

## EXPERIMENTAL INVESTIGATION INTO THE EXTRACTION OF NICOTINIC ACID USING NATURAL NON-TOXIC AND CONVENTIONAL SOLVENTS

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Nicotinic acid and its derivatives are extensively used in the food, pharmaceutical and cosmetic industries. The technique for separating nicotinic acid from fermentation broth has a significant impact on overall production costs. The extraction of nicotinic acid was investigated using various kinds of natural non-toxic and conventional solvents such as sesame oil, rice bran oil, cyclohexane, 1-octanol and methyl isobutyl ketone (MIBK). The results were presented in terms of extraction efficiencies ( $E\%$ ) and distribution coefficients ( $K_D$ ). To determine these two parameters, experiments were conducted at  $298 \pm 1$  K. The maximum extraction efficiencies of nicotinic acid were found to be 19.550, 16.514, 13.719, 17.526 and 6.216% using MIBK, 1-octanol, cyclohexane, rice bran oil and sesame oil, respectively. The differences in the extraction efficiencies of nicotinic acid using these solvents were explained in terms of dipole moment, dielectric constant and refractive index. Further attempts were made to correlate the extraction efficiencies with the other physicochemical properties of the solvents such as viscosity, density, molecular weight, etc.

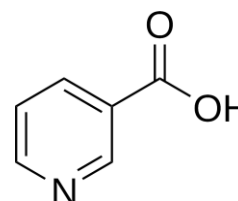
**Keywords:** nicotinic acid, extraction, extraction efficiency, natural solvents, conventional solvents

### 1. Introduction

Nicotinic acid (NA) - a carboxylic acid, namely niacin (3-pyridine carboxylic acid) ( $C_6H_5NO_2$ ) and referred to as vitamin B3 - is a solid with a white crystalline structure, translucent and contains a carboxyl side chain at position 3 as shown in *Figure 1* [1]-[3].

The market value of NA depends on several factors, including supply and demand, production costs and industry dynamics, as of the last update in January 2022. Among other sectors, the pharmaceutical and dietary supplement industries are the main users of nicotine, which may have an impact on the market value of this drug. The market for vitamin B3 was projected to reach US\$ 837.1 million in 2023. Between 2024 and 2030, this is projected to rise at a compound annual growth rate (CAGR) of 4.7%, reaching US\$ 1162.3 million [4]. The global demand for the following applications has a direct impact on the rise in the market value of niacin [4]. NA is found in avocados, peas, lentils, nuts and seeds as well as meals and meat, moreover, is an essential nutrient for humans that plants and animals produce. 34,000 tons of NA were produced worldwide in 2014, 63, 22 and 15% of which was intended to be used as an animal feed additive, food component for healthcare applications and

for industrial purposes such as in the pharmaceutical and cosmetic industries, respectively [5],[6]. NA is a potential analgesic and anti-inflammatory [7], moreover, has been widely used as a chelating agent in the food, pharmaceutical and beverage industries for over a century [8]. NA has anti-inflammatory properties used in various industrial applications, including biological systems, to stabilize metal ions in solutions used in industrial processes as well as to enhance the solubility and bioavailability of metal ions in agricultural applications [5]-[7]. Large commercial applications include in the pharmaceutical industry, e.g. chelation therapy and pharmaceutical formulation. It can also be used to produce skincare products, cosmetics and micronutrient fertilizers, which enhance plant growth and the productivity of personal care products and cosmetics.



*Figure 1:* Structure of nicotinic acid

Some of the applications of NA are presented in [Figure 2](#).

A deficiency of niacin causes pellagra, a nutritional disorder [3],[8]. Niacin is administered as a dietary supplement to prevent and treat pellagra. The primary method for avoiding niacin deficiency is through the consumption of food or nutritional supplements as the human body is unable to produce niacin [9]. Nicotinamide and nicotinic acid can be produced more effectively by converting 3-cyanopyridine enzymatically [8],[10]. 60-70% of the production costs in this fermentation-based process come from downstream purification costs, including product recovery, concentration, acidity and purification expenses [6],[11]. For over a century, organic acids have been produced via fermentation technology, leading to the production of aqueous solutions [9],[12]-[15]. Therefore, a cost-effective acid recovery process from the fermentation broth is required [3],[14]-[17]. Extracting carboxylic acids can be done in several ways from aqueous streams or fermentation broths, including dialysis, ion exchange, membrane technology, adsorption, precipitation, distillation and reactive extraction [1],[12],[18]. The technique of liquid-liquid extraction (LLE), frequently referred to as solvent extraction and partitioning, is the process of dividing substances or metal complexes according to how soluble they are in two distinct liquids that are immiscible, commonly polar water and an organic solvent [19].

