

Technische Universität München Analytical Research Group PD Dr. Thomas Letzel; PD Dr. Johanna Graßmann	<b>Exam Questions – Part 2</b> <b>Analysis procedures, sample</b> <b>preparation and reaction equations</b>
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**Questions from the “PAL Prüfungsbuch”**  
**Analysis procedures, sample preparation and reaction equations**

**307:** What does one understand by an analysis sample?

- 1) It is a homogenized mixture of representative individual samples
- 2) It is a sample of which the chemical composition is known
- 3) It is the final product of sampling that is completely prepared for the upcoming analysis
- 4) It is the purified final product of sampling
- 5) It is the united individual samples from sampling

**308:** What is referred to as validation?

- 1) It is the assessment of the validity of the analysis method
- 2) It is the first calibration of a testing agent
- 3) It is the conduction of multiple examinations in analysis
- 4) It is the controlling of the test by another testing laboratory
- 5) It is the briefing of employees in analytical methods

**309:** Which functions fulfil sampling and sample preparation in chemical analysis?

- 1) They ensure the supply of a representative analysis sample
- 2) They are only possible with liquid substances
- 3) They are only possible with solid substances
- 4) They end with taking the sample at the sample location
- 5) They ensure the supply of large sample quantities

**310:** Which of the following individual operations is called aliquoting?

- 1) Taking of defined parts of an overall solution
- 2) Taking of partial solutions for the standard addition method
- 3) Preparation of a calibration series by different amounts of weight portions
- 4) Addition of an inner standard to check measuring instruments
- 5) Staining of the analyte for photometry

**311:** With a surplus of sodium carbonate calcium carbonate is precipitated from an aqueous hydrochloric calcium chloride solution. The calcium carbonate then is separated by suction filtration. The suction filtration cake is washed to clean it. Which measure is *not suitable* to examine the success of the washing process?

- 1)  $\text{Na}^+$  detection in the draining off washing water
- 2)  $\text{Cl}^-$  detection in the draining off washing water
- 3) Controlling the draining off washing water with pH-paper
- 4)  $\text{Ca}^{2+}$  detection in the draining off washing water
- 5) Adding  $\text{Ag}^+$  ions to the washing water



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**312:** For which working operation sample segregation is of minor importance?

- 1) Digestion (pulping)
- 2) Weighing in the sample for analysis
- 3) Examination of an analyte species
- 4) Sieve analysis
- 5) Weighing in the sample to determine bulk density

**313:** Which of the named cases describes a contamination in the context of chemical analysis?

- 1) Sample solution, analyte or parts of the sample are adsorbed onto the sample vessel wall
- 2) During the sample preparation the analyte escapes from the sample solution
- 3) Parts of a dissolved analysis sample hydrolyse
- 4) Substances that beforehand were adsorbed onto the sample vessel wall dissolve into the analysis sample
- 5) Interfering matrix elements are adsorbed onto activated carbon

**314:** In chemical analysis what is understood by digestion (pulping)?

- 1) Adding acid or base to an analyte
- 2) Burning of a sample in a stream of oxygen
- 3) Transformation of an analyte into a soluble form by a chemical reaction
- 4) Dissolving a soluble analyte
- 5) Separation of an analyte from the sample matrix

**315:** In which of the named reactions *no* salt is formed?

- 1) Acid anhydride + base
- 2) Acid anhydride + water
- 3) Acid + base
- 4) Metal oxide + acid
- 5) Non-noble metal + acid

**316:** A spectroscopic measurement is conducted at a measuring wavelength of  $\lambda = 560\text{nm}$ . What is the correct name of the technique this operation belongs to?

- 1) VIS-spectroscopy
- 2) UV-spectroscopy
- 3) IR-spectroscopy
- 4) Micro wave-spectroscopy
- 5) Raman-spectroscopy

**317:** In which of the following cases the named measuring method is not suitable?

- 1) Determination of the melting point to test the chemical purity of acetylsalicylic acid
- 2) Refractometry to determine the chemical purity of ethyl ethanoate after distillation
- 3) Polarimetry to determine the concentration of butan-2-ol in ethanol
- 4) Measurement of conductivity to determine the overall salt content in seawater
- 5) Measurement of the pH-value to determine the heavy metal concentration in the ground after acidic rain

