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Application of wet effluent diffusion denuder for measurement of uptake coefficient of gaseous pollutants

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ABSTRACT

The comparison of theoretical approaches describing the collection of analyte in the cylindrical wet effluent diffusion denuder (CWEDD) with experimental data is presented. Various absorption liquids were tested for the collection of formaldehyde (distilled-deionized water, H_2SO_4 solution), acetaldehyde (distilled-deionized water) and nitrous acid (distilled-deionized water, sodium carbonate and sodium bicarbonate solutions of various concentrations and sodium phosphate pH 6–8) in CWEDD. pH of absorption liquids significantly influences the collection of formaldehyde as well as nitrous acid. The collection efficiency of formaldehyde for 0.05 M H_2SO_4 as absorption liquid was generally higher than for distilled-deionized water. Absorption liquid pH markedly affected the collection efficiency of HONO too (with increasing pH the collection efficiency increase). Data derived by Gormley–Kennedy equation for all investigated compounds were overestimated especially for higher flow rates of air, data calculated with respect to Henry constant are not in good agreement with experimental data and are considerably depended on a determination of the Henry constant value. The CWEDD can be alternative tool for the determination of uptake coefficient. Obtained uptake coefficients were in good agreement with data found in other literature.

1. Introduction

Diffusion denuders are often-used devices for a preconcentration of various analytes from air. Denuder tubes with the layer of a solid sorbent preconcentrate an analyte from a gas stream passing through the denuder via a selective collection on a specially prepared surface. When a sample stream is passed through the tube, the analyte accumulates on the wall with a rate governed, in part, by its diffusion coefficient. After exposure, the adsorbed species or a derivate is removed by a change of wall temperature or by solvent action and then analysed. Moreover, diffusion denuders are widely used when it is necessary to discriminate between gases and aerosols. The mechanism of separation in the denuder sampling depends on the large difference in diffusion coefficients between gases and particles. Gases with high value of the diffusion coefficient diffuse to the coated surfaces of denuder tubes while heavier particles follow the laminar flow and pass through the denuder tube without the collection [1–3].

Continuously wetted diffusion denuders are especially attractive because the collection surface is constantly renewed and their use also facilitates the development of automated analyzers. Gaseous analyte is collected into a thin film of absorption liquid flowing continuously down on the inner wall of the denuder tube and the denuder concentrate can be subsequently on-line analysed by suitable analytical method [1,4].

The most of mathematical solutions describing a mass transfer in cylindrical diffusion denuders, for analytical purposes expressed as a collection efficiency (CE), suppose that a limiting step of the mass transfer is a diffusion of analyte molecules to the denuder tube wall in air. The collection efficiency, a ratio of analyte amount collected on the denuder wall and a total analyte amount entering into the diffusion denuder, can be calculated according to Gormley–Kennedy (GK) equation. The GK solution has been derived in accordance with Fick's diffusion laws for several basic assumptions. They are that (1) the analyte is a trace gas so that reactions do not affect the bulk flow, (2) laminar flow is fully developed and the viscosity is constant, (3) the temperature is constant, both radially and axially, (4) the axial velocity is small compared to the bulk transport velocity, (5) for denuder tubes, the amount collected on the tube wall is small compared to the tube capacity and the denuder wall represents a "perfect" sorptive surface, and (6) homogeneous chemistry is not creating or destroying the analyte in the tube [5,6].

The first of these assumptions is fully satisfied for typical application, such as ambient monitoring of pollutants at the concentration level of part per million by volume and below. For

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establishing of laminar flow a subduction zone that is a tube of the same diameter as the denuder tube but the wall of subduction tube is not coated by any sorbent (in the case of wet diffusion denuders, the subduction tube wall is not wetted). The concept of "perfect" sorptive surface supposes that each collision of analyte molecule with the denuder wall leads to irreversible sorption on the denuder wall. If all assumptions are satisfied the collection efficiency in the cylindrical diffusion denuder can be calculated by Gormley–Kennedy (GK) Equation [5]:

$$1-\frac{c}{c_0}=0.819\,\exp(-14.6272\Delta)+0.0976\,\exp(-89.22\Delta)+0.01896\,\exp(-212\Delta)+\ldots \quad (1)$$

$$\Delta=\frac{\pi DL}{4F} \quad (2)$$

where c is the analyte concentration after passing through the denuder, c_0 is the concentration of analyte entering into the denuder, D is the analyte diffusion coefficient, L is the length of denuder tube, and F is the volumetric flow rate. If the value of Δ is lower than 0.025 then only first term is meaningful and other terms can be neglected.

