

## EXPERIMENTAL AND MODEL BASED EVALUATION OF SODIUM PERBORATE PRECIPITATION KINETICS

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### Abstract

The influence of different process conditions on the kinetics of sodium perborate precipitation was studied using a dynamic model of a batch precipitation process. The subprocesses nucleation, growth, and agglomeration were included in the model. Specifically, the effects of temperature and addition of ionic surfactant were investigated. The solute concentration and particle size distribution were followed during a batch experiment. Using this information, model parameters were identified via non-linear parametric optimization. It was observed that temperature has a large influence on the course of supersaturation and crystal size distribution. At lower temperature smaller and more compact agglomerates are formed.

## THE INVESTIGATION OF ATMOSPHERIC CONSTITUENTS RELATED TO OZONE AT TWO RURAL SITES

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### SUMMARY

Measurements of several atmospheric constituents ( $O_3$ ,  $NO_x$ ,  $NO_2$ , NMVOC,  $HNO_3$ ,  $SO_2$ ,  $SO_4^{2-}$  and  $NO_3^-$ ) and meteorological parameters were performed at two rural sites in Slovenia; at the site Brnik (380 m a.s.l.) and at the elevated site Krvavec (1720 m a.s.l.) during a campaign from September 29 to October 1, 1996. The results showed distinct diurnal variations and different concentration levels of the measured compounds at each site. Local meteorological circumstances as well as weather changes on the synoptic scale influenced strongly the distribution of the air pollutants. The concentrations of ozone precursors ( $NO_x$  and VOC) were present in sufficient amount to permit a substantial photochemical ozone production during favourable meteorological conditions. It was shown that areas, which are at a specific distance from an urban area, like the city of Ljubljana, can be exposed to higher ozone concentrations than the polluted area itself.

# A TOOL FOR THE DETERMINATION AND CLASSIFICATION OF THE MAIN AND INTERACTIVE EFFECTS USING EXPERIMENTAL DESIGNS

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## ABSTRACT

The use of experimental designs to obtain information about the factors and their interactions that affect the experimental system under study are described. For the classification of factors and their interactions according to their main and interactive effects a new parameter  $R_i$  is proposed. The computer program called EFFECTS has been made to simplify the choice of experimental design, the construction of an effect's matrix, and the calculation and classification of main and interactive effects. The usefulness of the program is shown by two examples: the use of a complete and Plackett-Burman factorial design.

## INFRARED INVESTIGATION OF $[\text{Cu}(\text{sac})_2(\text{H}_2\text{O})_4] \cdot 2\text{H}_2\text{O}$ - A PSEUDO JAHN-TELLER COMPLEX

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### ABSTRACT

The infrared properties of the dihydrate of tetraaquabis(saccharinato-*N*) copper(II), a compound characterized by a typical inherent instability of the pseudo Jahn-Teller type, were studied. The FTIR spectra of the protiated compound and of its deuterated analogues with varying deuterium content were recorded at room and liquid nitrogen temperature. The OH and OD, as well as the C=O and SO<sub>2</sub> stretching regions were compared to the corresponding regions in the IR spectra of the other members of the isomorphous series. The hydrogen bonding properties in this compound were discussed. The applicability of the previously derived correlation equation of the  $\tilde{\nu} = f(R)$  type, where  $\tilde{\nu}$  is the wavenumber of the  $\nu(\text{OD})$  vibrations from the isotopically isolated HDO molecules and  $R$  is the corresponding crystallographically determined O<sub>w</sub>...O distance was tested for this compound. New regression analyses were performed, including the present data.

**Keywords:** Jahn-Teller instability; metal(II) saccharinates hexahydrates; tetraaquabis(saccharinato-*N*) copper(II); FTIR spectra, isomorphism.

## INTERMETHOD COMPARISON FOR QUALITY CONTROL AT THE ENVIRONMENTAL SPECIMEN BANK (ESB) OF THE FEDERAL REPUBLIC OF GERMANY

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### Abstract

Stringent quality control and harmonization of sampling protocols are fundamental requirements of long-term environmental monitoring as it is carried out at the Environmental Specimen Bank (ESB) of the Federal Republic of Germany for more than 15 years. Reliable data for legislation and recommendation need to be of the highest quality both from the analytical as well as from the acquisition point of view. Quality assurance measures, hence, always should start at the sampling site when errors due to the wrong specimen, contamination or loss, wrong containers or bio-degradation can influence the final result fundamentally. At the ESB specially designed sampling protocols have been developed to minimize such unwanted influences. To ensure a maximum of sample integrity the materials are deep frozen at the site of sampling and processed at liquid nitrogen temperatures so that the finally analyzed aliquot in the laboratory is well representing the particular sector of the environment which was intended to be studied.