**318:** Which statement regarding polarimetry is correct?

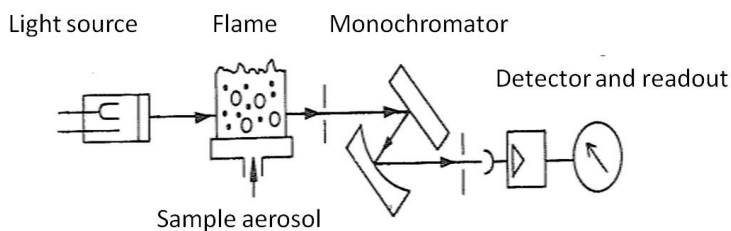
- 1) In polarimetry a specific wavelength is measured
- 2) In polarimetry the scatter of the Nicol prism is used
- 3) In polarimetry always polychromatic light is used for measuring
- 4) In polarimetry optical active substances are analyzed
- 5) In polarimetry one must measure discontinuous

**322:** Which of the following relations of method and physical principle in instrumental chemistry is correct?

Method	Physical principle
1) Atomic absorption spectrometry	Vaporization of the substance, excitation of the atoms, analysis of the emitted light
2) Partition gas chromatography	Vaporization of the substance, separation by adsorption and desorption processes, acquisition with a detector
3) IR-spectroscopy	Adsorption by electromagnetic radiation, which is necessary for the excitation of the electrons
4) Mass spectrometry	Vaporization and ionisation of the substance, separation of the ions regarding charge and mass
5) NMR-spectroscopy	Measurement of the magnetic moment of a dipole in a compound

**323:** What kind of equipment is depicted in this functional diagram?

- 1) Atomic absorption spectrometer
- 2) Flame photometer
- 3) Mass spectrometer
- 4) Infrared spectrometer
- 5) Ultraviolet spectrometer



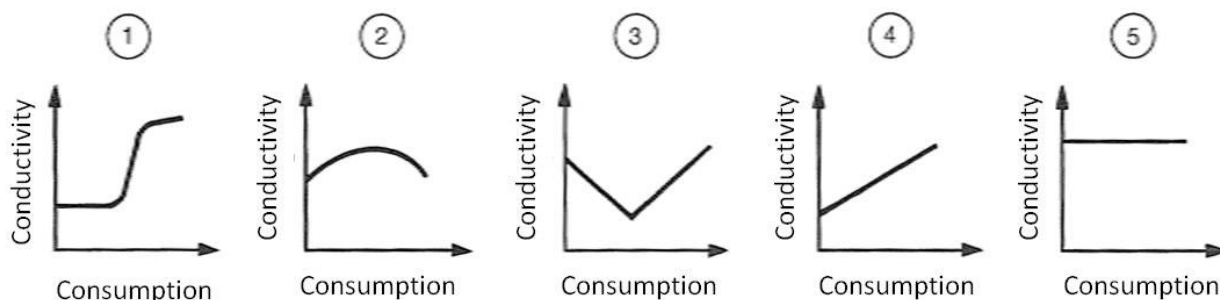
**324:** Which analytic procedure uses the conductivity of dissolved ions for their quantitative determination?

- 1) Paper chromatography
- 2) Photometry
- 3) Gas chromatography
- 4) Potentiometry
- 5) Conductometry

**325:** Which of the following statements about conductometric titration is correct?

- 1) It is mainly applied in complexometry
- 2) For measurement a glass electrode is necessary
- 3) A cell constant must be set at the potentiometer
- 4) One must work at temperatures  $> 80^{\circ}\text{C}$
- 5) It is suitable in precipitation titration for the determination of the equivalence point

**326:** Which diagram is characteristic for a conductometric titration?



**327:** In conductometric titration the electric resistance is measured. The electric resistance of an electrolyte solution does *not* depend on which of the named values?

- 1) Temperature
- 2) Distance of the electrodes
- 3) Size of the electrode areas
- 4) Applied voltage
- 5) Amount of substance concentration of the ions

**336:** What is crucial for the separation in mass spectroscopy?

- 1) Only the mass of the fragment
- 2) Only the electrical charge of the fragment
- 3) Mass and electrical charge of the fragment
- 4) Number of protons
- 5) Number of electrons

**337:** Which of the following statements about wavelength is correct?

- 1) The energy content of electromagnetic waves is directly proportional to their wavelength
- 2) The reciprocal value of wavelength is called wave number
- 3) On the interface between two phases the wavelength does not change
- 4) The product of wavelength and velocity of light is the frequency
- 5) UV-radiation has a higher wavelength than visible light

**338:** Which statement about photometry is *wrong*?

- 1)  $E$  is directly proportional to the amount of substance concentration
- 2)  $\varepsilon$  is substance specific
- 3)  $\varepsilon$  is independent from wavelength
- 4)  $E$  decreases with shrinking layer thickness
- 5) The Lambert-Beer law only applies for the linear range of the calibrating curve

