Tutorial Microwave Spectroscopy

Practical Course M

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Preliminary remarks

The preparation and execution of this experiment should give the students a basic insight in the main terms and operation of a high resolution and high sensitivity microwave spectrometer.

Basically the experiment contains all main components of a millimeter wave or THz spectrometer which are used in our institute to support interstellar detection of unknown molecular species. These measurements will allow a deep insight into the quantum mechanical description of transient molecules.

The experiment is thematically connected to major projects of the institute and should create interest for a bachelor work or dissertation in one of our work groups.

Some hints for the preparation of the experiment

Bold terms as well as all formulas should be explained respectively deduced in the written report. The items in section “Hints for the written report” should be followed. Supporting documents can be downloaded from the institute web page. It is necessary to read them carefully in order to be prepared for the experiment.

Tutorial

Introduction

A main task of modern astrophysics is the understanding of the formation of stars and planets. Therefore spectrally and spatially high resolved observations of star formation regions with their associated molecule clouds are important. Especially the electromagnetic radiation of molecules in the frequency region from 100 GHz to some 1000 GHz is of interest. From the observed spatial distribution of the gas, the excited states and the shape of the line profiles of different molecular species and transitions, the physical and dynamic state of the matter can be determined and compared with model predictions.

1. Your Preparation @ home

To be well-prepared you should study/explain the following key words:

- Rotation of diatomic molecules (Townes, Kap.1)
- Symmetric-top molecules (Townes, Kap.3)
- Ammonia molecule (Townes, Kap.12)
- Pressure broadening of molecular lines (Townes, Kap.13)
- General terms of microwave spectroscopy
- The principles of a lock-in amplifier (phase sensitive detection/synchronous demodulation/mixer)
- Microwave technology, waveguides, impedance matching (Hachenberg / Vowinkel)
- Frequency multiplication with nonlinear passive elements, e.g. diodes

2. Setup of the Spectrometer

The setup consists of a microwave synthesizer (Hewlett Packard HP8673E) with a frequency coverage of 2-18 GHz. The synthesizer frequency will be frequency doubled with a diode and finally connected to a K band waveguide (18-26 GHz). At the waveguide junction the power can be optimized / matched with a variable backshort.

The output frequency of the microwave synthesizer lies below the cut-off frequency of the waveguide, therefore it cannot propagate. Higher order harmonics, which are produced by the diode as well, are suppressed with a low-pass filter in waveguide technology, so that only the first harmonic wave (frequency x2) remains.

In order to control power matching, a 10 dB directive coupler is used to split part of the microwave signal into a power meter head. The absorption cell (brass waveguide) is vacuum-tightly connected with the other waveguide components by the use of Teflon window foils. At the right vacuum flange of the absorption cell, a small round bottom flask, in which a small quantity of aqueous ammonia solution is filled, is attached to a needle valve.

The vapor pressure of ammonia at room temperature (approx. 500 mbar) is sufficient to achieve sufficient gaseous ammonia over the measuring period. The left vacuum flange is connected with a rotary vacuum pump, a vacuum measuring gauge and an air ventilation valve. A one-way waveguide / isolator follows behind the absorption cell, which suppresses reflections back into the cell and thus prevents standing waves.

The detector diode follows, which can also be impedance matched by adjusting the backshort of the diode mount to the optimum waveguide impedance. The signal of the diode is connected to the input of the lock-in amplifier, whose DC output is connected to a digital voltmeter. The lock-in amplifier receives a phase reference from a sine-wave generator, which is also used for frequency or amplitude modulation of the microwave synthesizer via the modulation input ports.

Via an IEC bus and an IEC/USB adapter, the microwave synthesizer and the digital voltmeter are connected with the measuring PC, to allow for computer control and/or to recording of spectral data.