The samples are taken in large quantities (2.5 kg), stored in the vapor phase of liquid nitrogen ( $< -150$  °C) at the place of sampling, transported to the lab cryogenically, ground, homogenized and aliquotted (5-10 g fresh of each subsample) cryogenically and stored deep cool until use. Analytical quality control comprises the use of in-house (matrix identical) reference materials, analysis of CRM's, intermethod comparison and participation in inter-laboratory comparison exercises as well as participation in certification campaigns for candidate reference materials from national and international organizations. A number of environmental and toxicological important polycyclic aromatic hydrocarbons (PAH's), chlorinated hydrocarbons (CHC's) and several elements and species such as As, Ba, Ca, Cd, Co, Cu, Mch-Hg and total Hg, K, Mg, Mn, Ni, P, Pb, S, Se, Tl and Zn are determined routinely in the fresh, frozen, annual samples. Together with the biometric data and the sampling parameters a huge data set is being created which is the basis for chemometric and statistical evaluation such as trend analysis or significant differences between locations. The freshly frozen samples are ideal starting materials for speciation analysis of delicate organo-metallic compounds.

ARSENIC SPECIATION USING HIGH PERFORMANCE LIQUID  
CHROMATOGRAPHY SEPARATION AND ATOMIC FLUORESCENCE  
SPECTROMETRY DETECTION - APPLICATION TO WINE AND URINE SAMPLES

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**ABSTRACT:** A new chemical interface between a high performance liquid chromatograph (HPLC) and an atomic fluorescence spectrometer (AFS) utilising UV-persulphate decomposition and hydride generation (HG) was used for environmental arsenic speciation. On-line separation and decomposition of eight known arsenic compounds was investigated. The method developed was applied for arsenic speciation in several Slovene wines and in an urine sample from a person after seafood consumption.

**INVESTIGATION OF SUBSTITUTED R-PHENOL-QUINUCLIDINE  
HYDROGEN BONDED COMPLEXES WITH FT-IR SPECTROSCOPY IN THE  
MIR, FIR AS WELL AS <sup>1</sup>H NMR MEASUREMENTS AND QUANTUM  
CHEMICAL SIMULATIONS**

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**Abstract**

R-phenol - quinuclidine complexes were studied as a function of the  $pK_a$  of the phenols by MIR, FIR and <sup>1</sup>H NMR spectroscopy. The family of these complexes is very interesting due to the high  $pK_a$  of its base quinuclidine ( $pK_a = 11$ ). In the most cases the hydrogen bonds are middle strong. The most symmetrical systems (about 50% proton transfer) may have a pronounced barrier in their double minimum proton potential. The MIR spectra of these in the average almost symmetrical complexes show IR continua which extend over the whole region from 2700  $cm^{-1}$  to 300  $cm^{-1}$ . At least two bands with hydrogen bond vibrational character are observed in the FIR spectra (300  $cm^{-1}$  to 100  $cm^{-1}$ ). No correlation between the strength of these hydrogen bond vibrations and the acidity of the R-phenols was found. The reasons of this result are vibrational coupling and a strong influence of the environment. Nevertheless, SCF calculations show that the harmonic force constant of the pure FIR hydrogen bond vibration (R-phenol-quinuclidine) is a first order function of the  $pK_a$  of the R-phenols.

## KINETIC MEASUREMENTS OF LIGNIN PEROXIDASE ACTIVITY

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### Abstract

During cultivation of the white rot fungus *Phanerochaete chrysosporium* very high lignin peroxidase activities in extracellular fluid were observed. By applying the original method for determination of LiP activity too low values were measured. Therefore, a novel method based on derivatives was introduced. Substrate concentrations had to be increased (veratryl alcohol to 2 mmol/L, H<sub>2</sub>O<sub>2</sub> to 1 mmol/L) and the maximal value of the first derivative was found to be a proper measure of enzymatic activity.