**339:** What does the Lambert-Beer law state analogously?

- 1) Absorption increases with decreasing layer thickness
- 2) Absorption is proportional to the number of absorbing particles
- 3) Absorption is proportional to the amount of solvent
- 4) The absorbed quantity of light is equal to the non-absorbed quantity of light
- 5) The absorbed quantity of light is independent from the intensity of the radiation source

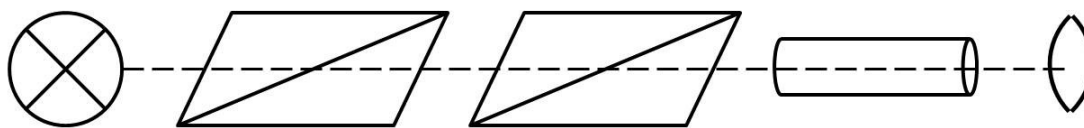
**340:** Which statement regarding the Lambert-Beer law is *wrong*?

- 1) The molar decadic extinction coefficient  $\epsilon$  depends on the concentration of the analyte
- 2) With a doubled layer thickness the extinction is twice as big
- 3) If, with unchanged layer thickness, the analysis solution is triply diluted resulting extinction is one third
- 4) A change of wavelength will result in a change of extinction
- 5) The extinction is directly proportional to the concentration of the analyte

**341:** What will cause a photometric analysis to turn out faulty?

- 1) The cuvette is filled only three quarters
- 2) There are finger prints on the blind side of the cuvette
- 3) A quartz cuvette was used for calibration and sample measuring
- 4) There is a scratch on the cuvette, which is located only on the lower half of the clear side
- 5) During the sample measuring the cuvette is sealed with a lid

**342:** Which components are interchanged in the following sketch of a polarimeter?



A) Light source

B) Polarizer

C) Analyzer

D) Cuvette

E) Observation lens

- 1) A with C
- 2) B with D
- 3) A with B
- 4) A with E
- 5) C with D

**343:** For the photometric determination of iron monochromatic light is necessary. Which component can be used as monochromator?

- 1) Detector
- 2) Cuvette
- 3) Chopper
- 4) Optical grid
- 5) Tungsten lamp

**344:** After adding ammonia solution an intense blue colouring developed in the test solution. Which ion was identified?

- 1)  $\text{Mg}^{2+}$
- 2)  $\text{Co}^{2+}$
- 3)  $\text{Cd}^{2+}$
- 4)  $\text{Cr}^{3+}$
- 5)  $\text{Cu}^{2+}$

**345:** Schematically the outline shows the passage of light through a photometric cuvette. Which equation for the given photometric units is correct?

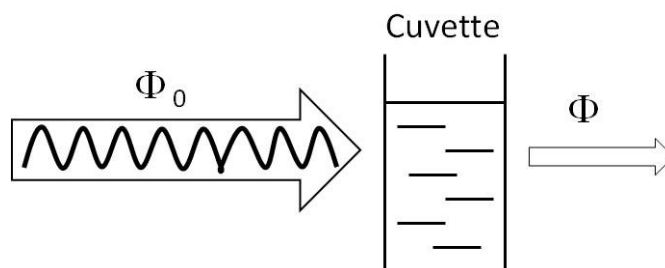
1)  $A = \frac{\Phi_0}{\Phi}$

2)  $E = -\lg T$

3)  $E = -\lg A$

4)  $T = \frac{\Phi_0 - \Phi}{\Phi}$

5)  $T = -\lg \frac{\Phi}{\Phi_0}$



$\alpha$  = Absorption  
 $A$  = Spectral absorbance  
 $T$  = Transmission

**346:** Which of the following components *cannot* be found in an UV/VIS-spectrophotometer?

- 1) Photodiode array
- 2) Deuterium lamp
- 3) Nicol prism
- 4) Entrance slit
- 5) Monochromator

**347:** Why do double beam photometer in UV/VIS-absorption spectroscopy work with two beam paths?

- 1) With the second optical path the impact of solvent and cuvette on the absorption are compensated
- 2) One optical path is needed for the UV-light, the other for the visible light
- 3) For exact measurement both beams that are emitted from the light source are necessary
- 4) The second light beam is necessary due to the used semi-permeable mirror
- 5) Because the test result has to be amplified two beam paths are necessary

