15. Gelation

The Problem

Hot gelatine solution becomes a gel upon cooling. Investigate the electric conductivity as a function of temperature as it gels. Explain the results obtained.

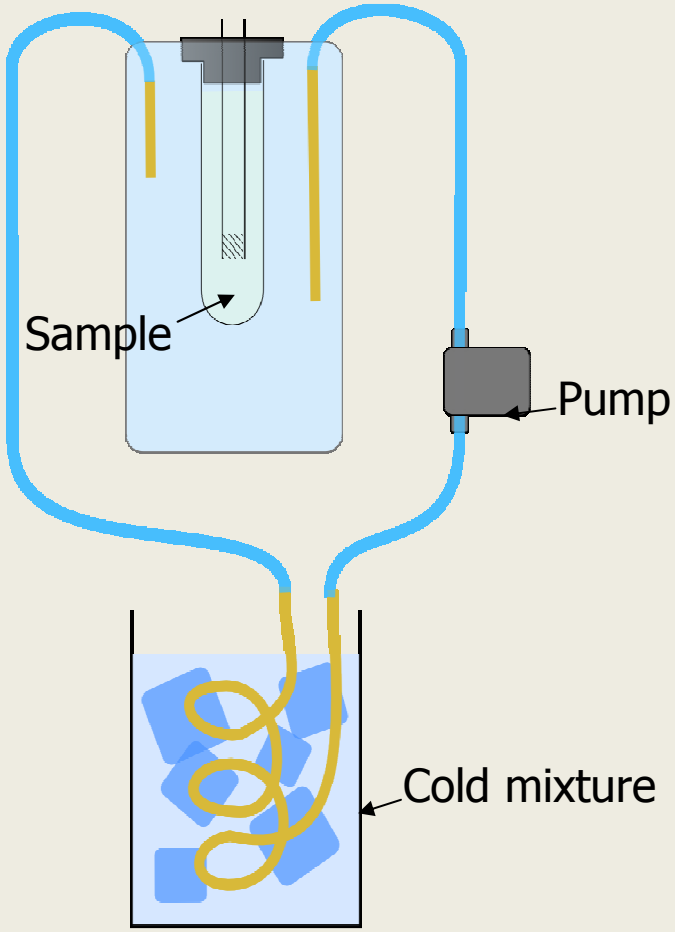
Outline

- Apparatus
  - Cooling mechanism
  - Conductivity measurement
- Apparatus 2
  - Temperature dependence at different frequences
- Structrue of gelatin
- Results
  - Conductivity - temperature dependence
  - Conductivity - voltage dependence
  - Conductivity - frequency dependence
  - Conductivity - temperature dependence measured at different frequencies
- Conclusion

Experiment

- Gelatin – in a closed glass test tube
- Warm gelatin cooled by antifreeze flow
- Temperature measured by a type K thermocouple
- Tinned electrode
  - surface: 1 cm^2
  - distance: 0.5 cm
- AC conductivity measured

