced mixer with TTL-driver, RHG DMK2-18
21 travelling wave tube amplifier, Hughes 8020H
22 see 16
23 see 7
24 coaxial sample cell, see text
25 limiter, HP 11867A
26 see 9
27 termination, Weinschel F1419
28 see 7
29 highpass filter, FXR HD-20N 2000 Mc
30 see 4
31 see 6
32 N-cabel, Amphenol RG-214
33 bandpass filter, Reactel 6B2-3000-2000 S22
34 see 11
35 see 4
36 directional coupler, MAC C 3204-20, 20 dB
37 see 15
38 mixer, MC ZEM-4300
39 amplifier, RHG ICFH 160 LN
40 attenuator 3 dB
41 see 39
42 bandpass filter, K & L 2B120-160/10-B/BP
43 mixer, MC ZAD1
44 frequency multiplier 10 to 130 MHz
45 lowpass filter, K & L 5L340-35-B/B
46 amplifier, Avantek GPD 461, 462, 464
47 variable attenuator, HP 355C
48 power divider, MC ZSC-2-1
49 oscilloscope
50 signal averager and control unit, [13]
51 frequency multiplier 10 to 100 MHz
52 personal computer
53 receiver, Rohde & Schwarz XKE2
54 regulated quartz oscillator, Rohde & Schwarz XSD2
55 frequency doubler

frequencies. With the power meter 15 and 14 the driving power of the TWTA 21 is monitored. With the PIN switches 17, 18, and 19, the polarizing pulses are produced according to the time diagram of Fig. 2 of [4]. The isolator 16 reduces reflections. The biphase modulator 20 provides the 0°/180° phase shift for the phase alternating pulse sequence (PAPS) [10], which has been reduced to a single 0°/180° phase alternation. The TWTA 21 amplifies the MW pulses.

It provides cw power up to 20 Watt. It is necessary to shield the instrument by an isolator 22 against high power reflections. The PIN switch 23 reduces the influence of the TWTA noise during emission of the transient signal which has been induced by the amplified MW pulses in the gas sample contained in a coaxial cell 24. Two such cells, *a* and *b*, are in use. They are used in the TEM mode. The cut off frequency of the next higher mode TE_{11} (H_{11}) is above 4 GHz.

a) A straight home made coaxial 50 Ω guide with 30 mm inner diameter and 13 mm outer diameter of the inner conductor, cross section 5.7 cm^2, length 10 m, volume 5.7 l. The ends of this line have been tapered to fit to N connectors, which provide also the vacuum seal.

b) A commercial flexible 50 Ω coaxial guide type HF 1 1/8″ Cu2Y made by Kabelmetall, Hannover, of 20 m length. The outer conductor is made of a corrugated copper tube with a mean inner diameter of 28 mm, whereas the inner conductor is a copper tube with an outer diameter of 12 mm. The volume is approximately 11 l. The ends of this guide are equipped with commercial N connectors modified for better vacuum pumping. The guide is rolled up in a circle of approximately 2 m diameter and can be cooled to − 50 °C by nitrogen evaporating from liquid nitrogen.

The arrangement of the cell 24, the limiter 25 and SPDT PIN switch 26 with a termination 27 is a result of a long trial and error procedure. First we enclosed the cell 24 in analogy to our waveguide set-ups with coaxial isolators. We observed variing transient signals of approximately 5 μs decay time not originating from the molecular ensemble. We give an example in Fig. 2. We traced it finally back to the circulators.

With the present arrangement the polarizing pulse is absorbed sufficiently in the termination 27, which is connected in the period of polarization to the cell 24 by the SPDT PIN switch 26. In the period of transient emission the molecular signal is guided via the PIN switches 26 and 28, the high passfilter 29 and the low noise amplifier 30 to the mixer 38. The PIN switch 28 is an additional protection of the detection system in the period of polarization. The mixer 38 is driven with the doubled frequency of 1, band filtered, 33, and adjusted in power by the attenuator 34, the amplifier 35 and the power meter 37.