**348:** Which statement regarding IR-spectroscopy is *wrong*?

- 1) Transmission spectra are unsuitable for quantitative analysis
- 2) One part of the IR-spectrum is called fingerprint area
- 3) In an IR-spectrum the components of a substance mixture can be identified
- 4) IR-spectra are suitable for qualitative and quantitative evaluation
- 5) In an IR-spectrum often it is possible to detect the presence of functional groups

**349:** Which of the following substances is *not* IR-active?

- 1) Hydrogen chloride
- 2) Oxygen
- 3) Methane
- 4) Ethanol
- 5) Toluol

**350:** Which fundamental condition must be met that an IR-spectrum of a molecule shows a band?

- 1) The concerning substance must be present as a liquid or gas
- 2) The molecule must be unpolar
- 3) Only polar substances show an IR-spectrum
- 4) With the vibrational excitation the polarity of the molecule must change
- 5) With the vibrational excitation the dipole moment of the molecule must change

**351:** Whereby absorption bands in IR-spectroscopy emerge?

- 1) Resonance vibration of atoms or atomic groups
- 2) Radiance of samples (luminescence)
- 3) Absorption of the visible light
- 4) Resonance vibration of  $\pi$ - electrons
- 5) Absorption of energy by electrons (quantum jump)

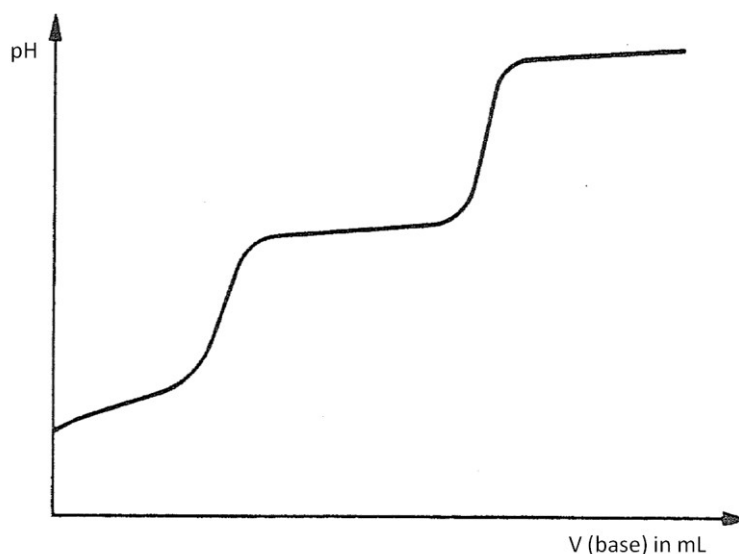
**352:** Which statement about IR-spectroscopy is *wrong*?

- 1) Molecular vibrations are a combination of stretching (valence) vibrations and deformation (scissor) vibrations
- 2) A complex molecule possesses a large number of vibration possibilities at which the whole molecule participates
- 3) The area below the wave number  $1500\text{ cm}^{-1}$  contains many bands and characterizes the molecule as a whole
- 4) Above the wave number  $1500\text{ cm}^{-1}$  absorption bands are located that can be allocated to certain functional groups
- 5) An IR-spectrum comes into existence by mutual vibration of electrons



354: In this diagram a titration curve is depicted. Which acid has been titrated?

- 1) Acetic acid
- 2) Formic acid
- 3) Nitric acid
- 4) Hydrochloric acid
- 5) Phosphoric acid



363: Which iodine compound is used as a reducing agent in iodometry?

- 1)  $I_2$
- 2)  $KIO_3$
- 3)  $KIO$
- 4)  $KI$
- 5)  $KIO_4$

364: Which statement about the water determination according to Karl Fischer is *wrong*?

- 1) The titer of the used solution must be regularly re-assessed
- 2) For indication a pH-electrode is used
- 3) In this determination method  $SO_2$  is oxidized by iodine in the presence of water to  $H_2SO_4$
- 4) The used solution consists of iodine,  $SO_2$  and an organic base in an anhydrous solvent
- 5) Next to water as primary standard substance it is possible to use oxalic acid dihydrate

365: Which statement about partition chromatography is *wrong*?

- 1) The separation of substance mixtures is accomplished by partition between a moving solvent and a stationary liquid, that is held by a solid matter carrier
- 2) The mobile phase can be a liquid
- 3) The moving solvent is called mobile phase
- 4) Substances that are readily soluble in the mobile phase remain at the starting point
- 5) The mobile phase can be a gas

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**372:** Columns that are used in GC have a theoretical plate number of approx. 10,000, HPLC columns have theoretical plate numbers of approx. 3,000. What kind of effects has this on the injection of a multi-component mixture?