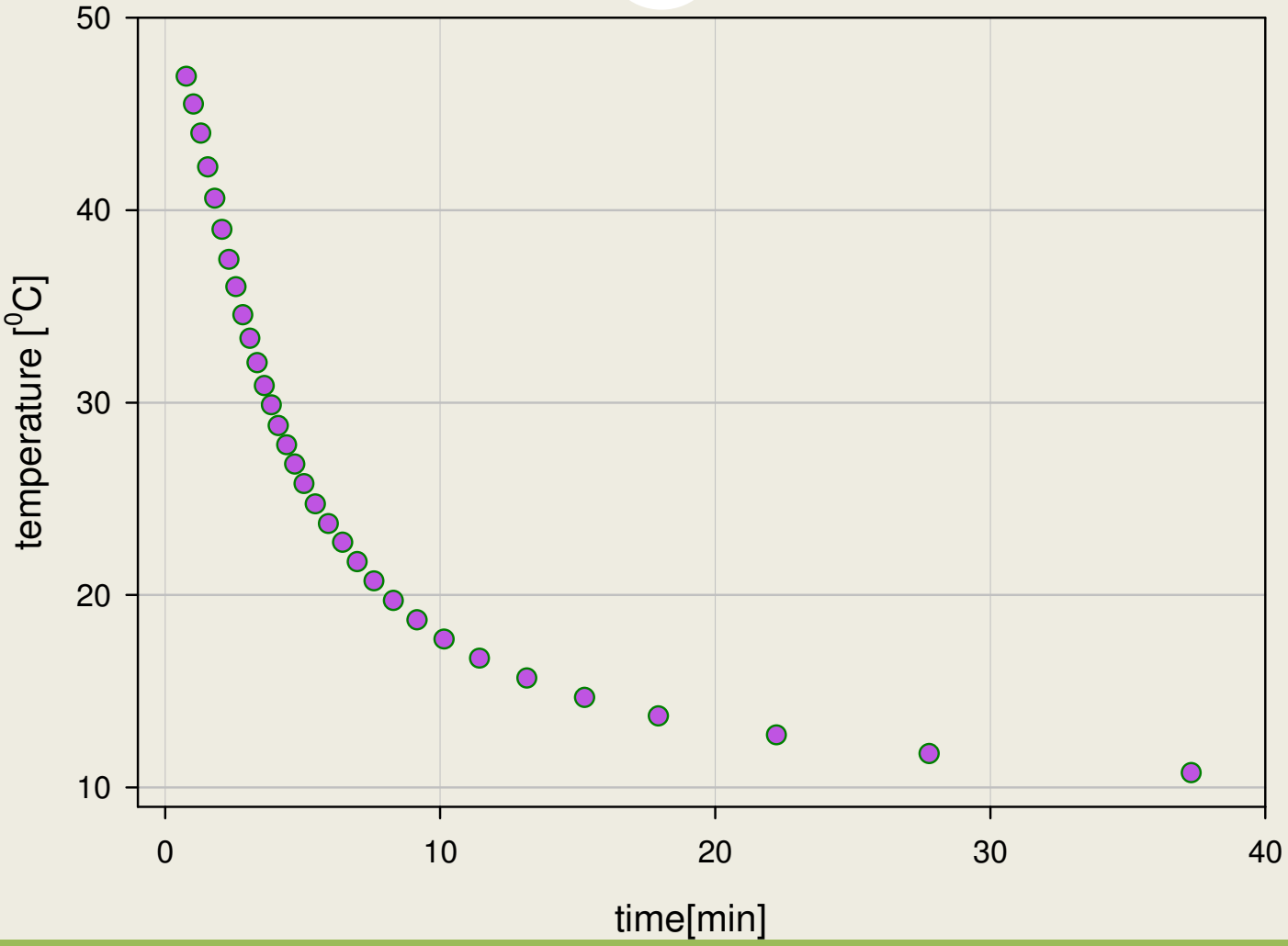
Cooling



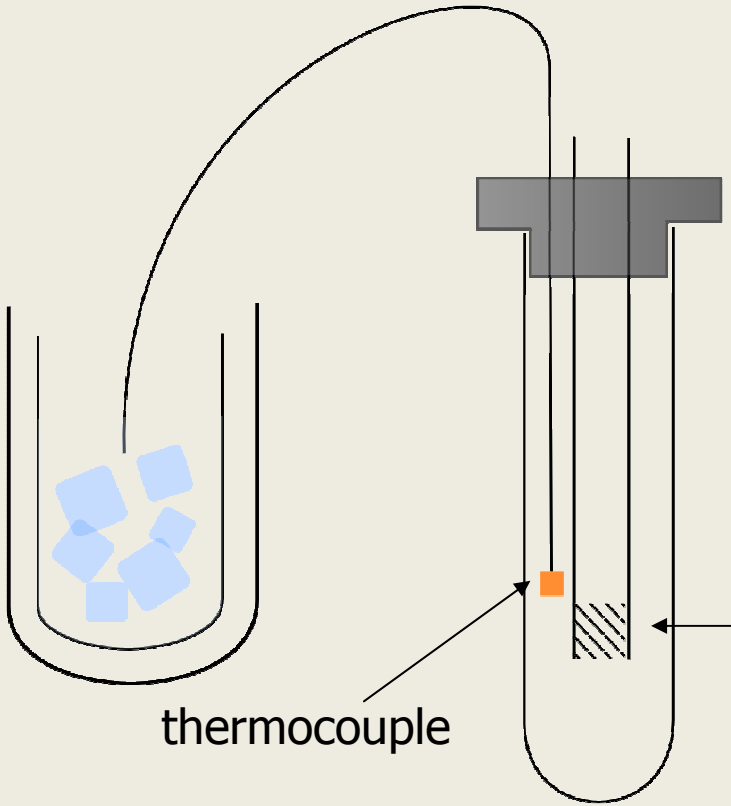
Cooling:

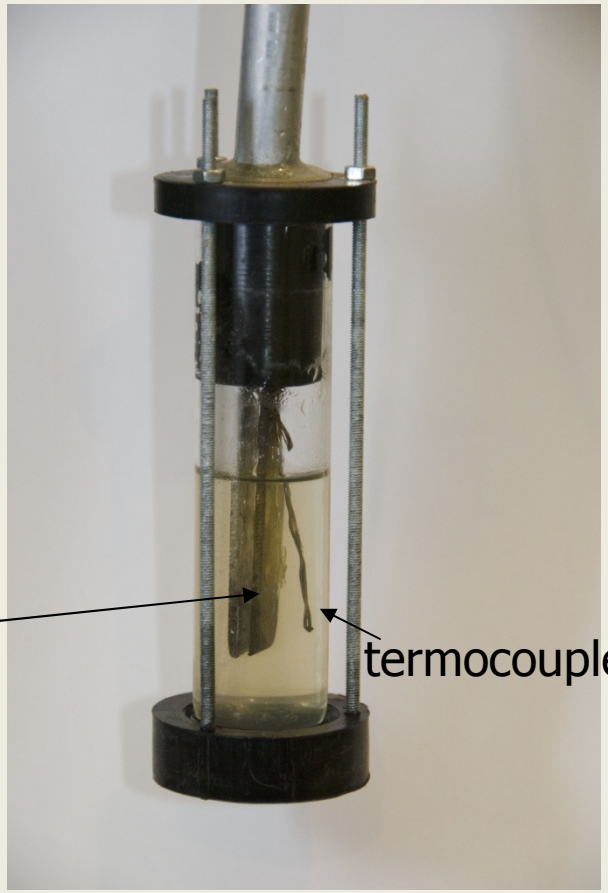
- Antifreeze flow
- Pump
- Cold mixture
  - mixture of ice and mixture mixture salt
  - t=-15°C
- Antifreeze temperature
  - constant
  - t=3°C

Cooling

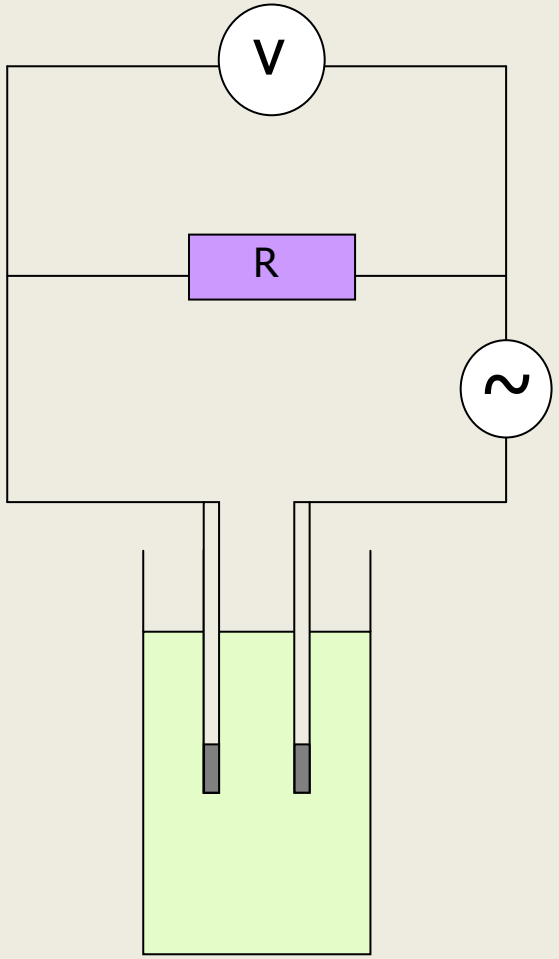


Conductivity measurement





Apparatus



- Voltage on a resistor connected in series measured
- Resistance of gelatin can be calculated:

$$R_g = R\frac{U_0 - U_1}{U_1}$$

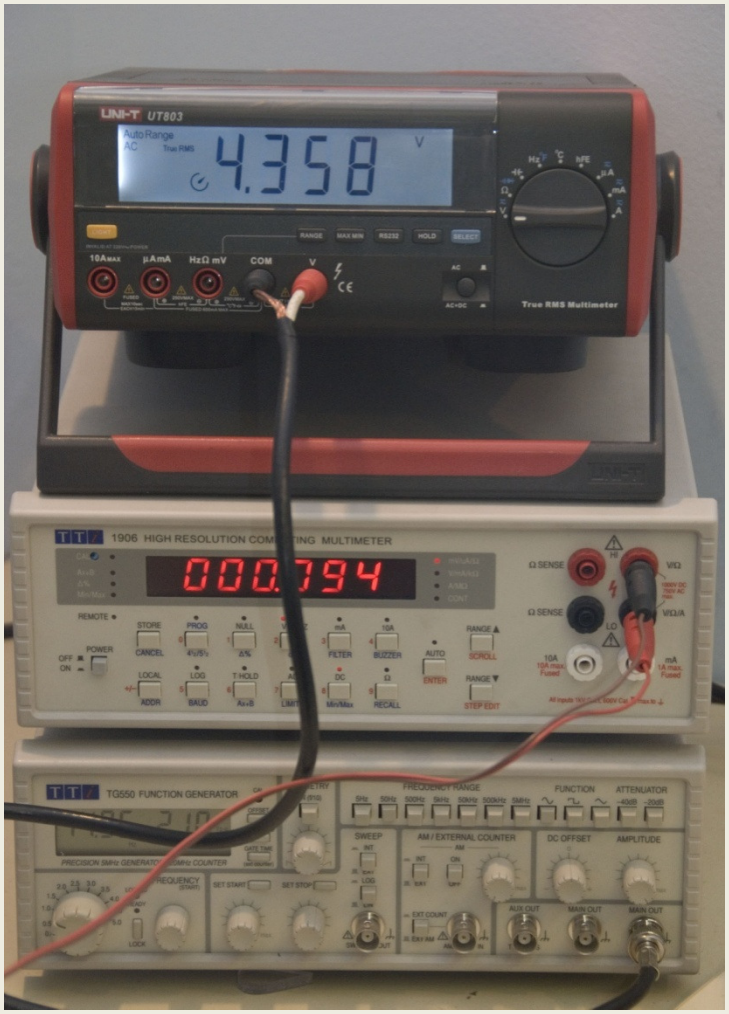
U_0 –source voltage
U_1 – resistor voltage
R_g – gleatin resitance
R – reistor reistance

- Conductivity:

