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Multi-step processing in a microstructured flow reactor: direct nitration of propane—a proof of principle

Abstract: The vapor phase nitration of propane was successfully investigated in a multi-step microstructured reactor. The concept integrates heating, evaporation of reactants, the reaction channel, and the quenching and cooling of the product mixture; all done in a reactor with one plate. The nitration of propane with evaporated nitric acid was carried out at 380°C–450°C. This radical chain reaction was subsequently quenched, in a fast manner due to the high surface-to-volume ratio of the reaction channel. Therefore, the conversion of propane was deliberately kept low (approx. 2%) but at favor of selectivity for 2-nitropropane which increases remarkably. The harsh reaction conditions require a sophisticated microreactor concept, the use of an exceptional construction material, that is, an uncommon stainless steel alloy, as well as a process design which avoids NO_x release into the environment.

Keywords: gas-phase; micro reactor; nitration; nitro alkanes.

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1 Introduction

The vapor-phase nitration of lower alkanes with nitric acid is an important chemical reaction to produce nitroalkanes. It proceeds by a free radical mechanism, where

nitric acid decomposes at temperatures above 350°C to give mainly •OH and •NO₂ or •ONO radicals. If propane is used as starting material the expected compounds 1-nitropropane or 2-nitropropane can be obtained by performing the reaction at high temperatures, for example, above 400°C. In addition, a cleavage of propane can be expected and therefore nitromethane and nitroethane were also found in the reaction mixture [1, 2]. Not much information about this process could be found in the literature, nevertheless, the vapor-phase process is used in industry [3, 4]. Some information is given about equipment and use of catalysts or additives, such as oxygen or halides [3, 5, 6], to enhance the nitration process. Owing to the fact that gas-phase nitration is a radical reaction, a thermal explosion of the reaction mixture should be avoided [7]. The equipment described in the literature is on a macro-scale, for example, a Pyrex™ glass tube with a diameter of 200 mm and a length of 8800 mm [5], or titanium tubes as upstreaming flow-through reactors [4]. The reported yield of nitrated products ranges from 30% to 40%. It should be pointed out that at a too high surface-to-volume ratio conversion can decrease because of radical extinction on the reactor wall, yet favorably shifting the ratio between the different nitropropanes towards 1-nitropropane [8].

In addition, nitrous oxide is used for nitration reaction of lower alkanes [9]. The yield becomes higher compared with nitration with nitric acid, but the authors claim that the presence of oxygen is a precondition. To add oxygen to the reaction mixture shifts the reaction into an explosive regime [10]. Oxygen as an additive gives, in certain cases, higher yields on nitrated alkanes but the amount of unwanted oxidized products increases remarkably [5, 11]. Oxidation can be limited by maximizing molecule-to-wall collision via providing high surface-to-volume ratio of the reactor [11]. To avoid possibility of explosion, a process for vapor-phase nitration of propane in a fused salt reactor was also developed [12].

A catalytic gas-phase nitration process was investigated using a Bi-Mo catalyst. Depending on the molar ratio of alkanes/nitric acid, reaction temperature and catalyst

contact time, yield of nitro paraffin increases nearly four times compared with the non-catalytic process [13].

Alternatively, liquid-phase processes can be applied. At low temperatures of approximately 100°C and a reaction mixture of nitric acid, propane, 1-trifluoromethyl benzene with N-hydroxyphthalimide as catalyst, high yields should also be achievable [14, 15]. Nitration of short chain alkanes (C1–C6) was carried out with nitronium hexafluorophosphate in methylene chloride or nitroethane solution. Results indicate direct electrophilic insertion of NO⁺ [16].

In all cases, a mixture of nitrated alkanes as well as of undisclosed products were obtained, caused by radical cleavage of C–C bonds (Figure 1). Therefore, the technical synthesis route uses low carbon alkanes mixtures instead of a single compound.

Naturally, further post-processing steps, for example, extraction and fractional distillation are required [17].

Four nitroalkanes, (3) to (6), are of great industrial significance and are mainly used as solvents or co-solvents, as intermediates for drug syntheses or even as additives for rocket fuels. In particular, 2-nitropropane (3) is widely used as solvent due to its excellent chemical and physical properties (detailed description in [18]). Therefore, syntheses routes with a high selectivity towards (3) are needed.