- 1) HPLC is three fold efficient
- 2) A GC column delivers narrower peaks in comparable retention times
- 3) A HPLC column has a better separation efficiency
- 4) In GC the peak areas are three times bigger than in HPLC
- 5) A GC column can separate about three times the amount of sample

**373:** Why is helium often used as carrier gas when using a thermal conductivity detector (main reason)?

- 1) Helium is inexpensive
- 2) It prevents side reactions (helium is inert)
- 3) There is a relatively high thermal conductivity difference between analyte and helium
- 4) With deployment of helium the thermal conductivity remains constant
- 5) The deployment of helium is first of all a safety-related issue

**374:** In a gas chromatogram the peak at 10.5 min is very wide and has a fronting. Which measure does *not* lead to an improvement of the peak form?

- 1) Decreasing the split from 80:1 to 40:1
- 2) Use of a column with a larger inner diameter
- 3) Increasing the velocity of the mobile phase
- 4) Decreasing the injected amount
- 5) Diluting the sample

**375:** Which measure extends the retention time of a substance in a gas chromatograph?

- 1) Increasing the oven temperature
- 2) Use of a longer column
- 3) Increasing the velocity of the carrier gas
- 4) Increasing the injection temperature
- 5) Application of a column with lower film thickness

**376:** The thin layer chromatographic separation of two substances is to be improved. Which measure does *not* influence the separation?

- 1) Choosing another mobile solvent
- 2) Use of another stationary phase
- 3) Double development
- 4) Using another means of detection, to make the two substances more visible
- 5) Using a HPTLC-plate

**377:** Which statement about thin layer chromatography is *wrong*?

- 1) It serves to monitor chemical reactions
- 2) It is used to control purity
- 3) It is more a quantitative than a qualitative method
- 4) It serves to analyse product mixtures
- 5) It can be used for the determination of the conditions for the preparative column chromatography

**378:** 100mL each of the named acids are available at 20°C with the pH-value 4. For which acid the highest amount of caustic soda  $c(\text{NaOH}) = 0,1 \text{ mol/L}$  is needed for its complete neutralization?

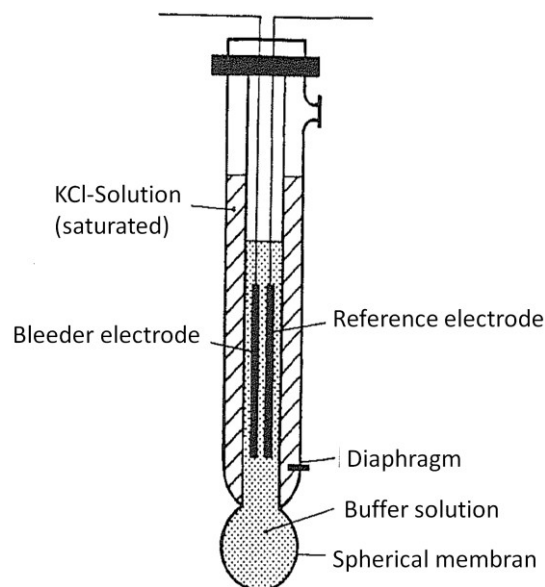
- 1) Acetic acid
- 2) Nitric acid
- 3) Hydrochloric acid
- 4) Sulphuric acid
- 5) For all acids the same volume is needed

**379:** An acid gets into in an acetate buffer solution. Which part of the buffer system is the reason for the very little change of the pH-value?

- 1) The  $\text{OH}^-$ -ions of the water
- 2) The cations of the salt
- 3) The water in the solution
- 4) The  $\text{H}_3\text{O}^+$ - ions of the water
- 5) The acetate ions of the salt

**380:** What item is depicted at the wrong place in this design of a combination electrode?

- 1) Bleeder electrode
- 2) Buffer solution
- 3) Reference electrode
- 4) Diaphragm
- 5) Spherical membrane



**381:** What is an ion selective electrode?

- 1) It is a special electrode, that can be used to prove if ions are (still) present in distilled water
- 2) It is a special electrode, with which it is possible to determine the concentration of a specific ion (e.g. fluoride concentration)
- 3) It is a special electrode, that is used to test for salts in oversaturated steam
- 4) It is a special electrode, that is used in mass spectrometers to count ions
- 5) It is a special electrode, that is used for the potentiometric measurement of ion concentrations without reference electrode