In real applications of diffusion denuders the assumption of "perfect" sorptive surface is not often fully satisfied. A more realistic boundary condition results from the assumption that the analyte is adsorbed with some characteristic probability on each wall collision. Therefore a solution (Cooney–Kim–Davis, CKD) supposing that only constant fraction γ_{CKD} of the wall collisions results in the sorption has been derived. Simultaneously, the effect of temperature and atmospheric pressure is taken into consideration [7,8]. The derived CKD equation is analogical to GK equation:

$$\frac{c}{c_0}=Q_1\exp(-A_1^2z*)+Q_2\exp(-A_2^2z*)+\ldots \quad (3)$$

where

$$z*=\frac{\pi D_0L}{2F}\left(\frac{T}{T_0}\right)^{\alpha-1}$$

D_0 is diffusion coefficient at standard conditions, T is temperature, T_0 is temperature at standard condition (273 K) and α is an exponent equal usually 2. Coefficients Q_n and A_n are a function of Sherwood number N_{Shw} which is a dimensionless parameter dependent on the fraction γ_{CKD}:

$$N_{\mathrm{Shw}}\approx\left(\frac{T}{m}\right)^{1/2}\left(\frac{T_0}{T}\right)^{\alpha}\frac{Rp}{D_0}\frac{\gamma_{\mathrm{CKD}}}{1-(\gamma_{\mathrm{CKD}}/2)} \quad (4)$$

where m is the analyte mass in atomic mass unit (amu), R is the gas constant.

In the case of the wet diffusion denuder the transfer of gases to the aqueous phase of the effluent solutions includes the diffusion to the walls, the accommodation to the surface, the solvation into solution followed by the dissociation and/or acid/base reactions. Both GK and CKD solutions deal only with a describing of mass transport to the denuder wall in gaseous phase, however, factors influencing an analyte collection from air into the film of absorption liquid are not taken in account. The gas solubility in liquids depends on the partial pressure above solution as well as on the temperature and chemical character of both components, i.e., dissolving gas and the solvent [9]. The relationship between partial pressure (p_i) of little soluble gas above solution and molar ratio (x_i) in liquid phase is linear and is described by the Henry's law $p_i = H_i \times x_i$, where H_i is Henry constant that is a good measure of the solubility of gas in liquid. The collection efficiency of low soluble analyte depends on the Henry's law constant, and the CE can be calculated as:

$$\mathrm{CE}=1-\frac{c}{c_0}=\frac{c_af_a}{c_gf_g+c_af_a} \quad (5)$$

where c_a and c_g are the concentrations in the aqueous and gaseous phases and f_a and f_g are the flow rates in the aqueous and gaseous phases, respectively. Substitution with the dimensionless Henry constant $\mathrm{H}^1 = c_a/c_g$ leads to the collection efficiency given by:

$$\mathrm{CE}=1-\frac{c}{c_0}=\frac{f_a}{f_g/\mathrm{H}^1+f_a} \quad (6)$$

In cases when acid/base reactions are involved, the effective Henry constant $\mathrm{H_{ef}}$ has to be taken into account, defined as

$$\mathrm{H_{ef}}=\mathrm{H}^1\left(1+\frac{K_a}{[\mathrm{H}^+]}\right) \quad (7)$$

where K_a is the dissociation constant of analyte [10].

Condensational growth, evaporation and heterogeneous chemistry are important phenomena in aerosol physics and technology, in atmospheric chemistry and in cloud microphysics and cloud chemistry. The mass transfer from gaseous phase into a condensed phase can be expressed by an uptake coefficient γ. The uptake coefficient is a measure of the ratio of the rate at which the molecules cross the interface region between the condensed phase and the gas phase including both physical and chemical processes in the gas and condensed phases as well as the interface. γ is defined in a similar way to γ_{CKD} as a ratio of number of gas molecules removed by the condensed phase in unit time and number of gas molecules striking the interface in unit time.

In this paper we compared three above mentioned theoretical approaches describing the analyte collection in the diffusion denuder with experimental data during the collection of formaldehyde, acetaldehyde and nitrous acid in the cylindrical wet effluent diffusion denuder into water. Selected compounds play an important role in tropospherical photochemistry and are key compounds in the understanding of chemical processes in atmosphere. The cylindrical wet effluent diffusion denuder frequently used as a suitable device for the continuous preconcentration of analytes from air into water can be employed as an effective tool for a measurement of uptake coefficients of gaseous pollutants into water.