2 Experimental

Propane (1) [CAS 74-98-6; 99.95% (3.5)] was delivered from Linde Gas (Munich, Germany). Nitric acid, red and fuming (2) (CAS 7697-37-2; >90%), and reference substances, such as 2-nitropropane (3) (CAS 79-46-9; >96%), 1-nitropropane (4) (CAS 108-03-2; >98.5%), nitroethane (5) (CAS 79-24-3; 99.5%), and nitromethane (5) (CAS 75-52-5; >96.5%), were purchased from Sigma Aldrich (St. Louis, USA).

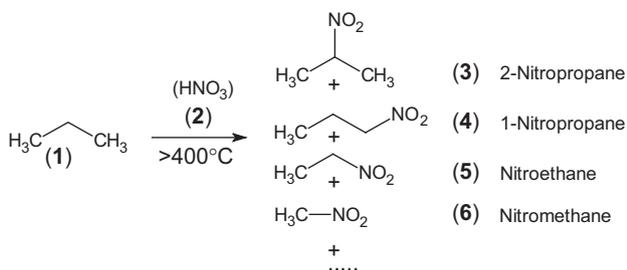


Figure 1 Expected product mixture for high temperature gas-phase nitration of propane. In addition, undisclosed compounds (marked as ...) are formed by C-C bond cleavage and induced reaction of $\cdot\text{OH}$ radicals.

Continuous and nearly pulsation-free feed of nitric acid was realized by using a syringe pump (Postnova PN1610, Postnova Analytics GmbH, Landsberg, Germany).

NiCrNi thermocouples were used for temperature measurements combined with a Voltkraft™ K204 (Conrad Electronic, Hirschau, Germany) data logger.

Two types of electrical heaters were used: 10 cartridge heaters (200 W each) to provide base load and two heating cables (250 W each) for temperature control management (both from Horst GmbH, Lorsch, Germany).

Mass-flow controllers were purchased from Bronkhorst-Mättig GmbH, Germany.

The product mixtures were characterized with GC (Varian CP-3900) methods. The GC detector was calibrated with 2-nitropropane (3) as standard.

2.1 Reactor set-up, general

The corrosive reactants and harsh reaction conditions applied, as well as the multi-step processing within one reactor, require a sophisticated reactor and process design. In addition to the actual chemical processing, material issues, heat management and safety considerations must be taken into account. In general, for multi-step processing different unit operations must be implemented: (i) preheating of propane (1), (ii) evaporation of nitric acid (2), (iii) mixing of gaseous reactants, (iv) quenching by mixing of the reaction mixture with water, and (v) simultaneous cooling of the mixture to avoid steam hammering (compare Figure 2). All in all, the reactor provides three different functional sections, that is, for evaporation and mixing (A), for the high-temperature reaction (B), and for quenching and cooling (C). The reactor plates (Figure 3) are clamped between two housing plates providing cavities for heating and cooling devices.

2.2 Process design and safety considerations

For safety considerations, the reactor can be purged with nitrogen at the propane inlet. Water is used to dilute nitric acid at the inlet and also to flush the whole reactor for cleaning and removing of possible blockage. Pressurized nitrogen is used to deliver water to the reactor.

While purging the whole system with nitrogen, both sections, (A) and (B) (Figure 2), are heated up to the appropriated temperature. In addition, section (C) is cooled down and the HPLC pump starts to feed water for later quenching of the reaction and diluting the reaction mixture. Once