$$\sigma = \frac{1}{R}\frac{d}{S}\frac{U_1}{U_0 - U_1}$$

d – electrode separation
S – electrode surface area
σ - conductivity

Apparatus



← AC voltmeter

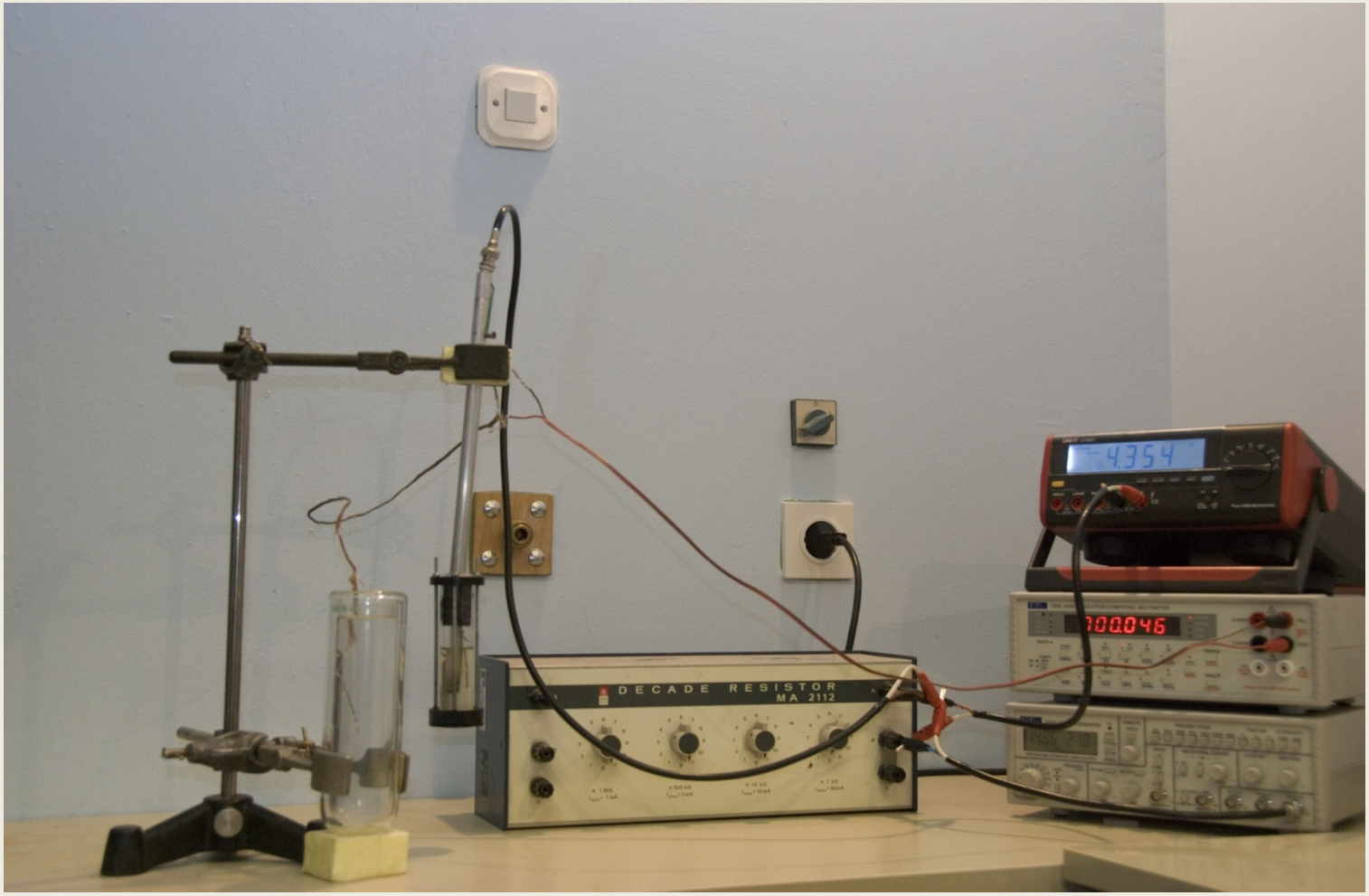
-measures voltage on the resistor

← Voltmeter

- Measures voltage on the thermocouple

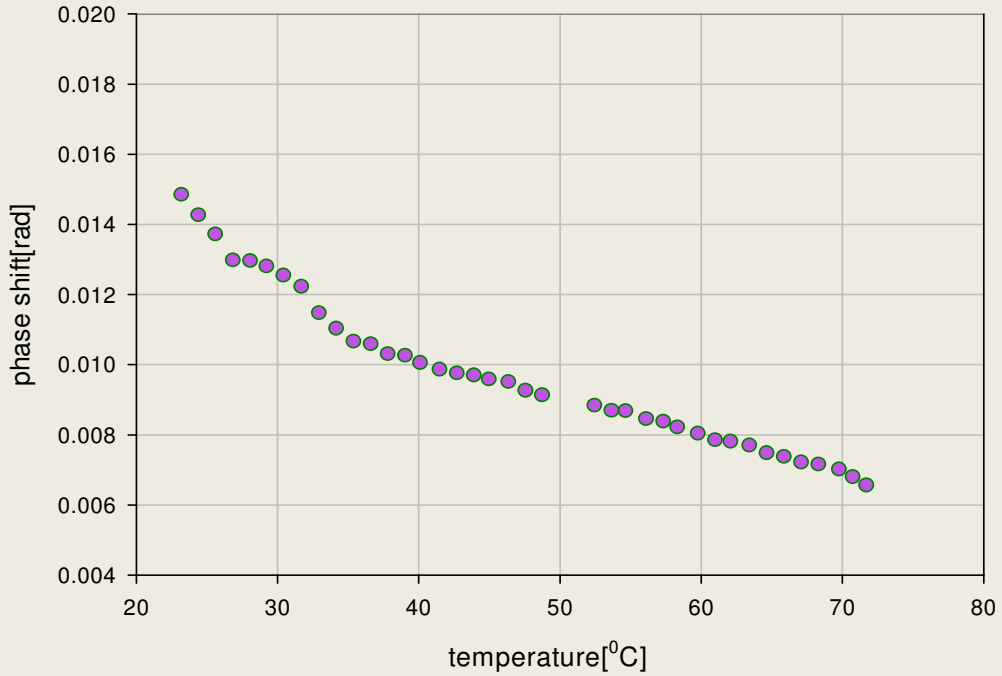
← Function generator

Apparatus



Experiment

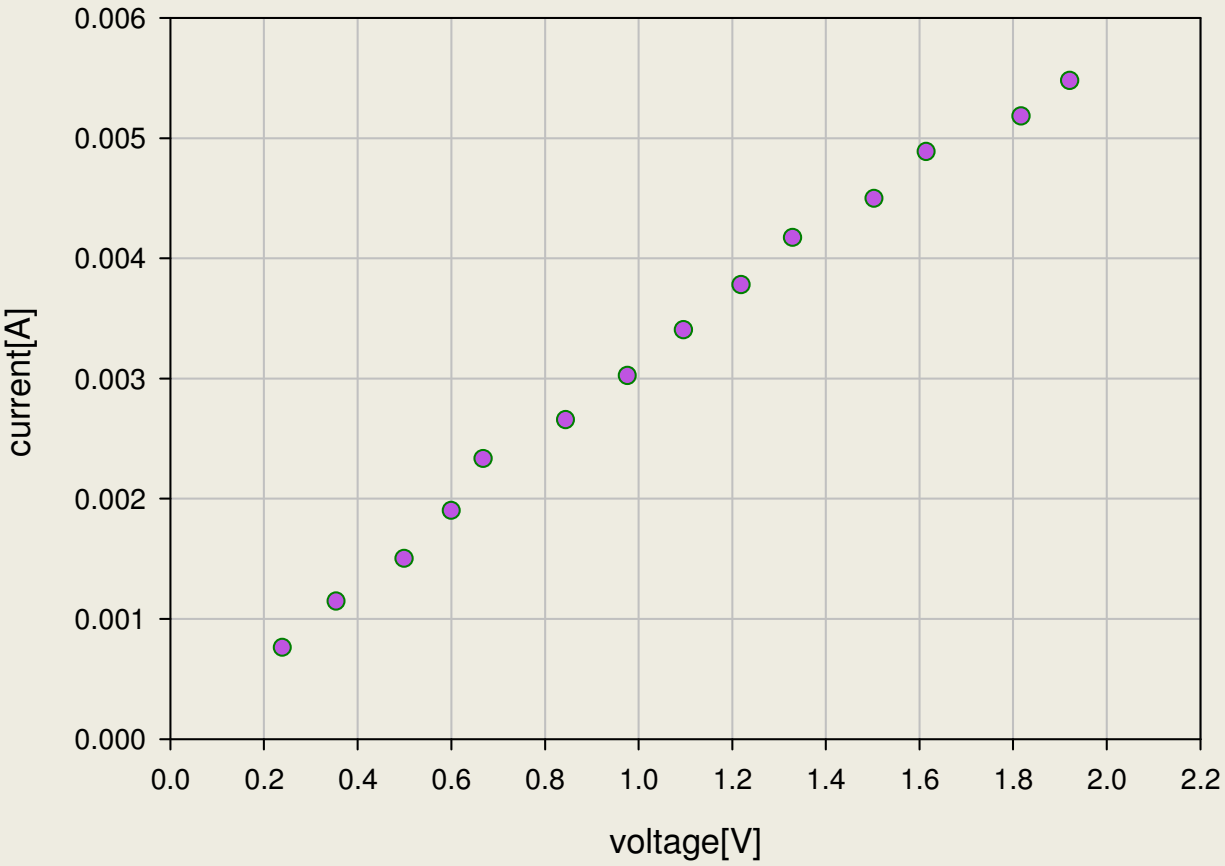
- Between the electrodes – capacatitave and ohmic resistance
- Capacitative resistance causes phase shift between current and voltage
- Phase shift measured by an oscilloscope:



- Phase shift – order of magnitude: 0.1 s
- Phase shift can be ignored at frequencies of order of magnitude 10 Hz

Conductivity

Voltage dependence



- Linear current – voltage dependence
- Resistance – the slope of the line
- Conductivity is constant at this voltage range