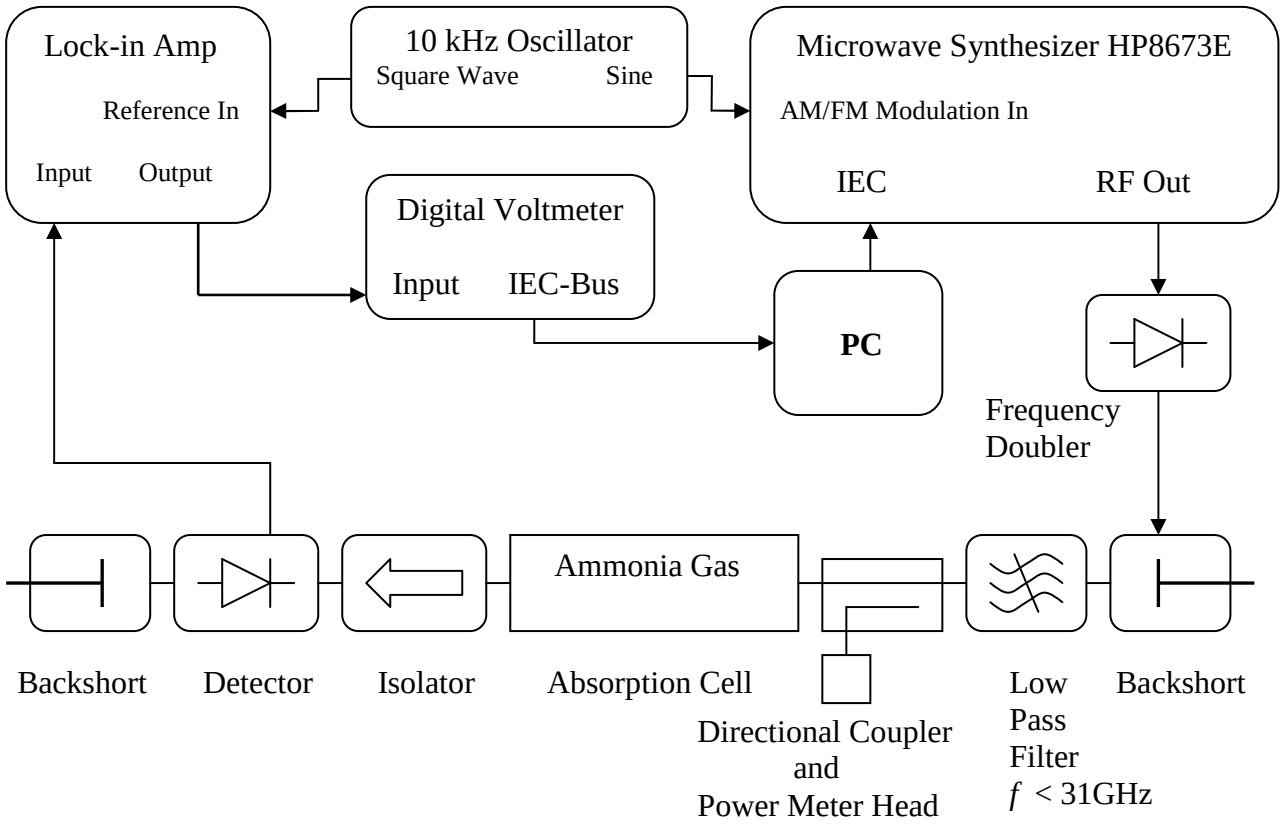

Figure 1: Schematic Diagram of the Spectrometer

3. Execution of the measurements

3.1. Principles of the measurements

The microwave synthesizer is PC controlled and will sweep stepwise over a given frequency range, additionally modulated with a 10 kHz signal. The signal can be used either for amplitude modulation (AM) or for frequency modulation (FM). The detector signal is demodulated synchronously to the modulation frequency reference with a lock-in amplifier, in order to improve the signal-to-noise ratio. The output signal of the lock-in amplifier is digitized with a digital voltmeter (DVM) and finally recorded by the PC. For further details of the measuring procedure please refer to Chap. 7 in the work of Sven Hees.

3.2. Bringing the vacuum components into service

Turn on the pressure gauge (Thermotron) and select system T2.