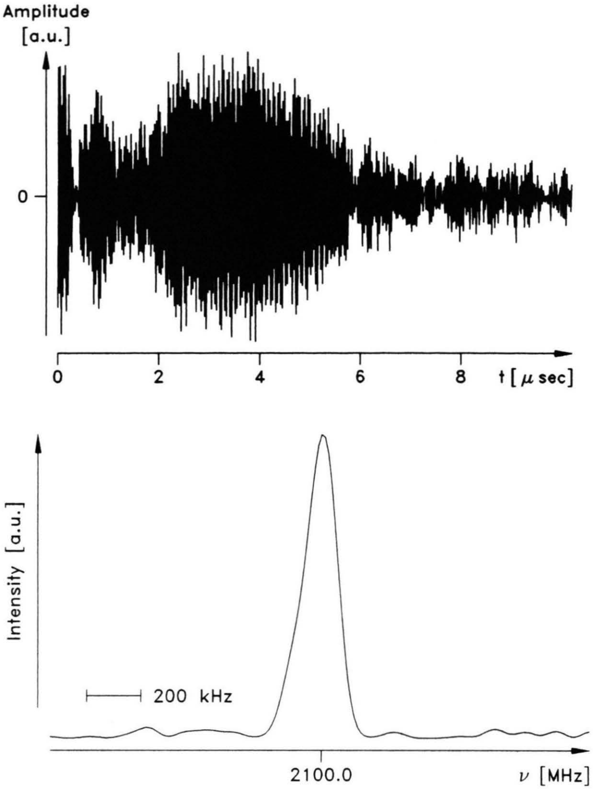

Fig. 2. Transient "decay" and a 2 MHz section out off its Fourier transform produced by storage of MW energy in circulators at the ends of the cell 24. They have been replaced by the set-up given in Figure 1.

The intermediate frequency branch starting with amplifier 39 is analogous to our other set-ups.

To reduce further coherent perturbations we switch off by the optional SPDT PIN switch 9 the modulating 160 MHz.

Experimental tests

We tested the spectrometer for sensitivity and resolution. In Fig. 3 we give a recording of the rotational line $J, K_-, K_+ = 24, 4, 20-23, 5, 19$ of sulphur dioxide, $^{32}S^{16}O^{18}O$, in natural abundance with an absorption coefficient α of approximately $3 \cdot 10^{-10}\ cm^{-1}$.

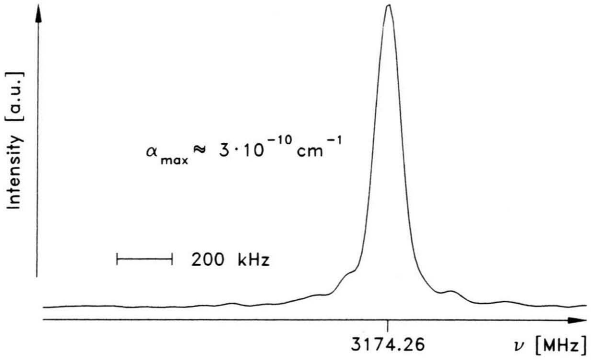

Fig. 3. A 2 MHz section out of a 50 MHz recording of sulphur dioxide, $^{32}S^{16}O^{18}O$, in natural abundance showing the $J, K_-, K_+ = 24, 4, 20{-}23, 5, 19$ transition with an absorption coefficient α of $3 \cdot 10^{-10}$ cm^{-1}. 10 m cell, sample interval 10 ns, 1024 data points, $9.8 \cdot 10^6$ experiment cycles, polarizing frequency 3174 MHz, 3072 zeros supplemented before Fourier transformation, measuring time 155 s, temperature 25 °C, pressure 3.6 mTorr (0.5 Pa).

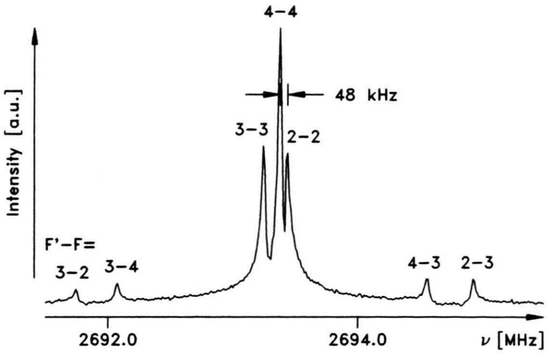

Fig. 5. A 4 MHz section out of a 50 MHz recording of cyanic acid, HCN, showing the $J = 3$ direct l-type transition. The F quantum numbers of the components are given. 10 m cell, sample interval 10 ns, 4056 data points, $9.8 \cdot 10^6$ experiment cycles, polarizing frequency 2694 MHz, no zeros supplemented before Fourier transformation, measuring time 155 s, temperature 25 °C, pressure 0.4 mTorr (0.05 Pa).