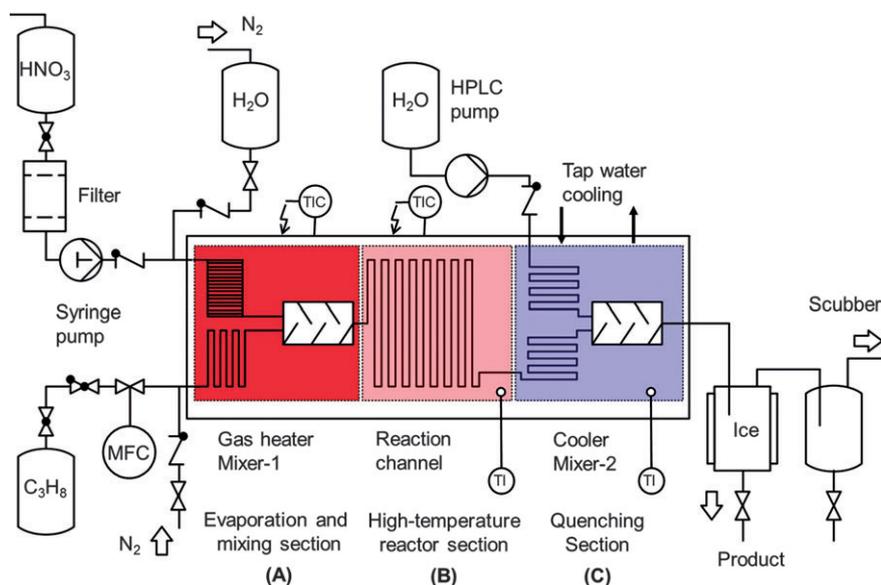


Figure 2 Scheme of the used reactor design.

the reaction conditions are stable, pressurized propane (1), provided from a commercial high-pressure gas cylinder, is fed into the reactor via heating up in section (A). The initial pressure was set to 6 bar and the gas-volume flow of (1) is controlled by a Bronckhorst mass-flow controller. An additional check valve secures the gas line. Nitric acid is pumped in the evaporator, section (A), by a corrosion-resistant glass syringe pump. The gaseous reactants are mixed in the integrated caterpillar micro mixer 1. After mixing the reac-

tion subsequently takes place along the electrically heated reaction channel. Having passed this channel, the mixture is cooled by heat dissipation to an outside pressed water cooler at section (C). The reaction mixture is diluted with water by integrated caterpillar micro mixer 2 and subsequently sparged through an ice-water filled flask to condense volatile nitric compounds. To avoid contamination of the environment, a scrubber filled with diluted NaOH solution removes gaseous nitric species.

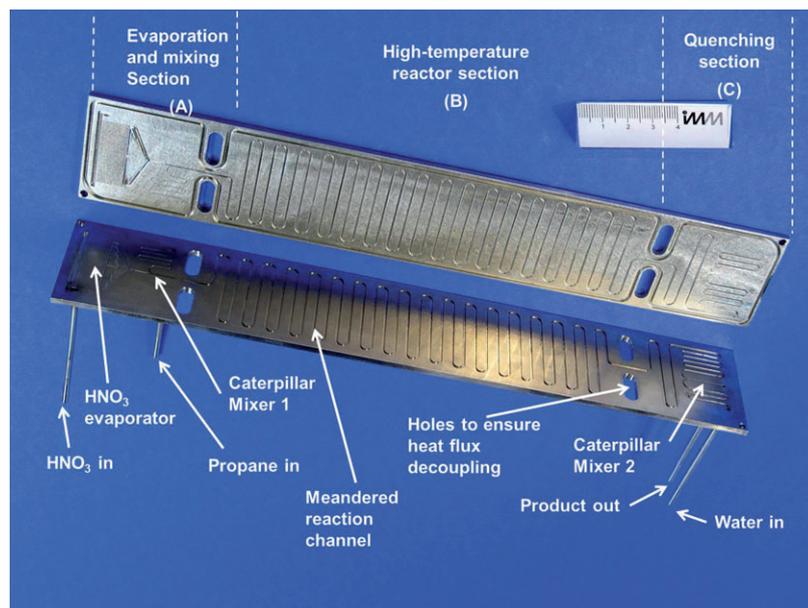


Figure 3 Structured reactor plates prior to welding. Channels were made by micromilling; the caterpillar-type mixers (mixer 1: CPMM 300; mixer 2: CPMM 600; IMM) were fabricated by laser ablation.