This study used conventional and natural solvents, namely methyl isobutyl ketone (MIBK), 1-octanol, cyclohexane, sesame oil and rice bran oil, to extract NA from the aqueous phase. Conventional and natural solvents extracted NA from the aqueous phase while reducing toxicity and creating an environmentally-friendly method. Several extraction parameters defined this experiment, including extraction efficiency ( $E\%$ ) and distribution coefficient ( $K_D$ ) [20]. Separating NA from

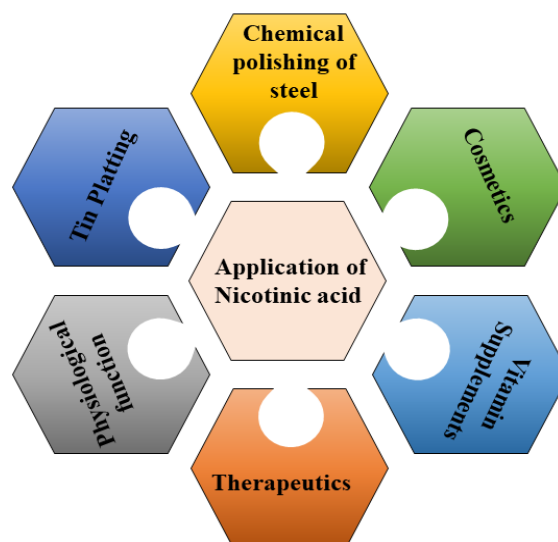


Figure 2: Applications of nicotinic acid

the broth is crucial and challenging because of the high cost of the process. Valuable carboxylic acid can be recovered affordably and sustainably using LLE [21]. Some authors have employed a LLE method using non-toxic solvents to separate valuable carboxylic acids [22].

## 2. Experimental

### 2.1. Samples and reagents

NA was purchased from S D Fine-Chem Limited, while the solvents rice bran oil, sesame oil, cyclohexane, 1-octanol and MIBK were procured from Patanjali Ayurved Ltd., India; Loba Chemie Pvt. Ltd., India and Merck Specialities Pvt. Ltd, India ([Table 1](#)). Sodium hydroxide was procured from Merck Specialities Pvt.

Table 1: Details of the chemicals used in the experiment

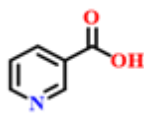

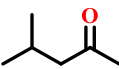

Chemical	MW (mol L <sup>-1</sup> )	Structure	Molecular formula	Supplier	Purity (%)
Nicotinic acid	123.10		C <sub>6</sub> H <sub>5</sub> NO <sub>2</sub>	S D Fine-Chem Limited, India	99.5
Rice bran oil	368.34	-	-	Patanjali Ayurved Ltd., India	99.0
Sesame oil	138.12	-	-	Patanjali Ayurved Ltd., India	99.0
Cyclohexane	84.16		C <sub>6</sub> H <sub>12</sub>	Loba Chemie Pvt. Ltd., India	99.5
MIBK	100.16		CH <sub>3</sub> COC <sub>4</sub> H <sub>9</sub>	Merck Specialities Pvt. Ltd., India	99.0
1-Octanol	130.23		CH <sub>3</sub> (CH <sub>2</sub> ) <sub>7</sub> OH	Merck Specialities Pvt. Ltd., India	98.0

Table 2: List of instruments in the present study

Instrument	Parameters	Model
Orbital Shaking Incubator	$T = 25\text{ }^{\circ}\text{C}$ rpm = 230	S-24BL, REMI, India
Benchtop Centrifuge	$T = 25\text{ }^{\circ}\text{C}$ rpm = 3930	REMI Centrifuge R-4C, India

Ltd., India for the titration, while oxalic acid was purchased from S D Fine-Chem Ltd., India. Oxalic acid was utilized to normalize the NaOH. All of the chemicals used in the study were not further purified nor pre-treated.

