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Surface chemical analysis — Determination of the minimum detectability of surface plasmon resonance device

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Foreword

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This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The surface plasmon resonance (SPR) is the term used for the real time chemical contents analysing device. The chemical ingredient dissolved in buffer solvent causes the dielectric constant change compared to the buffer solvent. Changes in the dielectric constant of the solution modify the resonance condition of the surface plasmon coupling at the interface between metal (mostly gold or functionalized gold) and solution channel. So the reflection from the interface has the dip corresponding to the surface plasmon components which is evanescent. The change of the reflection spectrum is analysed by a charge coupled device (CCD) and the change of the spectrum dip represents the absolute amount of the surface existing chemical component at the interface. The determination of the dynamic range of the chemical analysis depends on the upper limit and lower limit of the detectability of the SPR device. The objective of this document is to provide the standardized definition of lower limit of detection and experimental protocol of measuring the lowest detectability of the SPR device. To avoid the complex and unwanted chemical interaction between the metal surface and the analyte, a single chemical solute method is presented, suitable for use by non-expert operators. That provides users with the fundamental capability of the SPR device.

Surface chemical analysis — Determination of the minimum detectability of surface plasmon resonance device

1 Scope

This document describes a method for determining the minimum detectability of surface plasmon resonance device. This document is applicable to surface plasmon resonance devices of the white-light illumination type and the laser illumination type with the angle scanning capability.

2 Normative references

There are no normative references in this document.

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1.1
sensorgram
graph of responses versus time in surface plasmon resonance studies

3.2 Abbreviated terms

SPR surface plasmon resonance

RU response unit

CCD charge coupled device

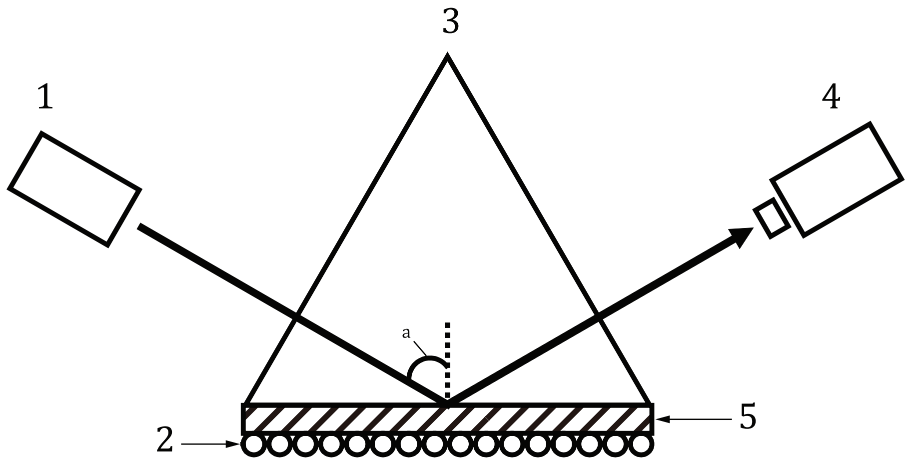
SD standard deviation

DI deionized

4 General information

4.1 Overview

Surface plasmon is the light-matter interaction due to the collective longitudinal coupling between the surface electrons and the excitation light at the metal/dielectric interface. The dispersion relation of surface plasmon mainly depends on the dielectric functions of metal and dielectric materials, thus the change of the dielectric constant of the dielectric material can change the resonant coupling of surface plasmon in different wavelength ranges. The coupled surface plasmon is basically an evanescent wave, so it does not propagate into the far field. Finally, spectral analysis of the reflected light reveals the wavelength range which the surface plasmon is coupled resonantly. The most widely-used geometry of the SPR is known as Kretschmann geometry. In the white light illumination type, the position of the dip tells the changes of the dielectric constant; and in the case of laser illumination, the reflected laser intensity or the resonant angle is changed by dielectric constant of the analyte. Both measurements provide with the dielectric constant changes of the targeted analyte. For example, the Kretschmann geometry is composed of a metal-coated prism as shown in Figure 1.