2. Materials and methods

Analytical methods applied for the determination of formaldehyde, acetaldehyde and nitrous acid in air have been described elsewhere [11–13]. Therefore only a brief description is presented here (Fig. 1). The cylindrical wet effluent diffusion denuder (CWEDD) and the flow-injection analysis (FIA) system were two main parts of the measuring apparatus. The cylindrical wet effluent diffusion denuder was used for the collection of formaldehyde, acetaldehyde and nitrous acid from air into water. The CWEDD operating at a vertical position consisted of a specially treated glass denuder tube (50 cm length × 1.1 cm i.d.) and untreated inlet and outlet glass tubes, assembled together with two polycarbonate fittings. The tubes were sealed by Viton O-rings to avoid leakage. The inlet tube (length 11 cm, i.d. 1.1 cm) adjusted the laminar flow of sampled air through the denuder. The absorption liquid was pumped (400 μL min^{-1}) by a peristaltic pump into the denuder tube through a porous PTFE O-ring located between the outlet tube and the wetted part of the CWEDD. The liquid spread over the inner wall of the CWEDD and flows continuously down under the influence of gravity. Analysed air (flow rate of 1 L min^{-1}) was sucked counter-currently through the denuder by means of a membrane pump. The denuder concentrate, i.e., the absorption liquid with a collected pollutant was aspirated at the bottom of the denuder tube

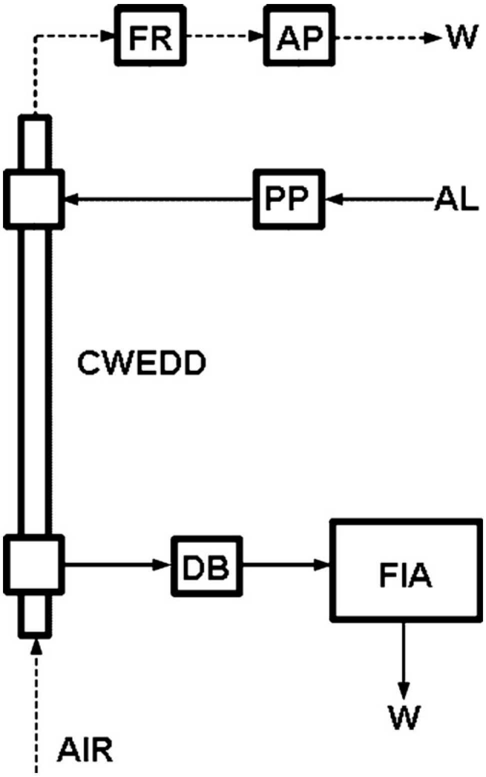

Fig. 1. The scheme of measuring apparatus. AL, absorption liquid; AP, air pump; CWEDD, cylindrical wet effluent diffusion denuder; DB, glass–Teflon debubbler; FIA, flow-injection analysis detection system; FR, flow regulator; PP, peristaltic pump; W, waste.

through the other porous PTFE O-ring using the peristaltic pump at the flow rate of 600 μL min^{-1} into a glass-PTFE debubbler with an inner volume of 200 μL.

Formaldehyde in the denuder concentrate was on-line detected employing a flow-injection analysis (FIA) method based on a chemiluminescence reaction of formaldehyde and gallic acid with hydrogen peroxide in an alkaline solution (modified Trautz–Schorigin reaction) [12] or, alternatively, a fluorescence 2,4-pentandione method [11]. Nitrous acid content in the denuder concentrate was determined by the FIA method based on oxidation of HONO into peroxynitrous acid that was detected by the chemiluminescent reaction with an alkaline solution of luminol [13].

For analytical parameters of applied techniques see Refs. [11–13].

3. Results and discussion

Formaldehyde, acetaldehyde and nitrous acid were chosen for the measurement of uptake coefficients using the CWEDD because of their important role in the chemistry of the troposphere. Formaldehyde and acetaldehyde are significant components of photochemical smog [13–15] and nitrous acid is known as a source of hydroxyl radicals after its photolysis at sunrise [16,17]. Therefore it is very important to keep at disposition reliable methods for the monitoring of these compounds. Moreover, the collection of formaldehyde, acetaldehyde and nitrous acid in the CWEDD is very advantageous technique for their preconcentration. Uptake coefficients for studied compounds were calculated from data obtained during the measurement of CE of formaldehyde, acetaldehyde and nitrous acid in various absorption liquids in the CWEDD.