## 2.2. Experimental procedure

Aqueous solutions of varying NA concentrations ( $0.010\text{--}0.102\text{ mol L}^{-1}$ ) were prepared by dissolving different amounts of NA in double distilled water. A stock solution containing  $18\text{ g L}^{-1}$  NA was used for the experiments. Nicotinic acid in the aqueous phase and solvents in the organic phase were combined in a 1:1 volume ratio of 10 ml each in 100 ml conical flasks. The mixture was shaken at 230 rpm for 5 hours at a constant temperature of  $298 \pm 1\text{ K}$  in an orbital shaking incubator (Model: S-24BL, REMI, India). The samples were placed in centrifuge tubes and spun at 3930 rpm for 5 minutes to achieve the desired phase separation of the aqueous and organic phases (REMI Centrifuge R-4C, India) (Table 2). A 0.01 M NaOH solution was prepared before each experiment. A titration with  $0.01\text{ mol L}^{-1}$  NaOH was used to determine the amount of nicotinic acid in the aqueous phase. The experimental procedure of the present work is summarized in the block diagram in Figure 3.

## 3. Results and evaluation

An experimental investigation was conducted into the extraction of NA from the aqueous phase using different solvents, namely cyclohexane, 1-octanol, MIBK, sesame oil and rice bran oil. The methodology is described below.

Regarding mass balance, the concentrations in the aqueous phase were used to determine those in the organic phase as follows:

$$[HNA]_{org} = [HNA]_{in} - [HNA]_{aq} \quad (1).$$

$E\%$  and  $K_D$  were determined using experimental data for the separation of NA. The distribution coefficient was defined as the ratio between the organic phase and the aqueous phase under equilibrium conditions:

$$K_D = \frac{[HNA]_{org}}{[HNA]_{aq}} \quad (2),$$

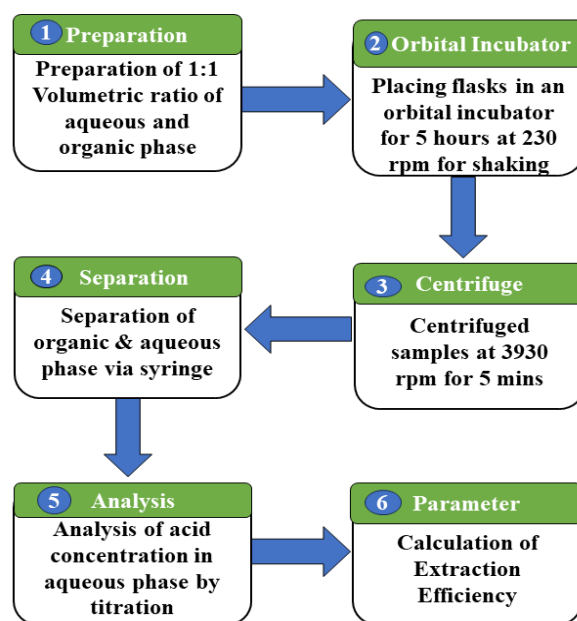


Figure 3: Flow chart of the experimental procedure using nicotinic acid

where  $[HNA]_{org}$  and  $[HNA]_{aq}$  stand for the concentration of nicotinic acid in  $\text{mol L}^{-1}$  in the organic and aqueous phases, respectively, at equilibrium.

The extraction efficiency is defined as follows in terms of the distribution coefficient:

$$E\% = \frac{K_D}{1+K_D} \cdot 100 \quad (3).$$

## Experimental uncertainty

To obtain accurate results, a few experiments were repeated twice as instrumental errors or any random fluctuations may cause the results to be uncertain. The calculations were performed using the mean values of the experimental parameters. An error of 1% was taken into consideration and repeated with an experimental error of less than 2%. It was determined that the average experimental uncertainty was close to  $x \pm 0.002$  using the following equation:

$$A(x) = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \quad (4),$$

where

$n$  represents the number of experimental observations,

$x_i$  represents an experimental observation,

$\bar{x}$  represents the mean of experimental values.

## 4. Discussion

The data obtained from the experiments performed to extract NA are presented in Table 3.