Key

1 light source
2 molecular layer
3 glass prism
4 photodiode
5 metal film
a Incidence angle.

NOTE Kretschmann gemeotry with total internal reflection in the glass prism. The evanescent field on the metal film interacts with the molecular layer via surface plasmon coupling.

Figure 1 — Schematic diagram of Kretschmann geometry

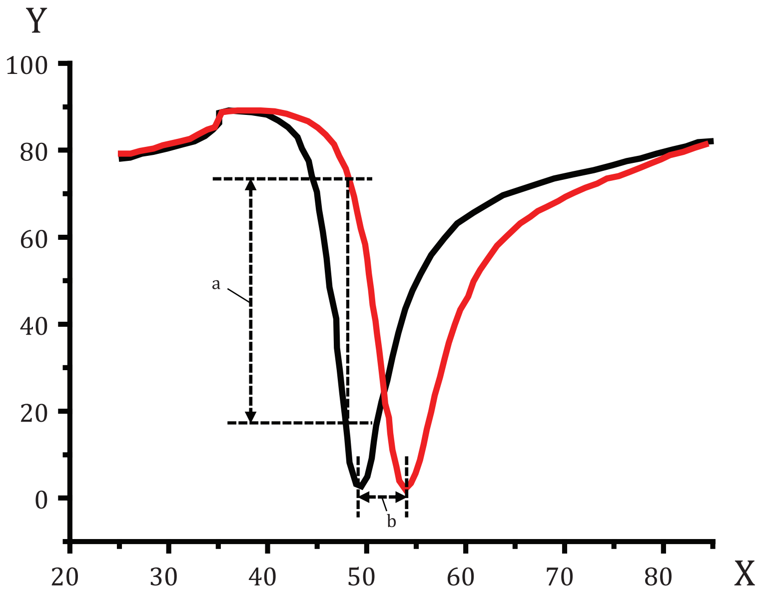
The excitation light is incident on the side surface of the prism and totally reflected at the interface between glass/metal film/dielectric materials. As mentioned above, the change of dispersion relation of the metal/dielectric interface due to the change of the analyte material is recorded with a CCD or a photodetector. The changes of the resonant wavelength or laser reflection intensity with incident angle are converted to the response unit (RU) in real time known as the sensorgram. The analysis of the sensorgram gives the absolute amount of the analyte in solution.

In this document, to provide the standardized method to measure the minimum detectability of the SPR, a protocol is provided to prepare the test specimen, acquire the data, analyse the data and extract the value of minimum detectability[2].

Subclause 4.2 specifies the basic principle of SPR measurement with the white light excitation. This document mainly applies to the Kretschmann geometry type of white light illumination[2]; however it is also applicable to the laser illumination type in the viewpoint of measuring the RU changes.

4.2 White light excitation type

The light source of the excitation covers the resonance wavelength range of the SPR. The generated SPR mode results in the dip of the response in reflected light as shown in Figure 2.



Key

X angle, θ_{spr}, in degrees

Y reflectance, R, in per cent

a dR, in per cent.

b dθ_{spr}, in degrees.

NOTE The dip of the angle changes depending on the amount of the analyte in the solution.