Apparatus 2

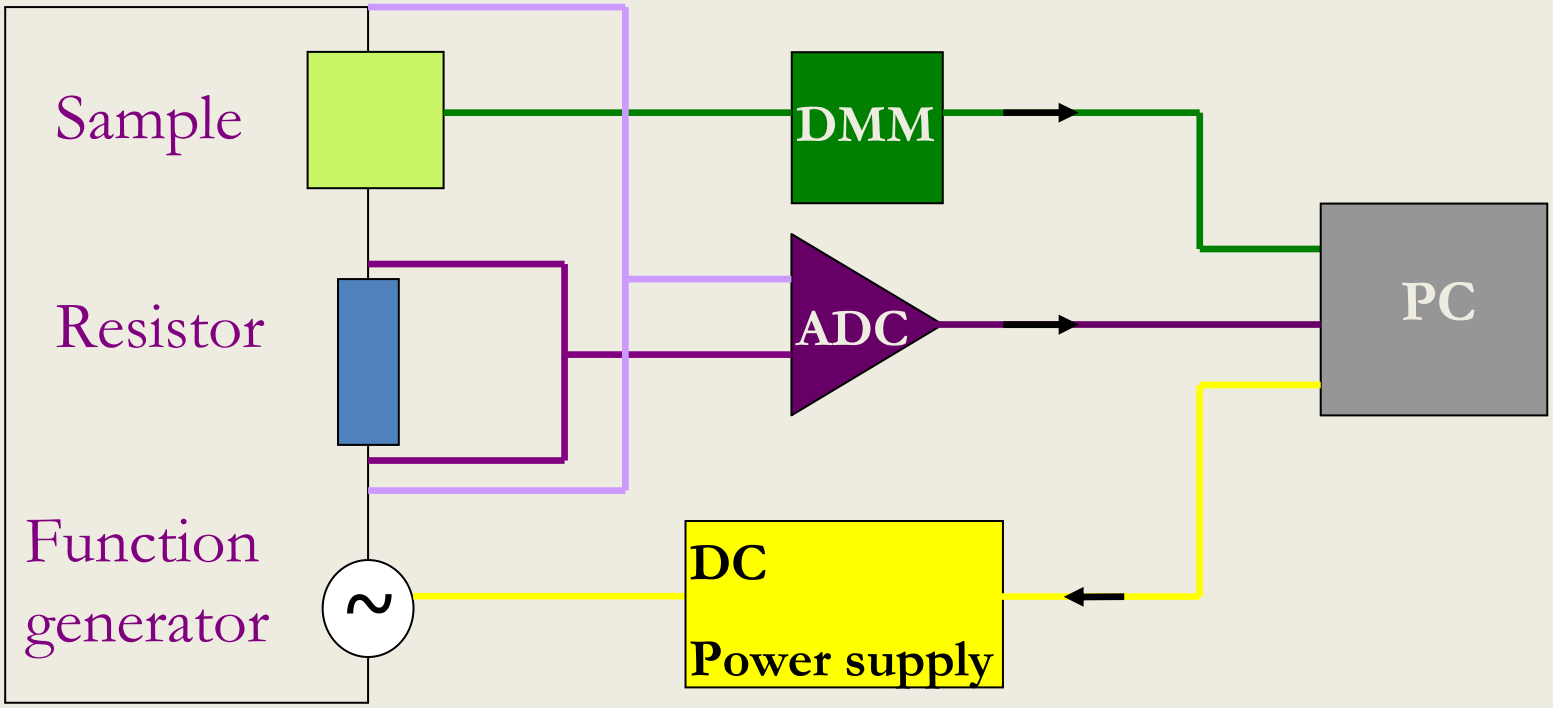
- Temperature dependence measured at different frequecies
- Necessary to measure during one cooling
- When temperature drops to a certain value conductivity measuret on the whole frequency range
- A computer program written in Visual Basic controlled the instruments

Apparatus 2

- Program
  - Temperature measured
    - DC power supply sets a frequency on the function generator
    - Voltage measured
    - New frequency
      - 30 frequencies
      - Range: 1 kHz – 5MHz
  - When temperature drops for 2 degrees the program starts over

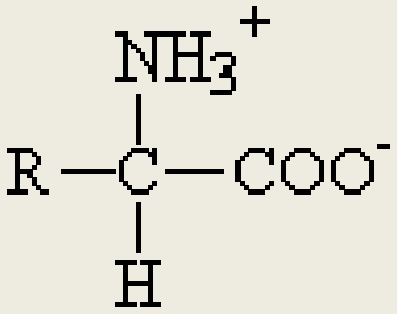
15 s

Apparatus 2


Sample
Resistor
Function generator
DMM
ADC
PC
DC
Power supply

Gelatin structure

- Gelatin is a protein
- Proteins – polymers build of aminoacids
- Aminoacid structure:



- Amino acids connect to form polypeptide chains:

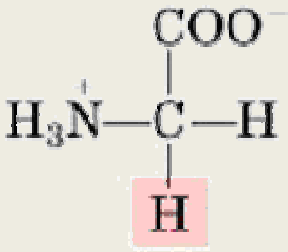


Gelatin structure

- R group
  - Defines the aminoacid
  - Can be polar, non-polar or charged
- In gelatin:

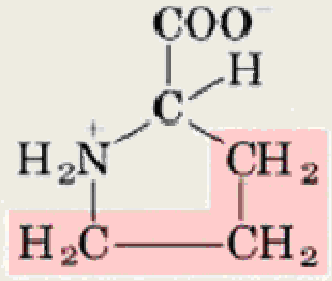
Glycine

27.2%



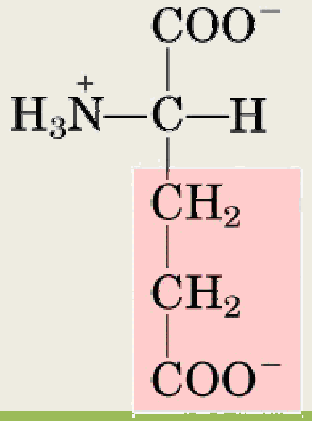
Proline

15.5%



Glutamic acid

11%

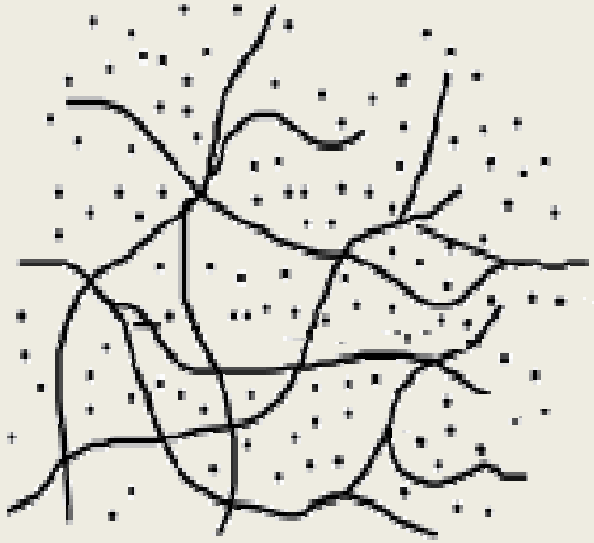


Gelatin structure

- Liquid phase
  - Protein molecule in water
  - Colloid solution
- Cooling
  - Smaller speed of thermal motion
  - Molecules don't have enough energy to resist attractive Coulomb forces between each other
  - molecules form chains