Table 3: Separation of nicotinic acid using natural and conventional solvents at  $298 \pm 1$  K

Solvents	$[HNA]_{in}$ (mol.L <sup>-1</sup> )	$[HNA]_{aq}$ (mol.L <sup>-1</sup> )	$[HNA]_{org}$ (mol.L <sup>-1</sup> )	$K_D$	$E\%$
Sesame oil	0.010	0.009	0.001	0.111	9.99
	0.025	0.023	0.002	0.086	7.91
	0.050	0.047	0.003	0.063	5.92
	0.075	0.071	0.004	0.056	5.30
	0.102	0.100	0.002	0.020	1.96
Rice bran oil	0.010	0.006	0.004	0.666	40.01
	0.025	0.018	0.007	0.388	28.01
	0.050	0.044	0.006	0.136	12.00
	0.075	0.070	0.005	0.071	6.63
	0.102	0.101	0.001	0.009	0.98
Cyclohexane	0.010	0.007	0.003	0.428	29.97
	0.025	0.021	0.004	0.190	16.00
	0.050	0.043	0.007	0.162	14.00
	0.075	0.070	0.005	0.071	6.67
	0.102	0.100	0.002	0.020	1.96
1-Octanol	0.010	0.006	0.003	0.639	39.01
	0.025	0.020	0.005	0.247	19.60
	0.050	0.041	0.009	0.219	18.00
	0.075	0.072	0.003	0.041	4.00
	0.102	0.100	0.002	0.020	1.96
MIBK	0.010	0.008	0.002	0.162	14.00
	0.025	0.021	0.004	0.190	15.96
	0.050	0.040	0.010	0.250	20.00
	0.075	0.059	0.016	0.271	21.32
	0.102	0.075	0.027	0.360	26.47

Table 4: List of pretreatment physicochemical properties of various solvents used for the extraction of nicotinic acid

Solvents	$MW$ (mol L <sup>-1</sup> )	$S_{water}$ (g L <sup>-1</sup> )	$\mu$ (cP)	$\rho$ (kg m <sup>-3</sup> )	$BP$ (K)	$MP$ (K)	$\epsilon$	$D_m$	$RI$	Ref.
Sesame oil	848.00	immiscible	52.50	914	483	267	3.00	-	1.474	[22],[23]
Rice bran oil	867.90	immiscible	58.70	916	540	253	2.14	-	1.467	[23],[24]
Cyclohexane	84.16	55.00 (25°C)	0.98 (20°C)	0.7781 (20°C)	354	280	2.02	-	1.426	[22],[23],[25]
MIBK	100.16	19.10	0.58	779	390	188	13.11	4.20	1.396	[22],[26],[27]
Octanol	130.23	0.46	7.36	824	468	257	10.30	1.68	1.429	[22]

The trend in the extraction efficiency values of nicotinic acid was as follows:

rice bran oil > 1-octanol > cyclohexane > MIBK > sesame oil.

This pattern can be rationalized by two factors that affect extractability, namely the level of NA hydration and the energy required by the molecules to bond with water. As a result, oxygenated solvents like MIBK extract more effectively than natural and other conventional solvents but are advantageously non-toxic unlike conventional solvents. Under equilibrium conditions, the extraction data show that as the concentration of NA in the aqueous phase rises, NA molecules remain relatively soluble in the organic phase.

The extraction results can be related to different physicochemical properties, e.g. dipole moment ( $D_m$ ), dielectric constant ( $\epsilon$ ), etc., listed in Table 4. Natural solvents have low relative permittivities ( $\epsilon$ ) and cannot donate a hydrogen bond. Since the relative viscosity ( $\mu$ ) and density ( $\rho$ ) of natural solvents are also high and immiscible in the aqueous phase, their  $E\%$  values are low and their  $K_D$  values are less than one. As the initial concentration of NA increases, their  $K_D$  and  $E\%$  decrease, accounting for the separation caused by the stronger NA association with water as naturally occurring solvents contain fatty acids. Conventional solvents such as cyclohexane and 1-octanol are non-polar. As the initial concentration of NA increases,  $K_D$  and  $E\%$  decrease. The polar protic solvent MIBK

contains a hydrogen atom bonded to the (C-O) electronegative group, which serves as a donor of hydrogen bonds. This behavior intensifies as the charge-to-volume ratio of the anion to be solvated rises. The highest  $K_D$  and  $E\%$  values are 0.162–0.360 and 14.00–26.47% using MIBK, respectively. Given that as the concentration of MIBK increases,  $K_D$  and  $E\%$  increase, MIBK can facilitate LLE more stable in the aqueous phase because of its strong polarity as well as high relative permittivity (Table 4) and extraction efficiency. As a result of its capacity to create hydrogen bonds, it is a more effective solvent for anions.