Move the valves into the following positions

- Outlet valve: **open**; turn lever to the right, inner part will be lifted.
- Needle valve: **closed**; turn right carefully until it stops.
  Caution! *Never apply force! The valve is spring-actuated. Closing further harms the valve!*

Turn on the vacuum pump by pushing the green button for more than 2 seconds. As soon as the pump works continuously, close the outlet valve. The pressure should drop below 10^{-2} mbar within 5 minutes and down to 10^{-3} mbar (green scale) within 30 minutes. Fill ammonia solution into the round bottom flask (fill level of about 3 mm) if not already done. Attach the flask to the metal flange and close the spring.
Now starting from the stop, turn the needle valve counterclockwise until the needle is lifted (resistance). Continue turning carefully and watch the pressure gauge.

The pressure should drop down to about 10^{-3} mbar within the next half hour. During this time setup the electronic measurement equipment.

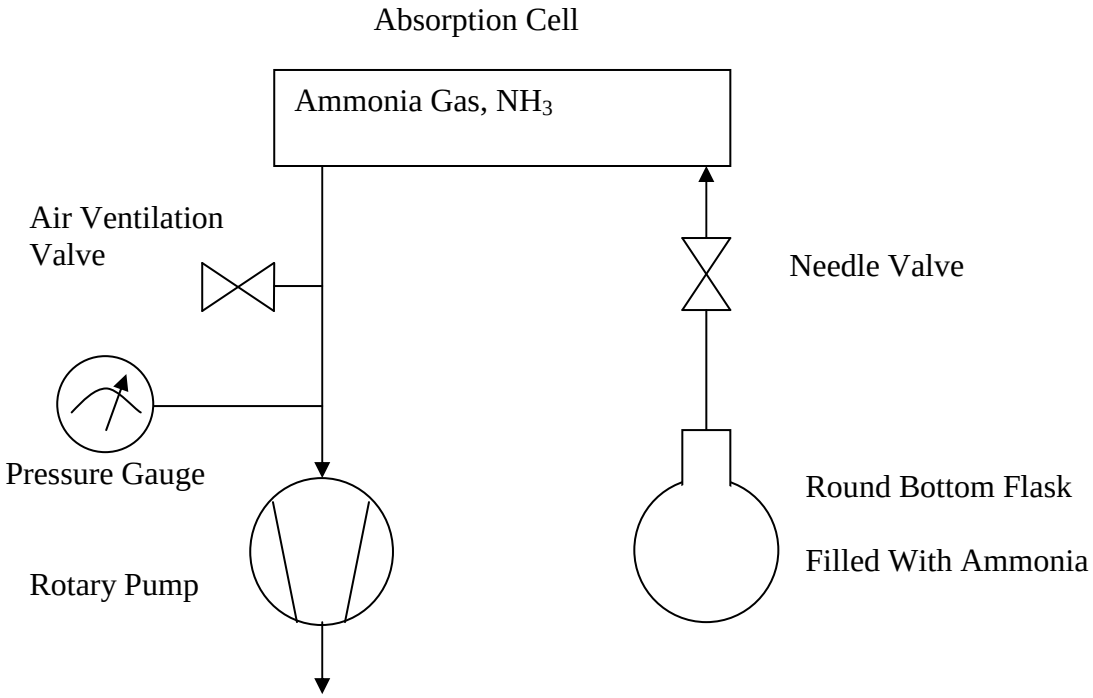

Figure 2: Schematic diagram of the vacuum setup

3.3. Setting up the electronic measurement equipment

Turn on microwave synthesizer, 10 kHz generator, lock-in amplifier and digital voltmeter. In case the microwave synthesizer displays the error message "95" (button "Message" is blinking; when pushing the button "95" is shown on the display), you have to manually switch to bus address 20. Push the buttons 20, STO, and LOCAL subsequently. Otherwise, the synthesizer will block the IEC bus.
Next, tune the synthesizer to 11935.056 MHz (half of the frequency of the NH_3 3,3 transition): Push the frequency button, then type in 11935.056 and press MHz. Turn off AM/FM modulation if turned on by pushing the respective OFF buttons.
Set the level to maximum, i.e. +13 dBm.
If the synthesizer is not accessible, it might be in "remote" operation. In this case, press "LOCAL" to activate front-side usage.