Figure 2 — Response of the reflected light after the surface plasmon coupling

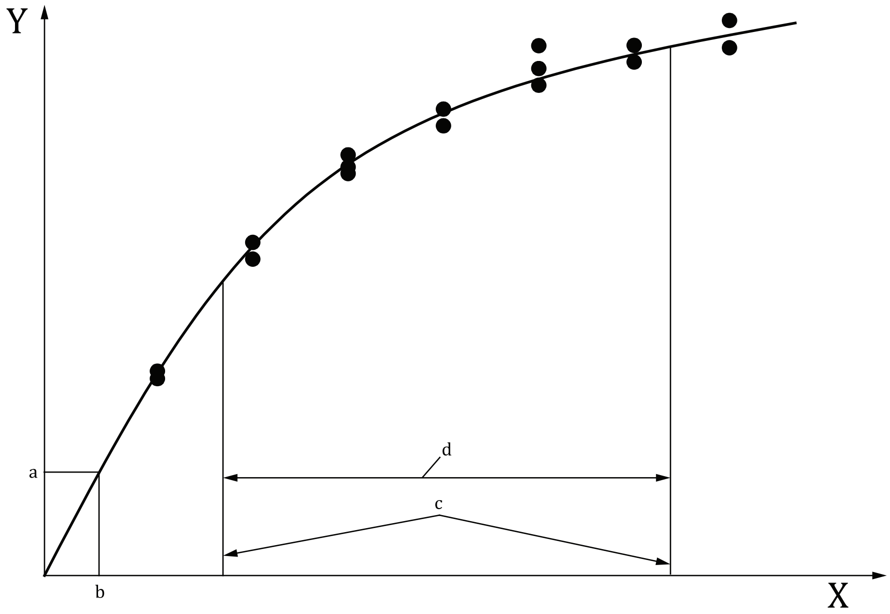
In the reference solution, the position of the dip is predetermined by the dispersion relation of the metal/dielectric interface. Thus the change of the analyte in solution is recorded in terms of the change of the spectral dip of the reflected light according to the coupling angle. The measurement speed is reasonably fast when the analysis of the reflected light is done with a CCD or a photodetector. So it is suitable for the real time measurements of the chemical reactions at the metal/dielectric interface.

4.3 Laser illumination type

The laser illuminates the interface and measures the change of the resonant angle by scanning the angle of the incident laser. It can provide high resolution of the SPR. In the viewpoint of the minimum detectability of SPR measurement, the laser illumination type can be used to find the change of the RU of analytes.

5 Outline of proposed method

The proposed measurement of small response of the signal is based on the 3-sigma rule. The 3-sigma rule is defined as a statistical calculation that refers to data within three standard deviations from a mean. Three-sigma limits (3-sigma limits) are used to set the lower control limits in statistical quality control. Statistically the meaning of the 3-sigma rule is to find the S/N ratio as 1. In this document, the various concentrations of the standard solutions are analysed by the SPR device to determine the lower limit of the detectability. To avoid the complexity in biomaterials, the absolute determination of the device quality by using simply a single neutral analyte (ethylene glycol in DI water, sucrose in DI water, etc.) on the bare gold film is proposed. The standard analysis of the 3-sigma rule is depicted in Figure 3.



Key

X concentration

Y response unit

a 3σ.

b Limit of detection.

c Limit of quantitation.

d Acceptable precision and accuracy.

NOTE The series measurement of solution's concentrations has their standard deviation (SD); and the crossing point of the SD value with expected extended signal determines the lower limit of detection as lowest detectability. The response is directly proportional to the concentration of biomolecules on the surface. For instant, 1 000 RU corresponds approximately to a surface concentration of 1 ng/mm^2 for an average protein ligand.

Figure 3 — Data acquisition of 3-sigma method

6 Instrument of operation conditions

6.1 General

The minimum detectability is determined by many affecting factors which include ambient condition such as temperature, air pressure and device parameters such as the flow rate, the measurement time.

6.2 Alignment of optics including incident light

Due to the self-contained nature of the internal optical path in the SPR, most SPRs require only the minimum degree of optical alignment by users. This factor depends on the superiority of the SPR system made by the manufacturers.

6.3 Sensor chip

This is one of the most important parameters of the measurement. In many cases, SPR manufacturers provide the functionalized sensor chip for varieties of the analytes. To determine the absolute minimum detectability, the bare metal sensor chip provided by the manufacturer shall be used. Commonly, the bare metal sensor chip is the bare gold coated chip and its thickness is around 100 nm.