The experimental results yielded the highest values of the  $K_D$  of 0.666, 0.639, 0.428 and 0.111 using rice bran oil, 1-octanol, cyclohexane and sesame oil, respectively, at a concentration of 0.01 mol.L<sup>-1</sup>, while in MIBK, 0.360 was recorded at a concentration of 0.102 mol.L<sup>-1</sup> of NA (Figure 4).

The distribution coefficient was influenced by the initial concentration of NA. As the initial concentration of NA increased,  $K_D$  decreased in all the solvents, while in the case of MIBK,  $K_D$  increased as the initial concentration of NA increased. When MIBK was used with 0.102 mol L<sup>-1</sup> of NA, a higher  $K_D$  was observed. Compared to conventional and natural solvents like 1-octanol, cyclohexane, sesame oil and rice bran oil represented in Figure 5, MIBK exhibits a higher distribution coefficient.

Due to their high viscosity and density in the aqueous phase, conventional and natural solvents are immiscible and unable to form free hydrogen bonds, moreover, these solvents have low  $K_D$  values. Since the aqueous phase is unable to dissolve the organic phase, the organic and aqueous phases are separated by these solvents, resulting in oils with lower distribution coefficients. As the molecular weight and solubility influence the distribution coefficient, the distribution coefficient of acids rises as molecular weight increases.

When using all conventional and natural solvents (except for MIBK), the initial concentration of NA increased and the extraction efficiency decreased. For MIBK,  $E\%$  rose as the initial concentration of NA increased (Figure 6). From an experimental study, it was found that a maximum extraction efficiency of 26.47% was reached using MIBK, whereas the lowest was 1.96% using sesame oil. Although their extraction efficiencies decreased compared to other solvents, the lowest was 1.96% using sesame oil. Their extraction efficiencies decreased more than the other solvents because of their long fatty acids and non-polar nature. The absence of a dipole moment in natural solvents prevented them from forming complexes with the acid in the organic phase thanks to free electrons as well as from acting as hydrogen donors in solvents other than 1-octanol and MIBK. Since the polar solvent MIBK is positively charged, its use increased the extraction efficiency compared to when 1-octanol was applied. As a result, it dissolves more readily in the organic phase of nicotinic acid, thereby increasing the extraction efficiency. On the other hand, because of its large hydrophobic group,

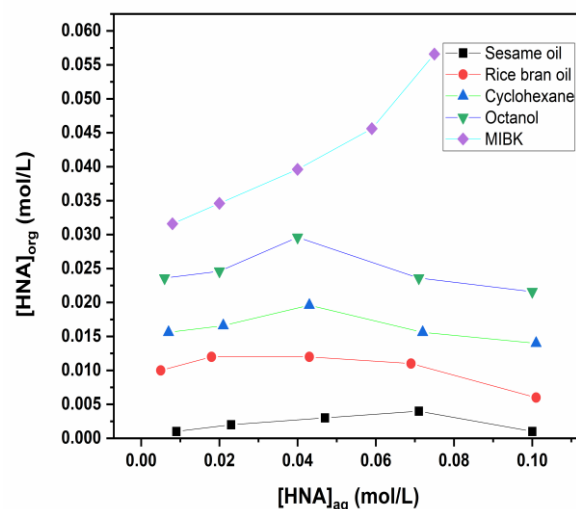


Figure 4: Equilibrium data for the extraction of nicotinic acid at  $298 \pm 1$  K with different solvents

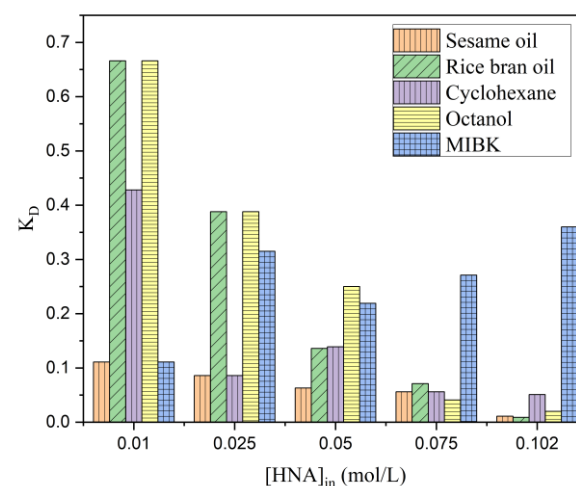


Figure 5: Variation in the distribution coefficient against the initial concentrations of nicotinic acid at  $298 \pm 1$  K