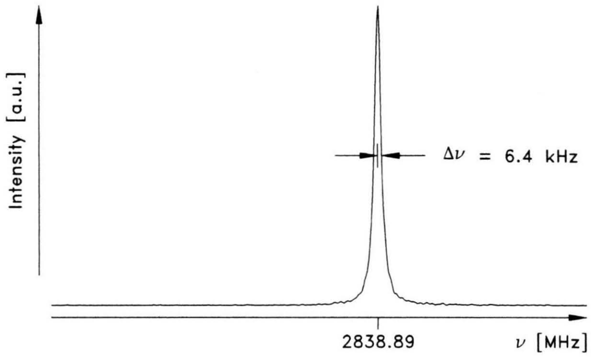

Fig. 4. A 1 MHz section out of a 10 MHz recording of sulphur dioxide, SO_2, showing the $J, K_-, K_+ = 27, 2, 26{-}26, 3, 23$ transition. Half width at half height = 6.4 kHz. 10 m cell, sample intervall 50 ns, 2048 data points, $9.8 \cdot 10^6$ experiment cycles, polarizing frequency 2838 MHz, 2048 zeros supplemented before Fourier transformation, measuring time 155 s, temperature 25 °C, pressure approx. 0.1 mTorr (0.013 Pa).

The measuring time of the $9.8 \cdot 10^6$ experiment cycles was 155 s. This shows that the sensitivity is comparable to the waveguide MWFT spectrometers in the other frequency regions.

In Fig. 4 a 1 MHz section of a 10 MHz recording of the transition $J, K_-, K_+ = 27, 2, 26{-}26, 3, 23$ of sulphur dioxide, SO_2, is presented.

The pressure was lowered to approximately 0.1 mTorr. The half width at half height is $\Delta v = 6.4$ kHz. The calculated Doppler width [14] is $\Delta v_{\text{Doppler}} = 4.3$ kHz. So we are near the Doppler width limit. The influence of wall collisions is difficult to estimate for this geometry. If one approximates the cross section of the circular ring cross section of the cell by a rectangle of 0.85×5.34 cm^2 one gets $\Delta v_{\text{wall}} = 3.8$ kHz [15].

The Fig. 5 shows the direct l-type transition $J = 3$ of cyanic acid, HCN, where we are interested in details of the hyperfine splitting [16, 17]. All components can be seen. These recordings promise that the spectrometer will be a useful instrument for problems of rotational spectroscopy.

The given recordings were made with the 5.7 l cell. The measurements with the cell of 11 l volume yielded about the same results as far as sensitivity and resolution are concerned, although the cell volume is by a factor two larger. The reasons for this result are not obvious. An important point may be, that the delay time between the end of the MW pulse and the start of the measurement has to be increased for the longer cell, as the transit time for the MW pulse is longer.

We thank the members of our group for help and discussion, especially the mechanic workshop of our institute for the craftmanship necessary to set up the spectrometer.

The funds were provided by the Deutsche Forschungsgemeinschaft, the Fonds der Chemie and the Land Schleswig-Holstein.

[1] G. Bestmann, H. Dreizler, H. Mäder, and U. Andresen, Z. Naturforsch. **35a,** 392 (1980).
[2] G. Bestmann and H. Dreizler, Z. Naturforsch. **37a,** 58 (1982).
[3] G. Bestmann, H. Dreizler, E. Fliege, and W. Stahl, J. Mol. Struct. **97,** 215 (1983).
[4] W. Stahl, G. Bestmann, H. Dreizler, U. Andresen, and R. Schwarz, Rev. Sci. Instrum. **56,** 1759 (1985).
[5] Ch. Keussen, N. Heineking, and H. Dreizler, Z. Naturforsch. **44a,** 215 (1989).
[6] H. Ehrlichmann, J. U. Grabow, H. Dreizler, N. Heineking, R. Schwarz, and U. Andresen, Z. Naturforsch. **44a,** 751 (1989).
[7] Ch. Keussen, R. Schwarz, U. Andresen, and H. Dreizler, Z. Naturforsch. **45a,** 711 (1990).
[8] M. Krüger and H. Dreizler, Z. Naturforsch. **45a,** 724 (1990).
[9] V. Meyer, W. Jäger, R. Schwarz, and H. Dreizler, Z. Naturforsch. **46a,** 445 (1991).
[10] H. Dreizler, Molecular Physics, **59,** 1 (1986).
[11] C. H. Townes and A. L. Schawlow, Microwave Spectroscopy, McGraw-Hill Book Co., New York 1955, Chapter 15.
[12] P. Wolf and H. Mäder, Mol. Phys. **64,** 43 (1988).
[13] U. Andresen and B. Kleibömer, Rev. Sci. Instrum. **59,** 1008 (1988).
[14] W. Gordy and R. L. Cook, Microwave Molecular Spectra, J. Wiley, New York 1984, formula (3.39).
[15] cit. [11], formula (13.85).
[16] E. Fliege, H. Dreizler, A. P. Cox, and S. D. Hubbard, Z. Naturforsch. **39a,** 1104 (1984).
[17] to be published.