3.1. Collection of formaldehyde and acetaldehyde

The collection of formaldehyde in various absorption liquids was studied in dependence on the flow rate of air passing through the CWEDD. During dissolution of formaldehyde in water a hydration reaction proceeds to form methyleneglycol. Because the formation of methyleneglycol is catalysed by H$^+$ [18], diluted solution of sulphuric acid besides distilled-deionized water were tested as absorption liquid.

The collection of formaldehyde and acetaldehyde in the CWEDD has been discussed in more detail elsewhere [11,12]. The measured dependences of HCHO collection efficiency in the CWEDD on the air flow rate for distilled-deionized water, 0.05 M sulfuric acid as well as theoretical data calculated according to all three theoretical approaches are shown in Fig. 2. At the air flow rate of 0.5 L min^{-1}, the HCHO collection efficiency for both studied absorption liquids was quantitative. In accordance with the theory, with increasing air flow rate the HCHO collection efficiency decreased for both absorption liquids. The HCHO collection efficiency for sulfuric acid solution was generally higher than that for distilled-deionized water, which confirms a shift in the equilibrium of hydration reaction toward the formation of methyleneglycol in acid solution. The collection efficiency of acetaldehyde was much lower than that for formaldehyde. At the air flow rate of 0.5 L min^{-1}, the acetaldehyde collection efficiency was 70%.

The difference between experimental data and theoretical data calculated according to Gormley–Kennedy equation results from an invalidity of an assumption of “perfect” sorption (it means that each analyte molecule collision with the denuder wall leads to the sorption). The experimental data for formaldehyde and acetaldehyde (Fig. 2) were then smoothed by theoretical curves calculated according to Cooney–Kim–Davis Eq. (3) for γ_{CKD} of 0.011 (HCHO, distilled-deionized water), 0.018 (HCHO, 0.05 M H_2SO_4) and 0.0018 (CH_3CHO, distilled-deionized water), respectively. Parameter γ_{CKD} expresses a fraction of analyte molecule collisions with the denuder wall or with the absorption liquid film in the CWEDD case which lead to an absorption of analyte. The values show that only 1.1% and 1.8% of formaldehyde molecule collisions with the film of distilled-deionized water and 0.05 M H_2SO_4 result in the collection of formaldehyde in the absorption liquid. As mentioned above,

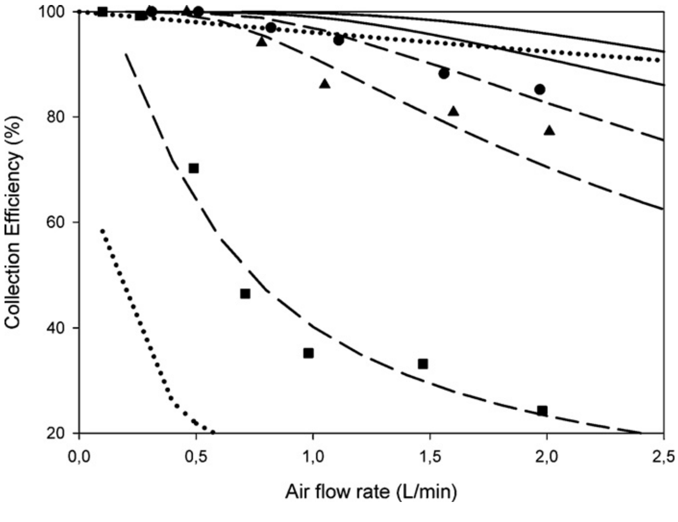

Fig. 2. The dependence of collection efficiency of formaldehyde and acetaldehyde on the air flow rate in the CWEDD. (▲) HCHO vs. distilled-deionized water; (●) HCHO vs. 0.05 M H_2SO_4 and (■) acetaldehyde vs. distilled-deionized water; (dashed curve) theoretical curve calculated according to CKD; (solid) theoretical curve calculated according to GK; (dotted) theoretical curve calculated with respect to Henry constant; 100 μg/m^3 HCHO, absorption liquid flow rate of 303 μL min^{-1}.

theoretical data calculated according to the Gormley–Kennedy equation data were markedly overestimated because of the invalidity of an assumption of “perfect” sorption for both HCHO and acetaldehyde, which results in strong difference from the experimental data.

Values of the collection efficiencies calculated according to Gormley–Kennedy equation for acetaldehyde at higher air flow rates were lower than those calculated for formaldehyde. This fact is in an agreement with experimental data. Especially distinct diffusion coefficients in gaseous phase for formaldehyde (0.1728 cm^2/s) and acetaldehyde (0.1284 cm^2/s) applied in Gormley–Kennedy equation caused difference between theoretical dependences.