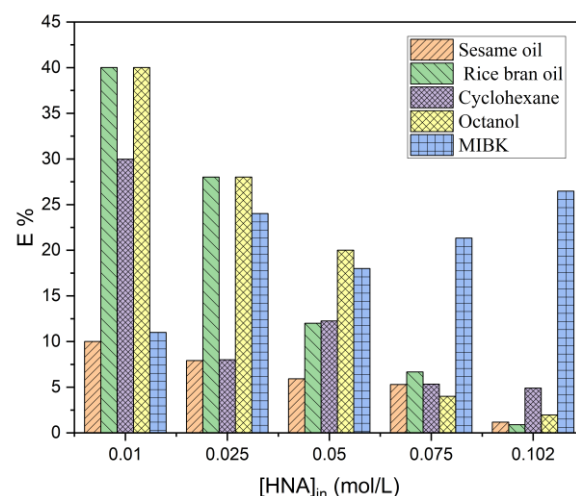


Figure 6: Variation in the extraction efficiency against the initial concentration of nicotinic acid at  $298 \pm 1$  K

Table 5: Summary of the extraction results for nicotinic acid

Solvents	Range of $K_D$	Average $K_D$	Range of $E\%$	Average $E\%$
Sesame oil	0.020-0.111	0.0672	1.960-9.990	6.216
Rice bran oil	0.009-0.666	0.2520	0.981-40.010	17.526
Cyclohexane	0.020-0.428	0.1742	1.960-29.970	13.719
Octanol	0.020-0.639	0.2332	1.960-39.010	16.514
MIBK	0.162-0.360	0.2466	14.000-26.470	19.550

1-octanol is a non-polar solvent unlike MIBK, limiting mass transfer from the aqueous to the organic phase. The maximum extraction efficiencies were 40.01, 39.01, 29.97 and 9.99% when rice bran oil, 1-octanol, cyclohexane and sesame oil were used, respectively, with 0.01 mol L<sup>-1</sup> of NA. In the case of MIBK, an extraction efficiency of 26.47% was observed when 0.102 mol L<sup>-1</sup> of NA was used (Table 5). Since the extraction efficiencies of MIBK increased as the initial concentration of NA increased, unlike for the natural conventional solvents, MIBK can be used with several kinds of extractants, e.g. trioctylamine, tributyl phosphate, Aliquat 336, etc., to enhance the effectiveness of NA extraction.

## 5. Conclusions

The recovery of nicotinic acid from aqueous streams through extraction using natural and conventional non-toxic solvents was investigated. The extraction process was analyzed in terms of extraction efficiency and distribution coefficient, moreover, correlations with the physicochemical characteristics of the solvents were found. Higher values of  $K_D$  and  $E\%$  were observed using MIBK with a large relative permittivity and dipole moment. Additionally, some kind of a relationship between the extraction equilibrium parameters and the relative viscosity and density of the solvents was observed, however, no generalizations were made because additional investigations are required. An extraction efficiency in excess of 29.47% was observed using MIBK. Although natural solvents yielded lower extraction efficiencies (1.96%), they are preferable compared to the toxicity of conventional chemical solvents. More stages can be employed to achieve complete extraction, moreover, reactive extraction may be another alternative to increase extraction efficiencies.

## SYMBOLS

$MW$	molecular weight
$S_{water}$	solubility in water
$\mu$	viscosity
$\rho$	density
$BP$	boiling point
$MP$	melting point
$\epsilon$	dielectric constant (relative permittivity)
$D_m$	dipole moment
$RI$	refractive index
$K_D$	distribution coefficient
$E\%$	extraction efficiency
$[HNA]_{in}$	initial concentration of nicotinic acid
$[HNA]_{aq}$	concentration of nicotinic acid in the aqueous phase
$[HNA]_{org}$	concentration of nicotinic acid in the organic phase

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