Reactor design	Channel parameter				
	Overall size (mm)	Heating zone (mm)	Water cooling channel	Channel dimensions (mm)	
Housing plates	330×80×20	190	6	7×3.9×4.5	
Reactor plates	Overall size (mm)	Channel dimension (mm)	Overall volume (mm ³)	Overall surface (mm ²)	Surface-to-volume ratio
	300×50×3	1.0×1.0×1 970	1 970	7 880	4
Evaporator	Number of channels	Channel dimension (mm)	Overall volume (mm ³)	Overall surface (mm ²)	Surface-to-volume ratio
	100	11.0×0.1×0.05	5.5	440	80

Table 1 Main dimensions of the used reactor system.

Short-chain nitro alkanes are slightly soluble in water, and therefore the mixture of condensed nitrated products in ice-water was extracted with methylene chloride. The separated organic phase was neutralized with diluted NaHCO₃ solution (10% in water), dried with sodium sulfate and directly used for GC analysis.

2.3 Description of the reactor system

The reactor consists of two microstructured plates made of corrosion-resistant special stainless steel (1.4361, X1CrNi Si18154) (Figure 3). The reaction channel, as well as in and outlet channels were made by precision machining. The two caterpillar-type micro mixers, a CPMM 300 type in section (A), with a 300 μm wide internally structured channel, and a CPMM 600 type in section (C) with a 600 μm wide channel (both IMM), and the small channels for

the evaporation part were made by electro-spark erosion techniques. All relevant dimensions are given in Table 1.

A more detailed view on the evaporation part is shown in Figure 4. The small eroded channels provide a high enough surface-to-volume ratio for continuous nitric acid evaporation. A comparatively big evaporation chamber avoids steam hammering and unwanted transfer of nitric acid droplets into the mixer. Slotted holes minimize a lateral heat flux between sections (A)–(B) and (B)–(C). In section (C), a second caterpillar-type mixer is used for cooling and diluting the reactant mixture with water. To avoid leaking of nitric acid and to ensure gas tightness, the reactor plates were laser-welded.

To provide suitable heat management, the reactor is press-fitted between two 20-mm thick stainless steel (1.4435) housing plates which contain the electrical heaters and two integrated water coolers (Figure 5). To ensure good heat flux and to seal both water coolers, a

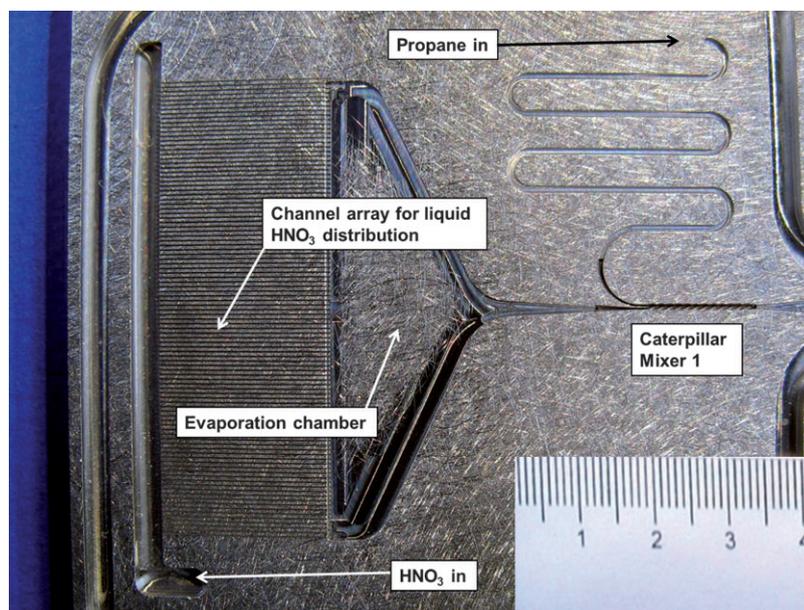


Figure 4 Magnified view of the evaporation and mixing section (A).