Values of uptake coefficient move up to 0.02 and 0.03 for formaldehyde and acetaldehyde, respectively [19]. Coefficients γ_{CKD} for formaldehyde found out by smoothing of experimental data (0.011 for distilled-deionized water and 0.018 for 0.05 M H_2SO_4) correspond to values of uptake coefficients found at the literature. Increasing of γ_{CKD} at low pH confirms acid catalysis of formation gem-diol form of formaldehyde and leads to higher solubility of formaldehyde in acid solution. γ_{CKD} for acetaldehyde was much lower ($\gamma_{CKD} = 0.0018$) than surprisingly high uptake coefficient for acetaldehyde.

Dependences of collection efficiency on air flow rate calculated in consideration of Henry constant according to Eqs. (7) and (8) are depicted in Fig. 2 as well. For formaldehyde the theoretical data of collection efficiency agree with experimental values at low air flow rates but calculated data of collection efficiency were overestimated for air flow rates higher than 1 L min^{-1}. Whereas in the acetaldehyde case theoretical curve of collection efficiency dependence on air flow rate lies below experimental data. The main reason of this fact is a very low value of Henry constant for acetaldehyde ($H = 11.4$ mol/atm [20]) in comparison with Henry constant for formaldehyde ($H = 2970$ mol/atm [19]). The high value of Henry constant for formaldehyde is related to high reaction rate ($K_{hyd} = 2300$) of the hydratation reaction leading to formation of gem-diol form of formaldehyde. While in the acetaldehyde case the reaction rate of hydration is low ($K_{hyd} = 11.4$) [20].

3.2. Collection of nitrous acid

In contrast to highly water-soluble gases for which the flow rate of air passing through the denuder is the main factor affecting the denuder collection efficiency, in nitrous acid case the collection efficiency depends on Henry constant as well. Henry constant covers other effects that can occur in mass transfer of nitrous acid from air into the absorption liquid (such as effect of air–water interface, solvatation, dissociation, acid–base reaction). Therefore the dependences of collection efficiency of nitrous acid on the air flow rate for various absorption liquids and pH were measured (distilled-deionized water, sodium carbonate and sodium phosphate solution of various concentrations and pH). The nitrous acid collection data were published elsewhere [13], therefore measured data will be discussed only briefly. The collection efficiency of nitrous acid in the CWEDD for all studied absorption liquids decreased with increasing air flow rate and at the flow rate of 1.0 L min^{-1} the CE was 100% (Fig. 3).

Theoretical Gormley–Kennedy data for HONO correspond to experimental collection efficiencies for alkaline absorption liquids, i.e. 1×10^{-2} M $NaHCO_3$ (pH 9.18), 1×10^{-4} M NaH_2PO_4 (pH 8.00) and 1×10^{-2} M Na_2CO_3 (pH 11.21). For these absorption liquids, we can assume the condition of “perfect” sorption was performed. For other studied absorption liquids, at higher air flow rates experimental values of collection efficiency were lower than corresponding theoretical values.

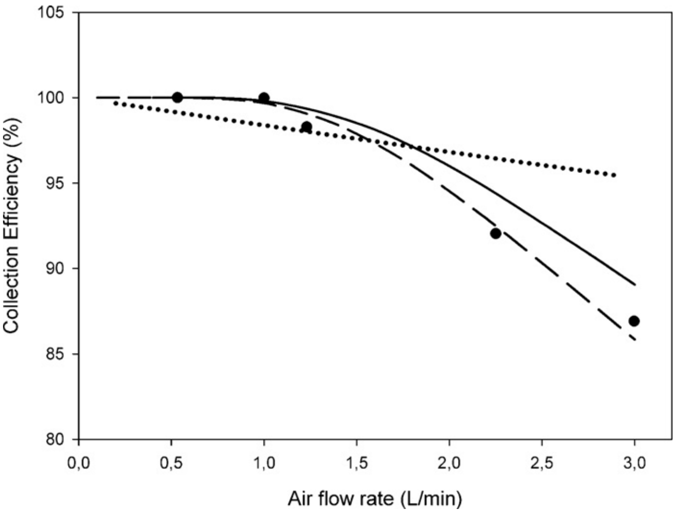

Fig. 3. The dependence of the collection efficiency of nitrous acid on the air flow rate for distilled-deionized water as the absorption liquid and theoretical dependences. (●) experimental data; (···) theoretical data according to equations (2.12–13) for pH 5,6; (—) theoretical data calculated according to GK; (– – –) theoretical data calculated according to CKD for $\gamma_{CKD} = 0.05$; absorption liquid flow rate of 400 μL min^{-1}; 11.21 μg/m^3 HONO.