Now tune the impedance of the waveguide.
Switch the power meter to –5 dBm range and zero it with the FINE-ZERO button. Activate the synthesizer output by pushing RF-OUT. You can tune to the maximum power with the backshorts. If this is not possible as you reach the limit stop, turn in reverse direction by $\lambda/2$ past the minimum.

Next, you have to tune the impedance of the detector diode.
The sine wave generator has to be set to amplitude modulation (AM) and tuned to maximum detector signal. Turn the amplitude of the sine-wave generator to zero and connect the sine output with the AM input of the synthesizer.
Tune the lock-in amplifier to 100 mV, 100 ms, phase of about 90°, and F. Switch the meter of the synthesizer to AM and activate AM with the 30% button. Increase the amplitude at the sine-wave generator until 15% of modulation is reached (pointer is upright). Use the back-

short at the detector diode to maximize the output voltage of the lock-in amplifier. Correct the phase simultaneously.

Detection of ammonia 3,3 transition in 2F mode.
Adjust the ammonia pressure, using the needle valve, to about $3 - 5 \times 10^{-3}$ mbar. Deactivate amplitude modulation (AM-OFF button) and switch the output of the sine generator to FM. Tune the lock-in amplifier to a sensitivity of 3 mV and switch to 2F. Activate frequency modulation (FM) with 0.3. Switch the display to FM and vary the amplitude of the sine generator until the pointer is upright, i.e. 1.5 or 0.5, respectively. Next adjust the phase at the lock-in to maximum. Use the PC for a functional test and record the 3,3 ammonia line transition. The backshorts have to be tuned each time another frequency is used.

3.4. Tasks

1. Measure the 3,3 ammonia transition via amplitude and frequency modulation in modes F and 2F. Describe the differences. Which method is preferable and why?
2. Measure 6 different ammonia transitions distributed over the whole K band and determine the constants υ_0, a, and b of the ansatz $\nu = \nu_0 - a(J(J+1) - K^2) + bK^2$.
3. Try to measure a reasonably faint transition. Increase the integration time per frequency and the time constant of the lock-in amplifier. Which line intensity is just detectable?
4. Determine the dependence of line shape and intensity from the amplitude of the frequency modulation (FM) signal in 2F mode. Measure a more intense transition with 4 different modulation settings.
5. Determine the dependence of line shape and intensity from pressure for 3 different pressure values. Notice that, in FM mode, the amplitude of the modulation signal has to be adjusted to the respective line width in order to measure the corresponding intensity correctly.
6. Measure the quadrupole splitting of the 3,3 ammonia transition.
7. Using the ammonia safety data sheet, determine the maximum permissible amount of aqueous ammonia solution in case the bottom flask breaks.

4. Notes on software

The software offers the possibility to scan and record a given frequency range. In this case, the user has to provide the central frequency, the width of the range, the number of sample points, and the integration time per frequency step. The latter value should be selected to be consistent with the time constant of the lock-in amplifier. Otherwise, the measured noise will be too large, or in case of jumps only the rising speed of the lock-in amplifier will be measured. Suggestions for improvements to the software are welcome.

5. Important hints for the written report

- Include the records of your measurements
- Include a schematic diagram of the experimental setup for each task
- Include a complete error calculation
- Discuss the results!
- Include your e-mail addresses

6. Literature

1. Staatsexamensarbeit Sven Hees, S.1–53, S.58f
2. Townes/Schawlow
   Chapter 1: Rotational Spectra of Diatomic Molecules
   Chapter 3: Symmetric-Top Molecules
   Chapter 6: Quadrupole Hyperfine Structure in Molecules
   Chapter 8: p 222f: Hyperfine-Structure of NH_3
   Chapter 12: The Ammonia Spectrum and Hindered Motions
   Chapter 13: Shapes and Widths of Spectral Lines
3. Merck Sicherheitsdatenblatt Ammoniak-Lösung 25%
4. G. Nimtz: Mikrowellen
5. Hachenberg-Vowinkel: Mikrowellen