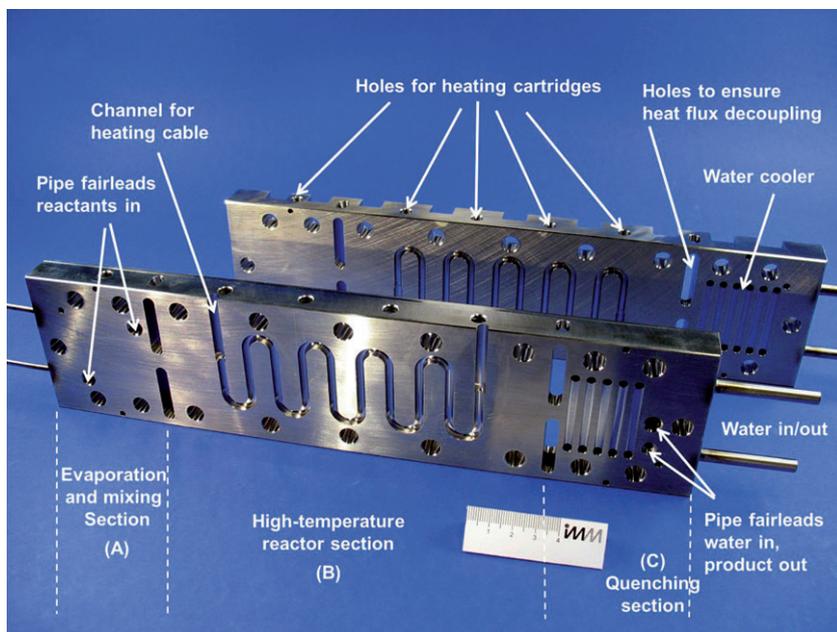


Figure 5 Housing plates with milled grooves for heating cables and cooling channels.

plane carbon seal tightens the housing and the reactor on both sites. The dimensions of the housing plates are given in Table 1.

Enough heating power is provided by 10 heating cartridges (200 W each) and two heating cables (250 W) inserted into the top and bottom housing plates. The heating cartridges ensure basic thermal load. They are steered by their internal thermocouples and a separate power supply unit. Three independent thermocouples

(green lines in Figure 6) placed on top of the welded reactor measure the temperatures at three points (entrance, middle, and outlet) of section (B). They are also used for temperature control and to control heating power of two heating cables to ensure equal temperature distribution.

The main function of water cooling in section (C) is to prevent steam hammering when the hot reaction mixture is quenched by diluting with water (a CAD drawing of the mounted reactor is given in Figure 6).

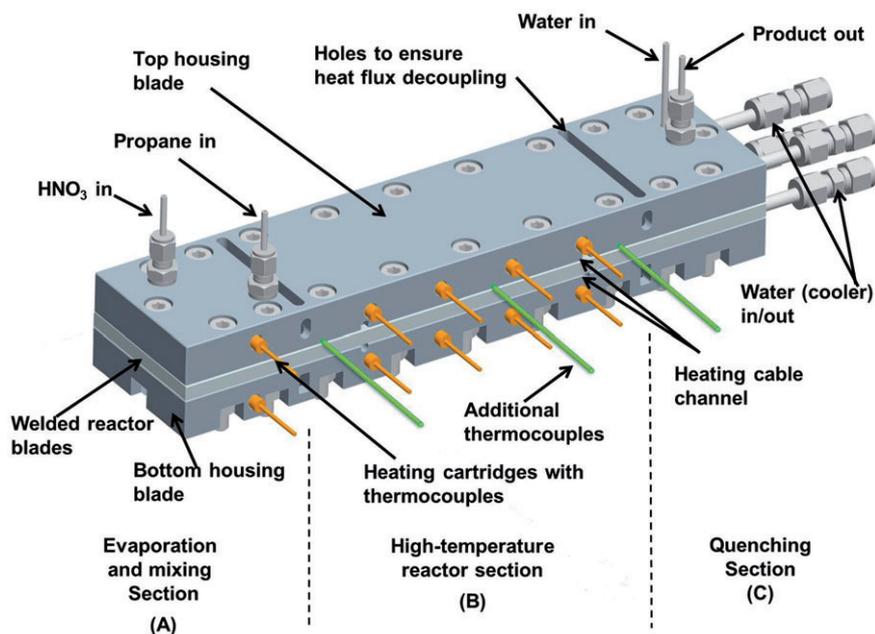


Figure 6 CAD scheme of the mounted reactor with inserted heating cartridges (yellow) and additional thermocouples (green).