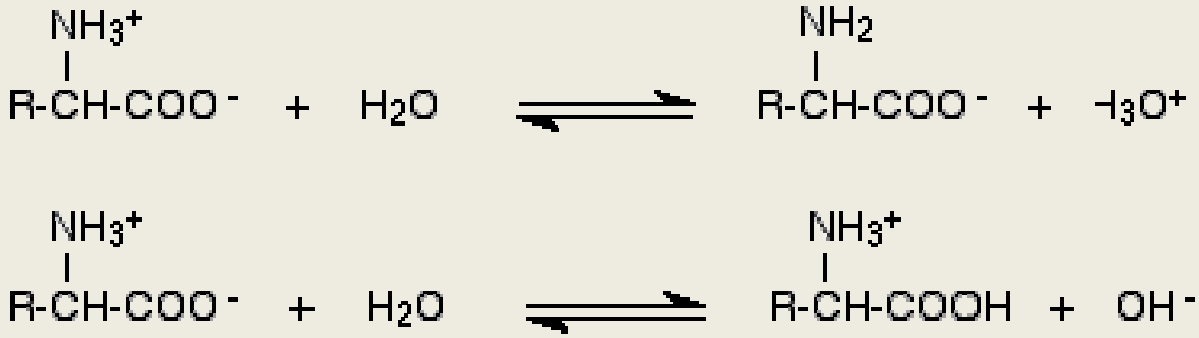
Gelatin structure

- Gel
  - The chains become longer
  - Long chaines form network
  - Rheological properties of olid bodies
  - Water molecules are in spaces between proteins



Reactions with water

- Aminoacids in water:



- Because of these reactions
  - some chain are positivly charged and some are negativly charged
  - Concentration of OH^- ions grows H_3O^+

Conductivity

Mechanism of conducting electrical current:

1) Moving of H_3O^+ and OH^- ions through the solution
2) Ion hopping

   -ions can hop from one chain to other when they get close
3) Oscillation of charged parts of protein chains

   - oscillation of parts of the chains increases conductivity at higher frequencies

Conductivity
temperature dependence

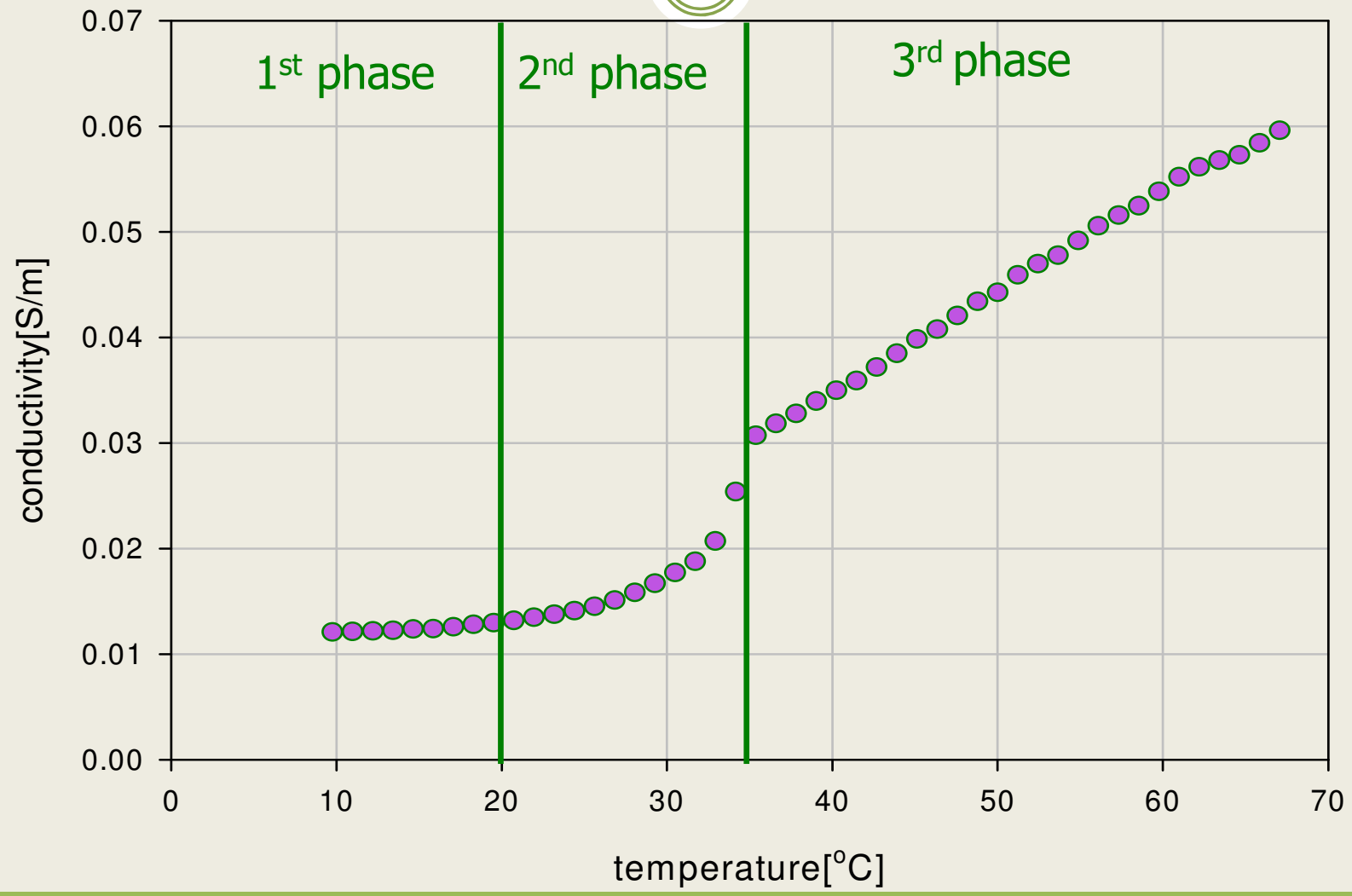
- Coductivity expected to grow with temperature
  - H_3O^+ and OH^- ions have more energy – they move faster
  - The protein chains can move more freely at higher temperatures
    - Easier for ions between them to move
    - Easier ion hopping
- Sol-gel transition - sudden conductivity growth expected
  - The structure untangles
  - Rotational movement of chain parts becomes possible
  - Transversal movement of H_3O^+ and OH^- ions is much easier
  - Ion hopping easier