From comparison of theoretical data of HONO collection efficiency and experimentally measured data for distilled-deionized water (Fig. 3) it is evident that Gormley–Kennedy data were overestimated as well as theoretical data calculated with respect to Henry constant for the air flow rate higher than 1.5 L min^{-1}. Whereas for lower air flow rates experimental collection efficiency of HONO were slightly higher. For calculation the effective Henry H_{ef} constant was applied (Eq. (8)) for pH 5.6 (lower pH value was probably caused by absorption of carbon dioxide from air). Experimental data were smoothed by curve derived according to Cooney–Kim–Davis equation for γ_{CKD} of 0.05. The value of γ_{CKD} accords with uptake coefficient for nitrous acid ($\gamma = 0.05$ [21]).

It is evident that for the air flow rates equal or smaller than 1.0 L min^{-1}, the collection efficiency of HONO in the CWEDD did not depend on the composition of the absorption solution and for all studied absorption liquids. At higher air flow rates the effect of absorption liquid composition was significant. In Fig. 4 theoretical as well as experimental dependences of nitrous acid collection efficiency on pH for air flow rate of 1.0, 2.5 and 3.5 L min^{-1} are

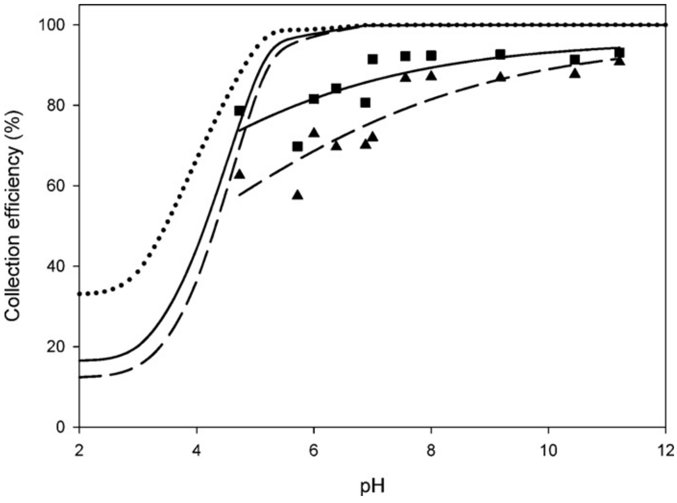

Fig. 4. The dependence of the collection efficiency of nitrous acid on pH of absorption liquid. (■) Air flow rate of 2.5 L min^{-1}; (▲) air flow rate of 3.5 L min^{-1}; (···) theoretical data calculated for the air flow rate of 1.0 L min^{-1}; (—) 2.5 L min^{-1}; (– – –) 3.5 L min^{-1}; absorption liquid flow rate 400 μL min^{-1}; 11.21 μg/m^3 HONO.

shown (Henry constant of 49 mol/atm [22] was applied in calculation). Even thought experimental values of collection efficiency were mostly lower than experimental data derived from Eqs. (7) and (8) in agreement with theory the collection efficiency increased with increasing pH (and increasing Na_2CO_3 and $NaHCO_3$ at the same time). At the flow rate of 1.0 L min^{-1}, the collection efficiency was 100% for all absorption liquids at various pH, which is in an agreement with theoretically calculated data (with exception of absorption liquid with lowest pH, i.e. 1×10^{-5} M $NaHCO_3$, pH 4.73).

4. Conclusion

The cylindrical wet effluent diffusion denuder is advantageous tool for continuous collection of various analytes in air. Our experiments prove that pH of absorption liquids significantly influences the collection of formaldehyde as well as nitrous acid.

In the study three theoretical approaches describing the analyte collection in CWEDD were compared with experimentally measured data for formaldehyde, acetaldehyde and nitrous acid as a model compounds with considerable importance in atmospherical chemistry.

From comparison of measured and calculated data these facts result: (1) data calculated by Gormley–Kennedy equation for both investigated compounds were overestimated for higher flow rates of air, (2) data calculated with respect to Henry constant were not in good agreement with experimental data and were considerably depended on a determination of the Henry constant value, (3) and finally, CWEDD can be alternative tool for determination of uptake coefficient. Obtained uptake coefficients are in good agreement with data found in other literature.

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