The nitration process was carried out between 380°C and 455°C in accordance with the temperature range given in the literature [2, 8]. The temperature of section (A) is always adjusted to the reaction temperature in section (B). Different molar ratios [C_3H_8 (1): HNO_3 (2)] from 0.5 to 6.0 were investigated. Gas composition and temperature dependent on volume expansion were roughly estimated to calculate residence times, which range from 0.4 to 2.5 s, depending on the flow rate used. Owing to the unknown molar amount of gaseous compounds (radicals and C–C bond cleavage), exact data could not be derived. Therefore, gas flow velocity is assumed to be between 5 and 0.9 m/s.

3 Results and discussion

In general, conversion of (1) was determined by volumetric determination of flue gas, which mainly contains unreacted propane and traces of unknown substances formed by C–C bond cleavage due to the radical reaction mechanism. In all cases, conversion was low and did not exceed 2%. An exact volumetric determination of such small differences between initial and flue gas volumes was impossible, and for a proof-of-principle it is also not stringent. Therefore, only nitrated products were taken into account, that is, the sum of all detectable nitro compounds was set to 1 (100%), and the molar fractions are used for further interpretation. A cleavage of propane C–C bonds results in the formation of nitroethane (5) and possibly nitromethane (6), but the latter could not be detected, not even in the raw product gas mixture.

3.1 Reaction temperature

Influence of reaction temperature was investigated for temperatures ranging between 385°C and 455°C. Volume flow of propane (1) was set to 32 ml/min (room temperature) and for nitric acid (2) 0.06 ml/min (liquid), that is, the molar ratio of (1) to (2) is 1.

A rough calculation gives an average residence time of approximately 1 s.

Unexpectedly, the molar ratios of the formed nitroalkanes are nearly independent from the reaction temperature (Figure 7). Decrease of 1-nitropropane can be explained by subsequent cleavage and formation of nitroethane. Values for 2-nitropropane, that is, molar ratio, remain high and independent of applied reaction temperature.

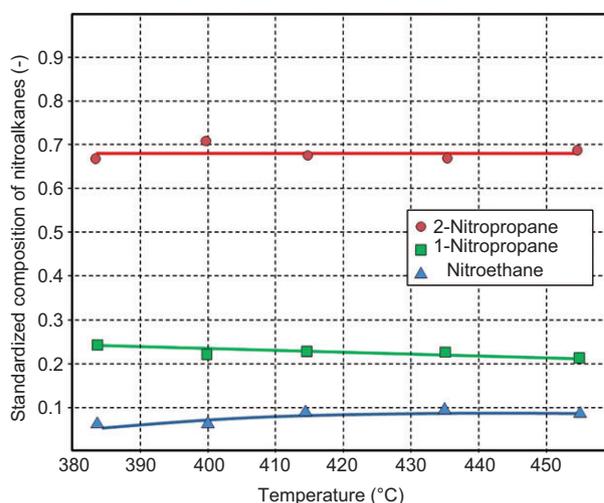


Figure 7 Standardized concentration ratio of nitroalkanes depending on the applied reaction temperature. (The concentration of all nitro compounds is set to one, i.e., the standardized composition is a fraction thereof.)

3.2 Residence time

With the assumptions made above, residence times were adjusted by a change in flow rates of the reactants. Flow rate of nitric acid was changed stepwise from 0.02 to 0.1 ml/min, and flow rate of propane was changed correspondingly to ensure a molar ratio of one between propane and nitric acid.

Calculation of exact residence times is impossible with the used experimental set-up. Owing to the radical mechanism of the reaction, neither the gaseous species nor their appearance and concentration are known. It also seems to be that water evaporation, that is, content of water from nitric acid, gives the main part for the temperature-dependent gas volume expansion. Therefore, only a rough estimation of residence times is possible, that is, assuming a range from 0.4 to 2.4 s depending on applied reactant flow rate. To make a comparison possible, liquid feed of nitric acid is used in Figure 8 instead of estimated residence times. At very short residence times, concentration of nitroethane tends to be zero. By contrast, highest values were achieved for 1-nitropropane at longer residence time, but the molar ratio of 2-nitropropane remains at high values of approximately 0.7 (Figure 8). This is in accordance with the results given in section 3.1.