Conductivity
frequency dependence

- Frequency expected to be low at low frequencies and to rise
  - Chain oscillations don't contribute to conductivity
  - Ions have to move a long way to transmit a signal
    - Whole distance between the electorodes for DC
- When frequencies are too large, the field changes direction very quickly
  - Charge carriers can't move fast enough
  - Conductivity drop
- A maximum expected

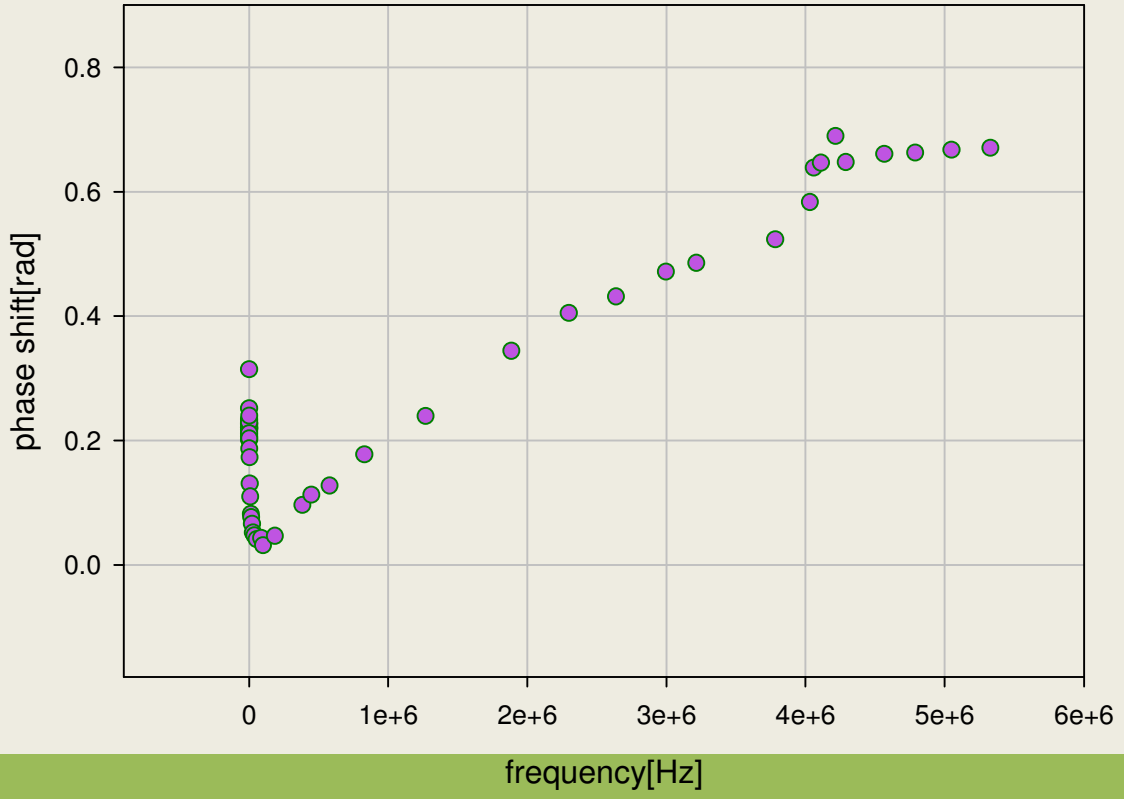
Conductivity

temperature dependence



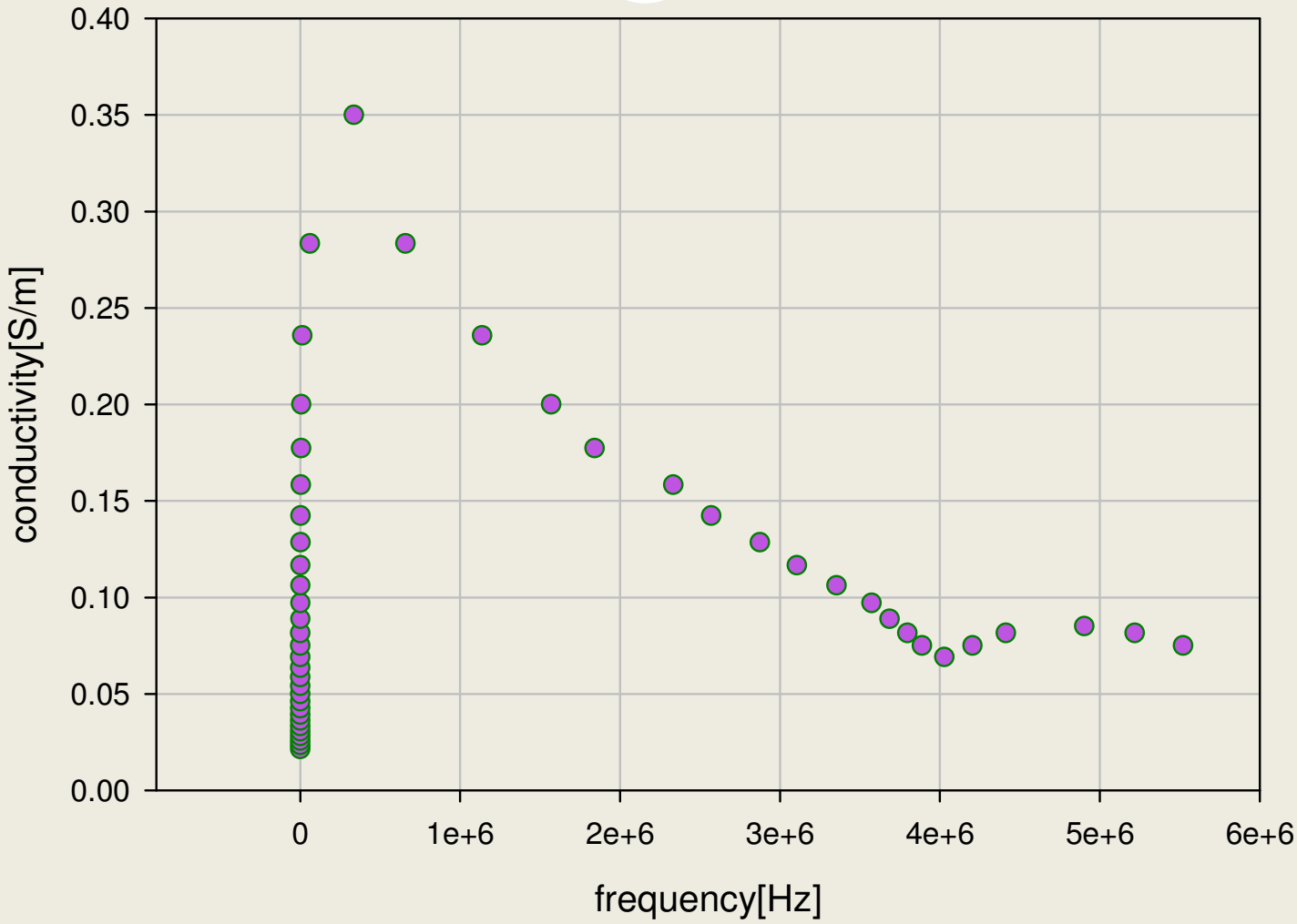
Conductivity
frequency dependence

- Temperature: 21°C
- Frequency range: 10Hz - 5MHz
- Phase shift frequency dependence:



- Phase shift order of magnitude 0.1 rad
- cosφ~0.999
- Phase shift can be neglected on the whole frequency range!

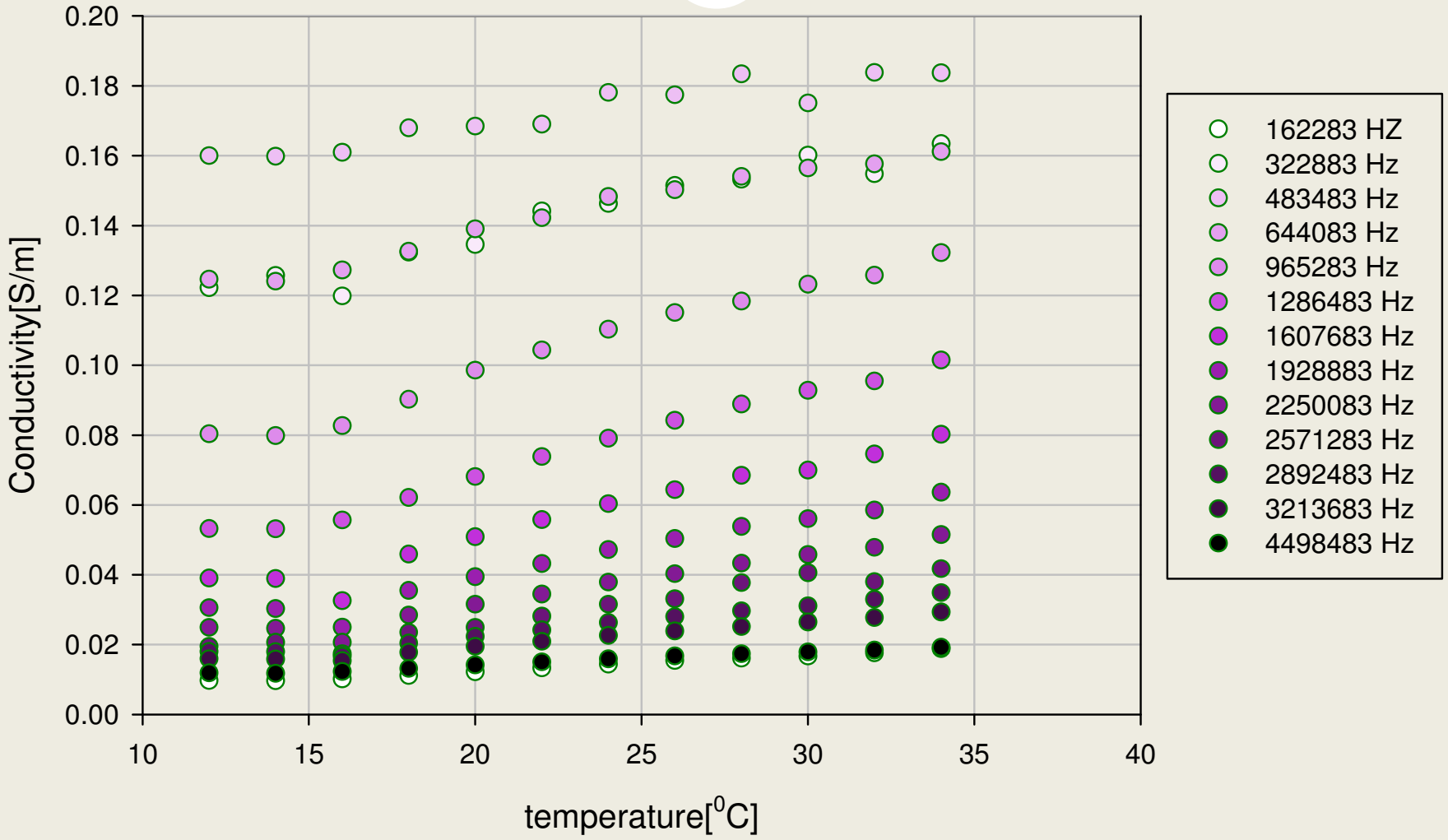
Conductivity
frequency dependence



t=21^{0}C

Conductivity

-temperature dependence - frequencies



Conclusion

- Gelatin structure considered
- 3 ways of conducting electrical current explained:
  - Movement of ions through the solution
  - Ion hopping
  - Oscillations of parts of protein chains
- Apparatus constructed
  - Cooling gelatin
  - Measuring condustivity
- Frequency dependence of conductivity measured and explained
  - Conductivity is very small at small frequencies
  - Coductivity grows until 100 kHz
  - After 100kHz conductivity slowly drops

Conclusion

- Temperature dependence of gelatin conductivity measured:
  - Conductivity grows with temperature
  - Sudden increase in conductivity observed at the sol-gel transition
- Temperature dependence explained
  - Charge carriers move more easily at higher temperatures
  - They move much more easily in sol than in gel phase
- Temperature dependence measured at differerent frequencies
  - The curve has the same shape
  - Conductivity values differ



Thank you for your attention!