3.3 Molar ratio

Molar ratio of propane (1) and nitric acid (2) was changed from 0.5 to 6. Volume flow of nitric acid (2) was kept

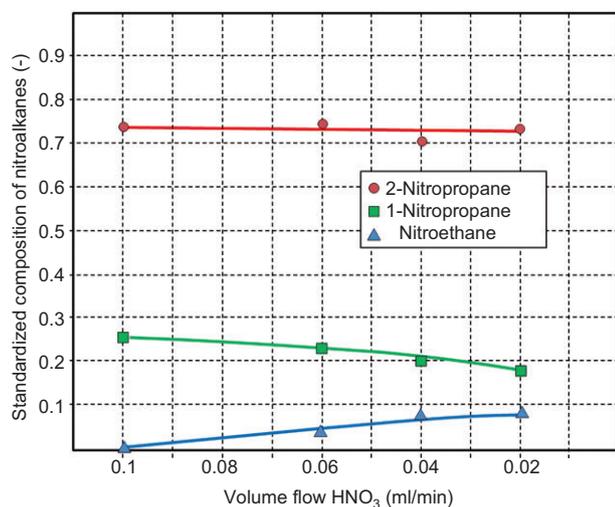


Figure 8 Standardized concentration ratio of nitroalkanes depending on volume flow of liquid HNO₃. Molar ratio between nitric acid and propane is set to one. Volume flow corresponds with residence time (increase of residence time from left to right).

constant (0.04 ml/min), and propane (1) concentration changed from 10.9 ml/min to 130.8 ml/min accordingly.

Reaction temperature is set to 415°C. At low molar ratios formation of 2-nitropropane (3) is preferred, whereas concentration of nitroethane (5) levels to zero (Figure 9). With increasing molar ratio, 2-nitropropane concentration drops down to 0.6. At the highest molar ratio of 1–6, concentration of 1-nitropropane is increased to 0.25. These results can be explained by the radical mechanism of the

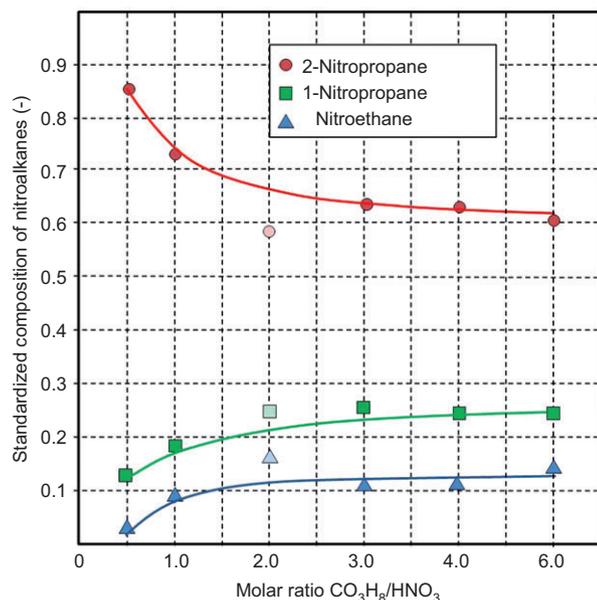


Figure 9 Nitroalkanes ratio dependent on the molar ratio of C₃H₈/HNO₃. 2-Nitropropane is the preferred product at low ratios.

reaction. The preferred compound from a thermodynamic point of view is 2-nitropropane (3) due to high concentrations of radicals when the molar ratio of (1) to (2) is low. With an increase of this ratio, propane (1) concentration exceeds concentration of nitric acid and the radical chain is terminated fast. Therefore, higher values for 1-nitropropane (4) could be achieved. The ratio of nitroethane (5) is nearly independent of reaction conditions, that is, temperature, residence time or molar ratio of reactants. It should be taken into account that the increase of propane (1) flow rate shortens residence time. Therefore, results should be judged carefully.

4 Summary

It can be concluded from the results described above that nitration of light alkanes with nitric acid in the vapor phase can be performed in a multi-step microstructured reactor – in this case as a proof of principle. It is well known that a free radical chain can be broken due to extinction of radicals at the reactor wall, when high surface-to-volume ratios are provided. In this case, microstructured reactors are not advantageous, because conversion should be low due to fast quenching of the radical chain mechanism. This is in accordance with experimental results attained. Conversion of propane was rather low and did not exceed 2%. However, in contrast to the results given in [8], 2-nitropropane (3) is the preferred product instead of 1-nitropropane (4). Co-feeding oxygen to increase conversion rate was not applied due to safety regulations, although it is interesting to test whether oxygenation reactions can be suppressed by high surface-to-volume ratio, as outlined in [11]. There are also some constraints concerning volume flow of propane. If flow becomes too high, it can be assumed that gas temperature is lower compared with measured reactor temperature. Maximum deliverable power for reactor heating (2.5 kW) is oversized, even though it does not ensure complete heat transfer due to estimated very short residence times below 0.5 s, especially not under laminar flow conditions. This might be the reason for the observed inconsiderable changes of product composition (nitroalkanes) at short residence times and higher propane (1) to nitric acid (2). In contrast to the literature [5], a temperature shift from 385°C to 450°C did not influence product ratio measurably.

Shortening residence time by a temperature-dependent increase of gas volume as well as fast extinction of the radical chain reaction by wall effects certainly avoids repeated bond cleavages and therefore a drop-down of

nitropropane concentration in favor of shorter chain nitroalkanes. An indication of fast breakdown of the radical chain reaction is also the absence of nitromethane in the product mixture. A continuous flow of liquid nitric acid below 0.05 ml/min, in particular constant continuous evaporation, could not be achieved and therefore an increase of residence time was not possible with the given set-up.

5 Conclusions on reactor and process design

Some constraints concerning the reactor itself must be taken into account. Because tightness of the reactor was not achievable by sealing and screwing, the reactor plates were welded together. In this case, maintenance of inner reactor structures was not possible anymore, and the reaction must be carried out carefully to avoid blockage and pressure overshooting. After every run, the reactor had to be cleaned by back-flushing with concentrated nitric acid and subsequent blowing dry with nitrogen to avoid corrosion by diluting nitric acid by feeding water or moisture air. It should be noted that some particles and rusty liquid came out of the reactor. We assume that the connectors could not sustain the harsh reaction conditions.

After finishing the experiments, the reactor was opened. As expected, rusty coverage and particles were found in the nitric acid evaporation part, but the microstructures were not destroyed (Figure 10). After an approximated time on operation of around 200 h, including functionality tests, etc., evaporation part, reaction channel and quenching section appeared unchanged.

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Figure 10 Magnified view of evaporation channels used. Microstructures are intact and not blocked, while some removable flash rust can be seen on the surface.

To summarize, proof of principle for the designed reactor was successfully done. The selected special steel (1.4361) was the right choice to perform nitration reactions at such harsh conditions. The radical reaction took place in a confined space; the conversion of propane was low. Such a demanding chemical process also necessarily needs follow-up processes, for example, environmental protection, waste disposal and wastewater treatment. A release of toxic or otherwise environmental harmful substances into the air must be avoided. Processing under micro-flow conditions is a priori not sustainable, or safe and environmentally friendly. It is necessary from early on to take a holistic process design into account and not focus on the “micro reactor” solely.

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Ma Jiang received his PhD in molecular engineering from the Kyushu University in 1997. In 2000, he secured the appointment of part-time professor and doctoral supervisor in Dalian University of Technology, and he was awarded as a specialist and state government advisor in 2005. He was engaged as concurrently supervisor of postgraduates of the Shenyang institute of chemical in 2006 and as a senior professional of the Sinochem Group in 2008. He is currently the general manager of Shenyang Cenkey Chemical Co., Ltd, and BMD Chemical Co., Ltd. His research interests involve studies of fine chemicals, in particular for fluorescent whitening agents.



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Christian Schütt received his baccalaureate at the Ludwig Erhard high school, Kiel. He finished his studies as a diploma engineer in biotechnology/process engineering at the Flensburg University of Applied Sciences in Flensburg and he received his PhD from the Institute of Phythopathology at the Christian-Albrechts-University, Kiel. Since 2005 he has been employee of the Institut fuer Mikrotechnik Mainz GmbH. The main tasks of his work are dimensioning, design, production control and testing of mixers, heat exchangers and reactors.